

Ni-based superalloys 625 and 718 fabricated through in situ interlayer hot forging arc plasma directed energy deposition – grain size refinement and heat treatment development

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*Enquanto este velho trem atravessa o pantanal
O povo lá em casa espera que eu mande um postal
Dizendo que eu estou muito bem vivo
Rumo a Santa Cruz de La Sierra*

*Enquanto este velho trem atravessa o pantanal
Só meu coração está batendo desigual
Ele agora sabe que o medo viaja também
Sobre todos os trilhos da terra*
Paulo Simões e Geraldo Roca, Trem do Pantanal, 1975.

To my parents

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ABSTRACT

Ni-based superalloys fabricated via fusion-based additive manufacturing (AM) typically exhibit a coarse and oriented microstructure, which may hinder their use in high responsibility applications. This is especially critical for arc plasma direct energy deposition (AP-DED) due to its lower cooling rate compared to laser-based AM, which may induce columnar millimeter-sized grains. AP-DED variants are emerging with the purpose of reducing grain size, particularly through interlayer mechanical deformation systems. Among these, forging stands out due to its lighter and more versatile systems.

The present work evaluates the effect of the in situ interlayer hot forging (HF) on the microstructure and mechanical properties of Inconel 625 and 718 fabricated via AP-DED. The Inconel 625 AP-DED process was described, followed by the development of a finite element model to optimize the HF tool. Subsequently, the effects of HF on the microstructure and heat treatment response of Inconel 625 AP-DED were comprehensively described. Inconel 718, in addition to coarse microstructure, also exhibits a lower heat treatment response due to the interdendritic segregation and eutectics formation. Thus, the present work also developed heat treatments for Inconel 718 AP-DED, which showed satisfactory performance, meeting the strength and ductility requirements of AMS 5662. However, the grain size problem persists. When printing Inconel 718 using AP-DED coupled with HF, significant changes in the grain size (millimeter-sized vs. 90 μm) and texture aspect (cube vs. random) were observed. Additionally, the anisotropy behavior was notoriously reduced (almost isotropic behavior). However, despite the grain size refinement, the final grain size is still quite larger in relation to wrought material, which induces a lower strain hardening and does not significantly affect the ultimate tensile strength and Young's modulus.

In conclusion, the main advantage introduced by HF-DED is its ability to reduce the material grain size and promote almost isotropic behavior.

Keywords: additive manufacturing, direct energy deposition, Ni-based superalloys, grain size, crystallographic texture, interlayer mechanical deformation, hybrid process

RESUMO

Superligas à base de Ni fabricadas por manufatura aditiva (AM) geralmente apresentam uma microestrutura grosseira e orientada, o que pode dificultar seu uso em aplicações de alta responsabilidade. Isso é especialmente crítico para a deposição por energia direta com arco de plasma (AP-DED) devido à sua menor taxa de resfriamento em comparação com as técnicas de manufatura aditiva baseada em laser, que pode induzir grãos colunares de tamanho milimétrico. Variantes do AP-DED estão a ser desenvolvidas com o propósito de reduzir o tamanho dos grãos, particularmente por meio de deformação intercamadas. Entre estes, o forjamento destaca-se por ser um sistema leve e versáteis.

O presente trabalho avaliou o efeito do forjamento a quente intercamadas (HF) na microestrutura e propriedades mecânicas do Inconel 625 e 718 fabricados por meio de AP-DED. O processo de deposição para o Inconel 625 foi descrito, seguido pelo desenvolvimento de um modelo de elemento finito para otimizar a ferramenta HF. Posteriormente, os efeitos do HF na microestrutura e na resposta ao tratamento térmico foram descritos. O Inconel 718, além da microestrutura grosseira, também apresenta uma resposta inferior ao tratamento térmico devido à segregação interdendrítica e à formação de eutéticos. Assim, o presente trabalho também desenvolveu tratamentos térmicos para o Inconel 718 AP-DED, que apresentou desempenho satisfatório, atendendo aos requisitos de resistência e ductilidade da AMS 5662. No entanto, o problema do tamanho do grão persiste. Ao imprimir o Inconel 718 usando AP-DED acoplado com HF, foram observadas mudanças significativas no tamanho do grão (milímetro vs. 90 μm) e textura cristalográfica (cubo vs. aleatório). Além disso, o comportamento anisotrópico foi notoriamente reduzido (comportamento quase isotrópico). No entanto, apesar do refinamento do tamanho do grão, o tamanho final do grão ainda é bem maior que o do material forjado, o que induz a um menor encruamento e não afeta significativamente a resistência à tração final e o módulo de Young.

Em conclusão, a principal vantagem introduzida pelo HF-DED é sua capacidade de reduzir o tamanho do grão do material e promover um comportamento quase isotrópico.

Palavras-chave: manufatura aditiva, deposição por energia direta, ligas à base de Ni, tamanho de grão, textura cristalográfica, deformação intercamada, processo híbrido

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1 INTRODUCTION

1.1 Context and Motivation

Additive manufacturing (AM), a layer-by-layer deposition process, has been considered a promising technology in industry 4.0 due to its design flexibility capacity [1], reduction in lead time and material waste (low buy-to-fly and topological optimization) [2], [3], and its ability to enable the development of new components and materials (e.g., one-piece parts and functionally graded materials) [4]–[6]. ASTM F3187 classifies the directed energy deposition (DED¹) processes, a subcategory of AM processes, according to the heat source (laser, electron beam, or arc plasma) used to melt and consolidate the feedstock material (powder and wire). Focused heat source DED processes (laser and electron beam) have a higher dimensional control [7], [8]; however, the commonly used powder feedstock material is difficult to handle, expensive, and causes a higher material wastage in relation to wire [9], [10]. Laser and electron beam DED processes are also more susceptible to defects such as porosity and lack of fusion [11], [12]. Otherwise, arc plasma DED processes use a typical welding processes as the heat source (i.e., gas tungsten arc – GTA, plasma transferred arc – PTA, and gas metal arc welding – GMAW), which significantly reduce the equipment and feedstock material costs (e.g., commercial wire spools) [1], [13]–[15]. In addition, arc plasma DED processes have the highest deposition rate (2–10 kg/h), albeit at the expense of dimensional accuracy and resolution [16], [17].

Despite the advantages of the DED processes in relation to the traditional (e.g., machining from a billet) manufacturing routes (e.g., lower lead time and supplier chain decentralization), the material properties and behavior of directed energy deposited (DEDed) parts have still not been optimized [18]–[20], which can prevent the industrial scalability of this technology. Additionally, DEDed parts often do not meet the specifications of the traditionally manufactured parts (e.g., wrought + machining) and standards requirements [21]. Further, the

¹ The terminology adopted in the present work followed the ASTM F3187 – 16 and ISO/ASTM 52900 – 2021.

higher residual stress developed requires post-deposition heat treatments [22], which must be applied even for non-heat-treatable alloys. The complex and multiple thermal cycles and non-equilibrium solidification conditions compounded with the higher heat input (e.g., arc plasma DED processes) or localized heat source (e.g., DED-laser) explain the relatively high residual stresses and distortions reported [23]. In addition, the epitaxial grain growth during the melting pool solidification usually generates a coarser and highly oriented microstructure, which results in anisotropic properties. This phenomenon is especially observed in alloys that do not undergo allotropic transformations, i.e., alloys that show the as-solidified (primary) microstructure at room (e.g., Ni- and Al-based) [24]–[26]. Furthermore, alloys that undergo allotropic transformation (e.g., low-alloy steels and Ti–6Al–4V) are less sensitive to solidification textures, i.e., the oriented and coarse primary grains (parent phase) are refined (product phase) due to the solid-state phase transformation; however, the crystallographic relationships between the parent and product phases (e.g., Kurdjumov-Sachs and Burgers) can still promote some preferential orientation in the product phase and anisotropic behavior [27]. In addition, brittle or soft points generated in the reheated regions (multiple thermal cycles) [28]–[32] can induce ductility and toughness anisotropy, acting as preferential crack nucleation sites [33].

To overcome the aforementioned problems, interlayer (cold or hot) plastic deformation coupled with the feedstock material consolidation has proven to be a promising alternative to control the microstructure, residual stresses, and mechanical properties of the DEDed parts [34]–[36]. These interlayer deformation methodologies also improve the material heat treatment response [37]. Deposition + interlayer mechanical deformation (e.g., rolling, hammer peening, and forging) were first applied in arc-welding [38]–[43] to relieve residual stresses and improve the fatigue life. In addition, commercial alloys (Ti- [44]–[46], Al- [47], [48], Fe- [49], [50], and Ni-based alloys [51], [52]) showed good formability in the as-built (as-printed) condition [53], reinforcing the use of interlayer deformation during fusion-based additive manufacturing processes [54]–[56]. Furthermore, arc plasma DED variants and hybrid processes have been also developed to promote grain size refinement (e.g., columnar to equiaxial transition [57] or recrystallization [53]) and reduce solidification segregation [58], e.g., deposition assisted by ultrasound vibrations [59]–[61], external magnetic field stir [62], forced cooling systems [63], the addition of inoculants in the melt pool [64], and redesign the feedstock material [65]. Among these alternatives, the interlayer mechanical deformation stands out for simultaneously relieving the residual stress (local yielding), inducing grain size refinement (dynamic and static recrystallization), and not altering the feedstock material specification [66].

Considering the complex geometrical aspects related to AM, DED + interlayer deformation has its own challenges, primarily associated with the non-linear path planning and the coupled deposition and deformation parameters (e.g., avoiding the entire remelt of the deformed zone). In addition, interlayer deformation processes can be classified by the temperature (cold – below – and hot – above the recrystallization temperature, respectively) and deformation mode (almost homogeneous and quasi-static – rolling – and localized and dynamic – peening/hammering/forging) [34]. Interlayer rolling had a high deformation and an efficient grain size refinement; however, this variant has path planning restrictions (almost linear deposition), which reduces its industrial scalability potential. In contrast, peening/hammering/forging had a less efficient grain size refinement and a more flexible path planning (they can be robotized), making them suitable for industrial miscellaneous part fabrication. Recently, the Manufacturing and Non-Destructive Testing research group of the Department of Mechanical and Industrial Engineering – NOVA FCT, specifically Duarte et al. [35] (Patent number WO2020261233), developed an *in situ* interlayer hot forging (HF) system that hot deformed the just deposited material (900 - 1100 °C) and allowed a free planar path planning, promoting a finer and less texturized microstructure. Therefore, considering the coarse and oriented microstructure typically associated with Ni-based superalloys fabricated via fusion-based AM processes, the present work proposed to study the effect of the *in situ* interlayer hot forging on the microstructure and mechanical behavior of the Ni-based superalloys 625 and 718 (commercially known as Inconel® 625 and Inconel® 718, respectively) fabricated by arc plasma DED.

1.2 Objectives

The present work aims to refine, through *in situ* interlayer hot forging [35], the coarse and cube texturized microstructure typically observed in the Ni-based superalloys 625 and 718 fabricated via arc plasma DED in order to meet the industrial grain size and mechanical properties requirements (e.g., API 6ACRA, AMS 5662, and API 20S). Additionally, for the Ni-based superalloy 718 (precipitation-strengthened alloy) fabricated via arc plasma DED, the standard homogenization/solution + aging heat treatments applied to cast and wrought material (e.g., AMS 5383 and AMS 5662) had a low response due to pronounced interdendritic segregation and eutectics precipitates (e.g., Laves and MC-type carbides). These precipitates consume especially the Nb alloying element (γ'' promoter), reducing the γ'' content (primary alloy strengthening phase), i.e., the effectiveness of the aging heat treatment may be

compromised. Thus, in addition to refining the grain size and reducing the crystallographic texture, studying the post-deposition heat treatment is also fundamental to ensure the equivalence in quasi-static mechanical properties between the arc plasma DED and traditional manufacturing routes (e.g., cast and/or wrought). To achieve the aforementioned objectives, the following specific objectives have been established:

- i Perform a literature review to evaluate the applicability of the *in situ* interlayer hot forging to typical commercial alloys (e.g., low alloy and stainless steels and Ti-, Ni-, and Al-based alloys).
- ii Optimize the deposition parameter of the arc plasma DED coupled to in situ interlayer hot forging.
- iii Develop and validate a thermal-metallurgical finite element model to simulate the thermal distribution during the deposition and design an optimized deformation tool (coaxial crown hammer).
- iv Describe the *in situ* interlayer hot forging grain size refinement mechanism for Ni-based superalloys.
- v Propose dedicated post-deposition heat treatment for the Ni-based superalloy 718 fabricated via arc plasma DED.
- vi Analyze the effect of the *in situ* interlayer hot forging on the microstructure and mechanical properties of the Ni-based superalloy 718 fabricated via arc plasma DED.
- vii Perform a comprehensive microstructural characterization of the Ni-based superalloy 625 and 718 fabricated via arc plasma DED (with and without in situ interlayer hot forging).

1.3 Document organization

The present document is organized into seven sections:

- Section 1 (Introduction) – motivation and objectives;
- Section 2 (literature review) – highlights the current development and material response of the DED + interlayer mechanical deformation and the difficulties associated with the Inconel® 718 fabricated via arc plasma DED;
- Section 3 – results for the Inconel® 625 fabricated via arc plasma DED coupled with *in situ* Interlayer hot forging;
- Section 4 – Heat treatments developed for Inconel® 718 fabricated via arc plasma DED;

- Section 5 – Inconel® 718 fabricated via arc plasma DED coupled with the *in situ* interlayer hot forging.
- Section 6 – The main conclusions obtained in the present work.
- Section 7 – Suggestions of future works.

Figure 1.1 described the three steps (thesis sequence) of the present work, which are temporarily divided by year.

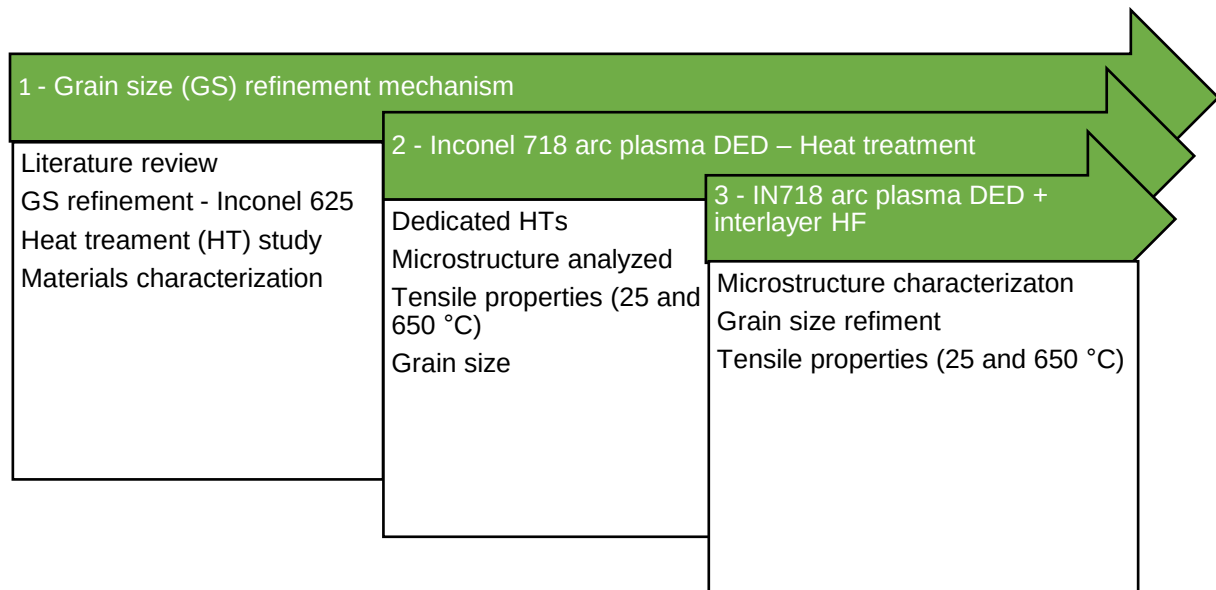


Figure 1.1 - Thesis sequence. GS and HT are the grain size and heat treatment, respectively. IN625 and IN718 refer to Ni-based superalloys 625 and 718, respectively.

1.4 Publications in indexed journals (ISI/Scopus) during the Ph.D.

1.4.1 Published

1.4.1.1 As first author

- Farias FWC, Duarte VR, Felice IO, et al. In situ interlayer hot forging arc-based directed energy deposition of Inconel® 625: process development and microstructure effects. Addit Manuf. 2023;66:103476. <https://doi.org/10.1016/j.addma.2023.103476>. IF: 11.
- Farias FWC, Duarte VR, Felice IO, et al. In situ interlayer hot forging arc plasma directed energy deposition of Inconel® 625: microstructure evolution during heat treatments. J Alloys Compd. 2023;952:170059. <https://doi.org/10.1016/j.jallcom.2023.170059>. IF: 6.2.
- Farias FWC, Duarte VR, Payão Filho J da C, et al. Arc-based directed energy deposited Inconel 718: role of heat treatments on high-temperature tensile behavior. Mater Res Lett. 2024;12:97–107. <https://doi.org/10.1080/21663831.2023.2297734>. IF: 8.3.

- Farias FWC, dos Santos TJG, Oliveira JP. Directed energy deposition + mechanical interlayer deformation additive manufacturing: a state-of-the-art literature review. *Int J Adv Manuf Technol.* 2024;131:999–1038. <https://doi.org/10.1007/s00170-024-13126-5>. IF: 3.4.
- Farias FWC, Duarte VR, Payão Filho J da C, et al. High-performance Inconel 718 fabricated via arc plasma directed energy deposition: effect of post-deposition heat treatments on microstructure and mechanical properties. *Addit Manuf.* 2024: 104252. <https://doi.org/10.1016/j.addma.2024.104252>

1.4.1.2 As co-author

- Rodrigues TA, Farias FWC, Avila JA, et al. Effect of heat treatments on Inconel®625 fabricated by wire and arc additive manufacturing: an in situ synchrotron X-ray diffraction analysis. *Sci Technol Weld Join.* 2023;1–6. <https://doi.org/10.1080/13621718.2023.2187927>. IF: 3.3.
- Rodrigues TA, Farias FWC, Zhang K, et al. Wire and arc additive manufacturing of 316L stainless steel/Inconel 625 functionally graded material: development and characterization. *J Mater Res Technol.* 2022;21:237–251. <https://doi.org/10.1016/j.jmrt.2022.08.169>. IF: 6.4.
- Rodrigues TA, Bairaão N, Farias FWC, et al. Steel-copper functionally graded material produced by twin-wire and arc additive manufacturing (T-WAAM). *Mater Des.* 2022;213:110270. <https://doi.org/10.1016/j.matdes.2021.110270>. IF: 8.4.
- Cota BS, Amendoeira DAE, Farias FWC, et al. High-strength low-alloy steel fabricated by in situ interlayer hot forging arc-based directed energy deposition assisted with direct cooling: Microstructural and mechanical properties evaluation. *J. Manuf. Process.* 2024;129: 273-291. <https://doi.org/10.1016/j.jmapro.2024.08.064>. IF: 6.1.
- Fonseca PP, Duarte VR, Farias FWC, et al. Enhancing manufacturing and post-processing properties of WAAM ER110 HSLA steel: in situ hot forging + post-deposited heat treatment effects on surface quality and specific cutting energy. *Prog. Addit. Manuf.* 2024; <https://doi.org/10.1007/s40964-024-00814-7>. IF: 4.4.

1.4.2 In progress

1.4.2.1 Under review

- Ni-based superalloy 718 fabricated by arc-based directed energy deposition: an experimentally-based strengthening mechanisms analysis. Materials Science and Engineering: A. Manuscript Number: MSEA-D-24-07481.

1.4.2.2 Under preparation

- Inconel 718 fabricated via arc plasma directed energy deposition: effect of the *in situ* interlayer forging on microstructure and mechanical properties.

2 LITERATURE REVIEW

Directed energy deposition (DED) additive manufacturing systems have been developed and optimized for specific materials and operational uses. However, due to the low material process response, i.e., low mechanical properties and corrosion resistance compared to typical material specifications and the anisotropic behavior, DED variants have been developed to overcome these critical issues. Among the DED variants, coupled DED + interlayer (cold or hot) mechanical deformation (e.g., rolling, hammer peening, and forging) systems stand out by refining the microstructure, reducing the crystallographic texture, improving the mechanical properties and heat treatment response, and reducing the residual stresses. In this regard, the present section reported the main DED + in situ interlayer mechanical deformation systems available, their characteristics, potentials, and limitations. Also, a detailed description of the grain size refinement mechanisms and their relationship with DED + interlayer deformation parameters were addressed. In addition, the effects of DED + interlayer deformation on the mechanical properties of common engineering alloys (e.g., Ti-6Al-4V, Inconel® 18, high-strength low-alloy steels, AISI 316L stainless steel, and 2xxx series aluminum alloy) are comprehensively analyzed. Furthermore, a critical review related to Inconel® 718 fabricated via arc plasma DED was also performed, which include the alloy general metallurgical, process and printability aspects, and post-deposition heat treatment.

2.1 DED + Interlayer mechanical deformation

2.1.1 Why deform the just deposited material?

Due to the almost unidirectional heat flux in AM processes [67], the remelting of the previously deposited layer (or substrate) [68], and the higher thermal gradient in the melting pool [69], epitaxial nucleation and growth are promoted instead of heterogeneous nucleation during the feedstock material consolidation. This results in coarse (millimeter sized) columnar grains with a preferential crystallography orientation (typically the cube texture, $\{100\}\langle 100\rangle$) [70], which can cause anisotropic properties [71] and lower fatigue resistance [72]. In addition, the numerous thermal cycles with steep thermal gradients can generate high residual stresses and subsequent distortions in the as-built part [73]. Solutions have been proposed to promote the columnar-to-equiaxed transition (CET) during solidification and generate a finer and non-oriented microstructure [74], such as the addition of inoculants [64], [75], [76], modification of alloy composition [77], [78], control of thermal cycles (thermal management) [79], [80], and vibration of the melting pool [81], [82].

The use of inoculants, as well as the modification of the feedstock material composition, can alter the material specifications (critical for high-responsibility applications, e.g., nuclear, oil & gas, and aeronautics industries), requiring several and costly tests (e.g., corrosion in operational environments, creep, wear, and fatigue) for feedstock material requalification and commercial adoption. Thermal management via an external cooling system (e.g., submersion in water and air jet) had limitations regarding parts size and geometry, low heat transfer efficiency, and it was almost restricted to wire-based DED (laser and arc plasma) processes, because the use of powder is incompatible with water or air currents [83]. Further, external cooling systems tend to be less effective in inducing the CET because their effect on the thermal condition at the melting pool (solidification rate and thermal gradient) is approximately negligible [67]. Besides, for reactive materials (e.g., Al and Ti-based alloys), and specific feedstock material/coolant conditions, a surface reaction can occur, forming scales and increasing the minor harmful element content in as-built alloys (e.g., H, O, C, or N). The main advantage of thermal management via an external cooling system is the lesser idle time and higher productivity in low-scale production. By contrast, for mass part production, the external cooling system becomes redundant because the 3D printer can continue to fabricate other parts while the just deposited one cools to the specified interlayer temperature.

Vibration-assisted deposition has been shown to be an interesting alternative to induce CET without altering the material composition and affecting the deposition process (final layer geometry and process stability) [60], [84], exhibiting intense grain size refinement. However, vibration is limited by the part geometry and size (wave attenuation) and alloy type [85], [86]. In addition, a vibration stress relief procedure was initially used for welded joints that could not be post-weld heat treated [87]. Using arc-based welding, Munsif et al. [88] and Hsieh et al. [89] highlighted that vibration-assisted welding synergistically induced a finer microstructure and reduced the residual stress levels. However, in additive manufacturing, similar studies related to vibration-assisted deposition have focused only on the microstructure namely grain size, grain morphology, and texture [90], [91], and the effect of vibration on the residual stress and distortions has not been addressed. It is worth mentioning that vibration-assisted welding stress relief had no significant effect on the maximum developed residual stresses and did not significantly alter the residual stress profile [87], [88], [92]. Thus, considering the higher residual stresses developed in the parts fabricated via AM and their coarse and oriented microstructure, there is a technological demand for DED process variants and/or hybrid processes that simultaneously refine the microstructure and relieve residual stresses. This can concurrently minimize the material waste and machining time (near-net-

shape primary part with minimal distortion), reduce the number of manufacturing steps (e.g., avoid heat treatments), and improve the material behavior.

The application of interlayer mechanical deformation during deposition evolved into a promising technology to overcome the challenges associated with DED processes, including the coarse and oriented microstructure, lower mechanical properties, and the requirement of residual stress relief heat treatments. Multiple deformation-thermal cycle processes (e.g., forging and/or controlled rolling + heat treatment) can transform the microstructure of an ingot (casting material with columnar grain morphology, such as materials fabricated via DED) into an equiaxial grain morphology via multiple cycles of dynamic/static recrystallization and reheating [93]–[96]. The low deformation level induced during low cooling rate solidification (e.g., cast, arc-based welding, and arc-based DED) is not able to induce significant grain boundary migration during the heat treatment or reheating thermal cycles, which may differ significantly from high-density laser-based (e.g., powder bed fusion) AM technology (very high cooling rate, i.e., enough internal defects to drive grain boundary migration during heat treatments) [21], [25], [97]–[112]. In this way, there are few alternatives to introduce in the as-print microstructure a substantial number of defects (i.e., makes is less stable) so that when supplying energy (e.g., increase the temperature), the material may come to a lower energy state (suffer some phase transformation). Among the most typical forms to increase the internal energy of a material, it can be highlighted the deformation and all technological processes associated with it (e.g., forging, extrusion, and rolling).

Thus, applying a similar principle used to transform the microstructure of ingot to AM processes, the cold or hot deformation between the deposited layers can induce grain size refinement and alter the phase transformation kinetics [66], as well as relieve the residual stress via local yielding [113]. In relation to cast, AM technologies typically exhibit a finer dendrite microstructure (fast cooling rate); however, the grain size developed due to the epaxial growth can be higher in AM, especially for DED technologies, which may induce a significant difference in final grain size since the initial grain size affects the dynamic recrystallization kinetics. In addition, the typical parameters used for “cogging” and thermomechanical process may be not efficient to design the interlayer mechanical deformation coupled with the deposition due to different grain size and oriented microstructure. Additionally, interlayer deformation is restricted by the deformation intensity (significant changes in layer morphology), equipment (hydraulic press vs. robot arm), and thermal gradient imposed by the deposition (i.e., deformation gradient). The fast DED thermal cycles in relation to conventional controlled rolling and the complex part geometry limit the use of non-adaptable routes (e.g., forging and

rolling), requiring the development of dedicated DED + interlayer deformation systems. Typically, these latter are composed of a deposition unit (heat source and feeding system) and a deformation tool that can work at high (hot deformation to induce dynamic recrystallization) or at low temperatures (cold deformation to induce static recrystallization during reheating thermal cycle or heat treatment).

2.1.2 Deposition + interlayer deformation – systems and process aspects

2.1.2.1 Rolling

The use of cold or hot rolling coupled with deposition originated from arc-based welding processes, as demonstrated by Adams et al. [39] and Coules et al. [43], and was used to improve the mechanical properties and relieve residual stress. In addition, the rolling process can alter the bead geometry and morphology (e.g., flattening the weld bead surface). The development of DED + interlayer rolling, especially cold deformation, began at the Cranfield University WAAM[®] group [66].

Deposition + cold interlayer rolling systems (Figure 2.1) are composed of two major components: a rigid moving system that supports a hydraulic pressure roller and a deposition system (welding torch + wire feeding and accessories). It is worth noting that the deposition system can use different welding processes (e.g., GMA and GTA), while the roller can have multiple profiles (flat and inverted, as detailed in Figure 2.1c). In addition, due to the high material strength at low temperatures (< 200 °C), high rolling forces are used (up to 160 kN) [114], which requires the use of robust and rigid structures, such as hydraulic systems. The main disadvantage of this interlayer rolling system architecture is its low versatility, which limits the part geometry and path planning. For example, the roller is restricted to approximately linear path planning (e.g., back and forth), differing from the deposition system (welding machine coupled to a robot arm or CNC machine), which allows an almost free path planning. Thus, the roller limits the deposition path planning flexibility and deposition strategies of the DED systems [115], [116].

Despite the abovementioned limitations, Hönnige et al. [117], [118] developed the side cold rolling technique, which applies a side surface rolling using a rigid support block. Figure 2.2 details the influence of the rolling force and rolling direction on the macroscopic aspect of the wall. Figure 2.2a–d demonstrates that when the rolling is applied in the top layer surface, a flat top layer is generated, followed by an increase on the layer width (volume conservation). Conversely, when rolling is applied to the side of the deposited part (Figure 2.2e–h), a reduction

in the part waviness and layer width is observed. Additionally, an increase in the rolling load results in greater deformation. Williams et al. [1] reported that the waviness of the DEDed-arc part surface limits its use in the as-built condition for structural applications due to the geometric stress concentration (i.e., reducing the fatigue resistance), requiring a post-deposition surface machine. Thus, as interlayer rolling reduces the waviness (Figure 2.2), the machining time and tool-life can be improved [17], [119]. Also, according to Dirisu et al. [114], the interlayer cold rolling promoted residual stress relief, refined the microstructure, and enhanced fatigue resistance.

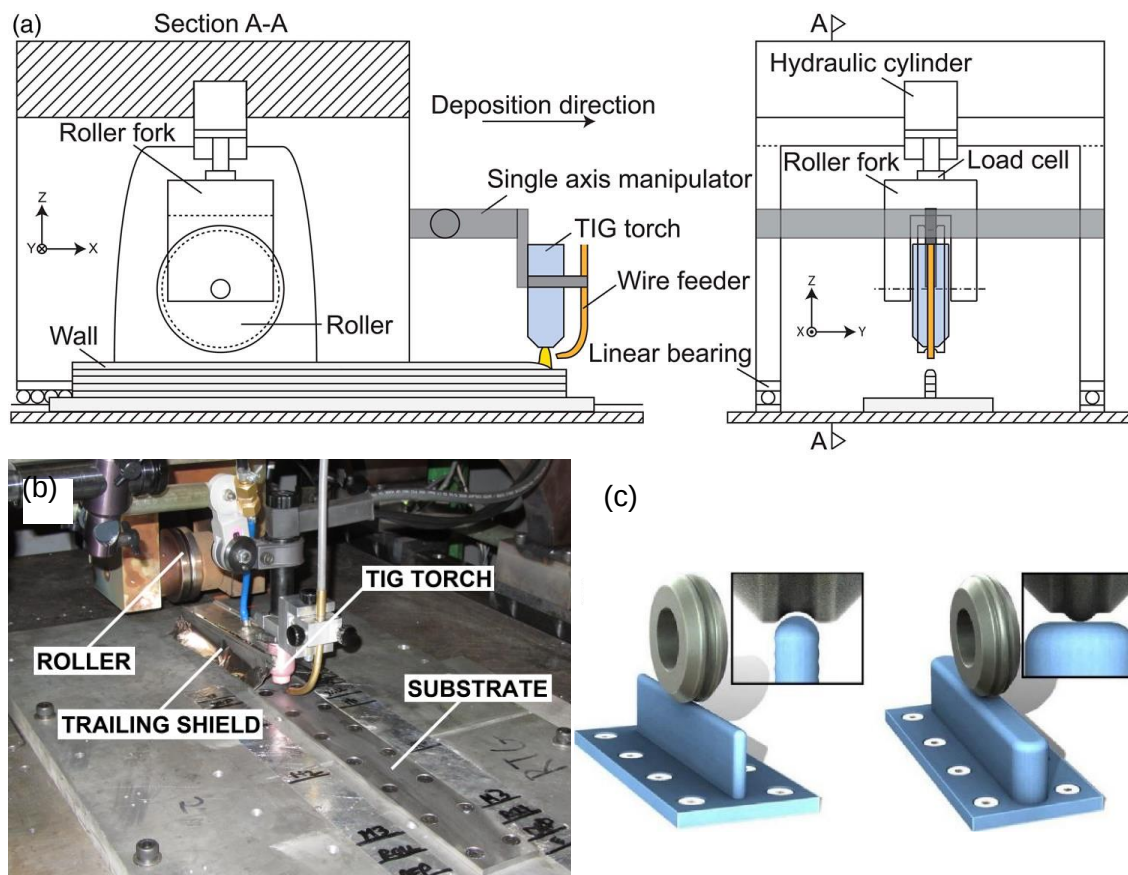


Figure 2.1 - Rolling + deposition experimental set-up: (a) schematic representation, (b) real image, and (c) different roller profiles (adapted from [116], [120], [121]).

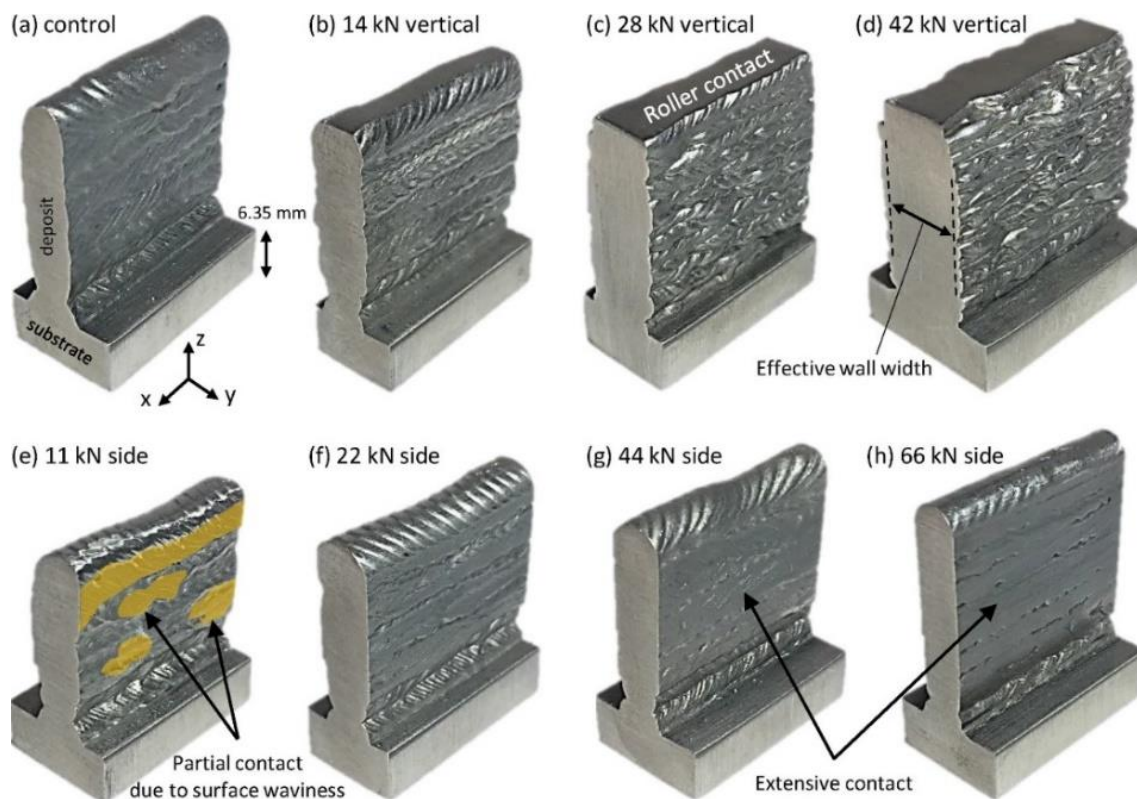


Figure 2.2 – Effect of rolling on part superficial aspect: (a) control sample, (b–d) vertical rolled, and (e–h) side rolled specimens (from [118]).

Gu et al. [122] reported that cold rolling also closed porosity during the deposition of aluminum alloys. The authors [122] showed that pore closure increased with the imposed rolling load. Figure 2.3 illustrates the inner morphologies of the flattened pores. In addition, the fraction of the smaller pores (radius lower than 2 mm) was reduced after rolling. However, pores with diameters greater than 5 mm and those located deeper in the solidified material, close to the fusion line, could only be closed with significantly higher rolling forces, resulting in considerable layer deformation. Additionally, as reported by Bercelli et al. [123], the presence of pores can drastically reduce the fatigue life of parts fabricated by DED-arc; these results were corroborated by Dirisu et al. [114], which observed that the fatigue life of the low-alloyed steel (AWS ER70S-6) fabricated via DED-arc + interlayer cold rolling increased with the interlayer rolling load, i.e., a higher rolling load reduces the pore volume, which increases the fatigue life.

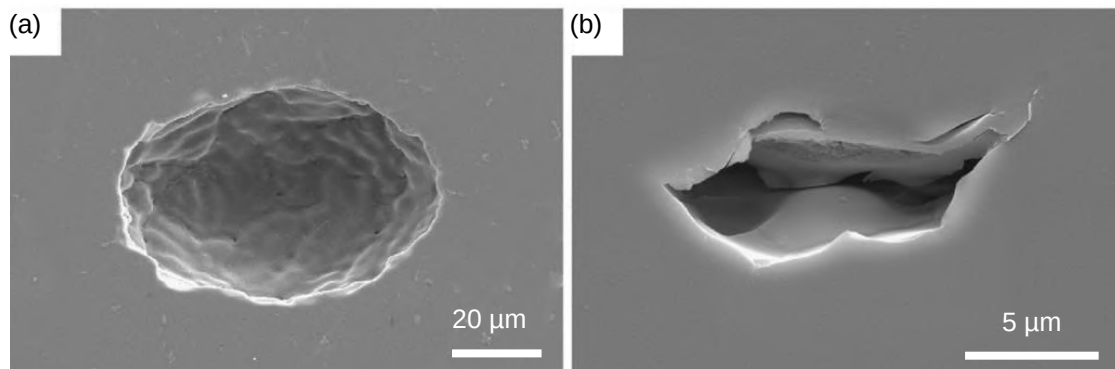


Figure 2.3 – Effect of rolling load on pore morphology: (a) 15 kN and (b) 30 kN (adapted from [122]).

Despite the effective grain size refinement and residual stress relief effects, the large equipment dimensions and limited path planning flexibility limited the interlayer cold rolling. In this context, other systems were developed, which considered the in situ deformation, i.e., immediately followed the feedstock material consolidation with the material still incandescent (i.e., hot deformation). Deforming the material at relatively elevated temperatures enables the use of lower rolling loads, simpler and less expensive systems, and increases process adaptability. However, other process challenges arise, such as the accurate rolling temperature control (distance between the heat source and the deformation tool), depth of recrystallized zone (ideally deeper than the penetration of subsequent layer), and the preservation of a flat and smooth layer surface.

Zhang et al. [124] developed a DED-arc + interlayer hot rolling system (Figure 2.4a), which consisted of a heat source (GMAW) and a micro-roller coupled with a hydraulic cylinder, both fixed and connected to a three-axis computer numerical control (CNC) machine. Therefore, deposition and subsequent deformation can follow the same path for simple curves trajectories (Figure 2.4b) and ensure a flat top surface. In addition, Zhang et al. [124] detailed the effects of the distance of the roller to the heat source (i.e., rolling temperature) on the final surface aspect (Figure 2.5). For short distances (Figure 2.5a), deformation occurs in or near the mushy zone (solid + liquid coexist), which can lead to material adherence (viscoplastic behavior) and potential fracture (elevated temperature ductility loss) at higher temperatures resulting in a poorer surface quality. By contrast, for longer distances (Figure 2.5c), the total deformation may be insufficient due to the high cooling rate (the just consolidated material strengthens as the temperature drops) and lower hot rolling loads (~ 3 kN). Hence, achieving precise process optimization becomes imperative. This must consider each material/deposition parameter pair, as well as the precise adjustment of the distance between the heat source and deformation tool to effectively refine the microstructure [125].

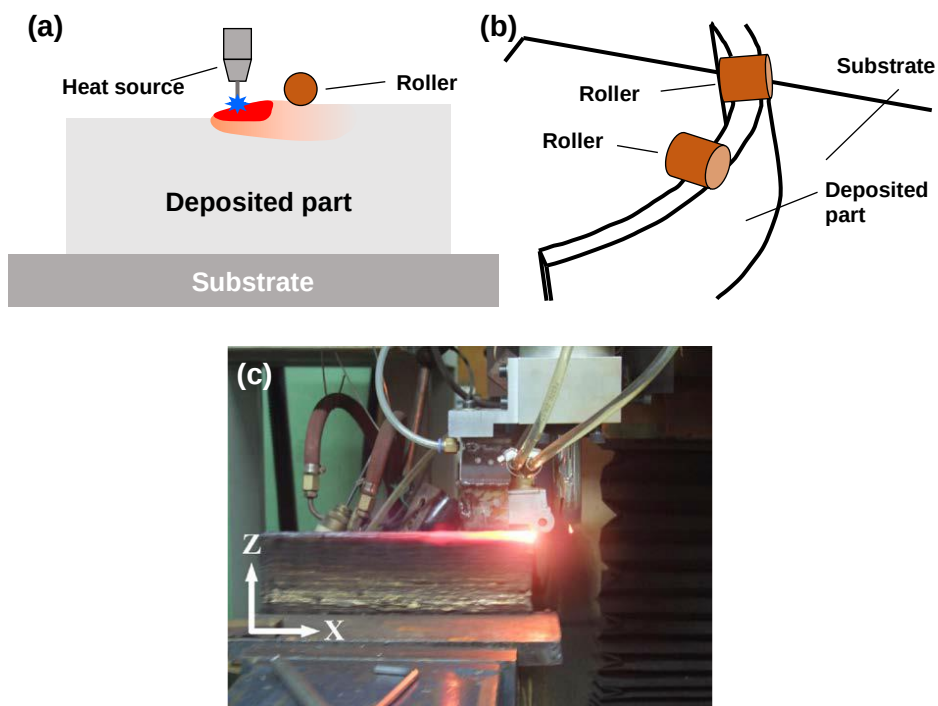


Figure 2.4 – (a) Deposition + interlayer hot rolling system, (b) similar path planning for both roller and heat source, and (c) system in operation (adapted from [124])

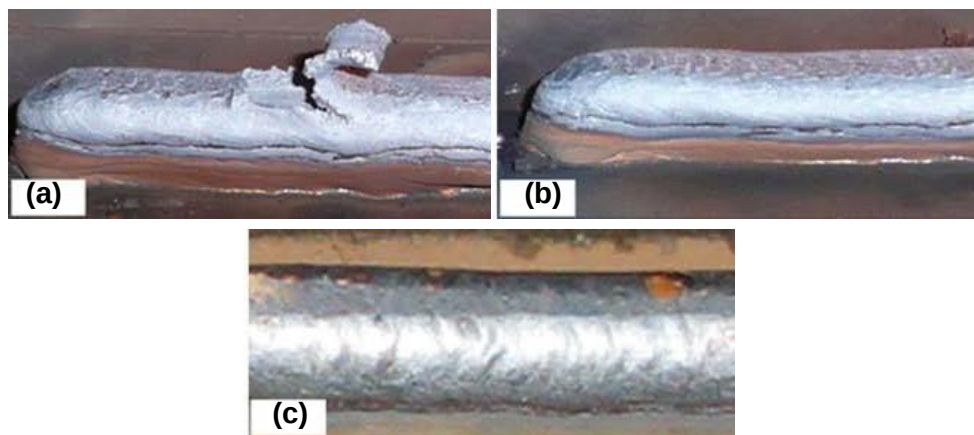


Figure 2.5 – Final surface aspect for different roller-heat source distances: (a) low, (b) appropriate, and (c) high (adapted from [124]).

Although the system developed by Zhang et al. [124] (Figure 2.4) provided good results, the control of layer width throughout the printed part was not guaranteed by the interlayer hot rolling because the thermal conditions (e.g., heat accumulation and interlayer temperature [67]) changed during deposition. Xie et al. [126] improved the layer geometrical accuracy using a metamorphic rolling mechanism (Figure 2.6), which consists of a system that is similar to that of Zhang et al. [124] (Figure 2.4) with the addition of two exchangeable side rollers. These side rollers improve the layer geometrical accuracy for multi-layer single-bead

and/or multi-bead DEDed parts. In addition, Xie et al. [126] reported that the absolute error in layer height remained constant during deposition, allowing an automatic correction of the contact-tip-to-work distance. Other authors [127]–[131] also developed deposition + interlayer hot rolling systems; however, the main idea and mechanism were similar to that originally proposed by Zhang et al. [124].

Zhao et al. [132] modeled the bead overlapping for Zhang et al. [124] system (Figure 2.4) using the bead-on-plate geometric fitting (sine function bead approximation) [133] and flat-top overlapping models [134]. The results showed that the best overlapping had a second-order polynomial correlation with the rolling deformation. For a deformation of 36.6% (layer height reduction), the optimized overlap is 74.24%. This value aligns closely with the multi-bead tangent overlapping model (73.8%) proposed by Ding et al. [135]. In addition, Zhao et al. [132] demonstrated that the bead overlapping performance, specifically its susceptibility to lack of fusion, remained unchanged even after layer surface flattening through interlayer hot rolling, corroborating the observation made by Xie et al. [126] regarding the ability of interlayer hot rolling [124] to print thick-sectioned parts (Figure 2.6).

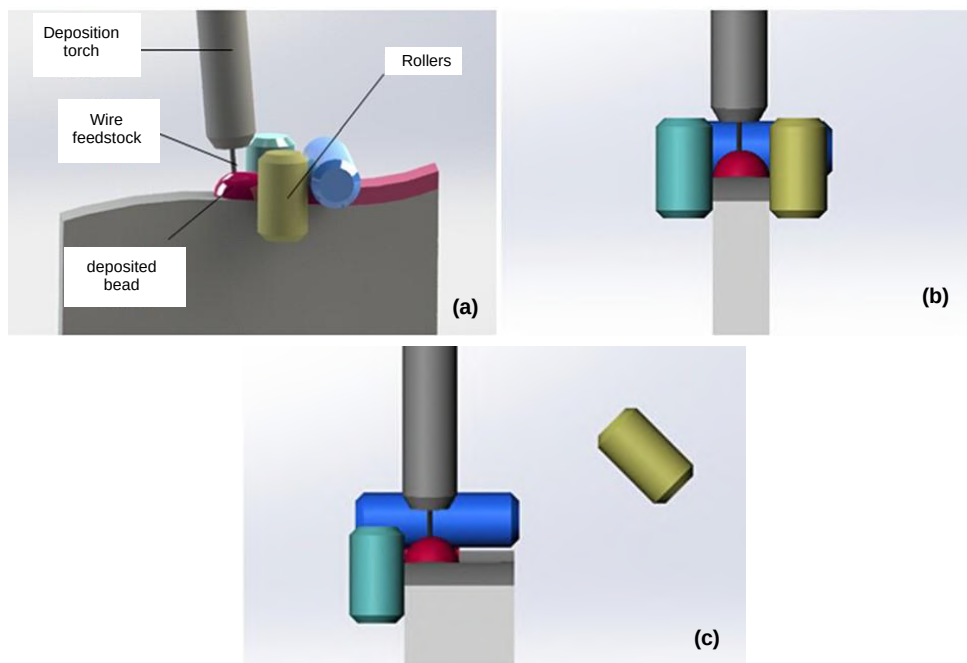


Figure 2.6 – (a) schematic diagram of the metamorphic rolling mechanism, (b) deposition of the multi-layer single-bead (thin wall), and (c) metamorphic rolling mechanism applied to multi-layer multi-bead part deposition (adapted from [126]).

The abovementioned deposition + interlayer (hot or cold) rolling systems used arc plasma as a heat source (i.e., typical welding machines). Li et al. [136] and Tain et al. [137] developed a deposition + hot rolling system (Figure 2.7) using a laser and powder as the heat

source and feedstock material (i.e., DED-laser), respectively. As opposed to the arc plasma-DED + interlayer rolling systems, in the systems proposed by Li et al. [136] and Tain et al. [137], the moving part was the substrate whose movement was controlled by three step motors (x, y, and z directions). The strain and strain rate were controlled via the z-axis movement (built direction) and the moving speed of the substrate, respectively. For the deposition of Ti-6Al-4V alloy and Ni-based superalloy 718, the load cells registered a rolling load of 1.3 and 3 kN, respectively, which are significantly smaller than the rolling loads used during cold rolling (up to 160 kN) [66], [115], [138], [139] and similar to that reported by Kan et al. [140] and Zhang et al. [124] (Figure 2.4). These rolling loads are even considerably lower than warm ($\sim 450\text{ }^{\circ}\text{C}$) rolling (50 kN) [110] applied to Ni-based superalloy 718 arc plasma DED, which reinforces the importance of a precise temperature control (distance between the heat source and deformation tool).

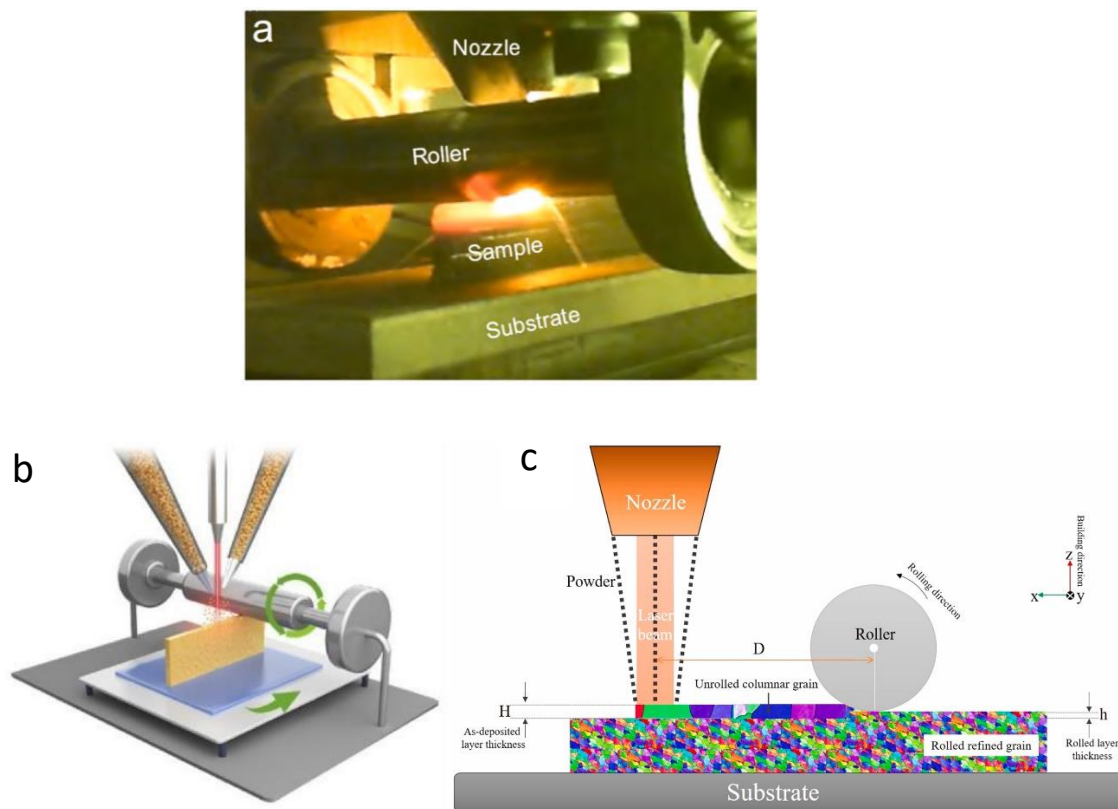


Figure 2.7 – (a) DED-laser + interlayer hot rolling, (b and c) schematic illustration (from [136], [137]).

Finally, both cold and hot interlayer rolling systems exhibited limitations in path planning, as the rolling system cannot rapidly alter the direction, thus constraining deposition freedom. However, due to its smaller equipment and lower rolling load, the interlayer hot rolling system could follow the heat source path along smooth curved trajectories (high radii of curvature). Conversely, the uncoupled characteristics of cold rolling system, where the heat

source and deformation parameters are independent, did not restrict the process window, allowing an improved layer aspect control (e.g., width and height). For instance, during part thickness transition, the deposition parameters can be adjusted without constraints. The precise control of the heat source-deformation tool distance in the interlayer hot rolling system restricts the deposition parameter window [141], making it difficult to control the geometric aspects of the layer during deposition. Thus, cold rolling had better deposition parameter control and limited path planning (almost restricted to linear deposition); by contrast, hot rolling showed more freedom in path planning and restrictive and difficult-to-control deposition parameters.

2.1.2.2 Hammering, Peening, and Forging

Similarity to rolling, peening/hammering/forging coupled with deposition also originated from arc-based welding [38], [142], [143]. In addition, to the best of the authors' knowledge, the Cranfield University WAAM group also pioneered the coupling cold peening/hammering with DED-arc. This innovation arose from the limitations of the aforementioned cold rolling system, specifically the heavy equipment and design constraints. Hönnige et al. [144], [145] developed a DED-arc + interlayer cold hammer peening variant to reduce residual stresses through surface compression plastic deformation, a mechanism previously described by Kobayashi et al. [146]. Hammer peening works with the tool (hammer) mounted on a robot or CNC machine using a moderate pin oscillation frequency (~ 200 Hz), enhancing the flexibility of the process in relation to rolling. Figure 2.8 illustrates the equipment and the possibility of their use in distinct positions (vertical, horizontal, and inclined) with complex path planning. These tool operated using an electromagnetic plunger coil (pneumatic system), delivering an impact energy of 750 mJ, thereby ensuring deep deformation zones [147]. Fang et al. [148] noted that coupling a pneumatic hammer with DED-arc can minimize porosity and alter pore shapes in Al alloys. Further, Shchitsyn et al. [149] revealed that Ti-6Al-4V alloy deposited using the cold metal transfer (CMT) and interlayer cold hammered met the mechanical strength of wrought Ti-6Al-4V.

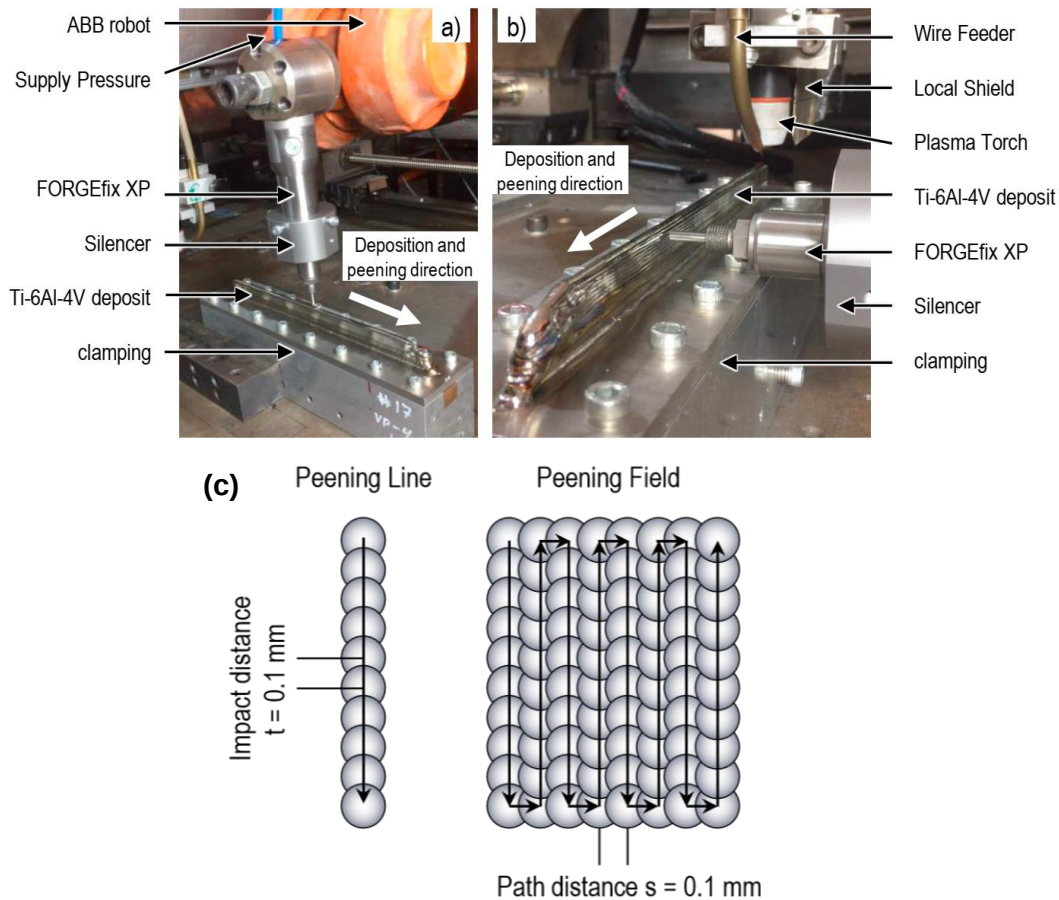


Figure 2.8 – setup of machine hammer peening: (a) vertical peening, (b) horizontal, and (c) flexibility path planning (adapted from [144]).

Similar to hammer peening, ultrasonic peening (Figure 2.9) is also a traditional deformation method used to reduce residual stresses and improve the mechanical properties and fatigue life of the welded joints [150]. An ultrasonic probe with a power of 100 W working at a high frequency ($\sim 20 \text{ kHz}$) was commonly used. Aligned steel impact tools with a needle-like aspect (diameter of 3 mm and spaced 2 mm; Figure 2.9b) deformed the bead surface. Gale and Achuhan [151] (AISI 316L stainless steel DEDed-laser) combined cycles of deposition and deformation. These authors deposited a specific sample height (2 layers) and subsequently conducted ultrasonic peening on the as-built material, observing a significant increase in the material hardness (from 2.4 to 4 GPa). Wang and Shi [152], [153] demonstrated that cold ultrasonic peening homogenized the residual stress distribution, altered the main residual stress component to a compressive state, and induced a finer microstructure of Inconel 718 DEDed-laser. Yi et al. [71] showed that even for the high penetration process (arc plasma-DED + interlayer ultrasonic peening –Figure 2.11), the ultrasonic peening mechanically affected zone (deep deformation) could induce microstructural refinement. In addition, due to the small

equipment size (Figure 2.9b) and low peening load (e.g., operated manually in welding), ultrasonic peening can be integrated within a gas protection chamber, allowing it to be coupled with the powder bed fusion (PBF). Zhang et al. [154] verified that interlayer cold ultrasonic peening could reduce and close discontinuities (Figure 2.10) by inducing top layer deformation, subsequently crushing and altering defects (e.g., porosity and lack of fusion) morphology, similar to observed for interlayer rolling (Figure 2.3) [122]. In addition, it was verified that interlayer cold ultrasonic peening prevents epitaxial columnar grain growth (refining the microstructure) and reduces the residual stress levels for Ti-6Al-4V fabricated via PBF. Therefore, ultrasonic and hammer peening can be considered suitable for commercial alloys DEDed.

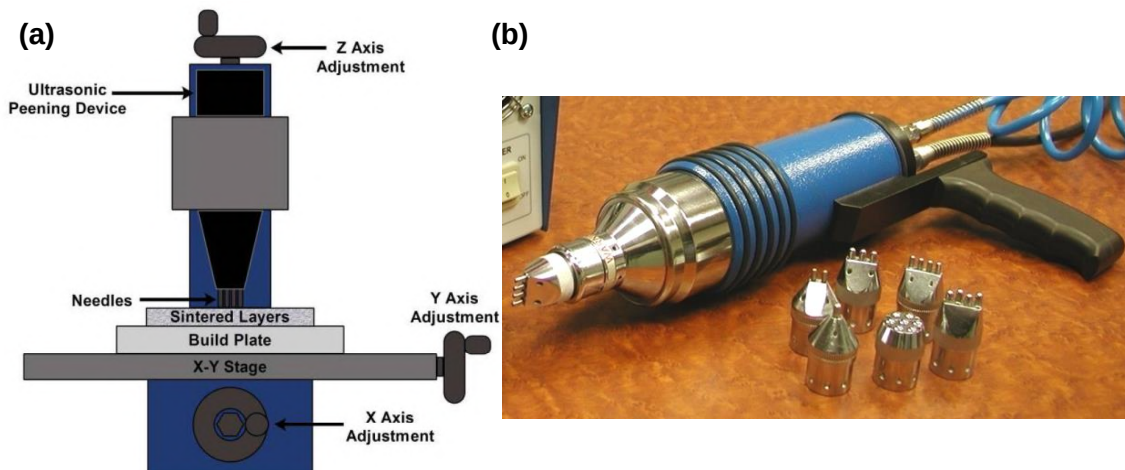


Figure 2.9 – (a) Schematic view of the ultrasonic peening setup (adapted from [151]) and (b) impact tools “needles” (adapted from [155]).

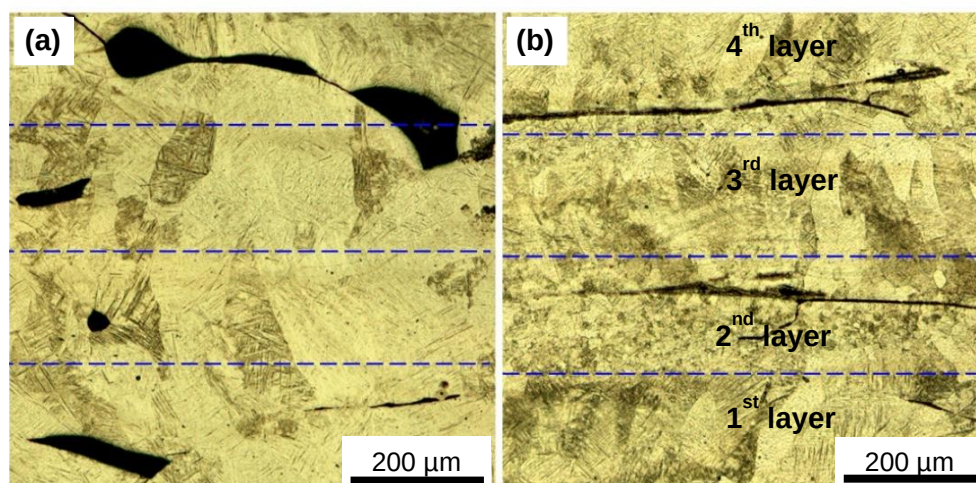


Figure 2.10 – Effect of ultrasonic peening on the PBF discontinuities: (a) without and (b) with ultrasonic peening (from [154]).

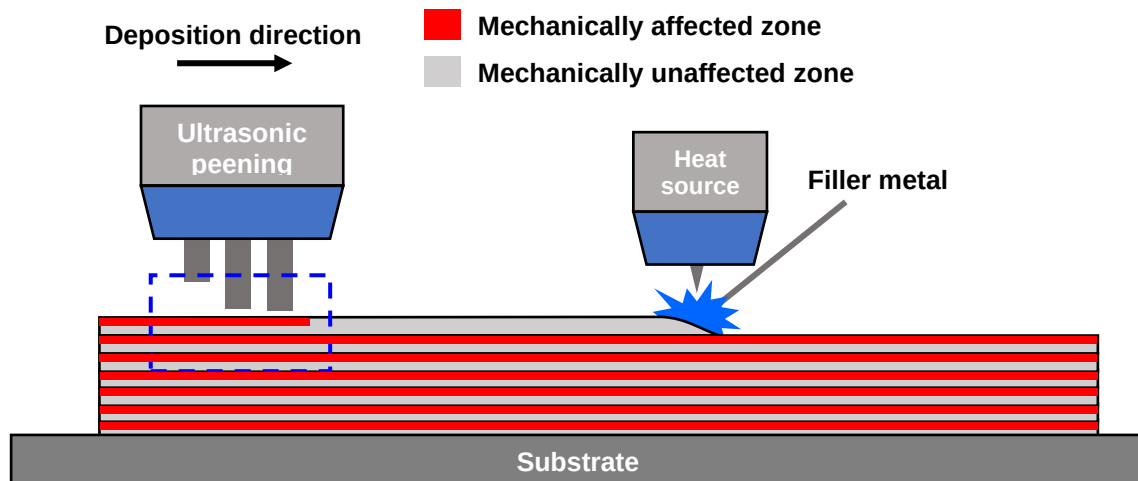


Figure 2.11 – Schematic representation of ultrasonic peening coupled with DED-arc (adapted from [71]).

Ye et al. [156] (DED-laser) and Li et al. [157] (DED-laser) developed a hot ultrasonic micro-forging system with an operating frequency of 20 kHz; Xiong et al. [158] (DED-arc), a hot hammer penning system with an operating frequency of 21 Hz. These systems [156]–[158] (Figure 2.11) resemble the cold hammer previously shown in Figure 2.9b, where the main difference between them is the peening temperature. Deformation occurred immediately after deposition, when the deposited layer was still at a higher temperature, as with those for the hot rolling variants. The abovementioned systems involve a conventional welding torch coupled with a separated plastic deformation device, i.e., uncoupled deposition and deformation.

Duarte et al. [35], [36] developed a DED-arc + interlayer hot forging variant (Figure 2.12a and Figure 2.12b), which consisted of a dedicated GMAW torch equipped with a hammer, activated by vibrating actuators (electromagnetic, solenoid, or pneumatic) that could operate at different frequencies (1 – 10 Hz) and loads (1.5 – 5 kN). The hammer path planning followed the torch, and the hammer size and distance from the welding pool control the forging temperature. Duarte et al. [35] and Farias et al. [159], [160] showcased that hot forging could reduce pore size, alter its morphology, and improve the mechanical properties of 316L stainless steel and Inconel 625 DEDed-arc. In addition, Gao et al. [161] customized a machine that combined DED-arc and single-point incremental hot forming, which operated at a maximum load of 1.5 kN, refined the microstructure, and improved the mechanical properties of 316L stainless steel DEDed-arc.

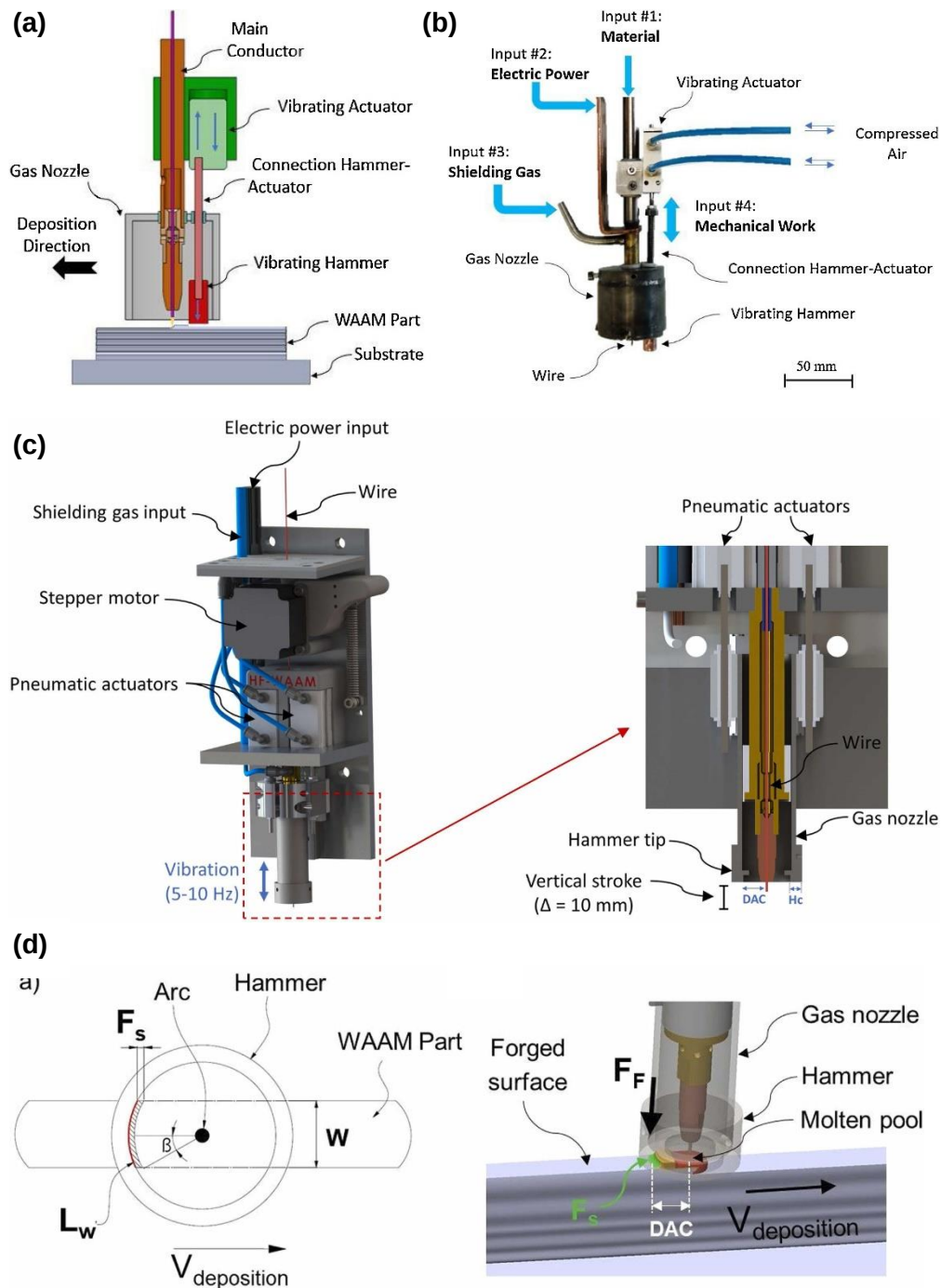


Figure 2.12 – DED-arc + interlayer hot forging: (a) schematic representation and (b) components (from [35]). (c) developed of coaxial hot forging, and (d) details of the circular crown concentric with the welding nozzle (from [36]).

Hammering, peening, and forging (cold and/or hot) showed advantages in relation to rolling, especially due to the lighter equipment, higher versatility and flexibility, lower loading forces, and freer path planning. However, the hot interlayer hammer peening and forging are limited to one-way deposition strategies, as they are challenging to use in oscillating (e.g., the deposition torch moves as a sine/square/triangle wave) and/or reversing deposition

strategies [67], e.g., even layer number from right to left ($\rightarrow$) and odd number layers with a reversed direction ($\leftarrow$). In this sense, Duarte et al. [36] improved their previous interlayer hot forging system [35] by changing the configuration of the forging tool from a cylindrical hammer to a circular crown shape directly connected to the welding gas nozzle (Figure 2.12c). Thus, the deposition and deformation became coaxial, thereby overcoming the path planning limitations, i.e., the forging region is independent of the direction of deposition (Figure 2.12d). Finally, for the first time, Karunakaran et al. [162] and Gupta et al. [163] integrated an interlayer cold hammering/peening system into a hybrid 3D printing machine (DED cell – laser, GTA, and GMA – and machining setup). These authors noted that hammering/peening/forging could be directly integrated into the manufacturing setup without altering it significantly, i.e., they did not need a specific manufacturing setup. This differs from the other interlayer mechanical deformation systems, such as laser shock peening and hot/cold rolling, which demand a dedicated setup.

2.1.3 Grain size refinement mechanisms

2.1.3.1 Rolling

2.1.3.1.1 Transformable solid-state alloys

The thermal conditions (intense thermal gradient and almost directional heat flux) and partially remelting of the substrate and/or previously deposited layer favor the epitaxial grain growth during DED deposition, which is commonly characterized by an oriented microstructure and anisotropic behavior, even for solid-state transformation alloys (e.g., Ti-6Al-4V). In this sense, the deposition + interlayer mechanical deformation can induce grain refinement (dynamic and/or static recrystallization), which can reduce the texture index (randomizing the microstructure) and homogenizes the properties throughout the part fabricated via DED.

Most studies on the effect of rolling on grain refinement focus on the Ti-6Al-4V alloy; however, the key grain refinement mechanism can be extended to other transformable alloys. The Ti-6Al-4V alloy solidifies as coarse and oriented β (body-centered cubic) grains, which during the cooling ($\sim 1000^\circ\text{C}$; β *transus* temperature), partially transforms into α (hexagonal close-packed) so that the final microstructure is composed of the α -phase (e.g., Widmanstätten and grain boundary) and some retained prior β -phase. For more details regarding the welding metallurgy of $\alpha + \beta$ Ti alloys, readers are referred to Short [164]. Similar allotropic transformations are observed in materials such as low-alloy steel (austenite $\rightarrow$ ferrite) and Cu-Al8% ($\beta \rightarrow \alpha$) [36], for example.

Martina et al. [139] observed that the prior β grain size of the Ti-6Al-4V reduced significantly (Figure 2.13) due to interlayer cold rolling. This grain refinement effect was notable when using a higher rolling load (75 kN). In addition, Martina et al. [139] tested the influence of the posterior layer on a previously rolled layer (Figure 2.14), identifying three zones: (1) columnar grains, (2) recrystallized zone, and (3) previous unaffected layer. In Figure 2.14, zone (1), closer to the top of the deposited layer, exhibited the columnar prior β grains aligned with the build direction. Zone (2) corresponds to the grains that grew epitaxially and were deformed (previous layer) and, in the subsequent layer deposition (reheating thermal cycle), statically recrystallized. Thus, despite zone (1) possessing a columnar aspect, the finer grains in the zone (2) inhibited prior β grains to cross several layers (some grains could reach up to 12 mm in the DED-arc condition – Figure 2.13a). The length of zone (3) depends on the deposition and deformation parameters since the recrystallization temperature is related to, in addition to other factors, the deformation stored energy, temperature, and time [165], where the first is a function of the rolling load and the others of the thermal cycle (i.e., deposition parameters). It is worth noting that, due to the higher cold rolling loads, the remaining deformation zone length – zone (3) – is superior to deposition penetration (Figure 2.14), which ensures an effective prior β grain size refinement mechanism in the entire deposited layer.

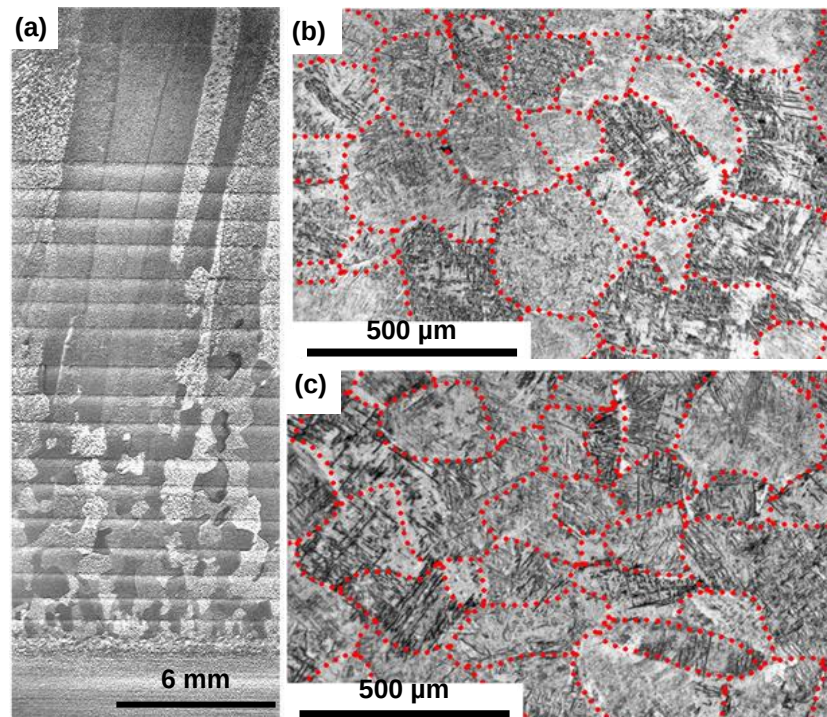


Figure 2.13– Prior β grain morphology of the Ti-6Al-4V DEDed-arc: (a) unrolled condition, (b) 50 kN rolling force, and (c) 75 kN rolling force (from [139]).

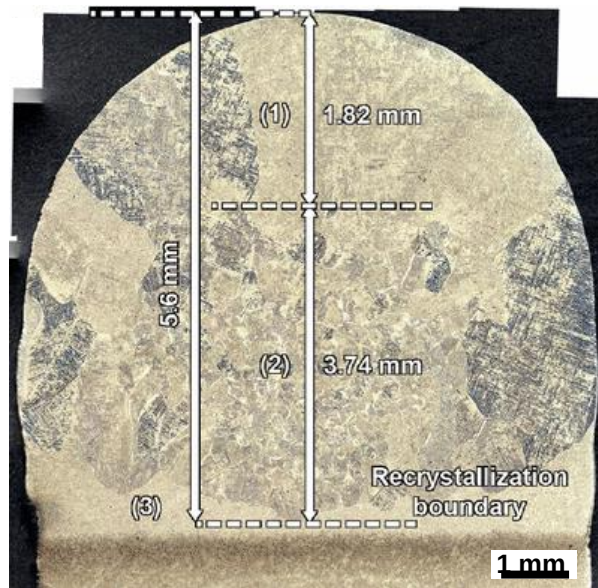


Figure 2.14 – Effect of the posterior deposition layer on the previous layer. (1) columnar grains, (2) recrystallized zone, and (3) previous unaffected layer (from [139]).

McAndrew et al. [116] reported that, for multi-layer multi-bead DEDed-arc parts, the grain size refinement, as observed by Martina et al. [139] (multi-layer single-bead), effectively did not occur. This was attributed to the lateral geometrical restraint of the multi-bead multi-layer thick part, i.e., the adjacent layer previously deposited prevents the deformation of the most recently deposited layer, inducing an insufficient stored energy required for static recrystallization. However, using an inverted roller profile (Figure 2.1c; deeper deformed zone), enough stored energy to promote static recrystallization can be achieved, expanding the interlayer cold rolling grain size refinement in thicker parts. McAndrew et al. [116] mapped the deformation (Figure 2.15) induced by rolling, showing that the maximum strain occurred at the edges of the rolled layer at a depth of 3 mm; close to the surface, the deformation was minimal due to friction between the roller and the surface, preventing material flow. Figure 2.15 also shows that the deformation penetration can extend up to 6 mm so that the deposition penetration (~ 1 – 2 mm) is always smaller than the deformation zone, which reinforced the results showed by Martina et al. [139]. Donoghue et al. [166] observed that the electron backscatter diffraction (EBSD) band contrast reduces (i.e., increase in the residual strain), while posteriority increases until reaching a constant value, corresponding to the region unaffected by cold rolling. Figure 2.16a and Figure 2.16b evidence that the maximum static prior β recrystallization coincides with the lowest band contrast region (highest deformation zone) and that the grain size refinement effect increases with the rolling load. Figure 2.16c and Figure 2.16d illustrate the effect of interlayer cold rolling in deposited layers, which indicated that the

grain size was refined throughout the part. Moreover, the lower rolling load (50 kN) generated a higher and scattered grain size (Figure 2.16e), i.e., it was less efficient.

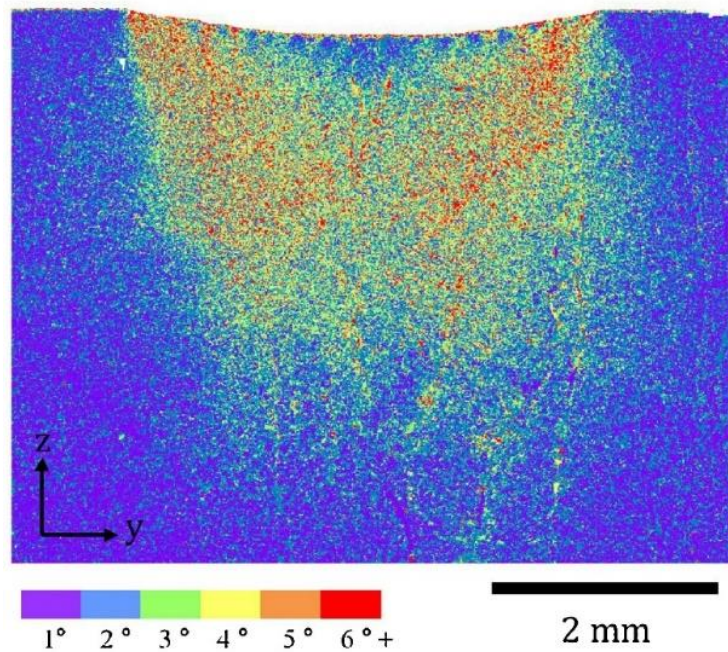


Figure 2.15 – EBSD map of the plastic strain of a Ti-6Al-4V DEDED-arc rolled (from [116]).

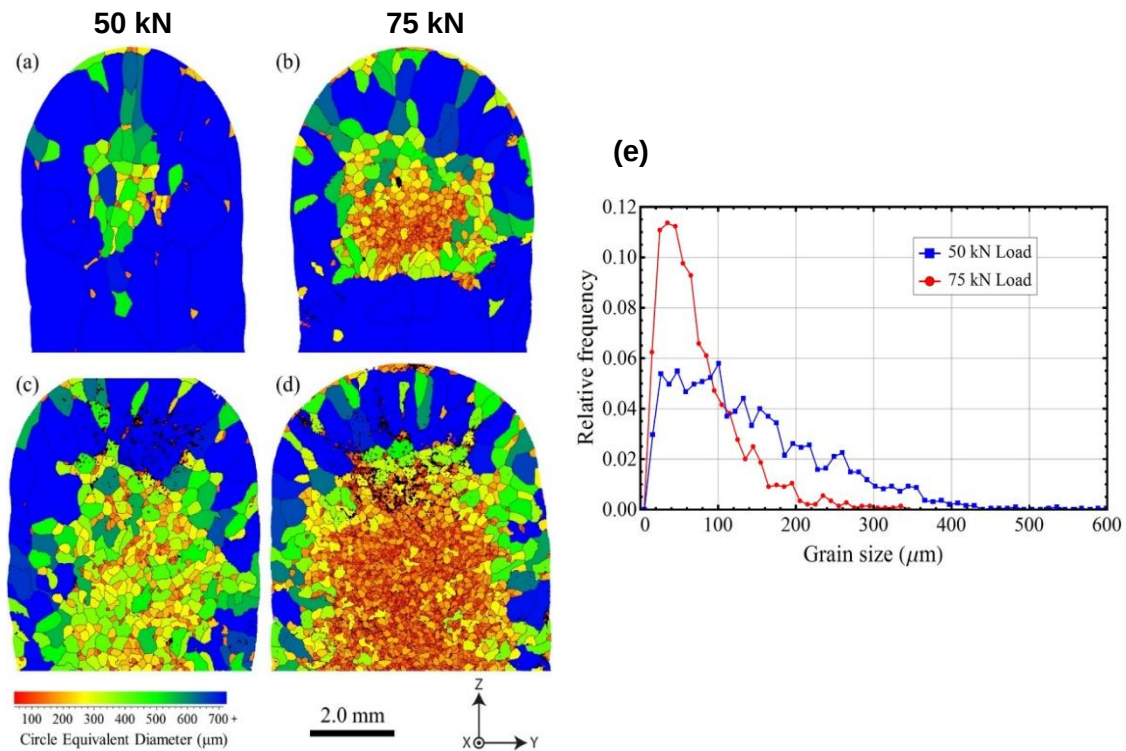


Figure 2.16 – Prior β grain size maps: (a) and (b) rolling applied in the penultimate layer; (c) and (d) rolling applied in the entire part; ϵ grain size distribution (from [166]).

According to Ding and Guo [167], the macroscopic compression (height reduction of 19% for 75 kN rolling load) observed by Donoghue et al. [166] is insufficient to induce the prior β grain refinement via static recrystallization during the reheating thermal cycle. Furthermore, β nucleation follows the Burgers orientation crystallographic relationship, which ensures that, during heating ($\alpha \rightarrow \beta$ allotropic transformation), the new generated β grains have the same crystallographic orientation of those prior β generated during solidification. This allows the next deposited layer to have almost the same crystallographic orientation (epitaxial nucleation and growth) as the previously deposited one, resulting in a typical coarse and texturized prior β grain [164]. For effective prior β grain size refinement, the interlayer rolling must induce the β nucleation without following to Burger's orientation [168]. Donoghue et al. [169] and Davis et al. [170] reported that interlayer rolling can induced the twinning of the remaining prior β (β' , Figure 2.17), which explains the prior β grain size refinement in DEDed-arc + interlayer cold rolling. The deformation induced the twinning of β (β'), which had a mobile grain boundary and an orientation different from the remaining β phase (Figure 2.17c), i.e., it did not follow the Burgers crystallographic relationship. Therefore, when the β' grains growth, they consume the prior β and α during the reheating thermal cycle. In addition, the β' grains growth is impinged by other β' grains (Figure 2.17d), which results in a fine and non-oriented microstructure. This proposed mechanism [169], [170] is in accordance with Martina et al. [139], which showed that a higher rolling load resulted in a finer β grain size, i.e., the higher rolling loads enhanced the twinning of prior β (β' formation) that growth without following the Burgers orientation.

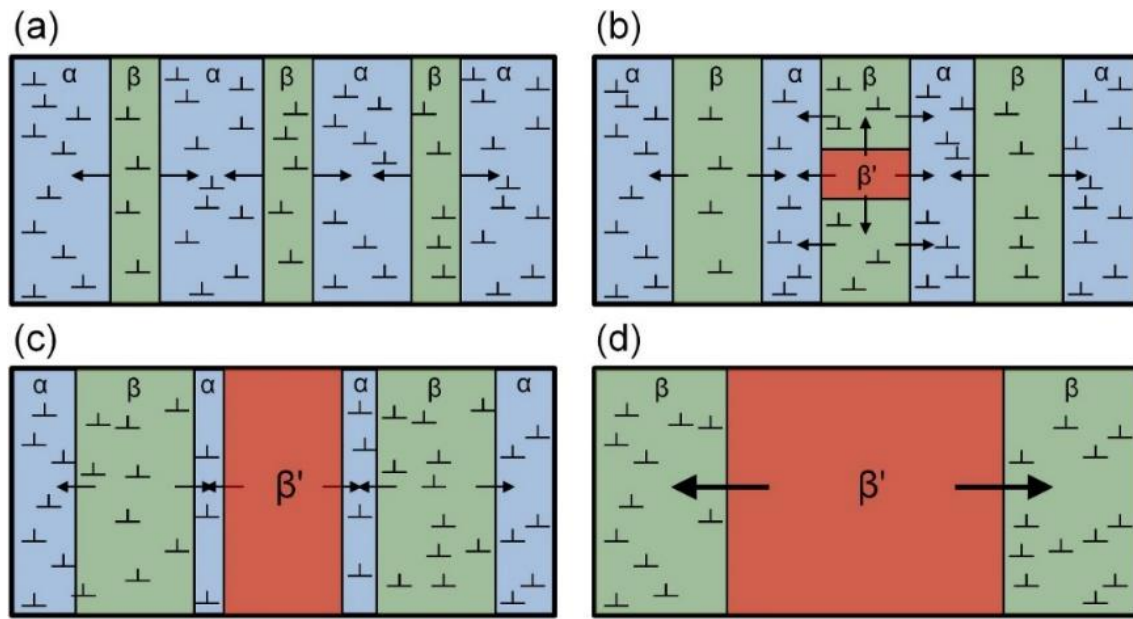


Figure 2.17 – β -grain refinement mechanism for Ti-6Al-4V DEDed-arc rolled: (a) stored dislocations due to cold rolling, (b) twinning β -grain (β') during heating thermal cycle, (c) β' grows into the neighboring deformed β phase, and (d) $\alpha \rightarrow \beta$ solid-state phase transformation and β' growing (from [169]).

In addition to the Ti-6Al-4V alloy, Colegrove et al. [66] and Dirisu et al. [114] also observed refinement of the parent phase (prior austenite) as a result of interlayer cold rolling in low-alloyed steel fabricated via DED-arc. The changes in the prior austenite grain size alter the kinetics of the austenite-ferrite phase transformation. With a finer grain size induced by interlayer cold rolling, diffusional transformations, such as ferrite formation (e.g., polygonal and acicular), is favored over displacive transformations (e.g., martensite). In other words, the finer prior austenite grain size reduces the hardenability of low-alloyed steel fabricated via DED-arc. As observed with Ti-6Al-4V (arc plasma DED + cold rolling), an increase in the rolling load also leads to finer prior austenite grain size. However, the thermal conditions during the thermal cycles were only slightly altered, which results in almost identical constituents (polygonal and acicular ferrites, low-alloy steel, and basketweave α , Ti-6Al-4V) for both conditions (interlayer rolled and unrolled). Due to the finer austenite grain size, the rolled condition showed a finer ferrite grain in relation to unrolled (8.6 vs. 14.5 μm) [114]. Colegrove et al. [66] suggested that the refinement of austenite grain size resulted from a high nucleation rate induced by the residual deformation. However, this phenomenon has not been thoroughly studied and requires further clarification. Given the low deformation during cold rolling (similar to Ti-6Al-4V), the crystallographic relationship between austenite and ferrite (e.g., Kurdjumov-Sachs), and the presence of residual austenite at room temperature (as observed in martensite-austenite constituents in weld metals) [33], it is possible that similar behavior of the prior β

grain refinement (Figure 2.17), i.e., retained austenite twinning, can occur for low-alloyed steels. Thus, interlayer cold rolling may not induce a higher nucleation rate of austenite during the heating as indicated by Colegrove et al. [66] but induces the twinning of the retained austenite, which will grow during reheating thermal cycle without following the Kurdjumov-Sachs orientation relationship.

Tian et al. [137] (DED-laser) and Gao et al. [131] (DED-arc) observed (Figure 2.18) a considerable prior β grain refinement, similar to cold rolling, using interlayer hot rolling, with a remarkable β phase texture intensity reduction (from 15.8 to 3.2). Gao et al. [131] explained the grain size refinement using the mechanisms proposed by cold rolling [169], [170] (Figure 2.17). In addition, Gao et al. [131] reported that dislocations, twins, and stacking faults are introduced during interlayer hot rolling, which supplies sufficient driving force for dynamic recrystallization in regions with peak temperatures higher than β *transus* and supported the grain size refinement mechanism proposed by Donoghue et al. [169] and Davis et al. [170].

Fu et al. [127] studied the behavior of bainitic steel parts fabricated via DED-arc + interlayer hot rolling (see Figure 2.4). Fu et al. [127] made an observation similar to Martina et al. [139] (interlayer cold rolling) regarding deformation-affected zones induced by interlayer hot rolling, i.e., a large deformation, small deformation, and undeformed zones. Furthermore, the selected roller-torch distance with a rolling temperature of 900 °C allows deformation but not austenite recrystallization (non-recrystallization temperature). This introduces deformation bands (austenite had a low SFE energy) that increase the ferrite nucleation kinetics during the cooling thermal cycle (similar to thermomechanical controlled rolling [171]), generating a homogenous fine ferrite grain size ($\sim 7 \mu\text{m}$). Ma et al. [130] reinforced the above results; the authors indicated that the roller-torch distance controls the final microstructure. Ma et al. [130], using a concept similar to that applied to arc welding, redefined the zones affected by interlayer hot rolling as a function of the peak temperature, where, at the medium-high-temperature (non-recrystallization austenite temperature), maximum prior austenite grain size refinement was observed due to the reheating thermal cycle ($\alpha \rightarrow \gamma$ transformation) and intense γ nucleation rate (deformation bands) [172]. In addition, the residual stored deformation energy enhances the ferrite nucleation sites; therefore, the synergic effect of finer austenite grain size and higher ferrite nucleation rate allows the development of a finer ferrite microstructure for low-alloyed steels fabricated via DED-arc + interlayer hot rolling concerning DED-arc.

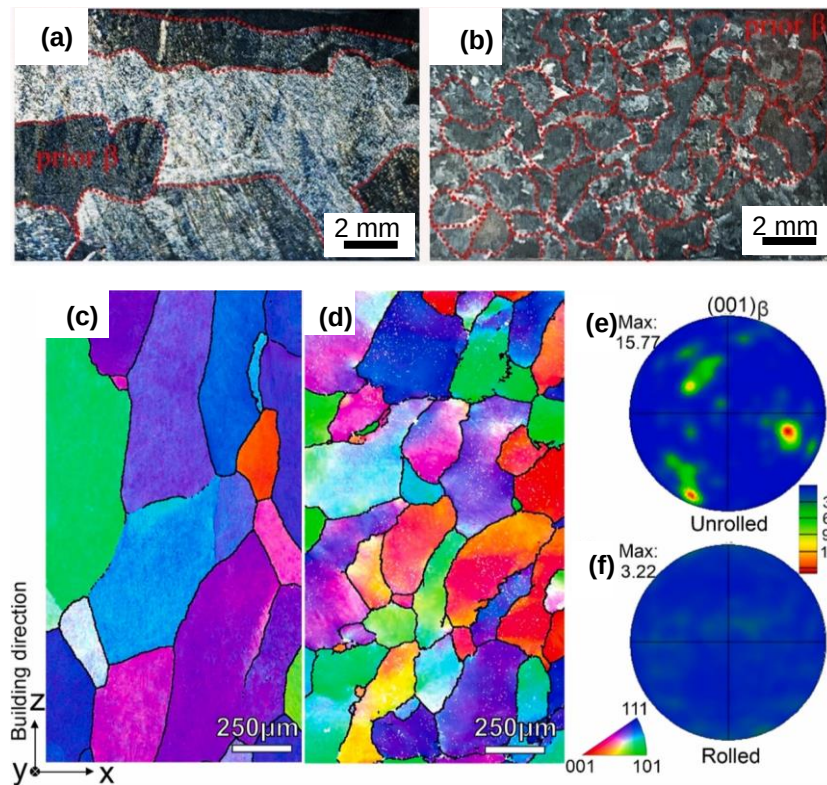


Figure 2.18 – Interlayer hot rolling grain refinement for the Ti-6Al-4V alloy: (a) DED-arc, (b) DED-arc + interlayer hot rolling (adapted from [131]), (c) DED-laser, DED-laser + interlayer hot rolling; (e) and (f) are the prior β pole figures referring to EBSD (c) and (d), respectively (adapted from [137]).

For the solid-state transformation materials, the final microstructure results from the solid-state transformations; thus, despite the interlayer deformation refining the prior microstructure (e.g., β and austenite, for Ti-6Al-4V and low-alloy steels, respectively), it is worth analyzing if the final microstructure is also affected (product phase morphology). In addition, for low-alloyed steels and Ti-6Al-4V, the solid-state transformation has a crystallographic orientation relationship between the parent (β and austenite) and product phases (ferrite – low-alloy steels – and α – Ti-6Al-4V). Thus, the grain size and crystallography of the parent phase directly affect the final microstructure since the grain boundary area plays a role in the transformation kinetics (e.g., finer austenite grain size reduces the hardenability). Zhao et al. [173] (low-alloyed steels) reported that the fine austenite grain size and the remaining residual strain enhance the formation of acicular ferrite. This was corroborated by Colegrove et al. [66], who observed a finer ferrite grain size when increases the rolling load, promoting acicular ferrite and decreasing the low toughness constituents, such as grain boundary ferrite. For a Ti-6Al-4V alloy, the prior β grain size refinement had a low influence on the α lath thickness [137]. However, the material at room temperature (α field) showed a low texture intensity, which approximately equalized the quasi-static mechanical properties (i.e.,

reduced the anisotropy). Gao et al. [131] (Ti-6Al-4V) observed microstructural changes due to interlayer hot rolling, where the common basket-weave and α grain boundary microstructure (DED-arc) became majority α acicular (DED-arc + interlayer hot rolling), corroborating the results of Martina et al. [139] (finer α grain size due to cold rolling). These authors [131] attributed this behavior to the increase in the length of prior β grain boundary (fine grain size), which enhanced the intragranular α nucleation rate and reduced the overall α lamella size.

2.1.3.1.2 Non-transformable solid-state alloys

The coarse grain and texture problems become more pronounced in alloys that do not undergo allotropic transformations during the cooling thermal cycle, i.e., the primary microstructure (e.g., dendritic, segregation, and coarse and oriented grains) remains at room temperature [25]. Thus, interlayer rolling can be an interesting alternative to inducing grain size refinement and overcoming the low performance of cast-like microstructures. Some examples of alloys with primary microstructures are Ni and Al-based alloys (e.g., Inconel 718 and Al-Mg4.5Mn alloy). The major grain size refinement mechanism for these alloys is the dynamic/static recrystallization induced by the deformation storage energy. In addition, two grain refinements can occur, static recrystallization during the heating thermal cycle (cold rolling) and in situ dynamic recrystallization (hot rolling) and subsequently static recrystallization during post-deposition heat treatment (both cold and hot rolling) [37], [109], [138], [174], [175]. Xu et al. [138] observed (Figure 2.19) these two grain size refinement mechanisms for Inconel 718 DEDed-arc + interlayer cold rolled, which resulted in a significant grain size refinement (15.7 μm vs. 26.7 μm – wrought material). Zhang et al. [109] (Inconel 718 DEDed-arc) showed that a higher rolling force and high deformation depth resulted in finer grain size. Hönnige et al. [37] (Inconel 718 DEDed-arc) optimized the rolling load (50 kN) and the solution heat treatment condition (AMS 5662), obtaining an approximately fully recrystallized equiaxial microstructure. Additionally, Hönnige et al. [37] highlighted that the two static recrystallizations, namely reheating thermal cycle and heat treatment, have similar proportions, where the first refined the region close to the fusion line (higher peak temperatures and strain energy) and the second one refined the regions with lower strain energy (from the middle to the bottom of the layer height).

Gu et al. [175] (DED-arc + cold rolling) reported that Al-6.3Cu alloy (2319; precipitation hardening) also showed the two grain size refinement mechanism mentioned above. Otherwise, the unheated-treatable solid-solution strengthened Al-Mg4.5Mn alloy (DED-arc + cold rolling), showed, as the major grain size refinement mechanism, the static recrystallization during reheating thermal cycle due to the non-post-deposition heat treatment. Thus, for alloys

that do not require post-deposition heat treatments (e.g., solid-solution strengthened–Inconel 625 and Al-based alloys series 5xxx), the interlayer cold deformation grain size refinement could be limited to the reheating zone, maintaining coarse and oriented grains, as showed in Figure 2.19.

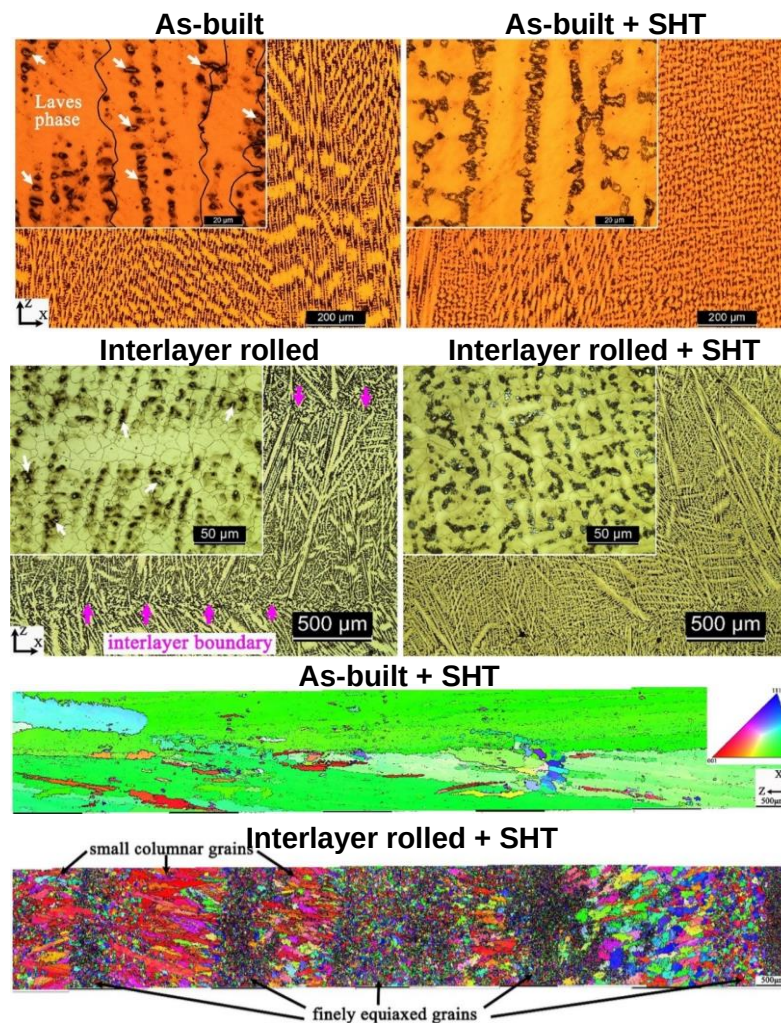


Figure 2.19 – Optical micrographs and EBSD orientation maps of Inconel 718 DEDed-arc with and without interlayer cold rolling (from [138]). SHT means solubilization heat treatment.

The hot rolling processes have a different grain refinement mechanism than cold rolling. In the first case, the rolling follows the deposition with the consolidated feedstock material still hot, leading to dynamic recrystallization. Furthermore, residual deformation where the region is hot deformed but not dynamically recrystallized can drive static recrystallization during the reheating thermal cycle and post-deposition heat treatments (similar to cold rolling). Zhang et al. [124], Hai-ou et al. [176], and Xi et al. [126] observed a change from the columnar and oriented grains to homogeneous equiaxial grains (Figure 2.20) for the 304

stainless steel DEDed-arc + interlayer hot rolling (Figure 2.5 and Figure 2.6). These authors attributed the grain size refinement to static recrystallization of dendrites microstructure, which was driven by the energy storage of plastic deformation and the reheating thermal cycles. This resembles the previously discussed cold rolling grain size refinement for primary (non-transformable) microstructure alloys.

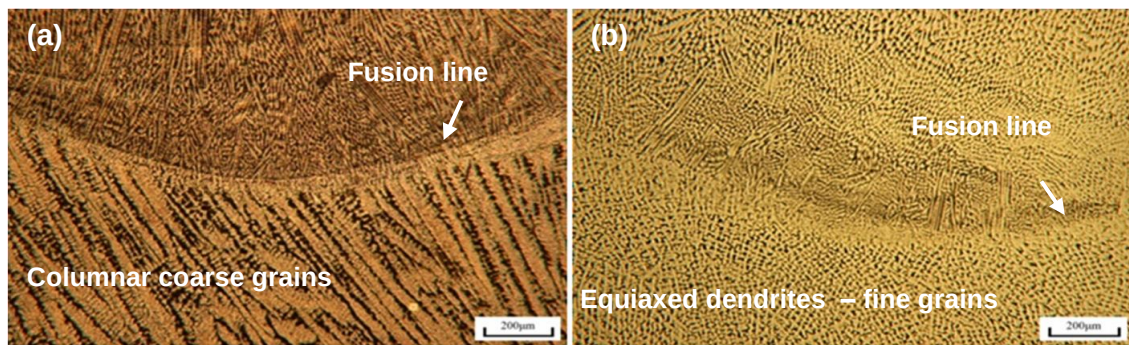


Figure 2.20 – (a) microstructures of the 304 stainless DEDed-arc and (b) DEDed-arc + interlayer hot rolled (adapted from [126]).

Li et al. [136], DED-laser + interlayer hot rolling (Figure 2.7), observed an intense grain size refinement for the Inconel 718 (Figure 2.21). The as-built sample had coarse ($\sim 113 \mu\text{m}$) columnar and oriented grains with a typical cubic texture ($\{100\}\langle 100\rangle$). In comparison, interlayer hot rolling changed the grain morphology drastically to a fine ($\sim 8 \mu\text{m}$) equiaxed grain structure. In interlayer hot rolling, it was reported both the dynamic (in situ) and static (reheating thermal cycle) recrystallizations, which refined and randomized the microstructure. Li et al. [136], Figure 2.21b, reveals two regions with different grain sizes (grains in region 1 are larger than those in region 2). In addition, the grain orientation spread (GOS; Figure 2.21c) depicted that the grains in region 2 were fresh recrystallized. The grains that formed a deposited layer (i.e., the columnar microstructure) were deformed by interlayer hot rolling. However, the deformation intensity was not homogeneous across the entire layer, where only the top layer surface region was dynamically recrystallized; the remaining grains continuous to have stored strain energy that can drive static recrystallization during reheating thermal cycles or heat treatments.

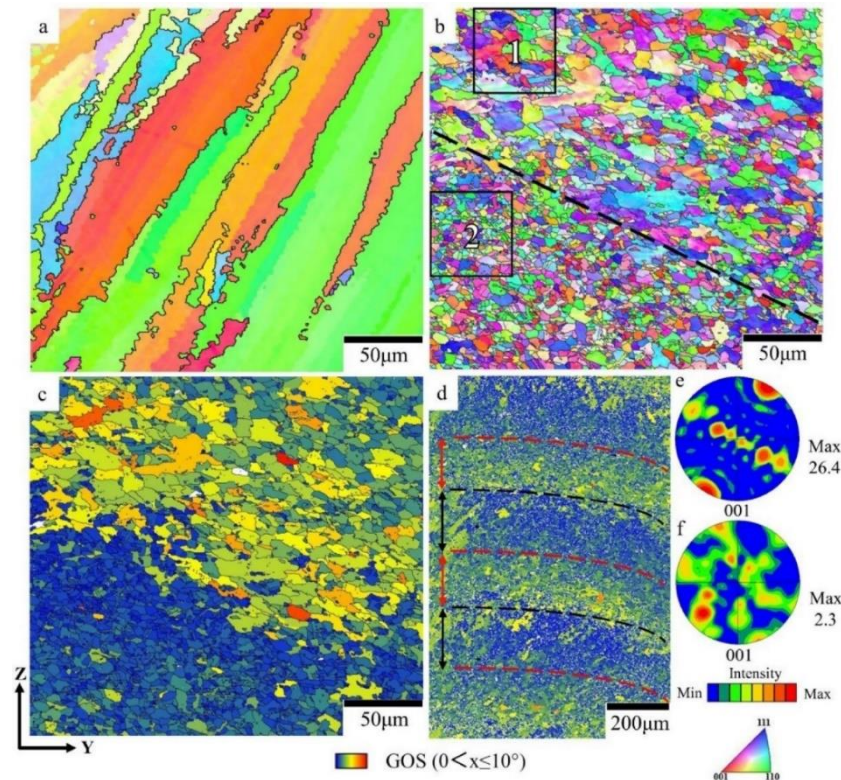


Figure 2.21 – EBSD maps of the Inconel 718 DEDed-laser: (a) as-built, (b) interlayer hot rolling grain refinement, (c) grain orientation spread (GOS), alternating bands of recrystallized and deformed grains, (e) (001) pole figure for as-built, and (f) (001) pole figure for interlayer hot rolled (from [136]).

Xie et al. [129] and Liao et al. [177], Al-Mg4.5Mn DEDed-arc, and Zhou et al. [178], AISi10Mg alloy laser-DEDed, reported that interlayer hot rolling (Figure 2.7) and in situ LSP had a lower grain size refinement effect (from 59 to 23 μm and from 9.8 to 8.6 μm , respectively) when compared to Inconel 718 (from 113 to 4.1 μm). Al alloys have a high stacking fault energy (SFE) [179], requiring high deformation to dynamically recrystallize since dislocations can easily reorganize and recovery at elevated temperatures. Therefore, it is expected that high SFE alloys have a lower response to interlayer hot rolling than low SFE alloys due to the low macroscopic deformation and recover processes, which are confirmed by the grain size refinement observed for low SFE alloys (e.g., Inconel 718 – grain size from 113 to 4.1 μm , $\sim 27 \times$) and high SFE ones (e.g., Al-Mg4.5Mn – grain size from 59 to 23 μm , $\sim 3 \times$). These results indicate that the interlayer hot deformation could have the better performance to specific alloy classes, namely solid-state transformable alloys and medium/low SFE non-transformable alloys.

2.1.3.2 Hammering, Peening, and Forging

Similar to rolling, the other deposition + interlayer mechanical deformation systems also have their own grain size refinement mechanisms and process characteristics due to the

different loading forces and deformation modes. It is important to note that topics addressed in Section 2.1.3.1 (Rolling) will not be revisited in Section 2.1.3.2. Instead, they will be only referenced.

2.1.3.2.1 Transformable solid-states alloys

Hönnige et al. [144], Ti-6Al-4V DEDed-arc, first described the effect of interlayer cold hammer peening on the prior β grain size refinement (Figure 2.22). The interlayer hammer peening achieves a pronounced prior β grain size refinement (from ~ 12 to 0.5 mm, $\sim 24 \times$). In addition, the authors claimed that the refined region is higher than the penetration deformation zone, which indicates that the prior β grains could continue to grow during subsequent thermal cycles. This aligns with the previously observed β grain size refinement mechanisms illustrated in Figure 2.17 and reinforces the idea that the new β (β') consumed the oriented prior β . Furthermore, despite the observed grain size refinement (Figure 2.22), the Ti-6Al-4V DEDed-arc + hammer peening grain size is larger than interlayer cold (~ 90 μm) and hot (~ 130 μm) rolled. Hönnige et al. [145] posteriorly detailed the hammer peening grain size refinement mechanism. The depth and distribution of the deformation zone (Figure 2.23a) have a steep strain gradient and depth of 2 mm. However, the effective deformation ($> 8\%$) for β refinement is reached only at 0.5 mm, which is lower than the deposition penetration (0.7 mm). These results suggested that the remaining deformed region (dotted black line in Figure 2.23a) can also generate β' , which grows during the reheating thermal cycle (Figure 2.23b). However, the refined prior β grain layer extends through the remaining deformation and solidification zones, suggesting that the refined prior β grain can grow upwards (epitaxial solidification) and downwards (recrystallization and grain growth into the remaining low deformation region). Thus, recrystallization takes place prior to solidification, i.e., during the heating cycle of the next deposited layer, so that the deposited layer grows epitaxially from these just recrystallized prior β grains.

Hu et al. [180] reported that the α dimensions were similar for DEDed-arc with and without interlayer cold hammer peening, which can be related to the larger prior β grain size compared to cold and hot rolling. However, Yi et al. [71] and Shchitsyn et al. [149] (Figure 2.11) observed that the interlayer cold hammer peening reduced the α grain boundary content and α lath size, which proves to be a topic that demands more investigation for a complete consolidation and comprehension. Finally, the hammer peening grain size refinement mechanism is almost similar to cold rolling (twinned β' growth [169], [170]; Figure 2.17),

diverging by the presence of a solidification process and lower deformation zone depth, which results in larger β grain size in relation to interlayer cold and hot rolling.

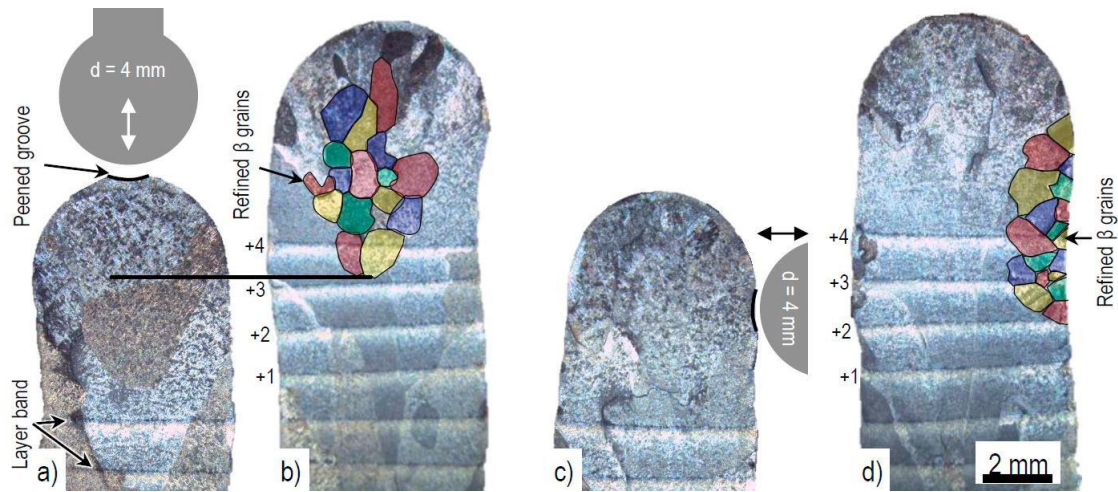


Figure 2.22 – β grain refinement of interlayer cold hammer peened Ti-6Al-4V WAAM walls (from [144]).

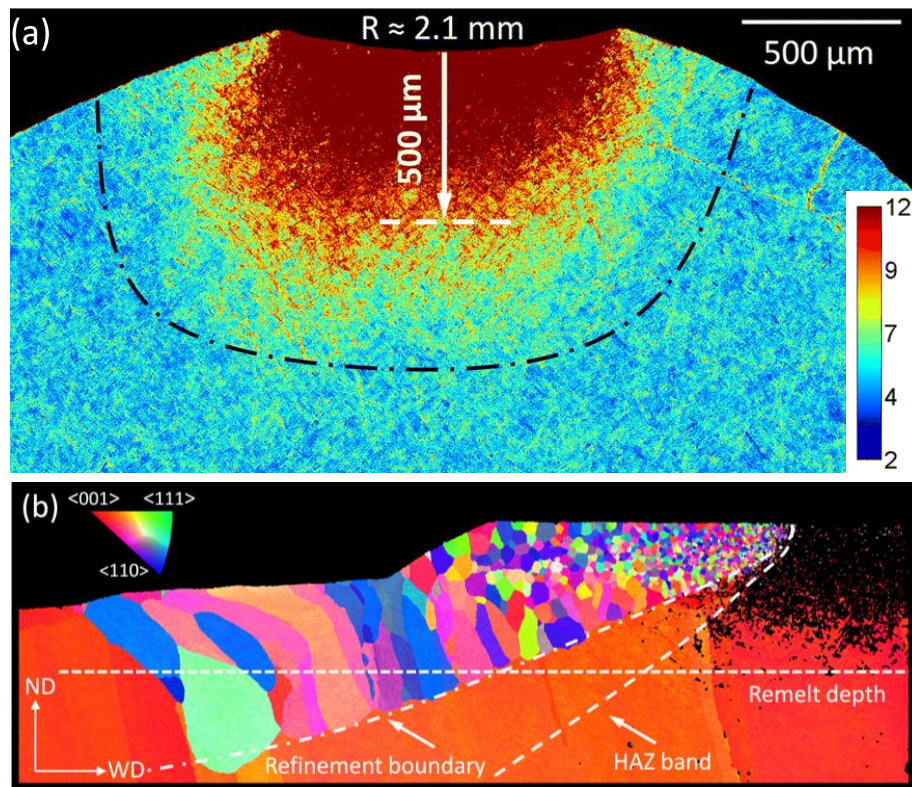


Figure 2.23 – (a) strain distribution map and (b) an β grain reconstruction EBSD map and interaction of thermal cycle with the deformed region (from [145]).

The use of interlayer hot peening/forging for solid-state transformation alloys is still scarce of publications due to its dynamic and localized energy characteristic, which can fracture the just deposited layer (hot material). Ye et al. [156], Ti-4Al-6V DEDed-laser + interlayer hot ultrasonic micro-forging (Figure 2.26), observed a slight prior β grain size refinement (from

~ 400 to ~ 320 μm); however, these data are statistically equal (large standard error). As proposed by Li et al. [157], the prior β grain size refinement was attributed to the synergistic effect of vibration inducing CET and some dynamic recrystallized β grains (at bead surface). Ye et al. [156] verified the dynamic recrystallization by the serrated grain boundaries (strain gradient) and the new β grains formed at a triple junction (high local strain regions). In addition, these new β grains can grow down and consume the columnar β grains, similar to that observed for cold hammer peening [145]. Xiong et al. [158], low-alloy steel (AWS ER70S-6) DEDed-laser + interlayer hot hammer peening, also observed the parent phase (γ) dynamic recrystallization at the layer surface; however, due to the $\gamma \rightarrow \alpha$ transformation, the final microstructure is not significantly affected. Although the prior γ grain size affects material hardenability and α formation/morphology, the mandatory effect on the final microstructure is governed by the cooling rates [181], which is almost insensible to the interlayer hot hammer (low effect on geometric aspects of the deposited layer) [158]. Thus, the interlayer hot hammer peening for transformable alloys showed insufficient grain refinement that justified its use.

2.1.3.2.2 Non-transformable solid-states alloys

Wang and Shi [152], [153], Inconel 718 DEDed-laser + interlayer cold hammer peening, highlighted that the major effect of interlayer cold hammer occurred on the interlayer regions (fusion line) and was related to an equiaxed dendrite zone (~ 200 μm) between the previously interlayer deformed and subsequently deposited layers (Figure 2.24). Inconel 718 DEDed-laser microstructure had typical coarse and oriented primary grains. Interlayer cold deformation generated a fine equiaxed grain size (~ 10 μm) band, which prevents continuous primary grains across several layers (Figure 2.24); however, the epitaxy persisted. Thus, the next layer will use the previously refined grains as a nucleation site, as suggested by Hönnige et al. [145] for DEDed-arc Ti-6Al-4V + interlayer cold hammer peened. Besides, the deformed region (intense kernel average orientation – KAM) must be higher than the melting pool penetration (0.1 – 0.5 mm for laser-based DED) to drive static recrystallization during post-deposition heat treatment (e.g., mandatory for Inconel 718) or reheating thermal cycles. Thus, similar to observed in cold rolling, non-transformable medium/low SFE alloys can undergo two grain refinement mechanisms when interlayer cold hammered: static recrystallization during deposition and subsequent heat treatment. In addition, Parvaresh et al. [182] observed grain refinement due to interlayer cold hammer peening for an AISI 347 stainless steel, which aligns with the findings proposed by Wang and Shi [152], [153].

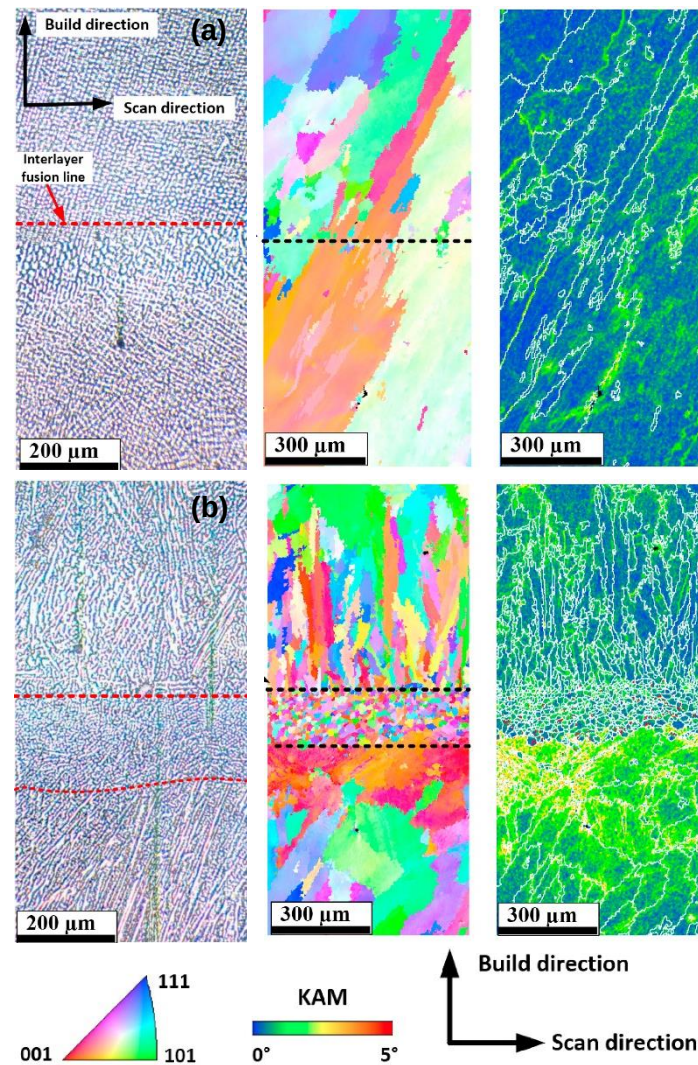


Figure 2.24 – Optical microscopy, orientation map, and kernel average misorientation (KAM) map: (a) Inconel 718 DEDed-laser and (b) Inconel 718 DEDed-laser + interlayer cold hammer peening (adapted from [153]).

As opposed to Wang and Shi [152], [153] (DED-laser), Fang et al. [148] (DED-arc + interlayer cold hammering) deposited the 2319 Al alloy, which required an intense and deeper deformation (high SFE alloy and arc-based process) to induce static recrystallization by the reheating thermal cycle. The primary grains of the 2319 Al alloy are mostly equiaxed, with a small number of columnar grains close to the substrate (high thermal gradient; Figure 2.25). As detailed in Figure 2.25, region A shows an intense deformation after hammering and had a fine grain size ($1.3\ \mu\text{m}$), which transformed into region B (reheating thermal cycle), characterized by static recrystallization and grain growth ($\sim 9\ \mu\text{m}$). Region C depicts the deformation depth of hammering ($\sim 2\ \text{mm}$), which was also characterized by static recrystallization; the other regions, closer to the substrate, were not affected by the hammering. Thus, the subsequent deposition + deformation cycles will reproduce this refined

microstructure pattern throughout, which proves the efficiency of hammering to refine the as-built microstructure of high SFE alloys (e.g., Al-based).

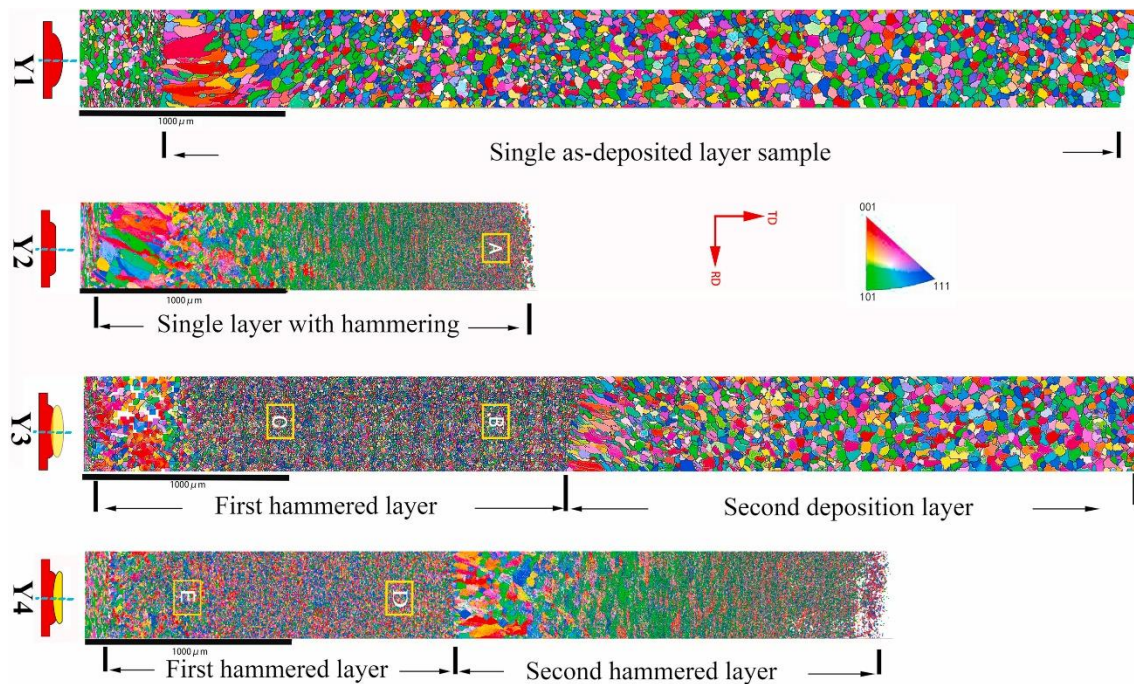


Figure 2.25 – Microstructure evolution of 2319 Al DEDed-arc + interlayer cold hammering. Single-layer + hammering; Two-layers + hammering on the first layer. (a), (b), (c) IPF-EBSD maps of the areas A, B, and C, respectively (adapted from [148]).

Duarte et al. [35] (AISI 316L stainless steel) demonstrated the grain size refinement of the DEDed-arc + interlayer hot forging (Figure 2.12) using 2D Debye-Scherrer diffraction patterns obtained via synchrotron X-ray diffraction; however, the authors did not investigate the phenomena that governed the grain size refinement. In addition, Duarte et al.[35] observed the increase in the cooling rate due to the higher bead width after the hot deformation, which promoted a finer primary microstructure (e.g., cellular and dendritic). Farias et al. [159], [160] clarified the grain refinement mechanisms for DEDed-arc + interlayer hot forging (Figure 2.12). The authors [159], [160], using in situ electron X-ray diffraction, reported that interlayer hot forging could generate enough crystal defects to induce static recrystallization during subsequent thermal cycles and post-deposition heat treatments. Li et al. [157], Ni-based superalloy (GH3039) DEDed-laser, reported that the interlayer hot ultrasonic micro-forging did not alter the typical columnar and oriented grains, which is mainly controlled by heat transfer aspects during part building. Additionally, Li et al. [157] highlighted that the CET depth, which is related to dynamic recrystallization, increased from 440 to 840 μm , which indicates that the interlayer hot ultrasonic micro-forging recrystallized the CET grains (Figure 2.26). In addition, the vibrations (forging strokes) also enhance the CET length [183]. Thus, hot forging can have

two simultaneous grain refinements: enhancing the CET due to its dynamic aspect (inducing vibration on the liquid) and dynamic and static recrystallization (solid-state).

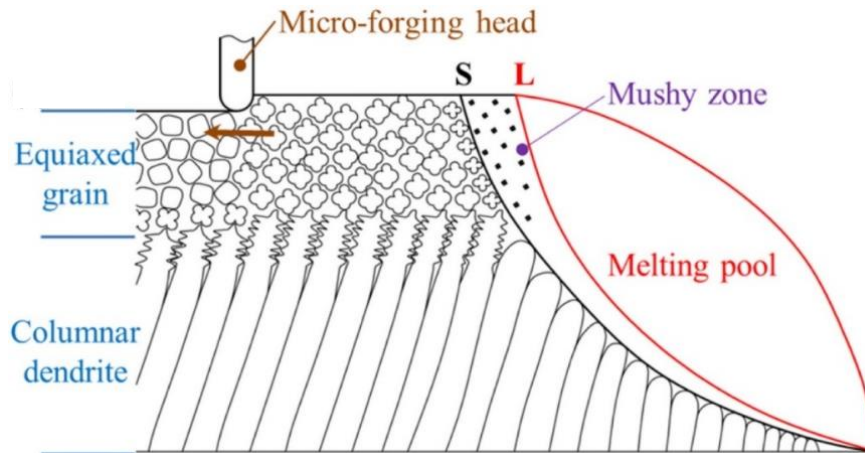


Figure 2.26 – Schematic illustration of the effect of DED-laser + interlayer hot deformation on microstructure (from [157]).

Figure 2.26 also indicated that the heat source-deformation distance could be a limiting factor since some materials can lose the ductility at higher temperatures, leading to cracking due to the intense and localized hot forging deformation, which explains the major use of the hammer and ultrasonic peening in cold conditions or with high heat source-deformation distance (far from the mushy zone). Hence, there is a minimum distance that is sufficient to stay out of the brittle temperature range [184] and a maximum distance (minimum temperature to drive dynamic recrystallization – hot deformation – or minimum temperature to ensure sufficient deformation for static recrystallization – cold deformation). For solid-state transformable alloys, the hot deformation must occur at a temperature superior to the transition temperature (e.g., β *transus* and austenite decomposition) to induce an effective dynamic recrystallization of the parent phase (grain size refinement), establishing a minimum deformation temperature. Additionally, each alloy possesses specific physical properties (e.g., thermal conductivity and density) and metallurgical behavior (e.g., recrystallization temperature and strength at elevated temperatures). Consequently, the combination of each material, DED process, and deformation system is almost unique. Therefore, parameter optimization should consider the stability of the deposition process, layer geometrical aspects (particularly penetration and width), and deformation parameters (heat source-deformation tool distance and deformation zone depth). This ensures that deposition + interlayer deformation effectively promotes grain size refinement and improves material behavior without compromising printability (e.g., instability, layer malformation, hot cracks, and lack of fusion).

2.1.4 Effects interlayer deformation on material mechanical behavior

A critical problem that can prevent the industrial scalability of DED processes relates to the poor mechanical properties of the part 3D printed. This included the inability to meet material specifications (e.g., API 20S and AMS 5662) and inferior performance compared to conventional manufacturing routes (e.g., wrought). In this context, Section 2.1.4 evaluates the effect of interlayer mechanical deformation on the mechanical properties of typical commercial alloys deposited via DED, comparing them with the standard industrial requirements and discussing the changes in mechanical properties based on the Section 2.1.3 (grain size refinement mechanisms).

2.1.4.1 Ti-based alloys

Martina et al. [121] (Ti-6Al-4V) first showed that the DED-arc + interlayer cold rolling improved both the yield strength (σ_{YS}) and ultimate tensile strength (σ_{UTS}) in both deposition (heat source path planning) and building (perpendicular to heat source path planning) directions; however, a slight drop in ductility was observed in relation to DEDED-arc. Further, Colegrove et al. [34] highlighted that Ti6Al4V DEDED-arc anisotropy, especially concerning the material ductility, was virtually eliminated by interlayer cold rolling. Figure 2.27 depicts the influence of interlayer mechanical deformation on the quasi-static mechanical properties of the Ti6Al4V DEDED. It is worth noting that the interlayer deformation systems (hot and cold rolling and cold peening) did meet the ASTM B265 (wrought Ti-6Al-4V) and AMS 4999 (additive manufactured Ti-6Al-4V) requirements, which was not observed for the DEDED condition.

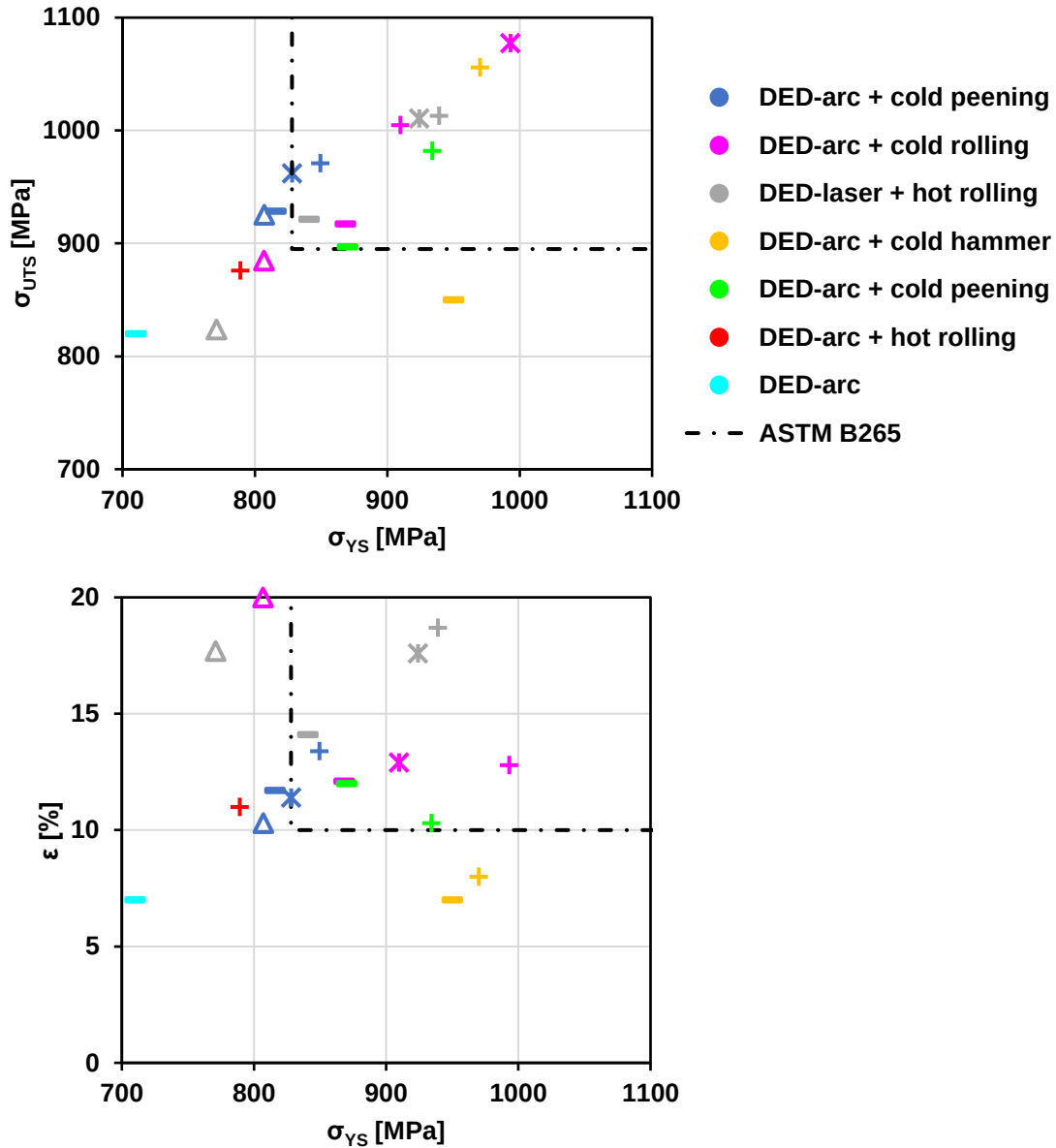


Figure 2.27 – Tensile properties of Ti-6Al-4V deposited by DED-arc + interlayer mechanical deformation. The symbols —, ▲, +, and * mean that the tensile specimens were taken from the DEDed part in the deposition direction (heat source path), built direction (90° from heat source path), interlayer mechanically deformed DEDed deposition direction, and interlayer mechanically deformed DEDed in built direction, respectively. In sequence, the references [71][122][137][145][185][186][187].

Yang et al. [188] (Ti6Al4V DEDed-arc) and Chi et al. [189] (Ti17 alloy DEDed-arc) showed that the interlayer cold ultrasonic and LSP, respectively, can improve both σ_{YS} and σ_{UTS} without affecting the ductility. Further, Huang et al. [185] highlighted that the finer prior β grain size reduced the allotriomorphic α content at the grain boundary, which can even increase the Ti-6Al-4V DEDed-arc ductility. These results are contrary to Yi et al. [71] and Martina et al. [121], both Ti6Al4V DEDed-arc, which observed an increase in σ_{YS} and σ_{UTS} and a reduction in ductility

due to the interlayer mechanical deformation. Besides, Tian et al. [137] and Hu et al. [186], Ti6Al4V DEDed-laser and DEDed-arc, respectively, reported that the interlayer hot rolling induced an isotropic behavior and increased both σ_{YS} , σ_{UTS} , and ductility.

Colegrove et al. [34] pointed out that the coarse and oriented prior β grain (DEDED-arc condition) induced the formation of aligned allotriomorph α -phase (soft region) at the prior β grain boundary, which had faster loading damage (i.e., strain accumulation and crack nucleation) than the Widmanstaätten interlocked α -phase matrix [190]. Thus, it is expected that the prior β grain refinement induced by the interlayer mechanical deformation enhanced the ductility. However, as depicted in Figure 2.27, Yi et al. [71] and Martina et al. [121] observed a decrease in ductility even considering the β grain refinement, which was associated with remaining deformation (higher dislocation density and increased work hardening). In addition, Bermingham et al. [187] reported that the smaller α colony size (i.e., basketweave microstructure) induced by the refined prior β grain can reduce material ductility due to the difference in mechanical properties of the prior β grain (α colonies) and grain boundary (allotriomorph α -phase). Hu et al. [186] correlated the lower ductility with the increase in the work hardening exponent due to the smaller prior β grain. Thus, considering that cold and hot interlayer deformation induced a finer prior β grain and smaller α colony size, the main difference between them is the remaining deformation (especially associated with deformation depth), which induced a lower ductility for DEDed + interlayer cold deformation concerning DEDed + interlayer hot deformation. This statement is corroborated by Tian et al. [137], who calculated the dislocation density for Ti-6Al-4V DED-laser and DED-laser hot rolling conditions, concluding that, due to the high-temperature deformation, these conditions had similar dislocation densities. In addition, the higher hardness and σ_{UTS} of the cold deformed condition reinforce the conclusion that its low ductility arose from the remaining deformation. Despite the lower ductility, cold deformation still ensures that the Ti-6Al-4V DEDed meets the standards and code requirements.

The increase in σ_{YS} and σ_{UTS} was usually associated with a finer α lath size; however, most of the literature [34], [137], [149], [186], [188] indicated that the interlayer deformation had a low effect on α lath size. Tian et al. [137] stated that the predominant factors for the increase in σ_{YS} and σ_{UTS} were related to the randomization and refinement of the prior β grain (from coarser and aligned to equiaxial), which also reduce the variant selection in the $\beta \rightarrow \alpha$ phase transformation and α phase texture index. Lütjering [191] reported that, for ($\alpha + \beta$) titanium alloys, the α colony and α lath size dictated the slip length. Thus, as the α lath size is less affected by the interlayer deformation, the α colony size was mainly responsible for the

increase in σ_{YS} and σ_{UTS} in relation to DEDED. Finally, the literature data converge (Figure 2.27) on the conclusion that both cold and hot deformation systems improved the σ_{YS} and σ_{UTS} of the Ti-6Al-4V DEDED, enabling it to meet the material specifications.

2.1.4.2 Low-alloy steels

Colegrove et al. [66] first studied the effect of interlayer cold rolling on low-alloy steel DEDED-arc (AWS A5.18 ER70S-6). The authors showed that the final microstructure (e.g., grain boundary ferrite and acicular ferrite) was not strongly influenced by the cold rolling. However, the remaining deformation (Taylor hardening) increased and homogenized the hardness profile so that a higher load rolling induced a higher hardness, which also converged to the Ti6Al4V alloy DEDED + interlayer mechanical deformation results [34], [137], [149], [186], [188]. Posteriorly, Dirisu et al. [114] carried out tensile tests (Table 2.1) that corroborated Colegrove et al. [66], showing that the σ_{UTS} and σ_{YS} were improved with the rolling load. Thus, for low-alloy steel DEDED + interlayer cold deformation, the major hardening mechanism was attributed to the increase in dislocation density (Taylor hardening), which was confirmed by Dirisu et al. [114] via transmission electron microscopy. In addition, the authors observed a finer ferrite grain size for a higher load rolling (160 kN; 14.5 vs. 8.6 μm), which improved the total strengthen (Hall-Petch). Similar to Ti-6Al-4V, the final microstructure of low-alloy steels arise from solid-state transformation so that the product phase (ferrite) and its constituents are less affected by the interlayer deformation, reinforcing the results of Colegrove et al. [66]. Ma et al. [130] (DED-arc + interlayer hot rolling) and Xiong et al. [192] (DED-arc + interlayer hot forging) also observed (Table 2.2) an improvement in mechanical properties with an almost isotropic behavior, which met the ASTM A182-19 standard. It is worth noting that both Dirisu et al. [114] and Ma et al. [130] (Table 2.2) observed a significant prior austenite grain size refinement and an increase in σ_{UTS} and σ_{YS} . However, due to the higher remaining deformation (high dislocation density), DEDED + interlayer cold rolling (160 kN) [114] induced a drop in ductility.

Table 2.1– Static tensile properties of a low-alloy steel (AWS A5.18 ER70S-6) DEDED-arc (adapted from [114]).

Rolling load [kN]	σ_{UTS} [MPa]	σ_{YS} [MPa]	Hardness, HV _{0.2}	Ductility [%]
0	402 ± 2.0	300 ± 1.5	186 ± 2.0	28 ± 1
50	552 ± 1.0	498 ± 5.0	225 ± 1.0	25 ± 2
75	546 ± 3.0	486 ± 2.6	219 ± 1.5	32 ± 2
160	600 ± 3.0	574 ± 2.6	233 ± 1.5	22 ± 2

Table 2.2 – Effect of the interlayer deformation on low-alloy steel DEDed quasi-static mechanical properties.

DED	Deformation		feedstock material	σ_{ys} [MPa]	σ_{UTS} [MPa]	ϵ [%]	References
	process	temperature					
arc	Rolling	cold	ER70s-6	574 ± 2.6	600 ± 3.0	22 ± 2	[114]
	—	—		300 ± 1.5	402 ± 2.0	28 ± 1	
arc	Rolling	hot	ER70s-6	387.8	530.9	36.9	[130]
	—	—		365.1	509.3	37.3	
arc	Forging	hot	ER70s-6	641.9	529.3	37.3	[192]
	—	—		297.4	469.6	42.2	
arc	Rolling	hot	Bainitic steel	1259.1	—	17.0	[127]
	—	—		1275.3	—	10.8	

Fu et al. [127] (Figure 2.6) verified the increase in ductility due to hot rolling for bainite steel (Table 2.2). In addition, these authors addressed the Charpy V-notch (CVN) impact energy absorbed. The interlayer hot rolling strongly increased the CVN impact energy (99 J/cm^2 – three times higher than DEDed-arc) in relation to both DEDed-arc and wrought material; the CVN impact energy had an isotropic behavior. Figure 2.28 shows that the interlayer hot forging induced a change in fracture mechanism from almost cleavage (brittle) to fine dimples (ductile) aspect. This behavior was directly related to the finer prior austenite grain size developed during hot rolling, which induced a finer sub-structure (packet and blocks), a smaller effective grain size [193] (similar to α colony size in Ti-6Al-4V), and a reduction in both grain boundary ferrite and martensite-austenite microconstituent content (both brittle) [194], ultimately enhancing the CVN impact energy.

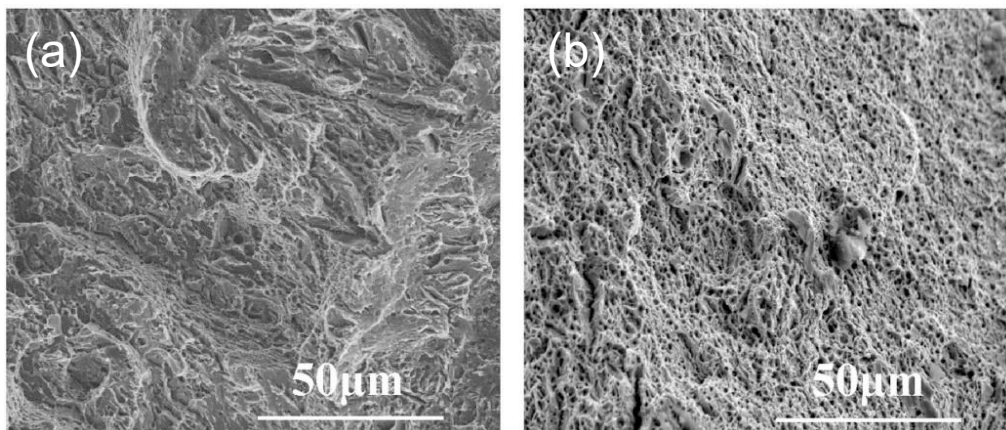


Figure 2.28 – Fracture surface of a bainitic still deposited by DED-arc: (a) without and with interlayer hot rolling (adapted from [127]).

2.1.4.3 Stainless steels

The AISI 316 L stainless steel DEDed (both laser and arc) usually met the materials specification (ASTM A240) in the as-built condition; however, as pointed out by Wang et al. [195], the as-built columnar microstructure results in an anisotropic behavior. In

this sense, Duarte et al. [35] (DED-arc), Gao et al. [161] (DED-laser), and Kan et al. [196] (DED-arc) reported (Table 2.3) an improvement in the mechanical properties and an isotropic behavior due to the interlayer hot deformation. Duarte et al. [35] and Kan et al. [196] observed that the hot forging and rolling, respectively, increased both σ_{YS} and σ_{UTS} (meet the ASTM A240 and ASTM A666 1/16 hard), which was associated with the finer grain size and the increase in the work hardening. These authors [35], [196] also linked the drop in ductility with the finer grain size, not meeting the ductility requirements (ASTM A240 and ASTM A666).

Parvaresh et al. [182] imposed a macroscopic interlayer cold deformation (similar to the pressing process) to the AISI 347 stainless steel DED-arc. As in the case of hot deformation systems [35], [161], [196], Parvaresh et al. [182] reported an increase in material strength (σ_{UTS} , from 576 to 803 MPa and σ_{YS} , from 395 to 697 MPa) at the expense of ductility (from 58% to 30%). Diao et al. [197], AISI 321 stainless steel DEDed-arc, verified that interlayer ultrasonic peening induced a finer γ grain size, which increased the σ_{YS} (from ~ 390 MPa to ~ 425 MPa) and did not alter ductility. Finally, although the interlayer deformation improved the strength of stainless steel, the low ductility problem persists, which is aggravated by the interlayer deformation. Thus, for alloys fabricated via DED that typically meet the strength requirements and fail in plasticity ones, interlayer deformation can be inefficient since the grain size refinement tend to increase the materials' work hardening exponent (Hollomon's equation).

Table 2.3 – Tensile properties of AISI 316L stainless steel DEDed + interlayer deformation.

DED process	Deformation		σ_{YS} [MPa]	σ_{UTS} [MPa]	Ductility [%]	References
	process	temperature				
arc	Forging	Hot	450	622	27.9	[35]
	—	—	360	574	32.5	
laser	Forming	Hot	254	597	65.2	[161]
	—	—	279	611	59	
arc	Rolling	Hot	340 ± 3	591 ± 4	38 ± 5	[196]
	—	—	265 ± 11	534 ± 4	49 ± 4	
ASTM A240			170	485	40	
ASTM A666 1/16 hard hot finished			310	585	35	
ASTM A666 1/8 hard cold finished			380	690	25	

Xu et al. [198] reported that the maraging steel (AISI Grade 18Ni 250) DEDed-arc had an inadequate heat treatment response, resulting in an inferior σ_{YS} (1270 MPa vs. 1720 MPa) concerning wrought material, which makes its industrial adoption unfeasible. Posteriorly, the same research group (Xu et al. [199]) observed that interlayer cold rolling (50 and 75 kN) promoted a significant improvement in the heat treatment response, which resulted in a substantial increase in σ_{YS} and σ_{UTS} (Figure 2.29) concerning DEDed-arc condition (σ_{UTS} from

1735 to 1345); however, the material requirements (ASTM A579) were also not met. Xu et al. [199] pointed out that the interlayer rolling induced an intense prior austenite grain size refinement (from primary microstructure – $\sim 200 \mu\text{m}$ – to equiaxed – $40 \mu\text{m}$), enhancing the length of high-angle grain boundaries and increasing the dislocation density, which strongly increases the atomic mobility during solubilization heat treatment (i.e., more solute is supersaturated in the martensite matrix). In addition, the higher dislocation density acts as precipitate nucleation site; thus, during aging, a higher content of hardening phase precipitated (1.4 vs. 2.2 %), which explained the significant improvement in the heat treatment response due to the interlayer cold rolling.

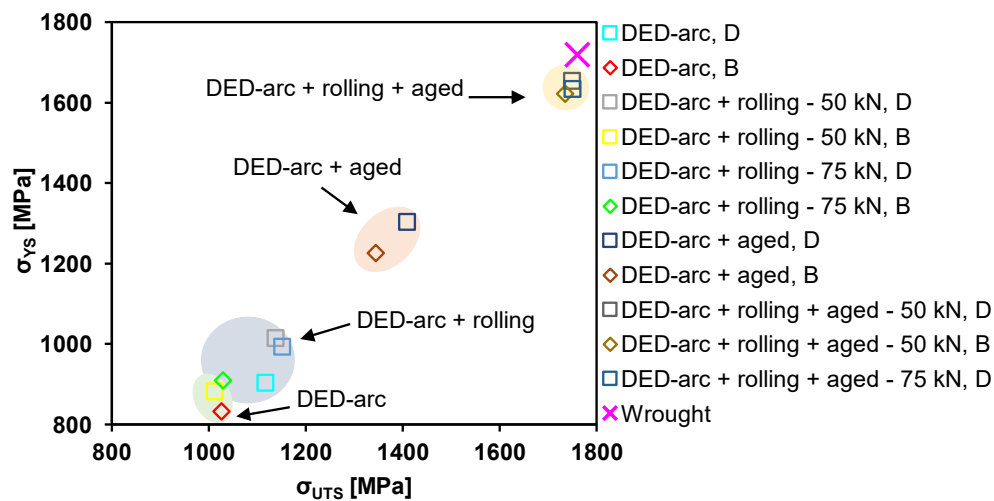


Figure 2.29 –Mechanical properties of the maraging steel DEDed (from Xu et al. [199]). B and D are the building and deposition directions, respectively.

2.1.4.4 Ni-based alloys

Ni-based superalloys fabricated via arc plasma DED typically did not meet the material specification (e.g., AMS 5662 and ASTM B670 standards) in the as-built and post-deposition heat-treated conditions, especially the Inconel 718 (a high-responsibility alloy used in the aerospace industry). This behavior was primarily related to coarse microstructure, intense interdendritic segregation (e.g., Nb and Mo), and deleterious eutectics precipitation (e.g., Laves phase and MC-type carbides), which reduced the material response to heat treatment (e.g., precipitation of γ'' and γ' phases) [25]. In this sense, Xu et al. [138] observed (Table 2.4) that the interlayer cold rolling can satisfactorily enhance the mechanical properties of the Inconel 718 DEDed-arc, meeting, for the first time, the AMS 5662 requirements. Since the Laves phase consumes Nb and reduces the γ'' and γ' content, and interlayer rolling has little influence on solidification conditions, the solution heat treatment ($\sim 980^\circ\text{C}$) cannot increase the γ'' and γ'

content (Laves phase dissolution temperature - 1146 °C). This explains the nearly identical aging response of the DEDed and DEDed interlayer-rolled samples (321 MPa and 391 MPa, respectively). Thus, the main difference between the rolled and unrolled DEDed-arc Inconel 718 mechanical properties rise from the grain size (1200 vs. 15.7 μm ; Figure 2.19) strengthening mechanism. According to Xu et al. [138], the difference in σ_{UTS} (295 MPa) between the condition rolled and unrolled can be attributed to grain size refinement (225 MPa, 76%) and the improvement in the material response to heat treatment (70 MPa, 24%). Therefore, the grain size refinement of Inconel 718 DEDed was critical for the material to meet the AMS 5662 requirements. Similar results were posteriorly obtained by Zhang et al. [109], which verified that the rolling force (50 and 75 kN) did not influence the quasi-static properties since the final grain size is almost similar (26.5 and 14.7 μm , respectively).

Table 2.4 - Mechanical testing results of the DEDed-arc interlayer cold (un)rolled Inconel 718 (adapted from [138]). AD and SA correspond to as-deposited and solution + aging heat-treated, respectively.

Condition		σ_{UTS} [MPa]			σ_{YS} [MPa]		Ductility [%]		AMS 5662
		H	V	Average	H	V	H	V	
AD	Unrolled	818 $\pm$ 13	756 $\pm$ 7	793 $\pm$ 32	525 $\pm$ 7	506 $\pm$ 2	33.3 $\pm$ 2.5	27.9 $\pm$ 1.3	✗
	rolled	1082 $\pm$ 13	1072 $\pm$ 6	1078 $\pm$ 12	763 $\pm$ 8	687 $\pm$ 1	26.2 $\pm$ 2.2	26.6 $\pm$ 1.3	✗
SA	Unrolled	1102 $\pm$ 78	988 $\pm$ 6	1056 $\pm$ 82	790 $\pm$ 9	791 $\pm$ 14	14.7 $\pm$ 1.3	12.8 $\pm$ 1.2	✗
	rolled	1348 $\pm$ 10	1356 $\pm$ 10	1351 $\pm$ 11	1057 $\pm$ 19	1035 $\pm$ 20	15.1 $\pm$ 3.3	17.4 $\pm$ 1.1	✓

Chen et al. [200] and Li et al. [136], DED + interlayer hot rolling (Figure 2.4 and Figure 2.7, respectively) also obtained a finer grain size ($\sim 25 \mu\text{m}$ [200] and $8 \mu\text{m}$ [136]) due to the interlayer hot deformation. Chen et al. [200] reported that the obtained mechanical properties met the AMS 5662 standard requirements in deposition and building directions. Wang and Shi [153], DED-laser, also pointed out better properties due to interlayer deformation (cold ultrasonic harmer peening); besides, the material (σ_{YS} and σ_{UTS} of 1037 and 1276 MPa, respectively) met the AMS 5662 requirements. Figure 2.30 summarizes the abovementioned works and compares them with the material specification, where it is possible to infer that the interlayer deformation can significantly increase the performance of the Inconel 718 DEDed. In addition, the tensile properties of Inconel 718 at elevated temperatures (649 °C) must also be considered for high-temperature applications, such as jet turbine blades and the nuclear industry [162]. In this scenario, only the warm rolling material [109]–[111] met the AMS 5662 requirements despite all DED + deformation conditions also improving the material properties concerning DED.

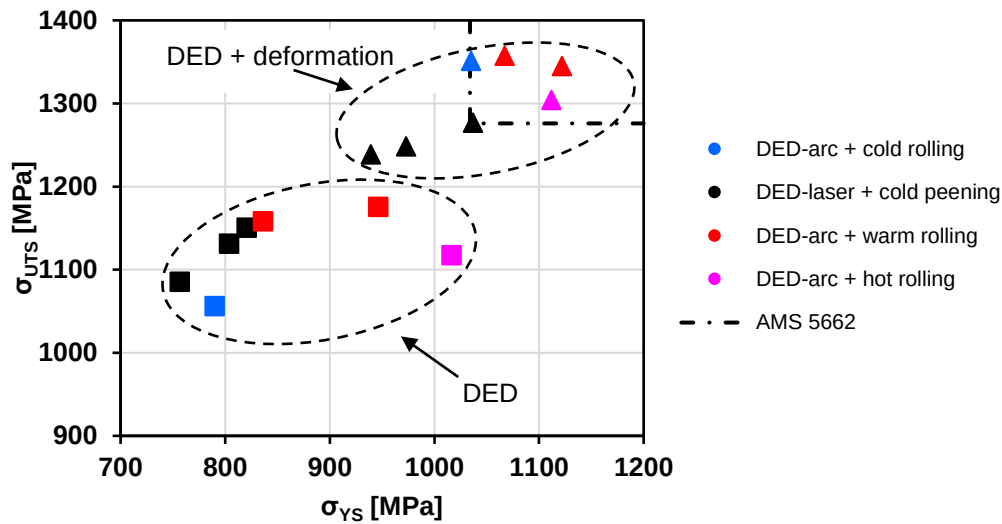


Figure 2.30 – Effect of the interlayer deformation on Inconel 718 DEDed yield (σ_{YS}) and ultimate tensile (σ_{UTS}) strength. Materials after post-deposition heat treatment. Cold rolling [138], cold peening [153], warm rolling [109], and hot rolling [200] references.

Zhao et al. [128] (Ni60 alloy) and Li et al. [157] (GH3039 alloy) deposited solid-solution strengthened Ni-based alloys and observed the effects of interlayer hot deformation (rolling and forging, respectively) on the hardness profile (Figure 2.31). The authors pointed out a banded hardness pattern related to the fine cellular microstructure close to the fusion line (peak hardness) and the coarse columnar dendritic (lower hardness) far from the fusion line. Using the deposition + interlayer mechanical deformation, the hardness profile became more homogeneous and average hardness increase. Zhao et al. [128] and Li et al. [157] associated the hardness increase with grain size refinement (Hall–Petch) and higher dislocation density (Taylor hardening).

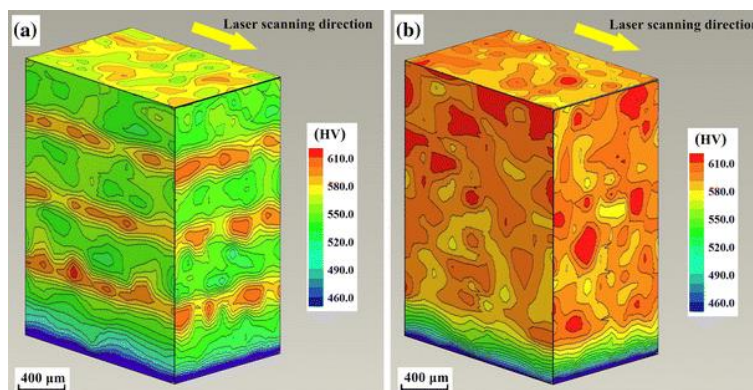


Figure 2.31 – Vickers microhardness contour maps of Ni60 alloy (a) unrolled and (b) rolled DED-laser (from [128]).

2.1.4.5 Al-based alloys

Fang et al. [148], Gu et al. [175], and Wang et al. [201] evaluated the effect of the interlayer cold deformation (hammering and rolling) on quasi-static mechanical properties of the 2319 Al DEDed-arc (Table 2.5). Fang et al. [148] reported that interlayer hammering increased the σ_{YS} and σ_{UTS} by 50.8% and 16.0% (238.9 and 332.8 MPa, respectively) in comparison to the un-hammered DEDed-arc in the as-built condition. However, this increase in material strength was followed by a reduction in ductility (from 18.1 to 13 %), i.e., there is a trade-off between the increase in σ_{YS} and σ_{UTS} and the reduction in ductility, which was also observed by Gu et al. [175]. Further, despite the grain size refinement ($\sim 9 \mu\text{m}$ vs. $89 \mu\text{m}$ [148] and $26.7 \mu\text{m}$ and $7.7 \mu\text{m}$ [175]) induced by the reheating static recrystallization, the authors [148], [175] reported that the grain boundary strengthening ($\sim 5 \text{ MPa}$) was negligible compared to the actual increase in material strength ($\sim 100 \text{ MPa}$). Thus, the major strengthening ($\sim 95 \text{ MPa}$) mechanism was associated with the Taylor hardening. However, 2319 Al alloy requires a post-deposition heat treatment to meet the full strength (solution + artificial aging heat treatment; T62) [202]. Gu et al. [175] reported that T62 heat-treated conditions (unrolled and rolled) met the ASTM B 247 requirements and no significant differences is observed among them (e.g., 142 vs. 144 HV, respectively). This behavior arose from the precipitation hardening (main strengthening mechanism) on mechanical properties. As mentioned, the grain size had no significant effect; also, due to the solution heat treatment ($535 \text{ }^\circ\text{C}/1.5 \text{ h}$), the Taylor hardening was negligible (recovering and static recrystallization). Therefore, the interlayer mechanical deformation had limitation effect of age-hardened Al alloys fabricated via DED.

Table 2.5 – Effect of interlayer mechanical deformation on the 2319 Al alloy DEDed quasi-static mechanical properties.

DED	Deformation		Condition	σ_{YS}	σ_{UTS}	ϵ	References
process	process	temperature		[MPa]	[MPa]	[%]	
arc	Hammer	cold	as-built	238.9	332.8	13	[148]
	—	—		148.5	289.2	18.1	
arc	Ultrasonic peening	cold	as-built	114	255	5.6	[201]
				64	224.5	8.1	
arc	Rolling	cold	as-built	247.5	319.8	8.5	[175]
	—	—		133.3	261.6	18.4	
	Rolling	cold	T62	321.2	458.1	17.0	
	—	—		333.9	461.2	14	
Wrought material				290	415	10	[203]

Xie et al. [129], Gu et al. [174], and Liao et al. [177] evaluated the behavior of the 5xxx series Al alloy (solid-solution strengthened – not indicated post-deposition heat treatment). Gu et al. [174] reported that the high-density dislocations (sub-structures and low angle grain boundaries) and the finer grain size are the main strengthening mechanisms of the 5087 aluminum alloy DEDed-arc + interlayer cold rolling. Furthermore, the material strength (σ_{YS} , σ_{UTS} , and hardness) increased almost linearly with the rolling load, which is related to the final grain size and work hardening (Hall-Petch relation; Figure 2.32a). Xie et al. [129] and Liao et al. [177] (Figure 2.32b) pointed out that the effect of interlayer hot rolling on the 5087 aluminum alloy DEDed-arc strength (σ_{YS} and σ_{UTS}) is almost insignificant; however, an increase in ductility (from 17 to 22%) was observed due to pore closure effect [178]. As previously mentioned, Al alloys had a high SFE energy, which is favorable for recovery during high-temperature deformation; thus, the deposition + hot deformation did not show appreciable improvement in the quasi-static mechanical properties of 5xxx series aluminum alloy despite inducing a fine grain size. In addition, for interlayer cold rolling, material strength increases due to high-density dislocation, a common mechanism of work-hardened alloys (e.g., 5xxx Al) [204]. Therefore, considering the results of the 2xxx ([148], [175]) and 5xxx [129], [174], [177] series aluminum alloys, the interlayer mechanical deformation did not show significant results and efficiency for Al-based alloys fabricated by DED.

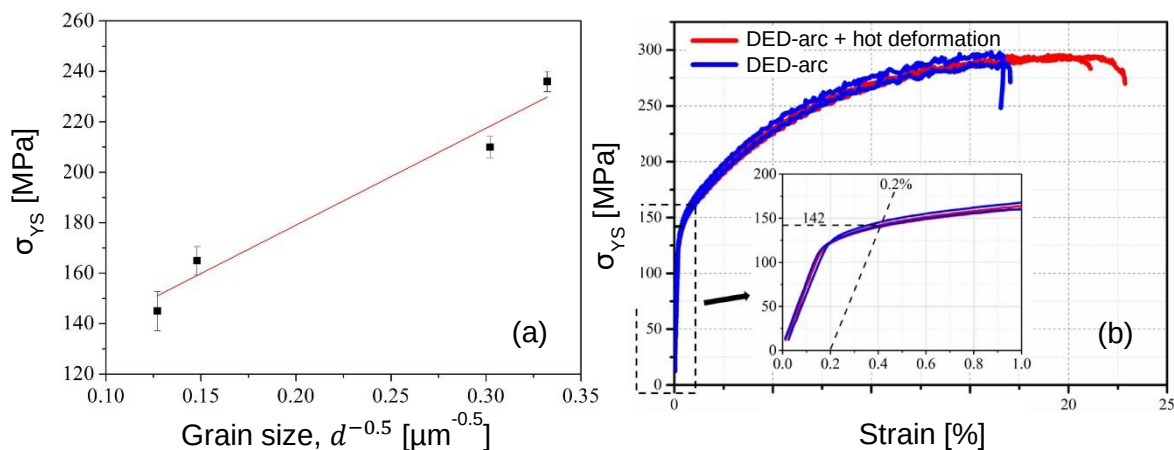


Figure 2.32 – (a) Hall-Petch relationship between the yield stress and the grain size ($d^{-0.5}$ [$\mu\text{m}^{-0.5}$]; adapted from [174]) and (b) stress-strain curve for the aluminum alloy 5087 (hot rolled and unrolled; adapted from [129]).

2.2 Inconel® 718 fabricated via arc plasma DED

Inconel® 718 parts are typically fabricated via casting or machined from a preform or slab (wrought material) [98], [205]. However, the cast Inconel® 718 had inferior mechanical properties (low double-aging heat treatment response) than wrought material [206], which

required an increase in parts thickness and, consequently, total weight (project requirement in aeronautics). In addition, in operational conditions that require fatigue resistance, cast material is not commonly recommended due to coarse grain size and susceptibility to defects, especially pores [207]. On the other side, Inconel® 718 has a low machinability, which results in a high tool consumption and long manufacturing time. Thus, the development of a suitable DED process, especially the arc plasma DED (high deposition rate), for Ni-based superalloys has industrial potential [208] since DED processes can reduce the material waste (near-net-shape process), part weight due to finer microstructure and better heat treatment response than casting, and also enables part topological optimization and redesign.

Moor et al. [209] and English et al. [210] estimated that DED could cost save 25 – 30% for Inconel® 718 parts fabricated via forging + machining (buy-to-fly higher than 10) by minimizing the chip volume (material waste), machining time, and enhancing the tool-life. For casting components, DED can cost save ~ 10% by reducing the rework (usually a problem in cast parts); further, the weight reduction (i.e., material cost) can be performed by replacing heavy cast structures (thicker walls) with arc plasma DEDed one. Therefore, from an economic point of view, the DED is not excessively superior to casting for large-scale Inconel® 718 parts production; on the other hand, when compared to forging + machining, the DED's near-net-shape capacity can reduce the fabrication time (high efficiency) and material waste (lower machining time and chip volume). In addition, it is worth emphasizing that the DED processes offer a flexible production chain (supply decentralization) and shorter lead time [211], which can be especially advantageous for spare parts fabrication and repair operations [212]. However, considering Inconel® 718 parts fabricated via forging + machining, the arc plasma DED has material requirements that are not fully meet, e.g., grain size (API CRA6A, API 20S, and AMS 5662 [105]), quasi-static mechanical properties requirements, and microstructure, preventing the adoption of Inconel® 718 parts fabricated via arc plasma DED [209], [210].

Despite the literature indicating that Inconel® 718 arc-welded had higher mechanical properties than the cast material (AMS 5383) [213], [214], the former usually did not accomplish the wrought material requirements (AMS 5662). Also, given that arc plasma DED processes use a commercial arc-welding machine (e.g., GMA, GTA, and PTA) as a power source and the distinct arc plasma DED boundary conditions and operational aspects (e.g., almost unidirectional heat flux, non-linear path planning, multi-axes load solicitation, absence of material specification standards and codes), the industrial absorption and scalability of the Inconel® 718 fabricated via arc plasma DED in mission-critical parts showed challenges and material requirements barriers. Thus, Graybill et al. [215], Johnson et al. [216], and

James et al. [217] stated that even considering the DED geometric freedom, shorter lead times, cost savings, and process advantages, the inequivalence performance of the Inconel® 718 fabricated via arc plasma DED concerning the wrought material in high temperature, dynamic loads/fatigue, creep, and sour environment limits the full adoption of the Inconel® 718 parts fabricated via AP-DED.

To the best of the authors' knowledge, Clark et al. [205], gas metal arc DED (GMA-DED), first presented results about the Inconel® 718 fabricated via AP-DED. Clark et al. [205] highlighted the presence of hot cracks (heat-affected zone liquation cracking), which were associated with the interdendritic eutectics (γ /Laves). Besides, these defects were also influenced by the part geometry, i.e., the residual stress distribution and heat flux during deposition are a function of part geometry, in which cylinder geometry showed lower printability (higher hot crack susceptibility). These data differ from Benn and Salva [218] (Inconel® 718 fabricated via GMA-DED), which observed a sound deposition (absence of defects) and the accomplishment of the casting material specification (AMS 5383); however, wrought material (AMS 5662) requirements were not met. Clark et al. [219] also observed that the low heat input of the gas tungsten arc DED (GTA-DED) results in a finer microstructure, absence of hot cracks, and a better heat treatment response in relation to GMA-DED [205]. However, due to the low deposition rate and non-coaxial wire feeding/heat source, GTA-DED can be limited to smaller and lower geometric complexity parts. Baufeld [220] evaluated the mechanical properties of the Inconel® 718 fabricated via GTA-DED (as-built condition), which were not addressed by Clark et al. [219]. The as-built GTA-DED outperformed the cast material mechanical properties; however, concerning laser-DED, electron beam-DED, and/or wrought material, the GTA-DED had an inferior performance. In addition, Baufeld [220] (GTA-DED) and Xu et al. [138] (GMA-DED), independently of the AP-DED process, did not observe printing defects (lack of fusion, pores, or hot cracks). Further, coarse and oriented columnar grains (extended through several layers and not meeting the AMS 5662, Figure 2.32) were verified, justifying the anisotropic behavior of Inconel® 718 fabricated via AP-DED. Similar results were also observed by Oguntuase et al. [221], ATI 718Plus® fabricated via GTA-DED, which reported inferior mechanical properties concerning the wrought and material specification.



Figure 2.33 - Tubular square Inconel® 718 GTA-DEDed and cross-section macrograph [220]. The electron backscatter diffraction map was taken from Xu et al. [138].

Xu et al. [105] investigated the effect of in-process oxide formation (environment oxygen reaction), wire feedstock material (composition variation from each supplier), and heat treatment on the mechanical properties of an Inconel® 718 fabricated via GMA-DED (cold metal transfer – CMT® – mode). The authors [105] reported that the first layers showed discontinuous oxide islands and the last layer (54th) a uniform oxide layer with a similar thickness to the first ones, indicating no significant oxide accumulation. In addition, the presence of an oxide layer during deposition unaffected the final mechanical properties, proving that the interlayer grinding is unnecessary. Additionally, independently of the wire supplier (both accomplished the chemical composition requirements of the AWS A5.14), the Inconel® 718 fabricated via GMA-DED (solution + double-aging – AMS 5662) met the cast and did not meet wrought material requirements, which followed the previous results showed by Benn and Salva [218]. Xu et al. [105] and Zhang et al. [222] attributed the lower mechanical properties of Inconel® 718 fabricated via AP-DED to the larger columnar grains ($> 200 \mu\text{m}$ vs. $26.7 \mu\text{m}$; Figure 2.34). Therefore, the main challenge about the Inconel® 718 fabricated via AP-DED was related to inferior mechanical properties concerning the wrought counterparts; this behavior is correlated with the typical coarse and oriented columnar grains and high volume of interdendritic eutectics formation, which reduces the material heat treatment response. Moreover, as previously described by Ling et al. [223], the higher cooling rate of the AP-DED process concerning casting will induce a finer microstructure and lower eutectic phases content, resulting in a better heat treatment response and justifying the superior mechanical properties of the Inconel® 718 fabricated via AP-DED in relation to casting material.

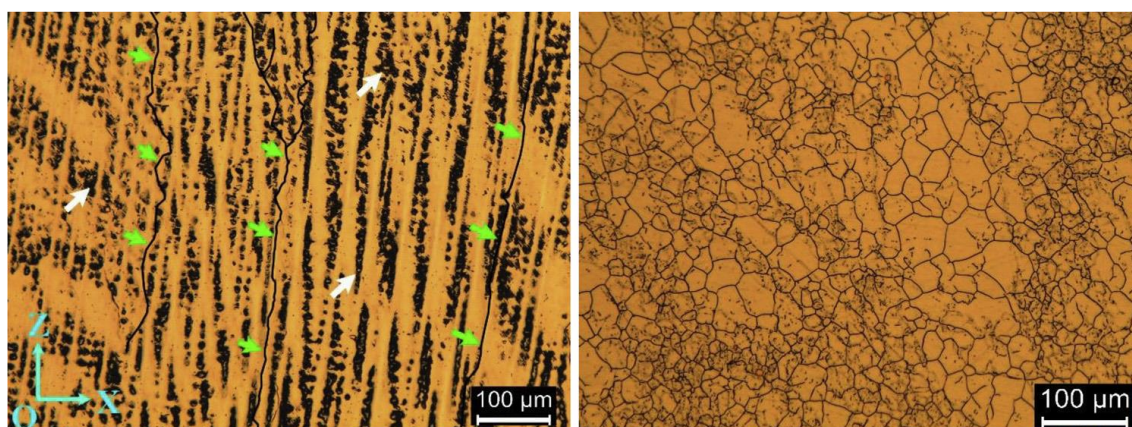


Figure 2.34 – Inconel® 718 microstructure: (left) arc plasma-DED and (right) wrought [105]. White and green arrows are the interdendritic precipitates and grain boundaries, respectively.

An important remark in relation to coarse and oriented microstructure typically observed in Ni-based superalloys fabricated via AP-DED must be reported. While such grains typically demonstrate inferior performance compared to the fine and non-oriented microstructure found in wrought materials, especially under cycle loading [224] and quasi-static mechanical solicitation [225], they offer advantages for creep applications. In high-temperature environments, grain boundary sliding becomes a significant deformation and damage mechanism [226], thereby potentially enhancing the performance of Ni-based superalloys fabricated via AP-DED. A similar observation applies to anisotropic or texturized microstructures, particularly in specialized applications like turbine blades [227], where aligning the maximum load with a specific crystallographic direction can ensure superior performance [228]. Therefore, despite the objective of the present work being to achieve a finer and non-oriented microstructure for Ni-based superalloys fabricated via AP-DED, it is worth noting that materials with a primary microstructure can excel in specific applications.

2.2.1 General metallurgical aspects and implications to additive manufacturing

Inconel® 718 was first commercialized in 1959 by Inco to overcome the low weldability of high-Al and Ti content Ni-based superalloys, which promoted a rapid precipitation and a high γ' content, leading to strain-age cracking; the current Inconel® 718 chemical composition was set around 1985 [229], [230]. This alloy is versatile and has been used in a wide range of environments and applications due to its high electrochemical and oxidation corrosion resistance, outstanding strength and ductility from cryogenic to high-temperatures ($\sim 650^\circ\text{C}$), and creep and thermal fatigue resistance [207]. These characteristics allowed it to be used in

vast industrial fields, such as aeronautics, defense, marine, and oil & gas. For instance, according to Sonar et al. [214] and Schafrik et al. [231], the General Electric CF6 engine consists of 34 % of Inconel[®] 718. This alloy is applied in turbine sections subject to elevated temperatures (ensuring creep and oxidation/wear resistance), such as blades, discs, and high-pressure regions of the compressor. Sims et al. [229] classified the 1980s as the processing era of Ni-based superalloys, i.e., numerous material process routes arose and allowed for Ni-based superalloys to expand their applicability and versatility. However, new challenges also emerged, especially in the additive manufacturing field (from 1990 onwards) [232], e.g., lower material properties and heat treatment response, anisotropic mechanical behavior, and printability problems (e.g., solidification and liquation cracks) [37].

Inconel[®] 718 (γ matrix; cubic; $Fm\bar{3}m$) is mainly strengthened by the fine precipitation of the metastable and coherent γ'' (Ni_3Nb ; $D0_{22}$; tetragonal; $I4/mmm$) and stable and coherent γ' ($Ni_3(Al, Ti)$; cubic; $L1_2$; $Pm\bar{3}m$) phases [233]. In addition, the γ matrix does not undergo allotropic transformations, i.e., this phase is stable from solidification to cryogenic temperatures (primary microstructure non-transformable alloy) [207]. Table 2.6 shows the specified (AMS 5663 and 5383) Inconel[®] 718 chemical composition limits, while Figure 2.35 depicts the crystal lattice of the γ , γ' , γ'' , and δ phases.

Table 2.6 – Inconel[®] 718 composition range (according to AMS 5663 and 5383 standards).

Chemical composition [wt. %]		
Element	Min.	Max.
Ni	50	55
Cr	17	21
Nb + Ta	4.75	5.5
Mo	2.8	3.3
Ti	0.65	1.15
Al	0.2	0.8
Co	–	1
C	–	0.08
Mn	–	0.35
Si	–	0.35
P	–	0.015
S	–	0.015
B	–	0.006
Cu	–	0.3
Pb	–	5 ppm
Fe	Balance	

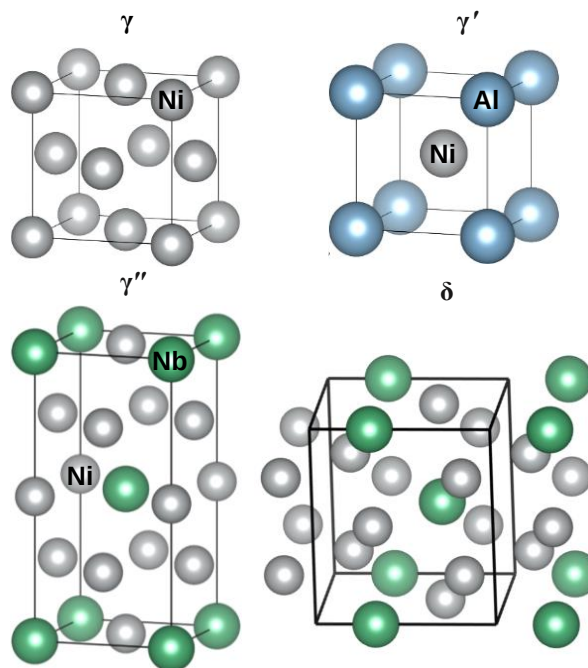


Figure 2.35 – Schematic representation of the γ (cubic; $Fm\bar{3}m$), γ' (cubic; $L12$; $Pm\bar{3}m$), γ'' ($D022$; tetragonal; $I4/mmm$), and δ ($D0a$, orthorhombic, $Pmmn$) [234] crystal structure.

The strengthening efficiency of the γ' and γ'' phases depend upon their size, volume fraction, and the strain induced at the coherency interface [235], [236]. Besides, the solid-solution strengthening generated by elastic stress fields due to differences in atom radius or lattice parameters between the Ni and alloying elements (e.g., Fe, Nb, Al, Co, and Mo) also acts in Inconel[®] 718. In addition, this alloy has a low/medium stacking fault energy (SFE), which restrains dislocation cross-slip, i.e., a high distance between the partial dislocation, which required a high energy to constrict them [237]. Manikandan et al. [238], supported by Dieter and Bacon [239], highlighted the following strengthening sources in Inconel[®] 718 due to precipitates/dislocation interaction:

- SFE difference between precipitates (e.g., γ' and γ'') and γ ;
- The shear modulus difference between the γ matrix and precipitates (e.g., γ' and γ'') generates a stress field around the interface, which interacts with dislocation motion during loading;
- Lattice misfitting (coherency strengthening) between the γ matrix and precipitates (γ' and γ'') generating elastic strain fields;
- Increase of the interfacial energy due to precipitate shearing (change in the morphology and increase the surface area);

- Order strengthening;
- Orowan bypassing mechanism (non-coherent precipitates, e.g., MC-type carbide and Laves phases).

In addition, precipitates with ordered crystal structures can also be strengthened by antiphase domains [240]. Chaturvedi and Han [241] reported that the γ' and γ'' had antiphase boundary energies of 175 and 296 mJ/m², respectively, which also reinforced the γ'' strengthening efficiency.

Polycrystalline Inconel[®] 718 is also strengthened by changes in grain size. The Hall-Petch relation indicates that a finer grain size (resulting in higher grain boundary density) induces a higher yield strength. High-angle ($> 15^\circ$) grain boundaries hinder the dislocation motion through adjacent grains since the deformation system ($\{111\}\langle 110 \rangle$ for FCC materials) continuity is interrupted. In addition, the grain boundaries have a low coordination number and atomic packing factor [242]. Also, due to the interruption of the deformation system, the grain boundaries can cause the dislocation pile-up, which locally increases the stress and promotes the dislocation generation and slip in the adjacent grains [235], [236], [239], [242], [243].

Zhang et al. [222] (Figure 2.36) discriminated the influence of the abovementioned strengthening mechanisms on Inconel[®] 718 fabricated via laser-based PBF. The Peierls-Nabarro (intrinsic) strength (17.5 MPa) was appointed as isotropic, i.e., equivalent in the vertical and horizontal directions. Due to the aligned and coarse columnar grains (leading to high aspect ratio), the grain size strengthening (via Hall-Petch mechanism) had a different effect on vertical (larger effective grain size; 145.4 MPa) and horizontal (178 MPa) directions, contributing to an anisotropic mechanical behavior. The solid-solution strengthening is a function of the atomic percentage of elements dissolved in the matrix; thus, for each material condition (e.g., as-built, direct aging, homogenized + double-aged), this mechanism will have different efficiency, ranging between 250 and 290 MPa [222]. In addition, Nb was appointed as the major solid-solution strengthening element; however, the inhomogeneous chemical element distribution (solidification segregation and deleterious second phase precipitation – Laves phase) reduced the efficiency of the solid-solution strengthening, being effective only in the as-built condition. The Bailey-Hirsch's relation [244] expresses the effect of the dislocation density (work hardening or Taylor strengthening), especially the geometric necessary dislocations for FCC materials. Thus, as Inconel[®] 718 typically undergoes a mandatory post-deposition heat treatment (e.g., homogenization + solution + aging following AMS 5383), this

mechanism has a low influence on the final mechanical properties, being expressive (similar to solid-solution) only in the as-built condition (85 – 170 MPa).

As previously described, the second-phase precipitation strengthening is the principal strengthening mechanism (~ 700 – 900 MPa), which is related to coherency strengthening associated with γ'' and γ' and the ordered structure strengthening, as well as the Orowan by-passing strengthening – Laves phases and MC-type carbides. In addition, the second-phase precipitation strengthening also had an anisotropic behavior, which was attributed to the Taylor factor difference between the vertical and horizontal direction and the allonged Laves phase morphology (ellipse-like; Orowan by-passing). As summarized in Figure 2.36, the yield strength anisotropy of the Inconel® 718 fabricated via PBF is correlated with the effective grain size (grains with a high aspect ratio) and Taylor factor. The latter is a function of the crystallographic texture, i.e., the presence of soft direction texture (e.g., typical cube texture observed in metal additive manufacturing) induces an inferior strength. Zhang et al. [222] results suggest that controlling the grain morphology and orientation, i.e., promote equiaxed grains with low texture is the fundamental point to mitigate the anisotropic behavior of the Inconel® 718 fabricated via AM.

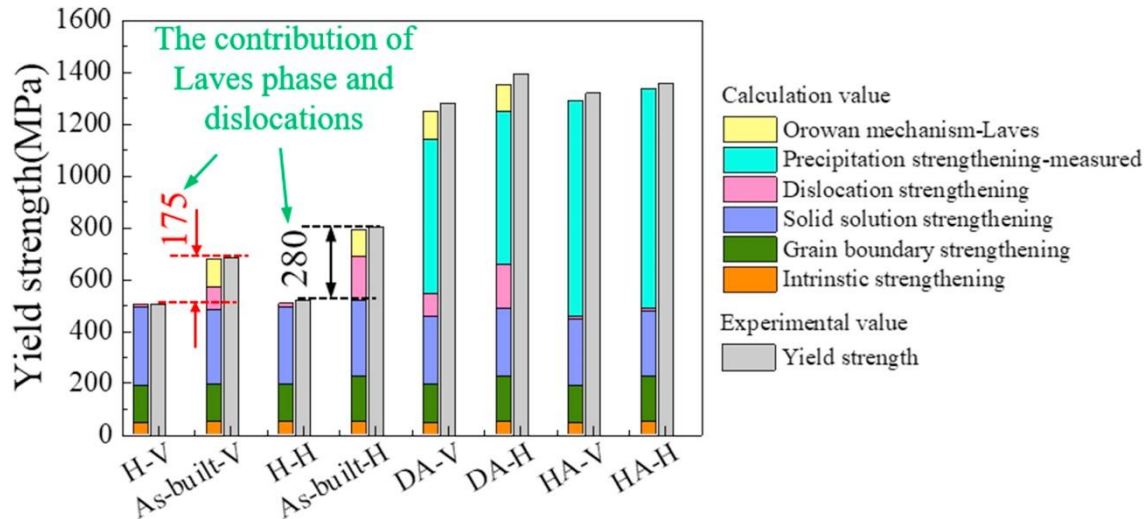


Figure 2.36 – Theoretical strengthening mechanisms contribution for the Ni-based superalloy 718 fabricated via powder bed fusion. H, V, DA, and HA mean horizontal, vertical, direct aging, and homogenized + aging, respectively [222].

The γ'' phase is metastable and can transform into a more stable phase (δ ; Ni_3Nb , D0_{19} , orthorhombic, Pmmn) when exposed to appropriate conditions (e.g., long exposition time at high temperatures) [245]–[247]. δ phase (Figure 2.35) is incoherent with the γ ; thus, its evolution at the expense of γ'' causes a drop in the yield strength. In addition, Medeiros et al. [248] and Azevedo et al. [249] reported that the δ phase works as voids and cracks nucleation sites during

mechanical loading, reducing the material toughness and ductility. At temperatures high than γ'' solvus temperature (~ 930 °C, e.g., during the DED cooling thermal cycle), δ can precipitate directly from the γ in high Nb content regions (e.g., interdendritic regions and γ /Laves eutectics) [250]; besides, previous cold deformation also improves the δ phase kinetics [251]. Figure 2.37 shows the typical Inconel[®] 718 microstructure and phases morphology in the additive manufactured (Figure 2.37a) and thermomechanically processed (Figure 2.37b) conditions, highlighting the effect of the manufacturing routes on the final material microstructure. Additively manufactured Inconel[®] 718 typically had a non-homogeneous distribution and less content of γ'' and γ' , as well as the presence of remaining Laves (undissolved after heat treatment) and δ at high Nb content regions. Otherwise, wrought material had a higher content and almost homogenous distribution of γ'' and γ' and absence of Laves phase, which explain the difference in mechanical strength for these conditions.

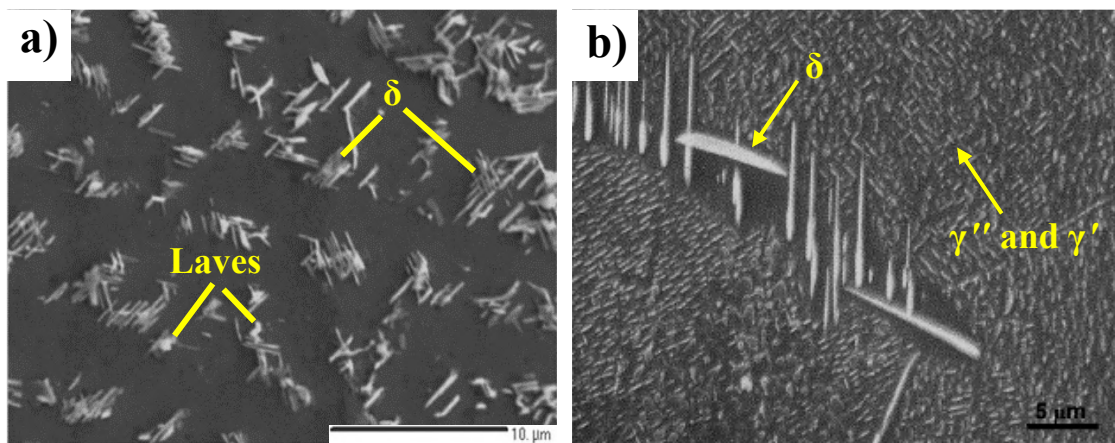


Figure 2.37 – Ni-based superalloy 718 typical microstructure: (a) additive manufactured [252] and (b) wrought material after heat treatment [253].

Qi et al. [254] reported that the ductility of the Inconel[®] 718 fabricated via laser-DED is related to the difference in mechanical proprieties between the dendric core (γ) and interdendritic (Laves and MC-type carbides) regions. Xu et al. [255] and Sui et al. [256] highlighted the effects of the Laves phase (volume, fraction, and morphology) on the fracture behavior of the Inconel[®] 718. These authors [255], [256] pointed out that the long-chain Laves (commonly observed in arc- and laser-based DED) fragmented and debonded (Laves/ γ incoherent interface) when loaded, which can induce crack nucleation and growth, similar to δ phase (incoherent interfaces). Granular Laves phase (e.g., low heat input deposition parameters) kept their integrity (i.e., do not fragment) and promoted the microvoids nucleation and coalescence (dimples generation), inducing a higher elongation [257]. Therefore, Laves

phase in Inconel® 718 fabricated via AM, in addition to consuming Nb and reducing the heat treatment response and mechanical strength, may also limit the alloy's deformation capacity.

2.2.1.1 Inconel® 718 - Solidification

The solidification of Nb-bearing Ni-based superalloys (e.g., Inconel® 718) begins by the formation of a γ nucleus, which grows and simultaneously segregates (Figure 2.38) Nb, Si, Ti, Mo, and C (partition coefficient – k – lower than unity) into the remaining liquid (interdendritic region) [258]–[261]; otherwise, alloying elements with $k \geq 1$ (e.g., Ni, Fe, and Cr) tend to concentrate on the dendritic core. Thus, the interdendritic liquid is enriched, especially by Nb, Mo, and C, until reaching the eutectic composition, and then the first eutectic reaction ($L \rightarrow (Ti, Nb)C + \gamma$) initiates, which is represented by the red line Figure 2.39. This reaction reduces the C content in the liquid; however, the Nb content continuously increases due to the γ formation. According to the Ni-Nb-C liquidus projection (Figure 2.39), the final steps of the Inconel® 718 solidification should be a ternary reaction ($L \rightarrow (Ti, Nb)C + \gamma + \text{Laves}$). However, if the ternary reaction occurred, the final microstructure should have an intermixed constituent forming by NbC, γ , and Laves phases, which was not experimentally observed (Figure 2.39). In this context, DuPont et al. [260], [261] proposed a solidification sequence based on experimental results, which showed that the Ni-Nb-C liquidus projection (Figure 2.39a) followed a class II reaction, i.e., the minimum energy in liquidus surface occurred on the γ and Laves separating line (red line in Figure 2.39). Thus, after primary γ solidification and posterior $L \rightarrow \gamma + (Ti, Nb)C$ reaction, the latter ceases, and the class II (divorced eutectic) reaction initiates, i.e., $L \rightarrow \gamma + (Ti, Nb)C$ finishes and $L \rightarrow \gamma + \text{Laves}$ starts, which results in two distinct eutectics morphologies (i.e., $\gamma/(Ti, Nb)C$ and γ/Laves).

In addition to phases present in DuPont et al. [260], [261] model, Formenti et al. [262] reported the TiN (higher melting point – 2930 °C) at the solidification sequence, which was formed prior to γ ; a similar result was also observed by Silva et al. [259] (Inconel® 625 cladding). Solecka et al. [263] and Farias et al. [264] also reported the presence of higher melting point refractory intermetallics formation in solidification sequence, e.g., TiC, TiN, and Al₂O₃. Yu et al. [265] simulated the Inconel® 718 solidification sequence using the computational Scheil-Gulliver model (Figure 2.39c), which considered the local equilibrium; the results corroborated the abovementioned experimental works and also indicated the presence of the δ and σ phases at the end of solidification. However, Oh et al. [266], high-speed in situ synchrotron X-ray diffraction, did not verify the δ and σ formation during solidification for additive manufacturing cooling conditions (10⁴ °C/s), which reinforces that the experimental

solidification sequence (class II reaction) proposed by DuPont et al. [260], [261] (model proposed for arc-welding conditions) and first reported by Knorovsky et al. [258] (differential thermal analysis) could also be applied for metal additive manufacturing processes (e.g., PBF and DED).

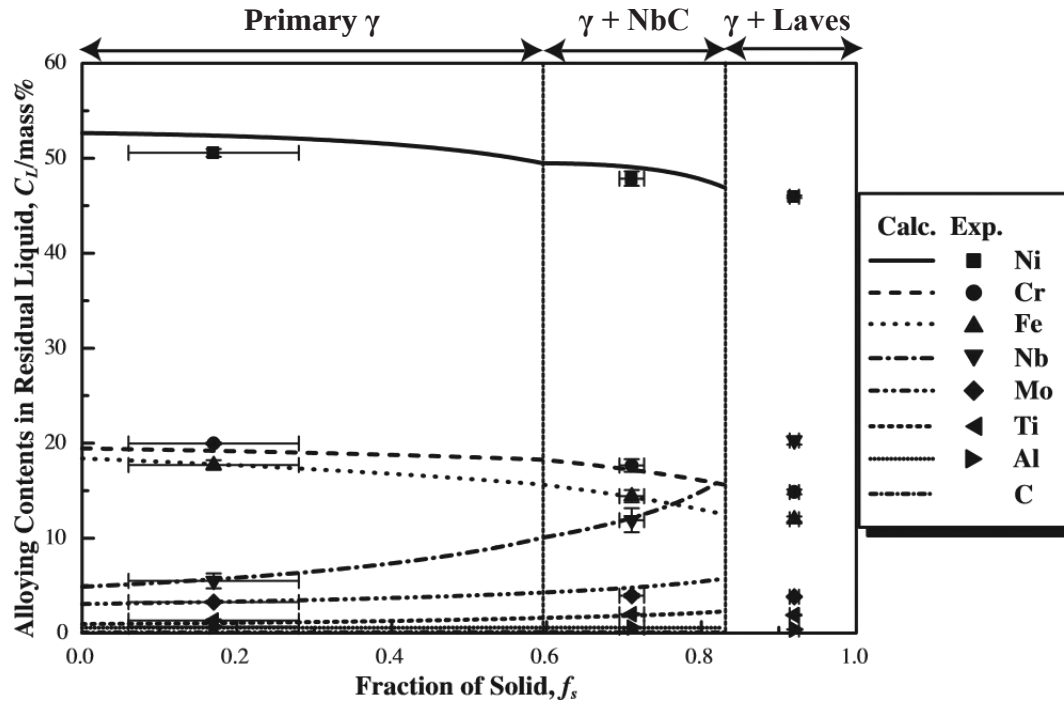


Figure 2.38 – Remain interdendritic chemical composition changes during solidification of the Ni-based superalloy 718 [267].

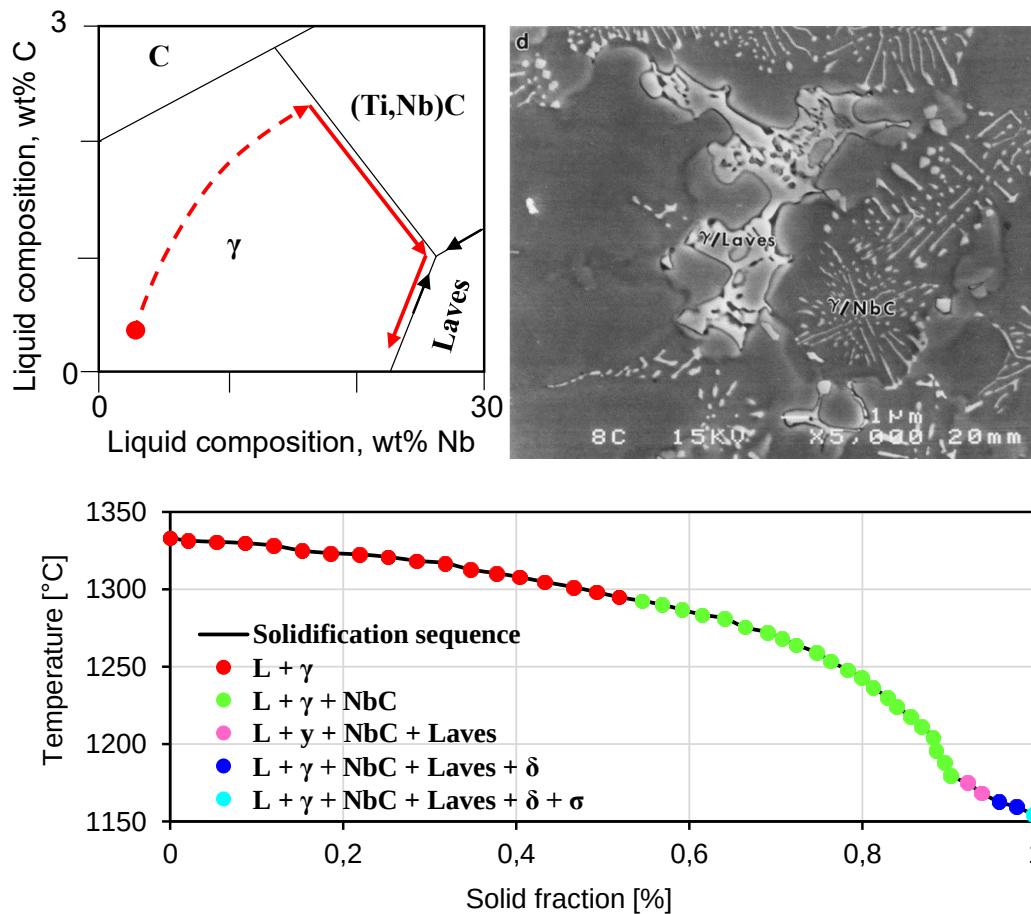


Figure 2.39 – Schematic representation of the solidification path (left) [260], microstructure (right) [261], and Scheil-Gulliver solidification model (below) [265].

The effect of cooling rate on the interdendritic precipitates, especially the Laves phase, was investigated by Antonsson and Fredriksson [268], Ling et al. [223], Manikandan et al. [269], and Shi et al. [270]. Antonsson and Fredriksson [268] showed that the Nb concentration in the dendritic core increased with the cooling rate, i.e., k_{Nb} increases, and the interdendritic segregation decreases with the cooling rate, which was related to the smaller second arm dendritic spacing (Kurz and Fisher [271] model). Ling et al. [223] indicated that the Laves content had a gaussian distribution with the cooling rate (maximum at 3 °C/s), which is also directly correlated with the secondary dendritic arm spacing. Besides, the welding, PBF, and/or DED processes had superior (> 3 °C/s) cooling rates during solidification [67], implying that the Laves phase amount will be lower than for casting material, as experimentally observed by Pereira et al. [272], i.e., it is expected that Inconel® 718 fabricated via A-DED had a better heat treatment response than cast material. Manikandan et al. [269] analyzed the influence of gas tungsten arc welding (GTAW) heat input on the microstructure of the Inconel® 718 (Figure

2.40). These authors showed that the heat input could drastically alter the weld metal microstructure. Higher heat input (lower cooling rate - Rosenthal's model) provided continuous interdendritic precipitates; lower high heat input (higher cooling rate), discrete and non-connected precipitates. These results indicated that the deposition parameters and, consequently, each AM process had a primary effect on the content, morphology, and distribution of interdendritic phases and chemical composition [273]. Thus, a heat treatment for the Inconel[®] 718 fabricated via DED must consider the microstructural aspects (secondary dendritic arm space and Laves phase content) and be developed for each specific AM process and parameters window.

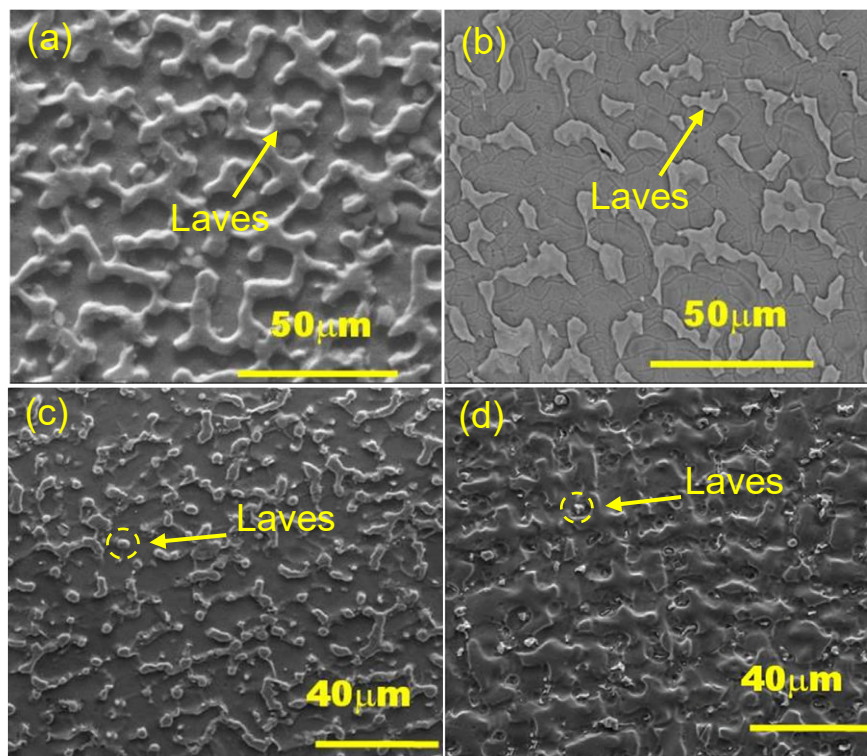


Figure 2.40 – Influence of cooling rate on Ni-saber superalloy weld metal: (a) 43, (b) 48, (c) 80, and (d) 394 °C/s [269].

Zhang et al. [274] compared the Laves phase formation mechanisms, morphology, volume fraction, and distribution for the Inconel[®] 718 fabricated via cast, laser-DED, and PBF. The authors highlighted that solidification mode (columnar-dendritic – additive manufacturing processes – and equiaxed –casting) would induce different Laves phase morphology and composition distribution. In addition, the Nb content in Laves phase is related to the solidification conditions, especially the solid/liquid interface velocity (i.e., solidification rate), in which a high solidification rate (laser-DED and PBF) had a lower Nb concentration in relation to casting (composition close to equilibrium). This explained the better heat treatment

response of the additively manufactured material due to the higher and most homogeneous Nb available to form γ'' during double-aging heat treatment concerning casting. On the other hand, the volume fraction of Laves phase followed an inverse tendency concerning cooling rate, i.e., cast (10^{1-2}), laser-DED (10^5), and PBF (10^7 °C/s) showed 2.8, 3.6, and 16.8% of Laves phase, respectively, which differ from Ling et al. [223], Manikandan et al. [269], and Fernandes et al. [275] results (higher cooling rate lower Laves phase content). Zhang et al. [274] reported that although PBF had a weaker Nb segregation, the finer cellular-dendritic microstructure creates a large interdendritic area with local Nb concentration enough to induce $L \rightarrow \gamma + \text{Laves}$, which can lead to a tiny Laves phase dispersion with an unexpectedly higher volume fraction than casting and laser-DED. Furthermore, Nabavizadeh et al. [58] also addressed, via phase-field model simulation, that the solidification mode influenced Nb distribution and the interdendritic precipitate volume fraction, supporting the Zhang et al. [274] results.

Therefore, as stated by Hosseini and Popovich [276], the Inconel® 718 primary microstructure (secondary dendrites arms space, interdendritic precipitates content, chemical composition distribution, and grain size) developed by the fusion-based processes (e.g., laser-DED, arc plasma-DED, PBF, welding, and casting) will dictate the strengthening phases (γ'' and γ') content and their distribution and the consequently quasi-static and dynamic mechanical properties, where finer primary microstructure processes (e.g., laser- and electron beam-based PBF) tend to perform better/similar to wrought material. Thus, considering the less energy density and higher heat source power by length (i.e., heat input) of the AP-DED in relation to laser- and electron beam-based additive manufacturing processes, it is expected that AP-DED has a lower heat treatment response and mechanical properties concerning laser-DED and PBF [214], [218], [276]–[284]. Thus, the Inconel® 718 fabricated via AP-DED parameters, e.g., deposition energy (heat input), energy density (arc plasma welding process), deposition rate, bead overlapping (layer geometry characteristics), and interlayer dwell time (or interlayer temperature), must be carefully optimized (an arduous and expensive task) to ensure an as fine as possible microstructure and the absence of defects (e.g., lack of fusion and hot cracks, which is not acceptable by ISO 5817) for the most typical part geometry, e.g., multi-layer multi-bead cylinder, wall, and S- and T-shape parts, following the ASTM 52922.

Kumara et al. [285], via a phase-field model, showed (Figure 2.41) that during solidification, even for additive manufacturing conditions (intense cooling rate, from 10^2 °C/s – AP-DED [67] – to 10^5 °C/s – PBF [286]), the dendritic core and interdendritic regions will have different phase transformation kinetics due to inhomogeneous composition, which rose from liquid segregation during solidification. This heterogeneous chemical composition distribution

increases the complexity of heat treatments response for the Inconel[®] 718 fabricated via AM, which can induce deleterious phases (e.g., coarse δ) and low content and heterogeneous distribution of the strengthening phases (γ'' and γ') [221], [287], [288]. Jang et al. [289] and Asala et al. [290], both Inconel[®] 718 fabricated via AP-DED, experimentally observed that the γ'' , γ' , and δ transformation kinetics were faster in the interdendritic region than in the dendritic core, which allowed the in-process precipitation (during the deposition), corroborating the simulation results [285].

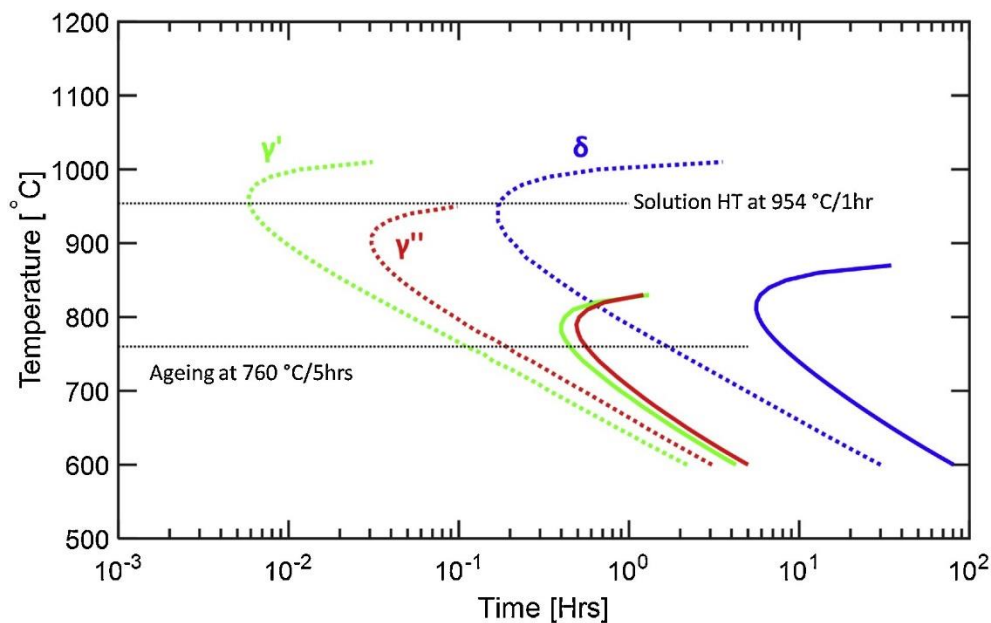


Figure 2.41 – Time-temperature-transformation (TTT) diagram of the Inconel[®] 718 fabricated via laser-DED. Dotted and solid lines represent the precipitation kinetics at the interdendritic region and dendrite core, respectively [285].

Finally, given that the thermal boundary conditions change during the deposition and the cooling rate decreases (e.g., increase of the interlayer temperature) [67], the dendrite arm (primary and secondary) spacing increases (Langer and Müller-Krumbhaar [291] and Kurz and Fisher [271] models) during the part built, which promotes different interdendritic segregation pattern throughout the part (e.g., bottom, middle, and top). Consequently, the time-temperature-transformation (TTT) diagrams (interdendritic and dendritic core regions) also changes during part building [292], which increases the complexity of heat treatment selection since each point of the part will have a different chemical composition distribution and heat treatment response. Besides, the in-process precipitation of hardening phases (γ'' and γ') and δ can generate during the long post-deposition direct double-aging heat treatment (~ 17 h, AMS 5662) an overaging condition (coarse and incoherence precipitates) [289], [293], which induced inferior mechanical properties concerning solution or homogenization + double-

aging conditions [294]. These results indicated that the use of as high as possible homogenization heat treatment temperatures to dissolve almost all precipitated phases formed during solidification and multiple depositions thermal cycles could be a suitable alternative to overcome the material heterogeneities and avoid the undesirable/deleterious phases (e.g., δ , Laves, and σ). However, it is worth noting that heat treatments with high peak temperatures (e.g., ≥ 1100 °C) can induce recrystallization (desirable) and secondary grain growth (undesirable) [153], [265], [295]–[297]; thus, the increase in the heat treatment temperature and soak time must be carefully designed for each DED and PBF processes.

2.2.2 Heat treatments

The age-hardening alloys, such as Inconel[®] 718, must undergo a mandatory (e.g., AMS 5662 and 5383) heat treatment (e.g., solution + double-aging) after part manufacturing (e.g., forging, casting, or AM) to improve the material strength via the hardening phases precipitation (e.g., γ'' and γ'). The heat treatment response depends on the initial microstructure; thus, each manufacturing route (e.g., casting, welding, and forging) will induce a different microstructure and, consequently, a heat treatment response. A similar approach can be extended to AM processes since the cooling rate, feedstock material type, and melting pool morphology differences between them, promoting distinct primary microstructures [287], [297]–[300].

Xu et al. [105] reported that during the solution + double-aging heat treatment (AMS 5662), the grain boundaries act as preferential nucleation sites for γ' and γ'' ; thus, the low grain boundary area (larger grain size; Figure 2.34) of the Inconel[®] 718 fabricated via GMA-DED tends to reduce the heat treatment response concerning wrought material (refined equiaxed grains). In addition, the non-homogenous composition (interdendritic segregation) and low solubilized Nb content (Laves and MC-type carbides consume the Nb) reduces the strengthening effect. Also, the multiple GMA-DED thermal cycles and the increase of precipitation kinetics in high Nb content zones (surrounding the Laves phase) induced the in-process precipitation of γ'' , γ' , and δ phases [292]. In-process precipitation can have deleterious effects during the post-deposition heat treatment, especially for direct aging heat treatment, since the previous γ'' and γ' precipitates can evolve to an overaged condition (reduce the heat treatment efficiency). Thus, although the part of Nb stays in solid-solution after the deposition (as-built condition), it is indicated to avoid the direct aging heat treatment due to its low

solubilization effect (i.e., not dissolve the secondary phase) and to prevent the overaging of the in-process precipitates hardening phases.

Seow et al. [25], PTA-DED, analyzed the Inconel® 718 heat treatment response using direct double-aging, standard homogenization + solution + double-aging (AMS 5383), and modified homogenization (1186 °C/40 min based on incipient melting temperature) + double-aging heat treatments. The authors [25] showed (Figure 2.42) that the AMS 5383 (434 HV) and the modified homogenization (392 HV) heat treatments induced significant changes in microstructure, especially dissolving almost completely the Laves content. On the other hand, due to low peak temperature, the direct aging (411 HV) had a microstructure similar to the as-deposited material (presence of Laves and MC-type interdendritic precipitates). In addition, the AMS 5383 heat treatment induced δ phase precipitation in grain boundaries during the solution step (980 °C/1 h), which must also be avoided, i.e., homogenization + double-aging is preferable to the commercial AMS 5383 heat treatment (homogenization + solution + double-aging). The modified homogenization heat treatment (absence of solution step and higher peak temperature) had a better combination of strength and ductility. However, none of the tested heat treatments met the wrought material specification at 25 and 650 °C (AMS 5662). Besides, the modified homogenization heat treatment induced static recrystallization and excessive grain growth (i.e., secondary grain growth), decreasing the crystallographic texture index and anisotropy concerning AMS 5383 heat treatment at the expense of grain boundary strengthening. In addition to the mechanical properties, the grain size requirement (AMS 5662 and API 6A718) must also be met by the Inconel® 718 fabricated via AP-DED, which was not accomplished by Seow et al. [25] (PTA-DED) and Xu et al. [105] (GMA-DED).

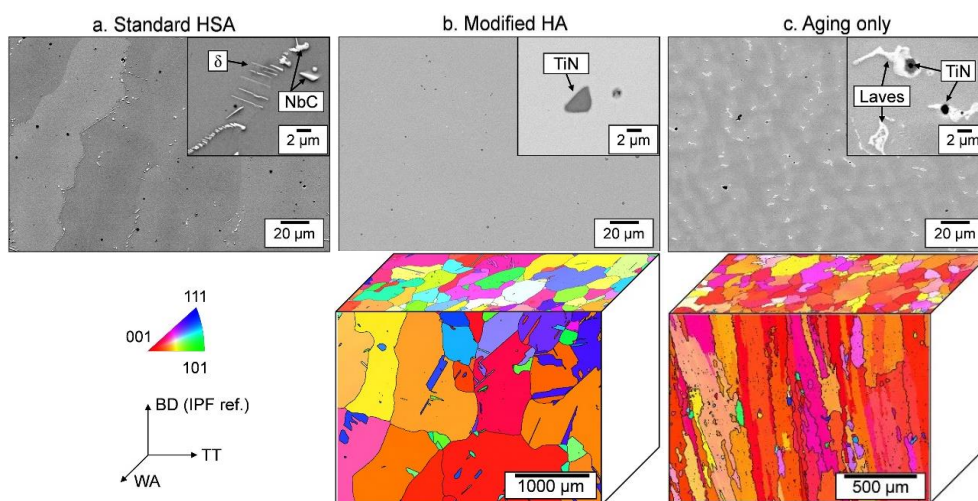


Figure 2.42 – Inconel® 718 fabricated via PTA-DED after post-deposition heat treatments [25].

Wang et al. [301], Inconel[®] 718 fabricated via pulsed PTA-DED, reported that the AMS 5662 heat treatment had a similar effect to those observed for other AP-DED, i.e., partially dissolved interdendritic Laves, precipitated δ phase (grain boundary), and induced a heterogenous distribution of strengthening phases. Besides, pulsed PTA-DED also did not meet the wrought material; however, the casting material specification was accomplished. Bhujangrao et al. [97] and Alonso et al. [98], both PTA-DED, analyzed the as-built behavior of the Inconel[®] 718 at high temperature (650 °C) and observed similar yield and ultimate tensile strength to wrought material; however, the ductility is quite inferior. These authors [97], [98] also indicated that the continuous interdendritic Laves act as crack nucleation and propagation sites, which result in a drop of ductility (absence of the specimen necking) concerning wrought material (no Laves phase).

Xi et al. [107], GMA-DED (CMT[®] pulse mode), using a similar Seow et al. [25] modified heat treatment (homogenization at 1185 °C/ 1 h), observed almost complete dissolution of the interdendritic precipitates (similar to Seow et al. [25]) and absence of static recrystallization and grain growth (different from Seow et al. [25]), which results in a significant increase in yield strength (most homogenous distribution and high content of hardening phases). Xi et al. [107] conducted the first study, to the authors' best knowledge, that met the Inconel[®] 718 yield strength requirements (AMS 5662 and AMS 5383) at room temperature using only deposition + post-deposition heat treatment. Posteriority, Xi et al. [108], using an oscillated deposition path planning, showed that a homogenization at 1150 °C/ 1 h followed by one-step aging (700 °C/ 14 h) is enough to ensure the almost total Laves phase dissolution and homogenous hardening phase precipitation, which promotes an intense strengthening and meet the wrought material quasi-static mechanical properties specification (AMS 5662).

The present authors believe that the main difference in the heat treatment response between PTA-DED and GMA-DED rises from the PTA constrict arc (high energy density process), which causes a higher cooling rate and residual intragranular strain level [302], [303]. Thus, PTA-DED can have enough residual strain and dislocation density to drive static recrystallization and posterior grain growth during a high-temperature homogenization heat treatment (i.e., > 1100 °C), similar to the behavior observed in Inconel[®] 718 fabricated via PBF (ASTM F3055 stress relieved heat treatment at 1065 °C/ 90 min) [287] and laser-DED (AMS 5383 1100 °C/2 h) [153], [304], [305]. These results emphasized the influence and sensibility of the DED processes and parameters selection on the final microstructure and behavior of the Inconel[®] 718. Thus, high-density energy processes (e.g., PTA- and laser-DED) had a superior heat treatment temperature and soaking time limits due to the undesirable

secondary grain growth. In contrast, GMA-DED had less restrictive limits, allowing homogenization temperature close to γ /Laves incipient melting temperature ($\sim 1185\text{ }^{\circ}\text{C}$) and longer soaking times without promoting secondary grain growth [258], [260], [261].

Kindermann et al. [21], [106] tested the AMS 5662 (980 $^{\circ}\text{C}/1\text{ h}$ + double-aging) and API 6A718 (1040 $^{\circ}\text{C}/1\text{ h}$ + double-aging) heat treatments response of the Inconel[®] 718 fabricated via GMA-DED (CMT[®] mode). The authors [21], [106] observed that the API 6A718 heat treatment dissolved almost all interdendritic Laves phase, generating a microstructure with a lower δ content (in relation to AMS 5662) and most homogenous γ'' and γ' distribution (Figure 2.43). In addition, Kindermann et al. [21], [106] did not verify static recrystallization and grain growth, reinforcing the inefficiency of the post-deposition heat treatment in changing the texture and grain size of Inconel[®] 718 fabricated via GMA-DED. AMS 5662 heat treatment had a low effect on Laves phase content, generated a heterogenous γ'' and γ' distribution (higher concentration close to Nb rich region) and higher δ content. Despite these microstructure differences, both heat treatments did not meet the material specification. In addition, Young's modulus was also lower than the wrought material (~ 150 vs. 207 MPa), which was related to the oriented microstructure since the soft direction ($\langle 100 \rangle$) had a higher texture index (cube texture). Besides, due to the typical larger grain size, a low strain hardening exponent (Hollomon's equation) [306] was observed, resulting in a low ultimate tensile strength.

These results reinforced that the standard heat treatments had low efficiency for Inconel[®] 718 fabricated via AP-DED and did not meet the quasi-static mechanical properties requirements. However, it is worth mentioning that an as high as possible homogenization peak temperature promoted a better performance, as observed when comparing, from the worst to the best performance, the direct aging [25], AMS 5662 [21], [106], API 6A718 [21], [106], AMS 5383 [25], and modified AMS 5383 (Xi et al. [107], [108], 1150 or 1185 $^{\circ}\text{C} / 1\text{ h}$) heat treatments responses. Thus, the study and development of dedicated heat treatments for Inconel[®] 718 fabricated via AP-DED become a decisive factor in accomplishing the material requirements and allowing its industrial scalability since the control of microstructure via the deposition parameters is limited (restrict window process, see Section 2.2.2) due to the intrinsic GMA-DED and PTA-DED thermal aspects (high thermal gradient and low solidification rate), which promotes columnar and oriented coarse grains (Figure 2.34a and Figure 2.42c).

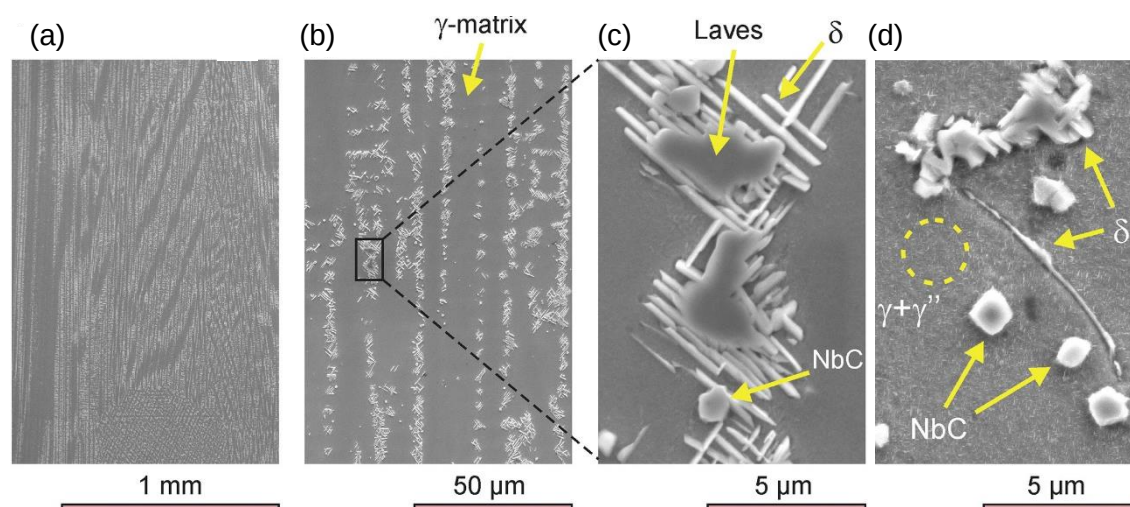


Figure 2.43 – Microstructure of the Inconel® 718 fabricated via GMA-DED (CMT® mode): (a – c) AMS 5663 and (d) API 6A718 heat treatments [21], [106].

Ferreri et al. [307] measured, using neutron diffraction and the Rietveld method, the volume fraction of γ' and γ'' in the Inconel® 718 fabricated via PBF after heat treatment (AMS 5662) and hot isostatic post-processing (1163 °C/ 100 MPa/ 4 h + AMS 5662 heat treatment; ASTM F3055). The authors [307] observed that Inconel® 718 wrought and fabricated via PBF (ASTM F3055) had almost similar volume fractions of γ' (6.5 and 5.4 %) and γ'' (17.8 and 17.7 %), which promoted a close mechanical behavior between these conditions since the precipitation strengthening mechanism is the principal strengthening for this material [222]. Otherwise, Inconel® 718 fabricated via PBF (AMS 5662) had similar γ' and less γ'' (8.4 %) content in relation to Inconel® 718 fabricated via PBF (ASTM F3055), which is related to the higher hot isostatic temperature (1163 °C) that promoted Laves dissolution and higher Nb content available to form γ'' [308]. Ling et al. [309], Inconel® 718 cast, showed that the AMS 5383 heat treatment (1100 °C) also generated a close γ'' content (~ 17.1 %) concerning PBF and wrought material; however, the as-casting coarse microstructure induced a lower strength in relation to wrought and additive manufacturing. Therefore, given the typical industrial heat treatments (e.g., AMS 5662, AMS 5832, and API 6A718) and the abovementioned results, it is expected that Inconel® 718 fabricated via AP-DED had lower mechanical properties concerning wrought material, as well as the non-compliance with material requirements.

Luna et al. [310] tested the effect of AM process and consequently primary microstructure (e.g., AP-DED, wire and powder electron beam DED, laser-DED, electron beam PBF and laser-PBF) on heat treatment response of the Inconel® 625. The authors [310] highlighted that the ASTM 3301 post-deposition procedure (stress relieving, 1066 °C/ 1.5 h,

followed by hot isostatic pressing, 1163 °C/ 3-4 h/ 100 MPa) succeeded by AMS 5666 (solution annealing, 1177 °C /1 h) induced an almost entirely recrystallized grain microstructure, meeting the material requirements (e.g., ASTM F3056) for all tested AM processes (i.e., independently of the primary microstructure). This procedure allowed the 3D printing manufacturer to select the most appropriate or available AM process for a specific application (e.g., part size and geometry, application criticality level, facilities available, and surface aspect) independently of the microstructural aspects.

Considering the Inconel[®] 718 major strengthening mechanism (γ' and γ'' precipitation), a heat treatment sequence that homogenizes the microstructure and chemical composition independently of the 3D printing process (i.e., primary microstructure) and ensures an equivalent γ' and γ'' precipitation (content, morphology, and distribution) becomes a great opportunity and challenge to Inconel[®] 718 fabricated via AM industrial scalability. However, differently from Inconel[®] 625, the Inconel[®] 718 solution annealing temperatures applied to wrought material are lower (1066 °C /1-2 h, AMS 22774), which may not ensure an equivalent γ' and γ'' precipitation between the AM processes (effect of primary microstructure). For instance, the Inconel[®] 718 fabricated via AP-DED heat treated (1052 °C /1 h, API 6A718) [21], [106] results (quasi-static mechanical properties) are inferior to laser-DED [265]. On the other hand, given the Inconel[®] 718 versatility and use in several industrial fields (e.g., turbine jet and spacer grids for nuclear reactor) and operation environments (acid mediums and higher temperatures), the use of a higher solution annealing temperature (> 1100 °C) after the ASTM F3055 procedure (stress relieving, 1065 °C/ 1.5 h, followed by hot isostatic pressing, 1185 °C/ 3-4 h/ 100 MPa) can induce secondary grain growth, which can jeopardize the quasi-static mechanical properties [25] and fatigue behavior [207], [208]. Thus, developing a general workhorse post-deposition heat treatment procedure for the Inconel[®] 718 appears to be a great challenge considering the precipitation strengthening mechanism aspects and the limitations imposed by the material standards and application requirements. However, to the best of the authors' knowledge, it is worth mentioning that no research has been published despite this literature-based analysis.

It is worth mentioning that despite it is possible to meet the Inconel[®] 718 quasi-static mechanical properties specification by selecting a correct/optimized heat treatment, as for the first and only time demonstrated by Xi et al. [158,159], the grain size requirement (ASTM No. 5 or finer) challenge persists. Therefore, during the dedicated heat treatment development for the Inconel[®] 718 fabricated via AP-DED, the present authors recommend considering the

majority/complete dissolution of the Laves and δ phases via as high as possible homogenization heat treatment temperature ($> 1100\text{ }^{\circ}\text{C}$) and soaking time (1 – 2 h), which should also not induce secondary grain growth. Also, avoid the solubilization step ($\sim 980\text{ }^{\circ}\text{C}/1\text{ h}$; acicular δ precipitation) and adopt the AMS 5662 double-aging step ($718\text{ }^{\circ}\text{C}/8\text{ h}$, cooling at $56\text{ }^{\circ}\text{C}/\text{h}$ to $621\text{ }^{\circ}\text{C}$ and $621\text{ }^{\circ}\text{C}/8\text{ h}$) [171].

2.2.3 Printability

The Inconel[®] 718 fabricated via AP-DED printability is mainly influenced by the alloy chemical composition (restricted by its chemical composition range - AWS A5.14) and the process type and their essential variables, which dictated the microstructure, thermal cycles, and thermal stresses [207], [214], [311]. Based on the ASME IX code, the essential variables for AP-DED are the wire feed speed (WFS), travel speed (TS), interpass/interlayer temperature, shielding gas, part geometry, and deposition strategy (e.g., fillet or oscillated pass).

Considering the Inconel[®] 718 fabricated via GMA-DED printability (Figure 2.44), Kindermann et al. [21], [106] (GMA-DED (CMT[®] mode)) used a fast travel speed (16.6 mm/s), resulting in low heat input and some liquation hot cracks and entrapped gas porous close to the fusion line, defects firstly observed by Clark et al. [205] (GMA-DED), which tested the highest travel speed (19 mm/s). Differently, Xu et al. [105] (GMA-DED (CMT[®] mode)), using a higher wire feed speed (7 m/min vs. 6 m/min), obtained sound parts due to lower travel speed (6 mm/s), i.e., slower cooling rate. Lee et al. [289] (GMA-DED (CMT[®] mode)), using a moderate wire feed speed (4.8 m/min) and travel speed (8.3 mm/s), also did not observe defects. Figure 2.44 shows that the Inconel[®] 718 fabricated via GMA-DED printability could be reached via proper process parameter optimization, especially avoiding higher travel speeds, which had an optimal range $5 - 8.3\text{ mm/s}$.

In addition, Zinke et al. [312] analyzed the effect of shielding gas on the Inconel[®] 718 fabricated via GMA-DED printability (WFS and TS of 9 m/min and 10 mm/s , respectively); the authors verified that shielding gas had no pronounced effect on printability, observing, for all tested shielding gas mixture (Ar/30He/2H₂/0.055%CO₂, Ar/30He/1H₂, Ar/2H₂, and Ar/30He/0.03O₂), hot cracks and lack of fusion, which are mainly associated with the travel speed used ($10 - 16\text{ mm/s}$). It is worth mentioning that most of the filler metal suppliers and AWS A5.32 recommended the use of an inert shielding gas mixture (e.g., typically used the mixture 75% Ar + 25% He) to improve the Inconel[®] 718 wettability, increasing the layer width

and reducing the lack of fusion susceptibility [313]. Also, pure argon shielding gas (AWS A5.32 I1 – Ar) is typically used for welding and 3D printing Ni-based superalloys [159].

The wire feed speed showed a wide range of operationally (4 – 9.5 m/min; Figure 2.44), corroborating Kindermann et al. [21], [106] results, which highlighted that only travel speed induced GMA-DED (CMT[®] mode) deposition instability and bead malformation (humping). It is worth emphasizing that not only the bead geometry and process productivity aspects, as considered by Clark et al. [205], Kindermann et al. [21], [106], and Le et al. [20], must be optimized, especially for alloys with a welding defects history, e.g., liquation and solidification hot cracks observed at Ni-based alloys [314], [315]. Besides, these data reinforce the low flexibility to control the secondary dendrite arm space, segregation level, grain size, and solidification conditions via the GMA-DED deposition parameters since the travel speed (associated with the solidification rate) had a narrow operational range.

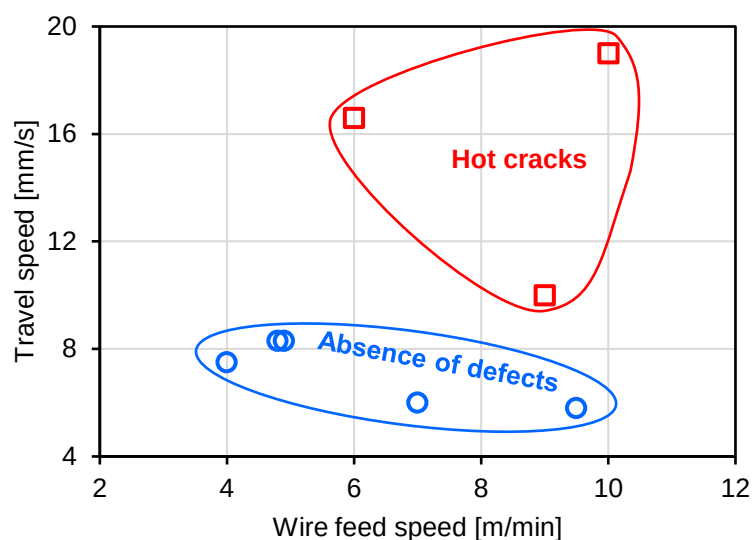


Figure 2.44 – Inconel[®] 718 fabricated via GMA-DED process window. Figure data was available in the Supplementary material.

Seow et al. [316] verified that the Inconel[®] 718 fabricated via PTA-DED fracture toughness had an anisotropic behavior, such as the yield and ultimate tensile strength; the authors [316], [317] attributed this behavior to the hot cracks (Figure 2.45) parallel to the build direction. Specimens with notch perpendicular to the build direction had fracture toughness ($J_{0.2BL(20)}$) comparable to the laser-DED and wrought material (197 ± 28 vs. 121 [265] and $221 \text{ kJ}\cdot\text{m}^{-2}$, respectively). These hot cracks were intergranular (growth through the high-angle grain boundaries) and located in the interdendritic regions, similar to observed by James et al. [318], [319] (Inconel[®] 718 fabricated via PTA-DED). In addition, the defects

occurred periodically throughout the part and were associated with the oscillating deposition path, implying that different part geometry or path planning had different hot crack susceptibility, as previously stated by Clark et al. [205]. Artaza et al. [320], Inconel® 718 fabricated via PTA-DED, mitigated the liquation hot cracks controlling the heat input (inferior to Seow et al. [316]) and idle time (interlayer temperature); the authors [320] described that a higher heat input or interlayer temperature induced a long mushy zone length (partially melted eutectics), which can induce crack nucleation. Further, despite the oscillated path planning resulting in a higher deposition rate, the heat accumulation and increase of the mushy zone size can jeopardize the material printability, being preferable for the Inconel® 718 a linear path planning [321]. Figure 2.46 summarizes the Inconel® 718 fabricated via PTA-DED process window, which did not show a clear operational zone (absence of defects).

Alvarez et al. [322] compared the Inconel® 718 hot cracking susceptibility (Varestraint testing) of GTAW and laser beam welding; the authors [322] concluded that the laser beam welding had low weldability due to the higher contraction strain (high-density energy process) and teardrop shaped melting pool morphology. Xu et al. [323] (PBF) reported that the increase in heat source density increased the defect area fraction (hot cracks), i.e., PTA-DED tends to have poor printability in relation to GMA-DED. In addition, to the authors' best knowledge, all the Inconel® 718 fabricated via GTA-DED showed good printability with the absence of hot cracks [219]–[221], [290]. Thus, from the literature survey, the PTA-DED, despite inducing a finer microstructure, is identified as more susceptible to hot cracks (lower printability) among the AP-DED processes (i.e., PTA > GMA > GTA). This was associated with the constricted arc with higher energy density concerning GTA- and GMA-DED, and the consequently localized thermal stress/strains [302], [324]. Therefore, given the high susceptibility to hot cracks of Inconel® 718 fabricated via PTA-DED and the non-coaxial wire feeding of the GTA-DED (path planning limitations), the GMA-DED appears to be the most suitable process to print Inconel® 718.

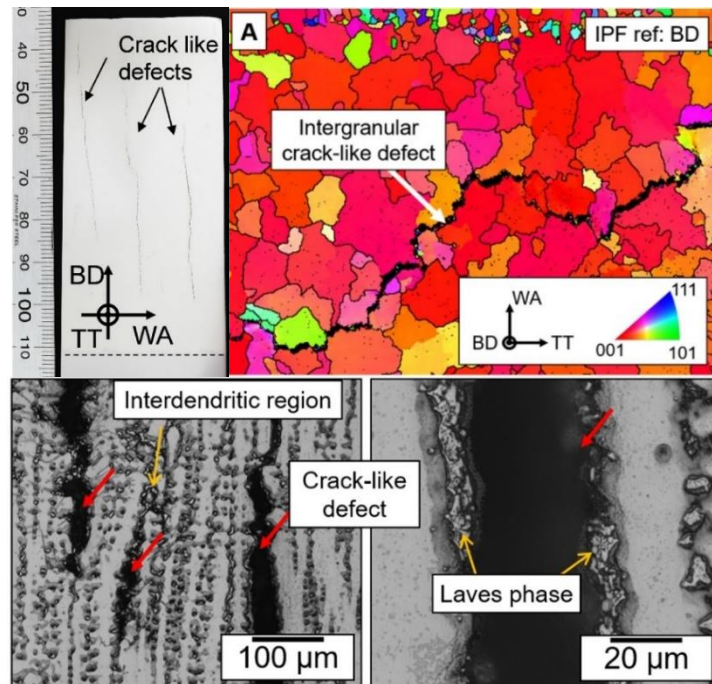


Figure 2.45 – Hot cracks on Ni-based superalloy 718 PTA-DEDed [316].

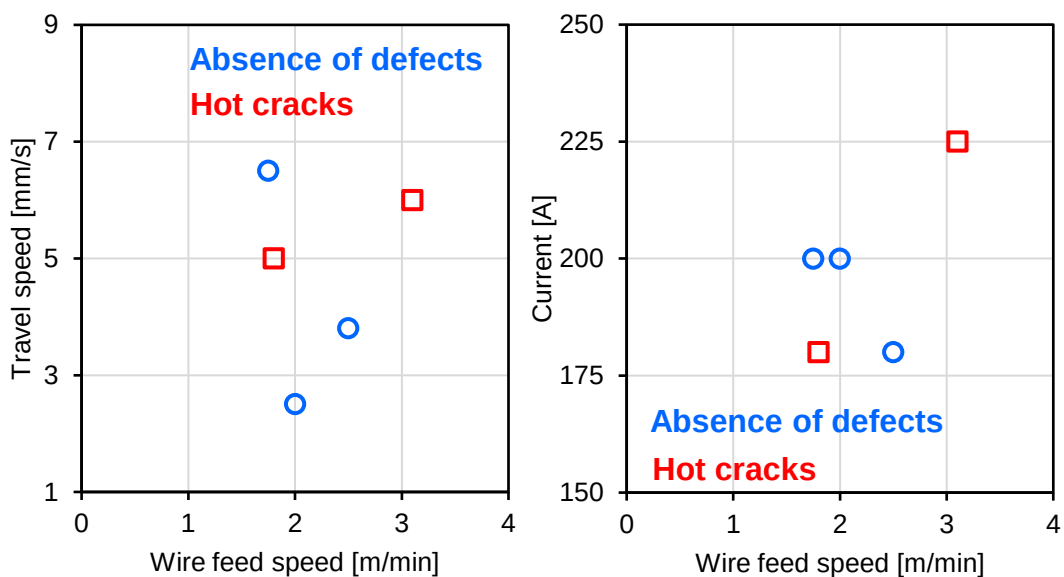


Figure 2.46 – Inconel® 718 fabricated via PTA-DED process window. Figure data was available in the Supplementary material.

2.2.4 Inconel® 718 fabricated via AP-DED + grain refiner systems

Given the abovementioned Inconel® 718 fabricated via AP-DED limitations (e.g., interdendritic segregation, eutectics formation, coarse and oriented grains, and inadequate heat treatment response), Xu et al. [138], Hönnige et al. [37], Zhang et al. [109]–[112], and

Chen et al. [200] applied during the deposition an interlayer deformation (GMA-DED + interlayer cold/hot rolling) to mitigate these drawbacks. The authors verified a significant difference between the GMA-DED and GMA-DED + interlayer rolling, especially in grain morphology and texture index (Figure 2.47), which affected the heat treatment response and material behavior (Table 2.7). For the first time, the Inconel[®] 718 fabricated via AP-DED accomplished both the quasi-static mechanical properties at 25 and 650 °C (AMS 5662) with an almost isotropic behavior ($0.93 < \text{anisotropy coefficient} < 1.07$) and fine grain size (meet the AMS 5662 requirements). These results opened a new research field, which can expand the use of AP-DED for high-responsibility engineering applications, similar to electron beam and laser-based PBF (e.g., ASTM F3055). However, the low path planning flexibility limits the application of the AP-DED + interlayer rolling to simple deposition strategies (e.g., linear walls), driving the development of new interlayer deformation systems, such as coaxial hot forging [35], [36], [159] and hammer penning [152], which had a less limited path planning and can be coupled to a robot arm. However, it is worth mentioning that the use of interlayer deformation makes the process control harder and restricts the process window since the deposition aspects (especially layer height and penetration) are coupled to the thermo-mechanical-affected zone (deformation distribution and depth).

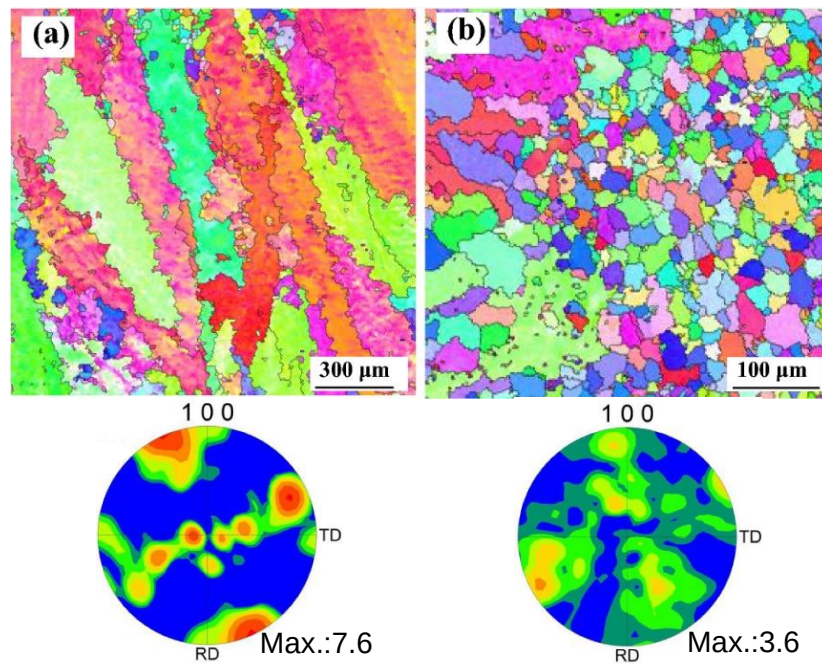


Figure 2.47 – Grain orientation map and pole figures of the Inconel® 718 fabricated via AP-DED and AP-DED + interlayer rolling [111].

Table 2.7– Effect of interlayer rolling on Inconel® 718 fabricated via AP-DED. I and SA mean homogeneous + solution + aging and solution + aging heat treatments, respectively.

	Deformation temperature	DED	Heat treatment	YS [MPa]	UTS [MPa]	Elongation [%]	References
Rolling	cold	GMA	AMS 5662	1035 ± 20	1351 ± 11	17.4 ± 1.1	[138]
Rolling	cold	GMA	HSA	1098 ± 31	1350 ± 23	18.6 ± 0.2	[109]–[112]
Rolling	cold	GMA	SA	1085.5 ± 23	1341.5 ± 20.5	15.7 ± 1.1	
Rolling	hot	GTA	AMS 5662	1112	1304	21.1	[200]
Wrought				1034	1276	12	AMS 5662

Figure 2.48 compares the quasi-static mechanical properties of the Inconel® 718 fabricated via AP-DED with other traditional manufacturing processes and material specifications. The laser-based processes (DED or welding) generally had higher mechanical properties and less anisotropic behavior than the AP-DED. Also, currently, Inconel® 718 fabricated via AP-DED, using the standard heat treatments, can compete with cast parts and fabricate non-critical spare parts (specific demands), which may remodel the Inconel® 718 cast parts production chain. In addition, Inconel® 718 fabricated via AP-DED currently has no comparable properties to forged parts. Thus, post-processing improvements (e.g., specific heat treatments) and the development and enhancement of hybrid processes (e.g., interlayer deformation) must be performed to guarantee that Inconel® 718 fabricated via AP-DED meets the wrought material specifications. In addition, the operational performance, dynamic and fatigue, and creep behaviors of Inconel® 718 fabricated via AP-DED, to the best of the authors'

knowledge, have not yet been addressed, which must be, at least, equivalent to wrought and/or cast material to ensure industrial scalability.

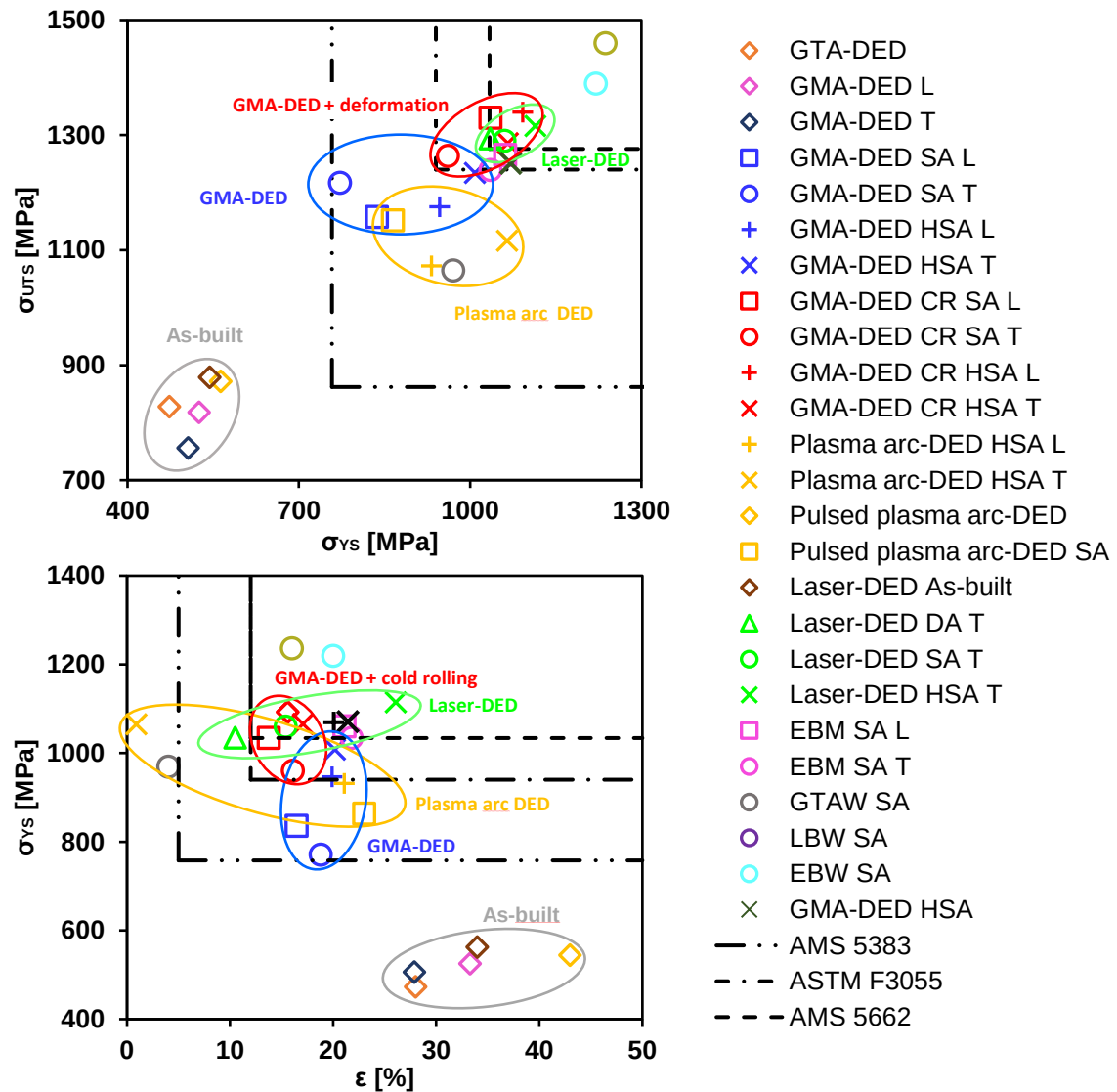


Figure 2.48 – Quasi-static mechanical properties of Inconel® 718 fabricated via AP-DED compared with the material specification and other manufacturing processes. SA and HSA are solution + aging and homogenization + solution + aging heat treatments. CR – cold rolling. L and T – longitudinal and transversal directions. Figure data used is available in the Supplementary material.

2.3 Summary

2.3.1 Interlayer mechanical deformation

- The cold rolling system operates with the highest loads (< 160 kN), requiring a large and rigid structure, which restricts it to linear path planning. However, the high-loading force confers an intense grain size refinement and residual stress relief. The hot rolling systems use lower rolling loads (~ 3 kN), have a higher path planning flexibility (allowing curved trajectory), and show similar grain size refinement.
- The hammering, peening, and forging (cold or hot) systems are more flexible than the rolling ones, use less robust and more adjustable equipment and have almost free path planning (allowing robotization). However, the low-depth deformation zone can induce a lower grain size refinement in relation to rolling, especially for DED-arc (high penetration).
- The interlayer cold deformation system are uncoupled in relation to deposition, allowing better control and reproducibility. The hot deformation system must control the distance between the heat source and deformation tool, which restricted the process window.
- For the cold interlayer deformation, the grain size refinement occurred by the accumulation of strain deformation and static recrystallization during the subsequent deposition thermal cycle and post-deposition heat treatment. The interlayer hot deformation can drive in situ dynamic recrystallization and refining the top layer surface grains, which can also function as nucleation sites during the solidification of the next layer. In addition, the remaining deformed zone can also induce static recrystallizing during post-deposition heat treatment.
- Ti-based alloys and high-strength low-alloy steels (solid-state transformable alloys) showed suitable responses to deposition + interlayer mechanical deformation processes with appreciable parent phases (prior β and austenite, respectively) refinement with a non-oriented primary microstructure, which resulted in almost isotropic quasi-static mechanical properties.

- DED + (cold and hot) deformation had lesser effect on the high SFE alloys, where 2xxx and 5xxx series aluminum alloys did not show significant changes in mechanical properties and microstructure aspects.
- Low to medium SFE alloys (e.g., AISI 316 stainless and Inconel 718) had the best response to interlayer (cold and hot) mechanical deformation. Arc plasma and laser DED + interlayer cold/warm deformation increased the Inconel 718 heat treatment response and reduced the anisotropy and texture index, meeting the requirements of the AMS 5662 standard.

2.3.2 Inconel 718 fabricated via arc plasma DED

- The gas metal arc DED (GMA-DED), especially the cold metal transfer – CMT®, showed a clear process window with lower hot crack susceptibility and better printability than the plasma transferred arc DED (PTA-DED).
- Dedicated post-deposition heat treatments for the Ni-based superalloy 718 fabricated via arc plasma DED must consider an as high as possible homogenization temperature ($\geq 1100\text{ }^{\circ}\text{C}$; dissolve the deleterious second phases) and soaking time (1 - 2 h). In addition, the solubilization step (e.g., $980\text{ }^{\circ}\text{C}/1\text{ h}$; AMS 5662) should be avoided (induce δ precipitation);
- The Ni-based superalloy 718 fabricated via arc plasma DED had an inferior quasi-static mechanical property at room and high ($650\text{ }^{\circ}\text{C}$) temperatures to wrought material (did not meet the AMS 5662 standard requirements) and superior to cast material. In addition, hybrid processes (arc plasma DED + interlayer deformation) increased the Ni-based superalloy 718 fabricated via arc plasma DED heat treatment response, allowing it to accomplish the AMS 5662 requirements.

2.4 Supplementary Material

2.4.1 Figure 2.44 data

		Wire feed speed	Travel speed	Arc plasma Voltage	Arc plasma Current	Aspect	References
		[m/min]	[mm/s]	[V]	[A]		
GMA	CMT®	7	6			Sound	[37]
	CMT®	4.8	8.3	13.4	120	Sound	[289]
	Synergic	10	19	35		Hot crack	[205]
	CMT®	4	7.5		180	Sound	[210]
	CMT®	7	6.0			Sound	[105]
	CMT®	9,5	5.8			Sound	[304]
	CMT®	6	16.6	15.4	143	Pores and hot cracks	[21], [106]
	CMT®	9	10			hot cracks	[312]
	CMT®	7	6			Sound	[109]

2.4.2 Figure 2.46 data

	Wire feed speed	Travel speed	Arc plasma Voltage	Arc plasma Current	Aspect	References
	[m/min]	[mm/s]	[V]	[A]		
PTA	2.5	3.8	20	180	Sound	[98]
	1.75	6.5		200	Sound	[301]
	3.1	6		225	Hot crack	[316]
	1.8	5		180	Hot crack	[318]
	2.5	3.8	20	180	Sound	[320]
	2	2.5	15,7	200	Sound	[321]
	2.5	3.8	24	180	Sound	[97], [322]

2.4.3 Figure 2.48 data

Process	Heat treatment *	Orientation	T	E	σ_{YS}	σ_{UTS}	ϵ	Reference
			[°C]	[MPa]	[MPa]	[MPa]	[%]	
GTAW	SA	T	25		719 ± 39	988 ± 12	26.9	[213]
GTAW	SA	T	650		970	1065	4	[325]
GTAW	SA	T	25		376.9	852	5.9	[326]
LBW	DA	T	25		1210	1424	12	[327]
	SA				1237	1460	16	
	SA (1080 °C)				1215	1405	19	
EBW	SA	T	25		1220	1390	20	[328]
	DA				1235	1375	16	
	SA		650		1070	1210	14	
	DA				1000	1200	12	
Casting	SA		25		940	950	23.1	[329]
			650		517	576	13.7	
Casting	HSA		25		651	909	10.3	[330]
Wrought	SA		25		1192	1380	19.1	[329]
			650		955	1061	13.9	[316]
Wrought	SA		25		1067 ± 30	1391 ± 27	14.8	[248], [265]
			650		1016 ± 37	1178 ± 9	24.6	[331]

Laser-DED	As-built	T	25	158	544	879	43	[265]
	DA			173	1035	1294	10.5	
	SA			185	1060	1290	15.4	
	HSA			222	1114	1316	26.1	
EBM	SA	T	650		793 ± 4	809 ± 14	1	[332]
		45°			757 ± 8	776 ± 12	16.2	
		T			815 ± 27	879 ± 27	8.5	
EBM	SA	L	25		1061	1266	21.1	[333]
		T			1035	1240	21.8	
GTA-DED	As-built	T	25	135 ± 4	473	828	28	[220]
GMA-DED	SA	L	25		836.3	1158	16.4	[110]
		T			771.9	1217.3	18.8	
	HSA	L			946.5	1175.4	19.9	
		T			1008.3	1234.6	20.2	
GMA-DED	SA	L	650		865.7	946.6	7.16	[109]
GMA-DED	As-built	L	25		525	818	33.3	[138]
		T			506	756	27.9	
GMA-DED + cold rolling	SA	L	25		1035	1330.4	13.8	[110]
		T			960.5	1264.4	16.1	
	HSA	L			1092.5	1339.85	15.6	
		T			1065.75	1285.8	17.1	
GMA-DED + cold rolling	SA	L	650		1009.1	1137	11.5	[109]
PTA-DED	As-built	L	25		421	646	17.7	[25]
	modified HSA				932	1073	21.1	
	T	As-built			431	756	21.3	
		DA			951	1072	2	
		HSA			1065	1116	0.9	
		modified HSA			856	1044	19.9	
Pulsed PTA-DED	As-built	L	25		563	872	34	[301]
	SA				864	1152	23	
PTA-DED	As-built	L	650		395 ± 38	628 ± 39	9	[25], [316]
	modified HSA				734 ± 18	821 ± 2	6.7	
	As-built	T			338	621	13.9	
	modified HSA				720 ± 21	800 ± 51	5.9	
GMA-DED	As-built	L	25		597	841	32.91	[107]
	HSA				1070	1249	20.1	
	As-built	T			613	890	33.2	
	HSA				1071	1251	21.4	
Casting	HSA		25		758	862	5	AMS 5383
Wrought	DA		25		1034	1276	12	AMS 5662
			650		862	1000	12	
PBF	DA		25		940	1240	12	ASTM F305
* DA – direct aging (AMS 6662); SA – solution + DA (AMS 6662); HAS – Homogenization + SA (AMS 5383). Longitudinal (L) and traverse (T) mean that the specimen was oriented parallel and 90° in relation to deposition direction, respectively. E, σ_{YS}, σ_{UTS}, and ϵ are Young's Modulus, yield strength, ultimate tensile strength, and elongation, respectively.								

3 IN SITU INTERLAYER HOT FORGING ARC-BASED DIRECTED ENERGY DEPOSITION OF INCONEL[®] 625

Inconel 625 fabricated via arc-based directed energy deposition (DED) has an anisotropic mechanical behavior, reducing the potential applications of arc-based DED in critical components. In this sense, the present work aimed to reduce the grain size and texture by applying an *in situ* interlayer hot forging (HF) combined with post-deposition heat treatments (PDHT). The produced samples were characterized through optical microscopy, scanning electron microscopy coupled with electron backscatter diffraction, synchrotron X-ray diffraction, and Vickers microhardness. Also, a dedicated deformation tool was designed and optimized via a finite element method model considering the processing conditions and thermal cycle experienced by the material. It is shown that the *in situ* interlayer deformation induced a thermo-mechanical-affected zone (dynamic recrystallized + remaining deformation, with a height of ≈ 1.2 mm) at the bead top surface, which resulted in thinner aligned grains and lower texture index in relation to as-built DED counterpart. In addition, the effects of solution (1100 °C/ 1 h) and stabilization (980 °C/ 1 h) PDHTs on the Inconel[®] 625 HF-DED parts were also analyzed, which promoted fine and equiaxed static recrystallized grains without cube orientation, comparable to wrought material. Therefore, the HF-DED process significantly refined the typical coarse and highly oriented microstructure of Ni-based superalloys obtained by arc-based DED.

3.1 Process development and microstructure effects

3.1.1 Introduction

Directed energy deposition (DED) additive manufacturing processes, especially arc-based ones (also known as wire arc additive manufacturing – WAAM[®]), have high deposition rates (2 – 6 kg/h), which makes them appropriate to fabricate medium to high volume metal parts as well as for being used in repair operations [1]. Arc-based DED systems consist of a heat source (a commercial welding machine, e.g., gas metal arc – GMA) that melts the feedstock material (in the form of wire) and a movement system (e.g., a robotic arm or CNC machine) that follows a previously optimized deposition strategy [334], [335]. Arc-based DED produces a primary part close to the final desired geometry, reducing the material waste and post-processing time and cost (e.g., by machining methods) [17], [336], [337]. Arc-based DED processes are especially advantageous for high-cost alloys and low-machinability materials (e.g., Ti- and Ni-based alloys). In this context, Moor et al. [209] and English et al. [210] assessed

that arc-based DED could promote savings of up to 25 to 30% in the fabrication of Ni-based superalloys parts with complex geometries (e.g., fittings, impellers, and spars for the aeronautic industry) [338] when compared to traditional manufacturing routes (e.g., machining from a billet) due to minimization of the buy-to-fly ratio and machining time [15], [339]. Additionally, due to the absence of dies and other dedicated tools, arc-based DED increases the production chain flexibility and promotes decentralization, which is especially advantageous for unconventional spare parts manufacturing (shorter lead times) [2].

Despite the abovementioned advantages, Ni-based superalloys parts fabricated via arc-based DED, especially Inconel[®] 625 and Inconel[®] 718, have critical material- and process-related challenges [340], [341]. Among them, it can highlight the coarse columnar grains and a highly oriented microstructure [342]–[345], high distortions and residual stress levels [37], low printability (susceptible to hot cracks) [317], poor heat treatment response [346]–[349], and inferior mechanical properties (i.e., does not meet the ASTM B 446 – 03) and corrosion resistance (both electrolytic and oxidation) [350]–[354], which can limit the full industrial adoption of arc-based DED in the oil & gas, aeronautics, nuclear, and naval fields for critical engineering parts owing to rigid standards requirements (e.g., API 6ACRA, DNVGL-ST-F101, and AMS 5662 standards).

In this scenery, arc-based DED variants, hybrid processes, and redesign of the feedstock material composition have been developed to induce grain size refinement (columnar to equiaxial transition [57] or recrystallization [53]) and reduce solidification segregation [58], which can be achieved by the use of ultrasound vibrations [59]–[61], external magnetic field [62], interlayer (cold or hot) deformation [35], [136], [199], forced cooling systems [63], the addition of inoculants in the melt pool [64], and increase the feedstock material alloying elements content [65]. Among these alternatives, the interlayer mechanical deformation variants stand out for simultaneously reducing the residual stress (promoting local yielding), inducing grain size refinement (via dynamic and static recrystallization mechanisms), and not altering the feedstock material specification [66]. Deposition + interlayer deformation (rolling, peening, or forging) was first employed in arc-based welding to improve the welded joint fatigue life (relieving residual stresses) and filler metal weldability [38]–[40], [43]. However, considering the typical geometric aspects associated with additive manufacturing (complex 3D geometries and no deformation restrictions), the DED + interlayer deformation has challenges related to higher residual stresses, non-linear path planning, and coupled deposition and deformation parameters (i.e., avoiding the entire remelt of the deformed zone and not induce deposition defects due to layer morphology modification). In addition, typical commercials

alloys fabricated by arc-based DED (e.g., Ti-6Al-4V [44], Al series 2xxx [48], AISI 316 L stainless steel [49], and Inconel[®] 718 [52]) showed adequate formability, reinforcing the potential use of arc-based DED + interlayer deformation.

Concerning Ni-based superalloys, to the best of the authors' knowledge, only Zhao et al. [128] (laser-DED + interlayer hot rolling) and Li et al. [157] (laser-DED + interlayer hot micro-forging) studied the solid-solution strengthened superalloys (Ni 60 and Ni80-Cr20 alloys, respectively) fabricated by DED + interlayer deformation. These authors [128], [157] observed that the interlayer deformation induced a finer and less oriented microstructure, increased the microhardness, and reduced the wear rate in relation to the deformation-free DED counterparts. For precipitation-strengthened superalloys, only Inconel[®] 718 was studied. Xu et al. [138] and Hönnige et al. [37] (GMA-DED + interlayer cold rolling), Zhang et al. [110] and Chen et al. [200] (GMA-DED + interlayer hot rolling), Li et al. [136] (laser-DED + interlayer hot rolling), and Whang et al. [153] (laser-DED + interlayer cold peening) observed that the interlayer deformation induced a grain size similar to that of wrought material (e.g., 8 [136], 12.7 [138], 25 ~ 30 μm [110], and vs. 15 μm ; both after the homogenization heat treatment) and a low crystallographic texture index (< 3), improved the post-deposition heat treatment response, and enhanced the quasi-static mechanical properties (meeting the AMS 5662 requirements). Despite the efficient grain size refinement and residual stress relieving due to interlayer cold or hot rolling, the low path planning flexibility may limit its industrial scalability [144]. In addition, both rolling, forging, and peening required a dedicated auxiliary facility with high structural rigidity to enable material deformation.

Recently, to overcome the path planning limitations and deposition strategies restrictions, Duarte et al. [36] developed a GMA-DED process coupled with an *in situ* coaxial hot forging mechanism, which deformed the newly consolidated feedstock material and allowed unrestricted changes in planar deposition directions. Furthermore, Duarte et al. [36] reported that the *in situ* interlayer hot forging reduced the residual stress and refined the microstructure of a CuAl8 alloy fabricated by GMA-DED. Thus, considering the commonly observed coarse and highly oriented microstructure of the Inconel[®] 625 fabricated by GMA-DED (both in as-built and post-deposition heat-treated conditions) [37], [317], [340]–[344], [346]–[354], the present work aimed to refine the grain size and reduce the texture of the Inconel[®] 625 fabricated by GMA-DED via an *in situ* interlayer coaxial hot forging variant. To optimize the forging temperature, the coaxial hammer was designed based on the thermal profile obtained through a thermo-metallurgical finite element method (FEM) model. Inconel[®] 625 GMA-DED parts (with and without *in situ* interlayer hot forging) were microstructurally

characterized through optical microscopy (OM), scanning electron microscopy (SEM) coupled with electron backscatter diffraction (EBSD), synchrotron X-ray diffraction (SXR) and microhardness. Finally, the effect of post-deposition heat treatments (solution and stabilization annealing) on Inconel® 625 fabricated by GMA-DED + *in situ* interlayer hot forging was also evaluated.

3.1.2 Materials and methods

3.1.2.1 Materials and process description

An AWS A5.14 ERNiCrMo-3 (UNS N06625; Inconel® 625) filler metal wire (1.2 mm) was deposited on an ASTM A36 Q235 hot rolled carbon steel plate (80 × 10 × 200 mm³) to build single-bead 10-layer walls. The arc-based DED machine consisted of a KEMPY PRO MIG 3200 power source (gas metal arc – GMA) coupled to an in-house developed 3-axis CNC machine [355]. The experimental apparatus was demonstrated in the Supplementary material. Table 3.1 summarizes the main deposition parameters, which were previously optimized considering the feedstock material supplier (ESAB®) guidelines and a design of experiments (Supplementary material). The electric current and arc plasma voltage oscillograms were acquired with a data acquisition system and a LEM LA 200-P current probe (sampling rate of 5 kHz). Figure 3.1 shows the final aspect of the part fabricated by Arc plasma-DED + *in situ* interlayer hot forging.

Table 3.1 - Arc plasma-DED + *in situ* interlayer hot forging parameters

Arc plasma-DED		
Electric current*	72.1	[A]
Arc plasma voltage*	17.1	[V]
Wire feed speed	3.5	[m/min]
Travel speed	5.0	[mm/s]
Heat input (η = 0.85) [60]	209.6	[J/mm]
Interlayer temperature	150	[°C]
Wire diameter	1.2	[mm]
Contact tip to work distance	10	[mm]
Shielding gas	Ar (Type II; Grade C)	
Shielding gas specification	AWS A5.32	
Shielding gas flow	15	[l/min]
Linear deposition length	100	[mm]
Layer numbers	10	
Substrate preparation	ISO 8501-1	
<i>In situ</i> interlayer hot forging		
Pneumatic pressure	5	[MPa]
Frequency	8	[Hz]

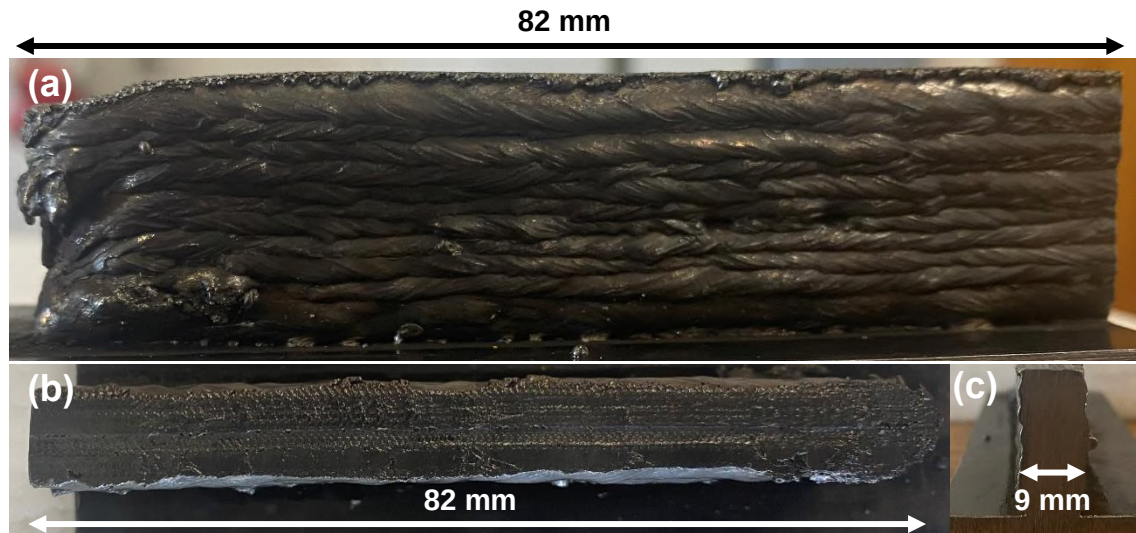


Figure 3.1 - Macroscopic aspects of the *in situ* interlayer hot forging arc plasma directed energy deposition of Inconel® 625: (a) side view, (b) top view, and (c) cross-section of a single-bead multi-layer 3D printed part.

3.1.2.2 *In situ* interlayer hot forging system

The *in situ* interlayer hot forging system, detailed in Figure 3.2, consists of two pneumatics actuators (Festo ADN-12-10-I-P-A) positioned symmetrically in relation to the welding torch that pushes/pulls the moving parts (gas nozzle and the circular crown hammer, which are mounted on a linear bearing), which hot deformed the material immediately after the deposition. The main *in situ* interlayer hot forging parameters are the forging force (operationally expressed by the pneumatic pressure) and the forging temperature (operationally expressed by the circular crown hammer radius). The smaller the hammer radius, the higher the forging temperature. For more details on the *in situ* interlayer hot forging design, parameters, and equipment, see Duarte et al. [36].

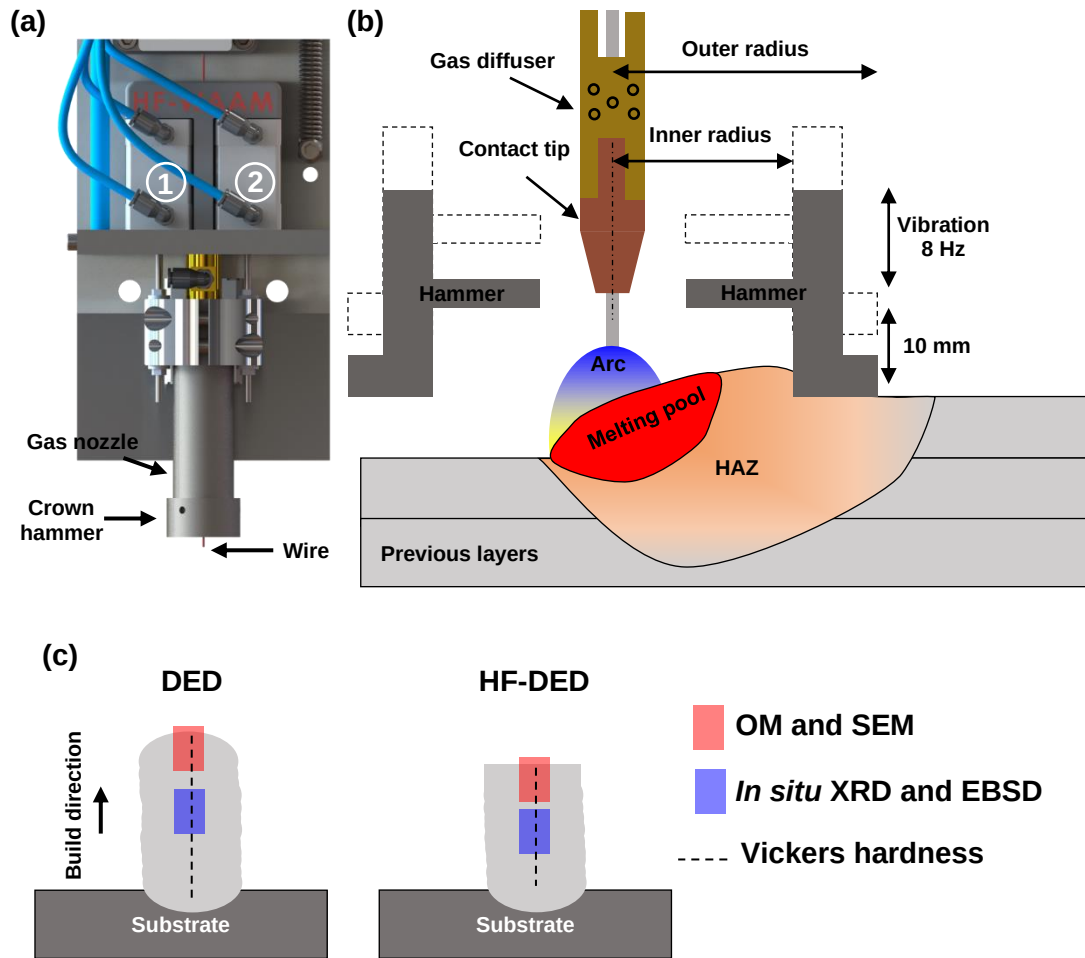


Figure 3.2– Schematic representation of the arc-based DED + in situ interlayer hot forging: equipment (a) and process (b), and (c) location where the microstructure characterization was performed at printed walls. HAZ corresponds to the heat-affected zone; ① and ② are the pneumatic actuators.

3.1.2.3 Post-deposition heat treatments

Post-deposition heat treatments (PDHT; Figure 3.3a) were performed in a furnace with a heating rate of 20 °C/s. The soaking time was 1 h at the peak temperature, and the cooling rate was 20 °C/s. The PDHTs (solution and stabilization annealing) followed the Inconel® 625 specification (ASTM B 446). The solution annealing (1100 °C/1 h) is used to promote the Laves phase dissolution [25], [346] and the homogenization of the solidification (as-built) interdendritic segregation. The stabilization annealing (980 °C/1 h) promotes the MC-type carbide precipitation (stabilization), preventing M₆C-type carbide precipitation and alloy sensitization [249], [356]. In addition, the literature [107], [138], [346]–[348], [357] highlighted that the PDHT had a negligible effect on the grain size, grain morphology, and crystallographic texture of Ni-based superalloys fabricated by GMA-DED, which was related to the low driving force (dislocation density and in-process developed deformation) for recrystallization or

significant grain boundary mobility. Thus, GMA-DED Ni-based superalloys retain the primary solidification grain microstructure even after PDHT. In opposition, high-density energy DED processes (e.g., laser-based) can undergo static recrystallization during the solution annealing due to the higher residual deformation developed during the deposition [25], [358], [359]. Thus, since it is consensus that PDHT does not promote significant grain size and grain morphology changes in Ni-based superalloys obtained by GMA-DED and considering the present objective (refining the grain size and reducing the texture), the stabilization and solution annealing PDHTs were carried out only in the arc-based DED + *in situ* interlayer hot forging (HF-DED) specimens.

3.1.2.4 Hot forging dynamic characterization

The hot forging system was dynamically characterized to assess the forging conditions by measuring the hammer speed and deceleration, which were used to calculate the forging force and strain rate during the *in situ* interlayer hot forging. A Photron FASTCAM Mini WX50 high-speed camera (operating at 10,000 fps and 512×256 pixels) with a Nikon AF NIKKOR 28–105 mm macro lens was used. Data post-processing was performed using a Python-based routine, where the frames were binarized, allowing acquired the instantaneous position of the moving parts (see section 2.1.1).

A Fluke TI400 infrared camera (7.5 – 14 μm wavelength) with an acquisition rate of 9 Hz and a resolution of 320×240 pixels was used to measure the forging temperature. To select the emissivity (0.76), the infrared camera thermal cycle was previously validated with thermocouples.

3.1.2.5 Microstructural characterization

The microstructure of the Inconel[®] 625 fabricated by GMA-DED was characterized via OM (Leica DMI 5000 M) and SEM (SU3800 Hitachi). The metallographic specimens were, in sequence, ground, polished, and electrolytically etched (10 wt.% chromium trioxides – Cr_2O_3 – in water, 5 V for 20 s). Figure 3.2c shows the position in the samples where the metallographic analyses were performed. The EBSD specimens followed the same metallographic procedure to that used for OM and SEM adding a final mechanical polishing step with colloidal silica, 0.04 μm , for 2 h (VibroMet[®]). The step size, voltage, and tilt angle used in EBSD analyses were 1 μm , 20 kV, and 70 °, respectively. EBSD data post-processing was carried out using the open-source MTEX[®] Toolbox [360]. Misorientations higher than 15 ° were considered high-angle

grain boundaries. The grain size was measured through the intercept method (ASTM E 112) using a square grid [222].

3.1.2.6 Vickers microhardness

Vickers microhardness (Mitutoyo HM-112) profiles (straight vertical line through the entire wall; Figure 3.2b) were performed using a load of 0.3 kgf ($HV_{0.3}$), a dwell time of 15 s, and an indentation distance of 0.5 mm, following the ISO 6507-1 standard.

3.1.2.7 Synchrotron X-ray diffraction

Synchrotron X-ray diffractions (SXR) were conducted at the High Energy Materials Science P07 beamline of PETRA III at the DESY synchrotron. A wavelength of 0.14235 Å (87.09 keV) was used. Rectangle samples ($10 \times 20 \text{ mm}^2$; Figure 3.2c) with a thickness of 2 mm were used, and the X-ray beam was focused on the middle of the samples. Before X-ray exposure, the samples were ground and polished. A 2D Perkin Elmer detector with a pixel size of $200 \times 200 \mu\text{m}^2$ was used to acquire the Debye-Scherrer diffraction rings, which were integrated along the full azimuthal angle to acquire the typical $2\theta \times$ intensity diffraction patterns. SXR beam spot size and exposure time were $1 \times 1 \text{ mm}^2$ and 4 s, respectively. The instrumental peak broadening was determined using LaB6 (calibrant powder). For more details about SXR set-up and data post-processing, see Rodrigues et al. [361]. Figure 3.3b details the experimental apparatus for in situ SXR during the heat treatments.

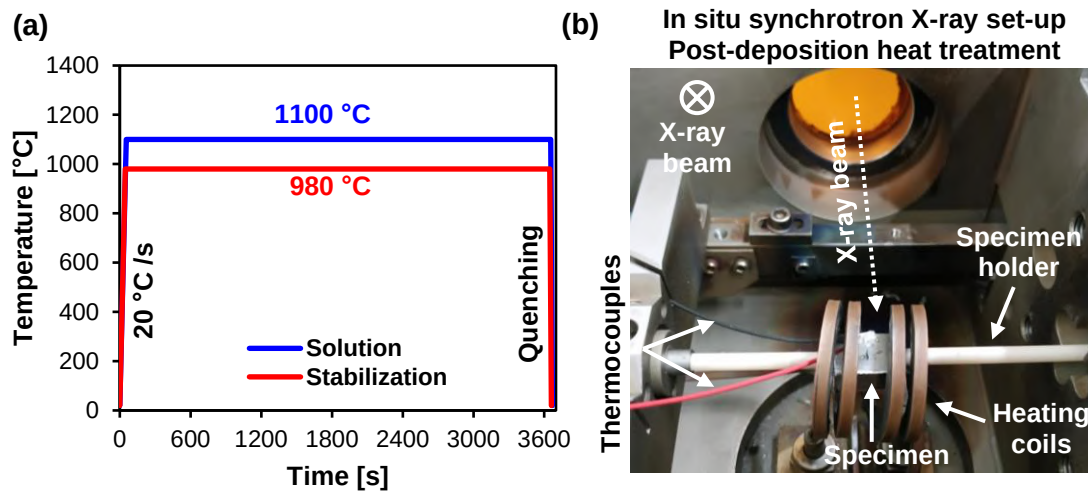


Figure 3.3 - (a) Solution and stabilization post-deposition heat treatments (PDHT), (b) *in situ* SXRD and PDHT apparatus.

3.1.2.8 GMA-DED modeling

To assess the thermal profile during deposition and estimate the ideal hot forging stroke position, a thermo-metallurgical finite element method (FEM) model was developed based on an Inconel® 625 10-layer single-bead wall deposited by GMA-DED. This model was developed using the commercial ESI Sysweld® FEM software. The actual physical dimensions of the substrate and GMA-DEDed wall and measured deposition parameters were used as model inputs. Figure 3.4 depicts the model configuration and mesh size. In addition, two k-type thermocouples, positioned at 9 and 14 mm (red dots in Figure 3.4) from the deposited bead edge, were used to validate the simulation results experimentally.

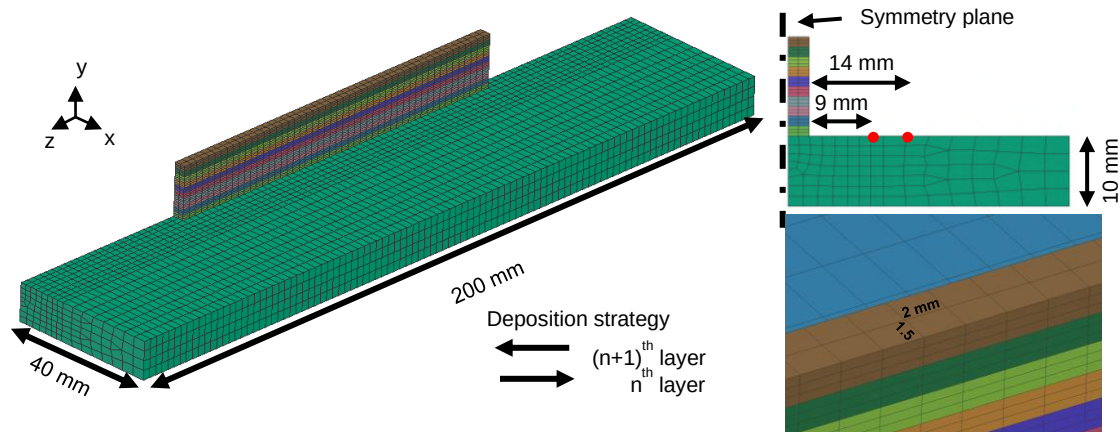


Figure 3.4 – Finite element model mesh. The red dots represent the position of thermocouples.

The present model was based on the energy balance and the multiphase transient heat flux with a volumetric and mobile heat source (modified Fourier equation; Equation 2.1) [362], [363]. T , v , k , ρ , c_p , and $\dot{Q}$ are the temperature, deposition velocity, thermal conductivity, density, specific heat, and the volumetric heat source, respectively. Both the feedstock material and substrate isotropic physical properties (k , ρ , and c_p) were extracted from the ESI Sysweld[®] material database (detailed in the Supplementary material). $\dot{Q}$ was modeled using the Goldak's double-ellipsoidal [364]. The heat source geometrical and deposition electric parameters are available in the Supplementary material. The initial ($t = 0$; Equation 2.2) and boundary (Equation 2.3) conditions considered that all the activated mesh nodes were at 27 °C (T_0) and the radiation and convection flux occurred on the model surface, respectively. $\vec{n}$, ε , σ , and h are a unitary vector perpendicular to the model surface, the Stefan–Boltzmann constant, the gray body emissivity (0.76), and the convective heat transfer coefficient, respectively. h was defined as 20 W·m⁻²·K⁻¹ for the surfaces exposed to air and 100 W·m⁻²·K⁻¹ for the substrate bottom surface (simulating the heat flux to the welding table) [67].

$$\frac{\partial}{\partial x} \left(k \frac{\partial T}{\partial x} \right) + \frac{\partial}{\partial y} \left(k \frac{\partial T}{\partial y} \right) + \frac{\partial}{\partial z} \left(k \frac{\partial T}{\partial z} \right) + \dot{Q} = \rho c_p \left(\frac{\partial T}{\partial t} - v \frac{\partial T}{\partial x} \right) \quad (2.1)$$

$$T(x, y, z, t = 0) = T_0 = 27 \text{ °C} \quad (2.2)$$

$$-\left\langle \frac{\partial T}{\partial x}, \frac{\partial T}{\partial y}, \frac{\partial T}{\partial z} \right\rangle \cdot \vec{n} = \varepsilon \sigma (T^4 - T_0^4) + h(T - T_0) \quad (2.3)$$

It is worth noting that multi-layered single-bead DED models are symmetric in relation to the plane formed by the deposition and built directions. Thus, a Neuman boundary condition along the heat source path was adopted to reduce the computational time. Also, although the GMA-DED layers are arc-shaped, rectangular-shaped beads reduce the simulation error and computational time and simplify the meshing process [365], [366]. The

model mesh had 12,800 3D hexahedral elements and 16,609 nodes. The layers were finely meshed ($2 \times 1.5 \times 0.46 \text{ mm}^3$, as shown in Figure 3.4) to precisely predict the temperature during the deposition, even considering the intense thermal gradient associated with the GMA-DED. The layers elements were simulated using the birth-and-death technique [367], and the substrate elements were progressively coarsened from the bead edge. Furthermore, the GMA-DED model simulation performance, documentation, and validation followed the ISO 18166 standard recommendations.

3.1.3 Results and discussion

3.1.3.1 Crown hammer design

3.1.3.1.1 GMA-DED modeling validation

Figure 3.5 compares the simulated thermal cycles with the temperature evolution measured by thermocouples. Proper matching in both heating and cooling thermal cycles was observed. Measured and simulated peak temperatures in all layers are similar, with maximum relative errors of 11.6 and 8.0 % (thermocouple at 14 mm, 1st and 2nd layers, respectively). For other layers, the maximum relative error is around 5 %. The small difference between the simulated and measured results can be attributed to three main sources: i) the not exact idle time (90 s) between layers, which can induce some difference in the observed peak temperature and a non-temporal matching; ii) intrinsic variations ($\sim 1 - 2 \text{ mm}$) of the contact to work distance during deposition, which can increase the arc power (maintained constant in the FEM model) [368]; iii) slight difference in physical properties (k , ρ , and c_p) of the feedstock and substrate materials compared to those obtained from the ESI Sysweld® database [369]. Also, Farias et al. [67] highlighted that discrepancies between the mesh node and the exact thermocouple positions could also be associated with minor peak temperature differences. Despite the differences between measured and simulated thermal cycles, the present model proved to be suitable for accurately estimating the thermal profiles during GMA-DED deposition.

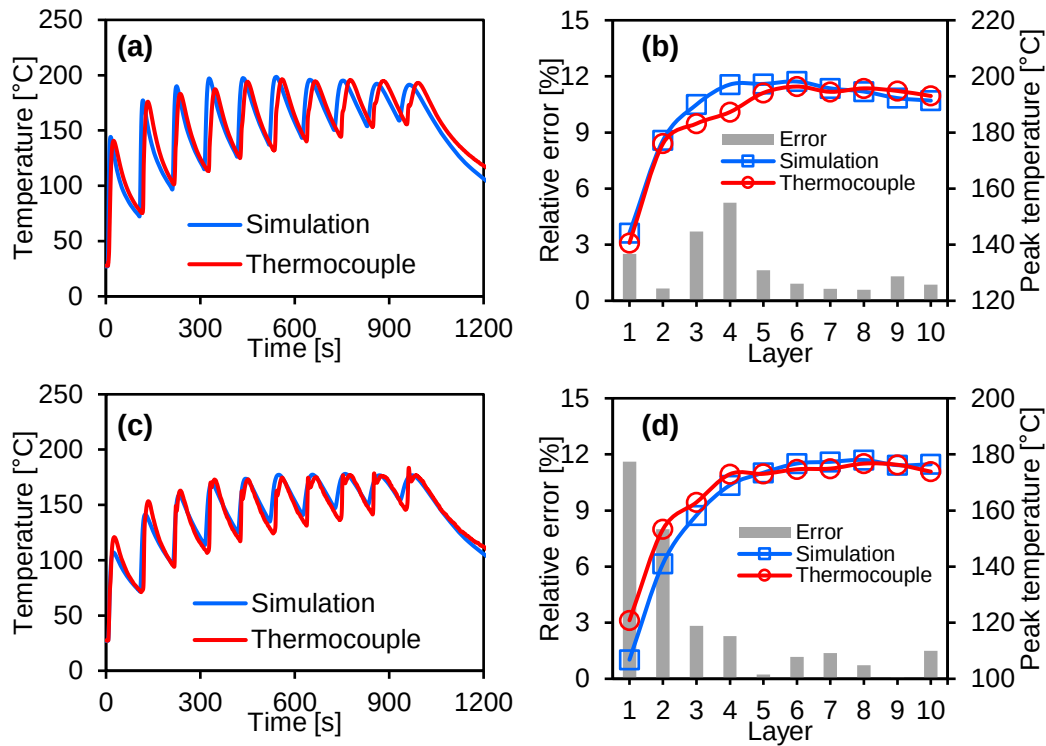


Figure 3.5 – Thermocouple and FEM model thermal cycles obtained at a point located 9 (a-b) and 14 mm (c-d) from the bead edge.

3.1.3.1.2 Forging hammer design

The validated thermo-metallurgical FEM model (Figure 3.4 and Figure 3.5) was used to estimate the thermal profile during the deposition (Figure 3.6a). Also, according to the Scheil-Gulliver solidification model (Figure 3.6b; Thermo-Calc® software), the Inconel® 625 solidification range is from 1340 °C to 1135 °C, which was corroborated by previous experimental data [207], [370], [371]. The sum of the melt pool (> 1340 °C) and mushy zone (1135-1340 °C) lengths defines the minimum distance (hammer radius) between the heat source (arc plasma) and the deformation tool. For a smaller hammer radius, the deformation tool will strike over the melt pool, resulting in liquid metal spattering and a poor layer surface [124]. Also, in the mushy zone, the material possesses a liquid film along the grain boundaries (brittle temperature range) and can fracture during localized deformation [184]. Thus, by combining the thermo-metallurgical FEM model and the Scheil-Gulliver solidification data, the minimum hammer radius can be defined (19.3 mm). In addition, the forging zone (5.9 mm, as detailed in Figure 3.6) was defined considering the typical Inconel® 625 forging temperatures (900 – 1100 °C) [52], [372], [373]. Therefore, to ensure that the in situ interlayer hot forging

occurs within the usual Inconel[®] 625 forging temperature range, the inner diameter was set to 20 mm.

Another important aspect of the hammer design is the outer diameter (Figure 3.2). Considering the hammer oscillation frequency and the torch travel speed (detailed in Table 3.1), the hammer forges only 0.625 mm (forging step, F_s) in each stroke [36], which means that the remaining surface of the hammer (up to the outer diameter) will act over the already forged material. Thus, the remaining hammer's surface will serve as a stopper since the hammer will stroke on the previously deformed material. Therefore, the outer diameter of the hammer was set at 25 mm to ensure that the deformation is limited by the previously deformed material, forming an upper flat layer surface. Otherwise, if the hammer's outer radius were equal to the inner radius plus the forging step, slight variations in the deposition conditions would change the minimum forging temperature, resulting in an uneven bead surface. Therefore, from the simulation results, the inner (20 mm) and outer (25 mm) hammer radii were designed, as detailed in Figure 3.2. In addition, the thermography (Figure 3.6c) showed that the minimum forging temperature (905 °C) matched with the one estimated by the FEM model (900 °C), reinforcing its accuracy. It is worth mentioning that smaller (17.5 mm; - 2.5 mm) and higher (22.5 mm; + 2.5 mm) inner radius hammers were also tested, and the results obtained corroborated the simulation approach methodology for the hammer design, i.e., 17.5 mm stroked the liquid metal and 22.5 mm induces a low deformation at layer surface.

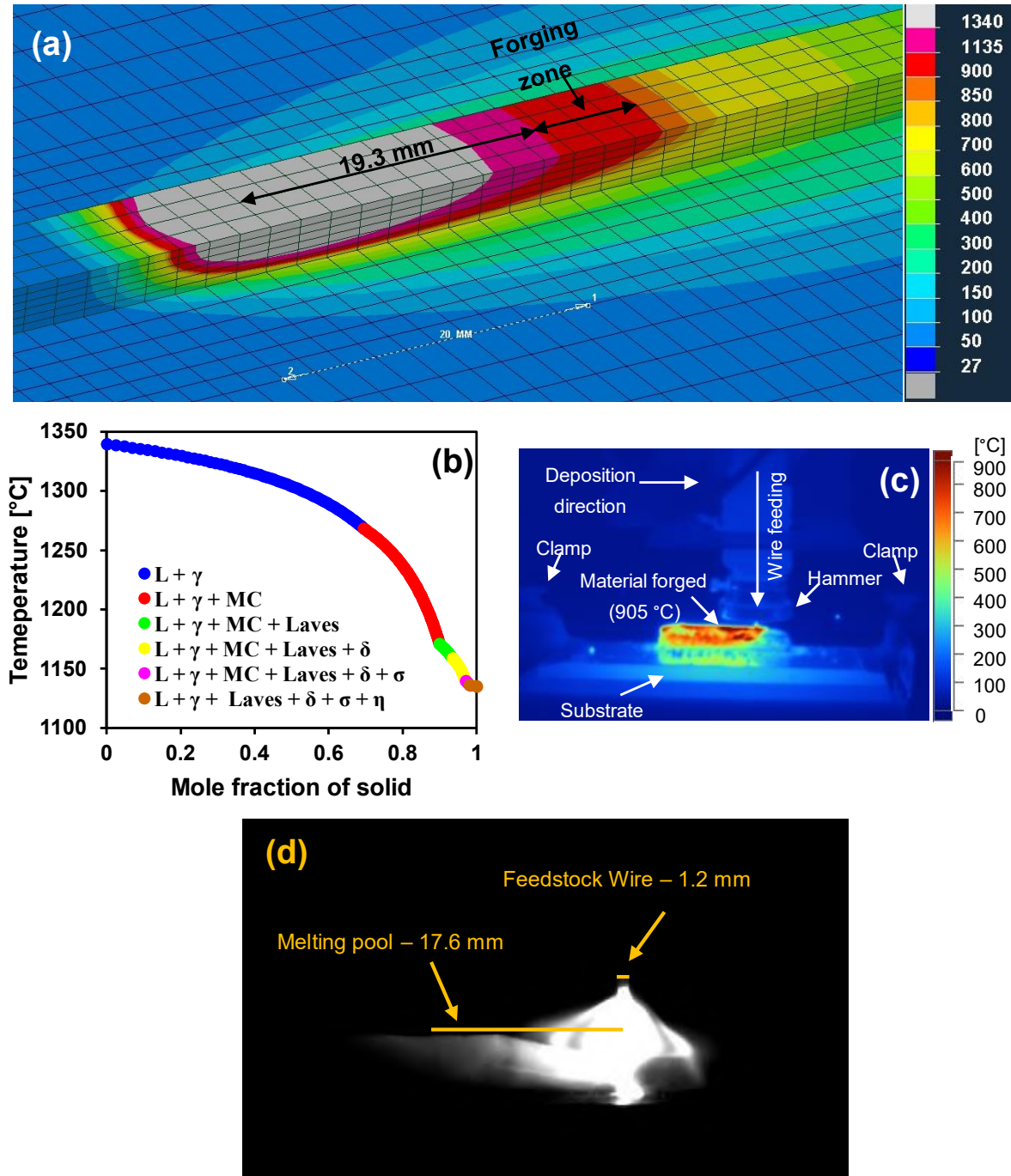


Figure 3.6 – (a) Simulated thermal profile showing the minimum hammer radius (19.3 mm) and forging zone, (b) Inconel® 625 solidification sequence obtained via Scheil-Gulliver model (Thermo-calc®), (c) thermal profile obtained via thermography during Inconel® 625 HF-DED, and (d) melting pool measurement (17.6 mm).

The forging force was calculated using the principle of impulse and momentum via high-speed camera filming. The forging force depends on the hammer speed before contact (1 m/s) and impact time (2.7 μ m) [36]. Therefore, the average measured impact force (1917 N) applied over the forging area (4.18 mm²) corresponds to an applied stress of 458.6 MPa.

Additionally, considering the impact time and true strain (0.11), the average strain rate was estimated (322.2 s^{-1}). Thus, combining the dynamic hammer characterization with the Inconel® 625 flow stress curves (Figure 3.7; JMatPro® software), it is possible to identify the forging conditions and the influence of the forging parameters on the amount of strain promoted.

In summary, it is possible to conclude that the HF-DED forging temperature and the deformation of the deposited layer can be adjusted by controlling the hammer design, keeping the deposition parameters constant. The inner radius of the hammer will determine the forging temperature, and the outer radius will ensure the deformation uniformity (stress and strain) and a flat final layer surface aspect.

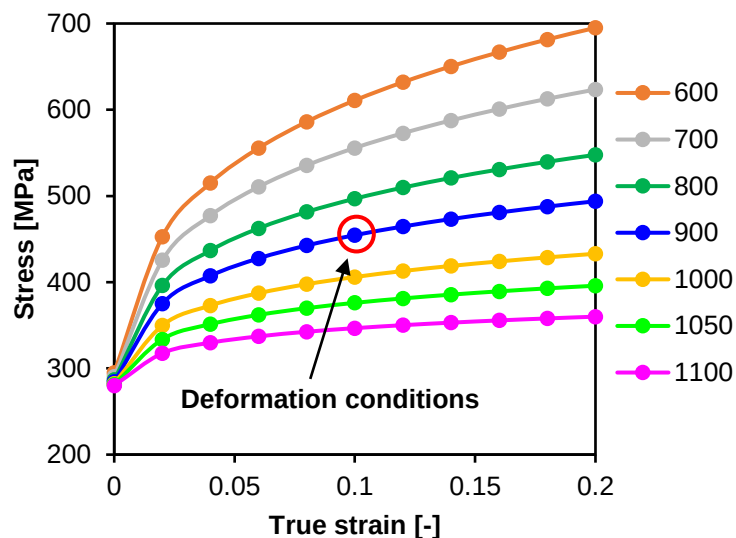


Figure 3.7 – Inconel® 625 flow stress curves (320 s^{-1}).

3.1.3.2 Effect of in situ interlayer hot forging on the microstructure

The last layer of the Inconel® 625 fabricated via arc-based DED had the typical Ni-based superalloy as-built microstructure (Figure 3.8a), which was in good agreement with the previously observed in the literature [37], [317], [340]–[344], [346]–[354], i.e., this layer was composed by coarse and aligned columnar grains almost parallel to build direction due to the nearly unidirectional heat flux. In addition, the upper region (close to the layer surface) possesses equiaxed dendrites due to the occurrence of the columnar to equiaxed transition (CET), which were induced by the reduction of thermal gradient in the melt pool [343], as described by the Kurz–Giovanola–Trivedi model [374]. However, the length of the CET region is smaller than the subsequent layer deposition penetration ($\sim 1.34\text{ mm}$), being observed only

in the last deposited layer. The HF-DED samples (Figure 3.8b) showed a larger equiaxed zone in relation to DED (1.2 vs. 0.2 mm), which derives from the dynamic recrystallization induced by the in situ interlayer hot forging, i.e., the equiaxed grain zone had different origins for the HF-DED (hot deformation) and DED (natural changes in thermal conditions). In addition, the interlayer hot deformation is not enough to completely change the layer microstructure (bead height, ~ 2.5 mm, higher than the dynamically recrystallized zone – 1.2 mm) so that the last deposited layer had two distinct zones: thermo-mechanical-unaaffected zone (primary microstructure without remain deformation, PM) and thermo-mechanical-affected zone (dynamic recrystallized grains and deformed primary microstructure).

Figure 3.9 compares the interlayer regions between DED and HF-DED conditions. Apart from the top of each sample (Figure 3.8), it was observed that the HF-DED promoted overall finer non-aligned dendrites close to the fusion line. However, due to the competitive grain growth in the melt pool, the microstructure turns to be oriented away from the fusion line, similar to that observed by Farias et al. [264] (Inconel® 625 claddings deposited by powder plasma transferred arc welding) and Li et al. [343] (Inconel® 625 parts fabricated by GMA-DED). These results suggest that the HF-DED improves the nucleation sites density at the fusion line, i.e., some remaining dynamic recrystallized grains (non-remelted) or those static recrystallized (reheating thermal cycle) in the thermo-mechanical-affected zone act as epitaxial growth sites, interrupting the coarse and highly oriented grains throughout several layers (as typically observed for the Ni-based superalloys fabricated by arc-based DED processes) and promoting finer and less aligned dendrites from the fusion line [152], [153].

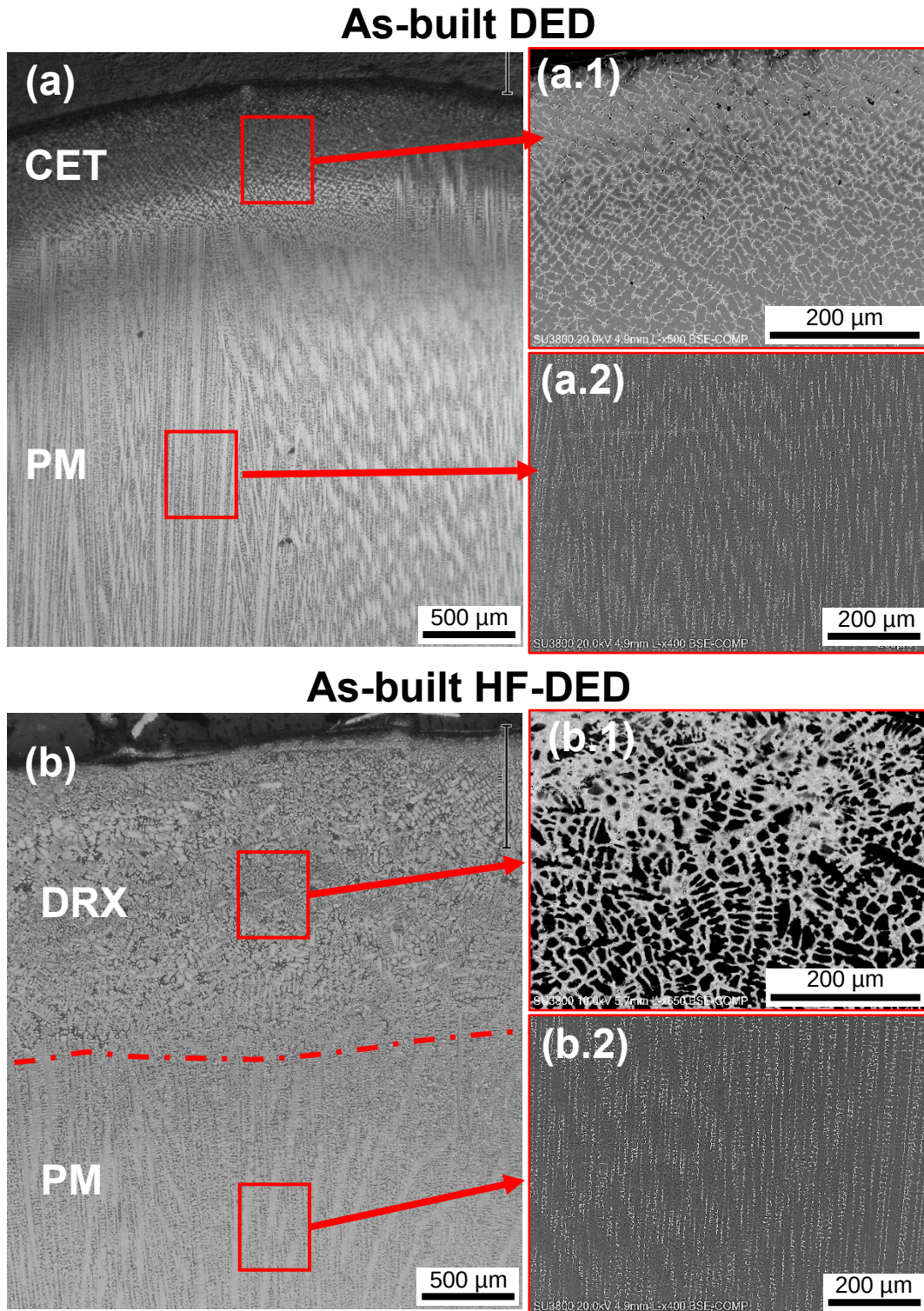
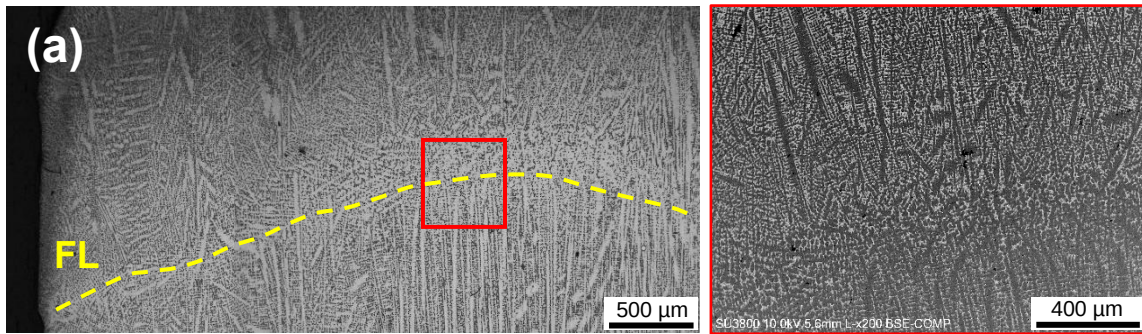


Figure 3.8 – Last layer (10th) upper surface microstructure: (a) DED and (b) HF-DED. The sub-figures (a.1, a.2, b.1, and b.2) correspond to red rectangles indicated in (a) and (b). CET, PM and DRX mean to columnar to equiaxed transition, primary microstructure, and dynamic recrystallized, respectively.

As-built HF-DED



As-built DED

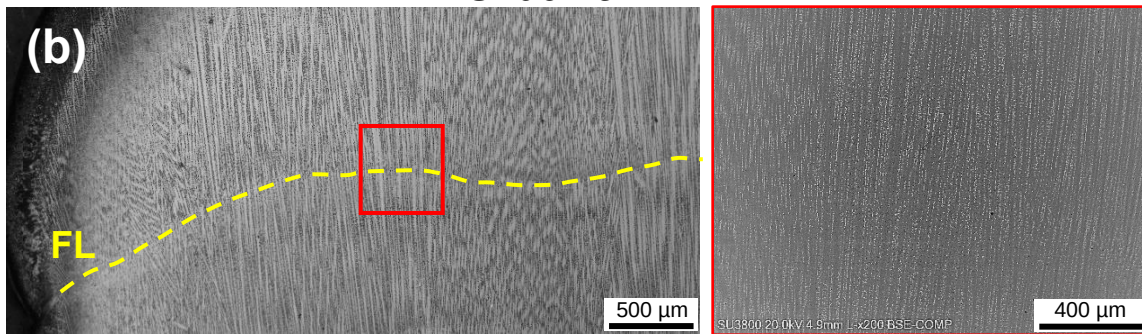


Figure 3.9 – Comparison of interlayer microstructure on Inconel® 625: (a) HF-DED and (b) DED. FL means fusion line.

The image orientation map (EBSD data detailed in Figure 3.10) corroborated the OM and SEM results (Figure 3.8 and Figure 3.9), showing that the DED fusion line had continuous columnar epitaxial grains. In contrast, the HF-DED material showed small equiaxed grains and a thinner and less oriented microstructure compared to its DED counterpart. This latter feature was attributed to the higher number of nucleation sites observed at the fusion line (Figure 3.9a and Figure 3.10b). In addition, the static recrystallization driving force and kinetics depend on the deformation level, peak temperature, and time [375]. Also, considering the high penetration of arc plasma-DED processes and fast cooling rate, the remaining deformation zone had low static recrystallization driving force and kinetics despite undergoing a high peak temperature (just below the melting temperature), which did not induce an intensity static recrystallization close to fusion line for HF-DED during the reheating thermal cycles (Figure 3.10d and Figure 3.10e), as typically visualized in the heat-affected zone of cold rolled plates welded [376] or interlayer cold deformed DEDed parts [37].

The meso-texture was evaluated using the EBSD data and expressed by the orientation distribution function (ODF; Figure 3.10c and Figure 3.10f) and pole figures (PF; Figure 3.11), which showed that the HF-DED had a less oriented microstructure (5.4 vs. 7.6 index). However,

both conditions had the typical fusion-based processes crystallographic texture (cube component, $\langle 100 \rangle \{100\}$) [138]. In addition, the HF-DED material also showed some rotation concerning the ideal cube component ($\phi_1 = 45^\circ$; HF-DED, $\phi_1 = 45^\circ \pm \sim 15^\circ$), which can be related to the change of top layer surface morphology (from the arc-shape – DED – to flat surface – HF-DED), inducing a deeper penetration at the melt pool center (similar to the first layer deposited at the substrate, i.e., flat surface), which tilted the grains and promoted some rotated cube aspects, as also previously reported by Gustafsson et al. [377] (Inconel® 625 GTA welded) and Eghlimi et al. [378] (duplex stainless steel GTA welded).

The HF-DED lower texture index was related to the recrystallized and equiaxed grains formed in the fusion line and the thinner aligned grains. Therefore, considering the overall maintenance of columnar grains and cube texture aspect, it is possible to indicate that the HF-DED slightly influences the solidification conditions. In addition, the kernel average misorientation (KAM; Figure 3.10b, Figure 3.10e, and Figure 3.10g), which is directly correlated with the geometrically necessary dislocations density [379], depicts that the next layer deposition did not entirely annihilate the HF-DED effects, showing a remaining thermo-mechanical-affected zone that can induce static recrystallization during the subsequent thermal cycles and/or post-deposition heat treatment. From KAM results, it is possible to confirm that the equiaxed grains close to the fusion line were recrystallized (KAM values below 1.5) [380]. Thus, the HF-DED induced thinner columnar grains, reduced the texture index, and promoted a remaining thermo-mechanical-affected zone.

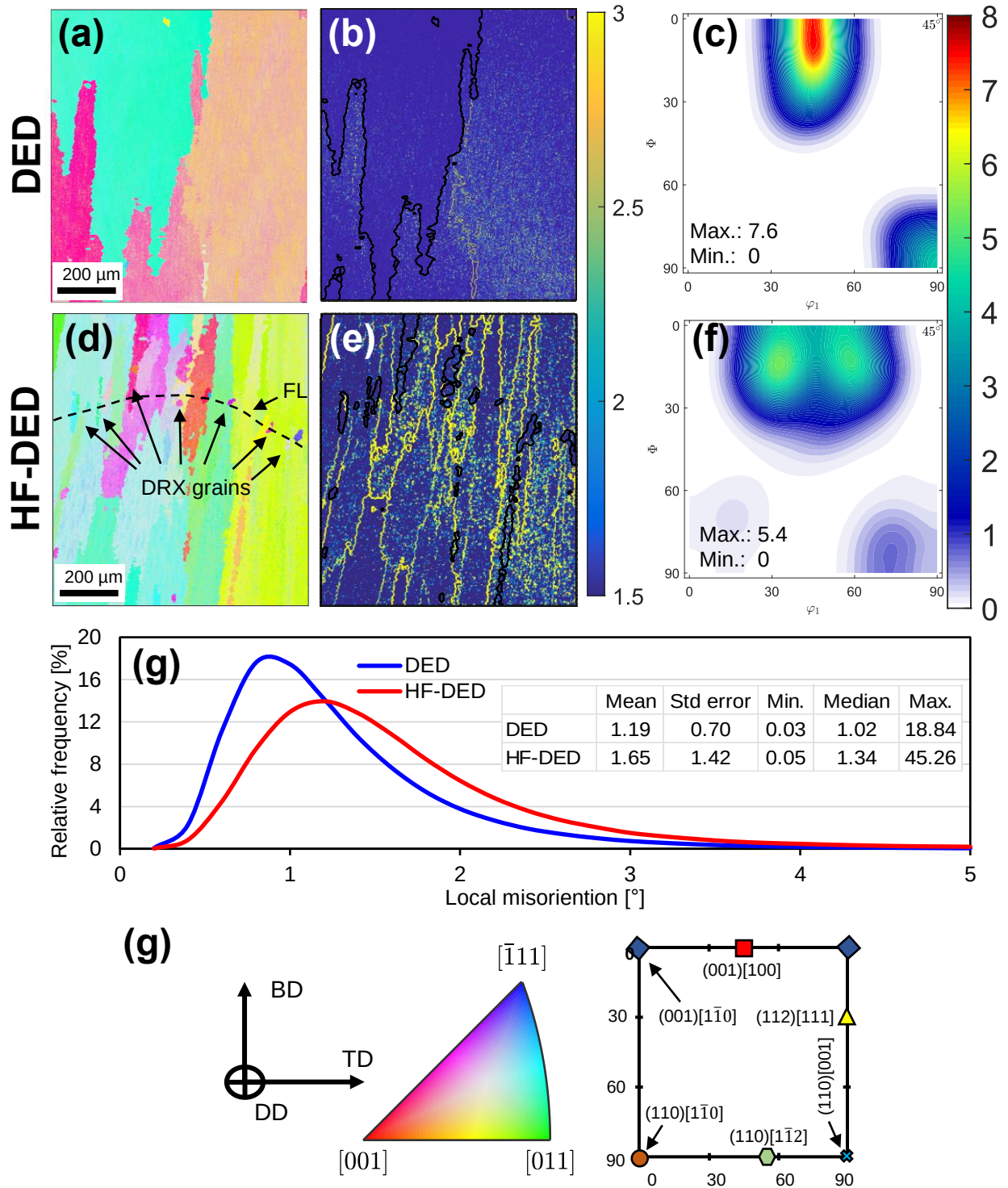


Figure 3.10 – EBSD analysis of Inconel® 625 DEDed (a-c) and HF-DEDed (d-f). (a) DEDed orientation image map (OIM), (b) kernel average misorientation (KAM) map, and (c) orientation distribution function (ODF; $\phi_2 = 45^\circ$); (d) HF-DEDed OIM, (e) KAM map, and (f) ODF; (g) KAM distribution; and (h) sample orientation, inverse pole figure color key, and ideal ODF texture components ($\phi_2 = 45^\circ$). In (g), BD, TD, and DD are the built, transverse, and deposition directions, respectively.

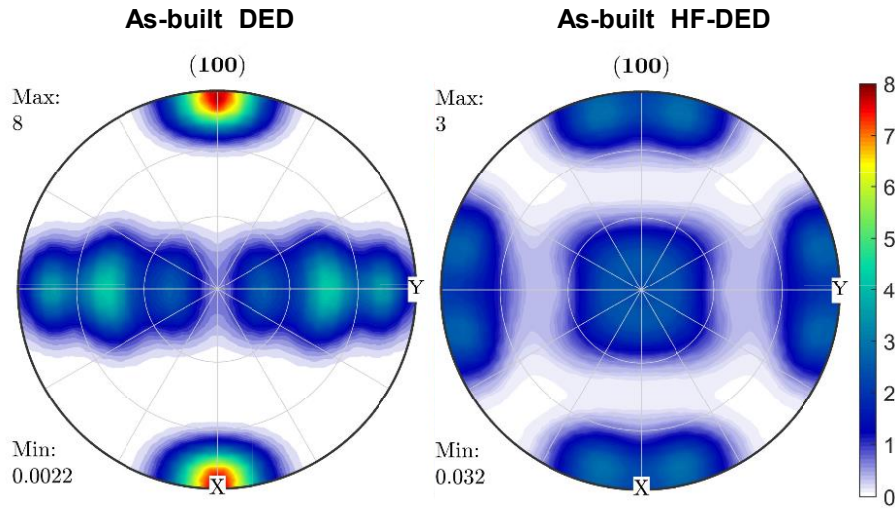


Figure 3.11 – Pole figure of the Inconel® 625 arc plasma DEDed and in situ interlayer hot forging DEDed, both in as-built condition.

The synchrotron X-ray diffraction, 2D Debye-Scherrer rings (Figure 3.12), were used to evaluate the meso/macro texture. It can be observed that the DED material did not possess any continuous rings but rather had spotty features, which is a clear indication of coarse grains and highly oriented microstructure. *In situ* interlayer HF induced the formation of almost continuous diffraction rings and reduced the “spotty feature”, indicating a finer and less oriented microstructure. The 2D Debye-Scherrer rings analysis supports the EBSD data (ODFs and PFs), i.e., the HF-DED has a less oriented and more refined microstructure. In addition, the synchrotron X-ray diffraction 2D Debye-Scherrer rings were fully integrated along the azimuthal angle (-180° – 180°) to evaluate the diffraction pattern (2θ vs. intensity) as shown in Figure 3.13, which detailed similar diffraction patterns for both DED and HF-DED. The main observed differences were related to peak intensity, where the HF-DED material showed (111) and (011) γ diffraction peaks with higher intensity (i.e., less oriented) in relation to DED.

According to the experimental solidification model of DuPont et al. [260], [261] and Silva et al. [259], the Nb-bearing Ni-based superalloys (e.g., Inconel® 625) solidification initiates with the formation of γ and the elemental segregation, especially of Mo, Nb, and C, to the interdendritic liquid. This is followed by the first eutectic reaction ($L \rightarrow \gamma + \text{MC-type carbide}$), consuming C, and finishes with the second eutectic reaction ($L \rightarrow \gamma + \text{Laves}$). These results were in good agreement with the simulated solidification sequence (previously shown in Figure 3.6b), where both MC-type carbides and Laves phase was confirmed by the synchrotron X-ray diffraction (Figure 3.13). In addition, Inconel® 625 Scheil-Gulliver solidification simulation predicted the formation of δ . Also, as Van et al. [292] reported, the multiple thermal cycles can induce the solid-state precipitation of δ . As reported by Oh et al. [266] using high-speed *in situ*

synchrotron X-ray diffraction, σ and η phases have not been observed experimentally. This indicates that although these phases are thermodynamically predicted under a solid/liquid local equilibrium condition (Scheil-Gulliver model), the thermal conditions (fast cooling rate) during DED suppress their formation. Thus, given the non-significant effect of the *in situ* interlayer HF variant on solidification conditions concerning the conventional DED [35], [36], [355], similar constituents (as shown in Figure 3.13), and the maintenance of cube texture component, the main differences between the HF-DED and DED processed materials were related to the remaining thermomechanical-affected zone (Figure 3.10c) and the KAM distribution, with high dislocation density in the HF-DED part.

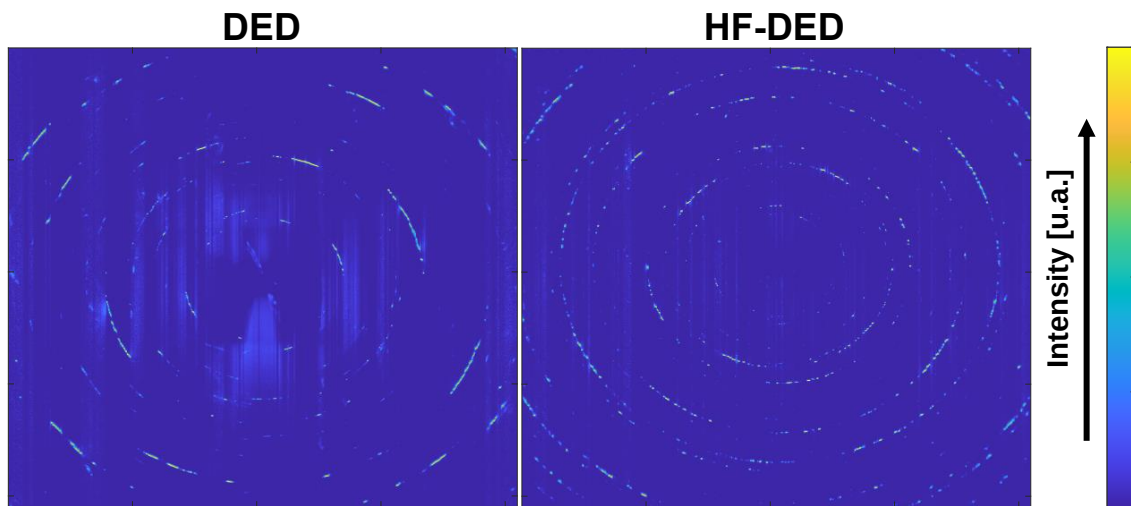


Figure 3.12 – 2D Debye-Scherrer diffractions patterns of DED and HF-DED materials.

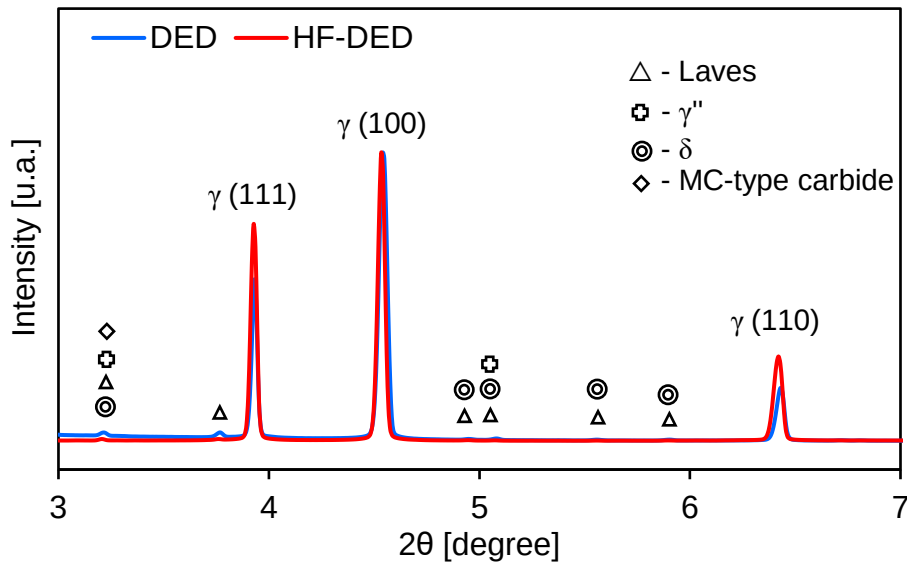


Figure 3.13 – Synchrotron X-ray diffractogram.

The Vickers microhardness profile (Figure 3.14) shows that the HF-DED material had a higher hardness than its DED counterpart. Close to the top bead surface (10th layer), the localized deformation and finer grain size due to dynamic recrystallization induce a significant hardness increase; however, due to the remelting and multiple thermal cycles, the hardness drops from the last layer surface to the substrate direction. The conventional DED material also has a higher hardness in the last layer surface, which was attributed to previously observed CET fine grains (Figure 3.8). Throughout the whole part, the HF-DED had a higher hardness than DED, which is related to the remaining deformation (as identified in the KAM map of Figure 3.10).

The Inconel[®] 625 is a solid-solution strengthened Ni-base alloy; thus, considering the non-significant effect of HF-DED on the solidification conditions and phase presents and the Zhang et al. [222] results (solid-solution strengthening mechanism did not change for different Inconel[®] 718 laser powder bed fused conditions), it can be stated that the solid-solution strengthening mechanism was similar for both DED and HF-DED conditions. Also, according to Gao et al. [381] and Li et al. [343], the Taylor strengthening (dislocation density) is preponderant in relation to grain boundary strengthening (Hall-Petch relationship) for Inconel[®] 625, which explains the hardness difference of ≈ 60 HV0.3 in the last layer. Given the higher KAM (Figure 3.10c; Taylor strengthening) and thin aligned and recrystallized interlayer

grains (i.e., finer grain size), a similar analysis can be extended to the remaining part, explaining the higher hardness of HF-DED samples compared to the DED material.

Correlating the hardness profile (Figure 3.14) with the HF-DED microstructure (Figure 3.8b), it is possible to estimate the total length (~ 2.1 mm) of the thermo-mechanical-affected zone (dynamically recrystallized grains – ~ 1.2 mm – and deformed primary microstructure – ~ 0.9 mm), which is higher than the subsequent layer penetration (~ 1.34 mm). Thus, the HF-DED microstructure showed, at the top layer (10th; Figure 3.8b), a dynamic recrystallization zone, which is remelted (Figure 3.9a), remaining the deformed primary microstructure between the layers (confirmed by the KAM map – Figure 3.10e) and some recrystallized grains that originated during the thermal cycles (Figure 3.6a). Therefore, the HF-DED produced a deformed microstructure, which can have a potential grain size refinement during annealing heat treatments, as observed at cold worked low/medium stacking-fault energy welded materials [382].

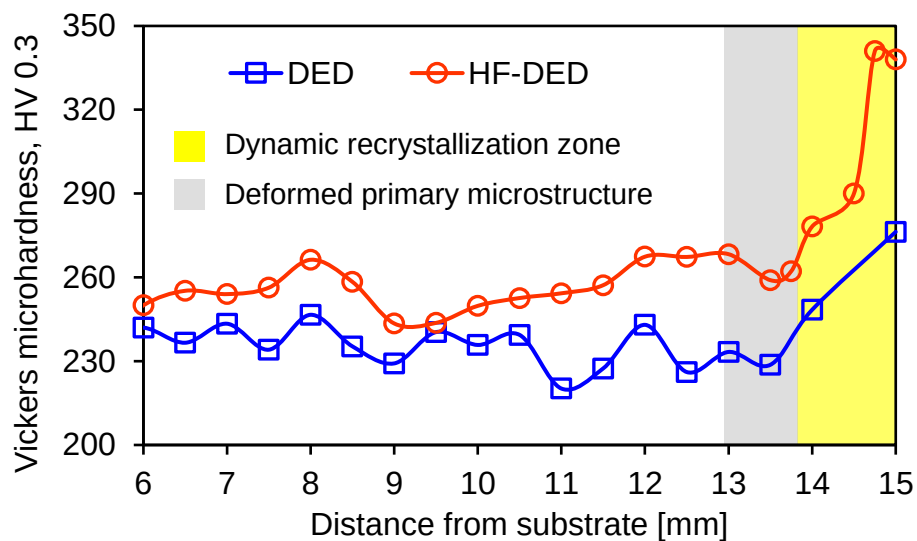


Figure 3.14 – Vickers microhardness (HV_{0.3}) profiles of the DED and HF-DED materials.

3.1.3.3 In situ interlayer hot forging effect on post-deposition heat treatment response

Given the remaining thermomechanical-affected zone expressed by the KAM distribution (Figure 3.10) and the higher hardness of HF-DED condition, the in situ interlayer hot forging can have two distinct grain size refinement mechanisms: in situ (via dynamic recrystallization) and post-process (static recrystallization – during reheating thermal cycles

and/or PDHT) [37], [110], [136], [138], [153], [200]. Thus, considering that the in situ grain size refinement was not efficient (completely remelted) to significantly alter the grain morphology and the main crystallographic cube texture (Figure 3.10), the present work also sought to improve the grain size refinement via static recrystallization of the remaining thermo-mechanical-affected zone using post-deposition heat treatments (solution and stabilization annealing). HF-DED (980 °C/1 h) and HF-DED (1100 °C/1 h) conditions are denominated as HF-DED-980 and HF-DED-1100, respectively.

Figure 3.15 details that the HF-DED-980 material had a significant grain size refinement in relation to as-built DED (61 vs. 375 μm ; Figure 3.16). However, some aligned non-recrystallized grains persisted. Moreover, the HF-DED-1100 showed a fully equiaxed (recrystallized grains) microstructure with a grain size comparable to wrought material and other arc plasma-DED + interlayer deformation processes (Figure 3.16). These results proved that the double grain size refinement mechanisms could occur in Inconel® 625 HF-DEDed, i.e., (i) thin columnar grains and some equiaxed grains can form at the fusion line due to the dynamic recrystallization and (ii) equiaxed grains due to the static recrystallization of the thermomechanical-affected zone during PDHT. Additionally, DED material (typical columnar microstructure; Figure 3.15a) had aligned grains (average aspect ratio > 3); PDHT reduced the aligned grains content and promoted equiaxed ones ($1 < \text{aspect ratio} < 2$). Besides, the HF-DED-1100 material had a better heat treatment response than the HF-DED-980 one, which can be related to its higher peak temperature that increased the static recrystallization kinetics and allowed less deformed zones (regions far away from the fusion line) also underwent static recrystallization [375]. In addition, despite the HF-DED-980 material possessing a low texture index (Figure 3.10), the solution annealing (HF-DED-1100) eliminated the casting/welding/additive manufacturing typical cube texture (Figure 3.15).

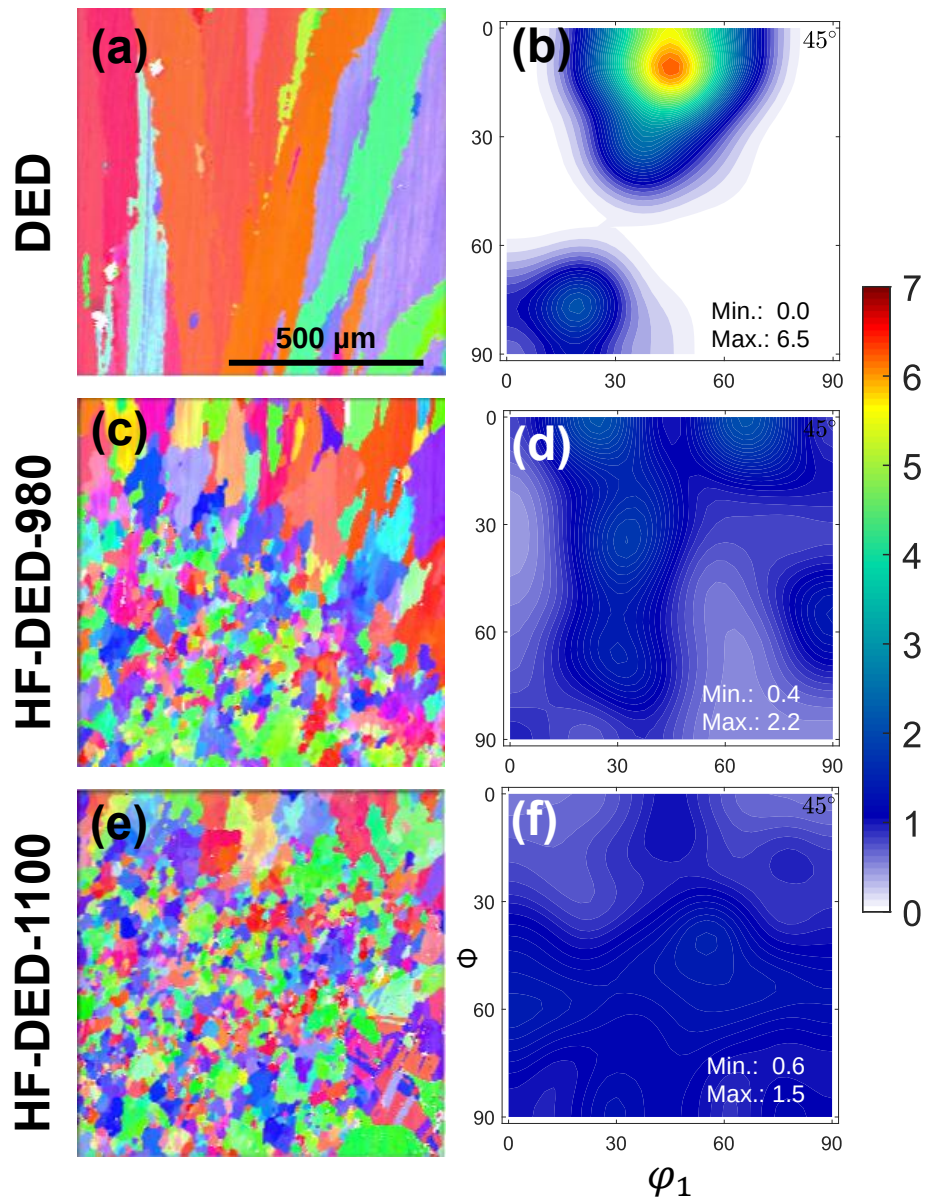


Figure 3.15 – Electron backscatter diffraction (EBSD) analysis of Inconel® 625 performed at interlayer region: (a) orientation image map (OIM) and (b) orientation distribution function (ODF; $\varphi_2 = 45^\circ$) in DEDed condition; (c) OIM and (d) ODF in HF-DED-980 condition; (e) OIM and (f) ODF in HF-DED-1100 condition. The EBSD data were acquired in the interlayer region, following the Figure 3.2c.

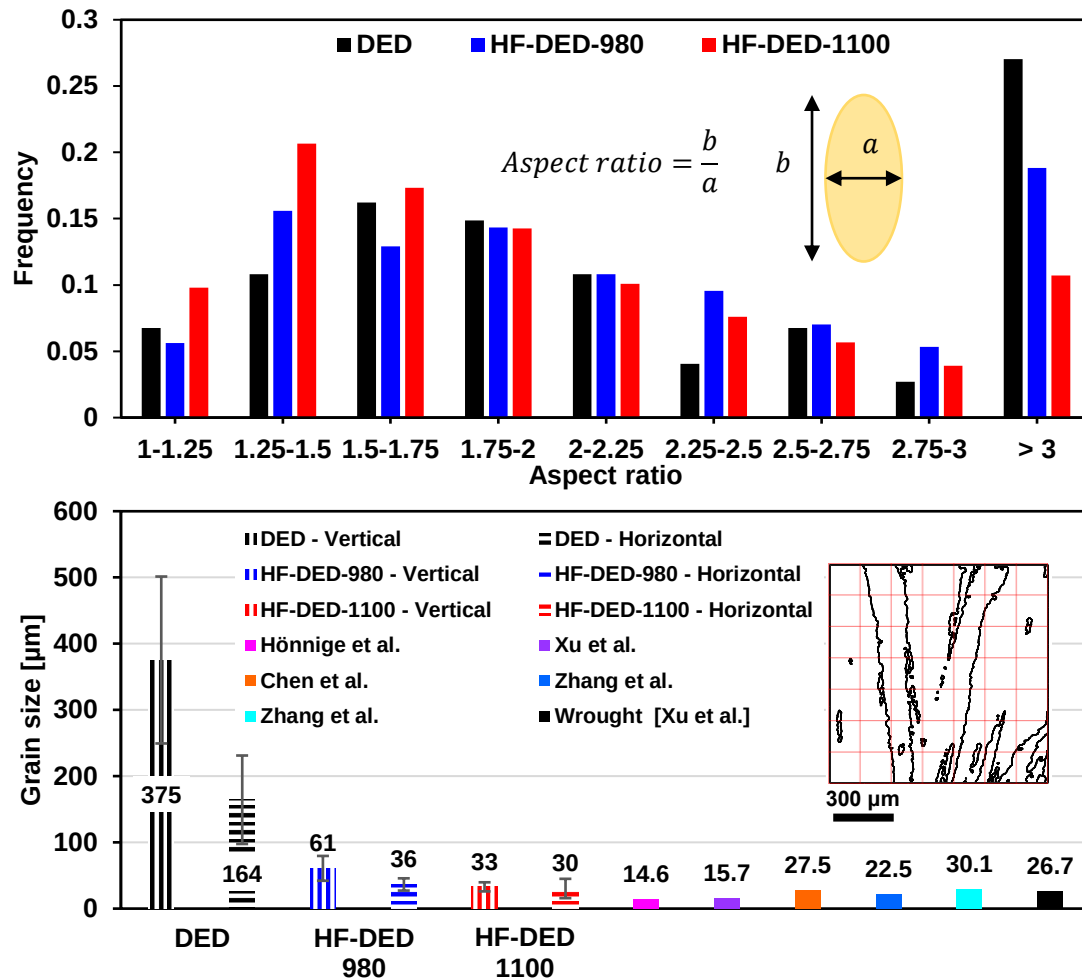


Figure 3.16 – Grain aspect ratio (upper) and size (bottom) for different Inconel® 625 DED-process conditions. The sub-figures exemplify the aspect ratio calculation and the procedure (intercept method) to measure the grain size, respectively.

Similar to the as-built conditions (DED and HF-DED), the synchrotron X-ray diffraction data (Figure 3.17a) did not significantly differ between the HF-DED-980 and HF-DED-1100. However, the intensity of the main γ peaks ($\{111\}$ and $\{100\}$) had a low difference, which is a clear indication of a low cube texture, reinforcing the ODF results (Figure 3.15). Also, the KAM distribution (Figure 3.17b) indicated that HF-DED-980 had a slightly higher remaining dislocation density (higher median local misorientation), which can be related to the low temperature and time (stabilization annealing – 980 °C/ 1 h) to complete the static recrystallization process. These results matched the hardness profile, which, even considering the HF-DED-1100 finer grain size, reveals that the Taylor strengthening mechanism was preponderant, resulting in a higher (~ 15 HV_{0.3}) hardness of the HF-DED-980 concerning the HF-DED-1100 material. Moreover, comparing the HF-DED-1100 and HF-DED-980 with their

DED counterpart (Figure 3.18 and Figure 3.14, respectively), it is possible to indicate that despite the significant grain size reduction (observed in Figure 3.16), the KAM median of HF-DED-1100 and HF-DED-980 is lower (0.82 and 0.89; Figure 3.10c) than that in the DED material (1.19; Figure 3.17b), which can explain the DED higher hardness compared to HF-DED-1100 and HF-DED-980 conditions. Therefore, Taylor strengthening mechanism (dislocation density) is superior to the grain boundary for Inconel® 625 fabricated via arc plasma DED. The heat treatment reduced the dislocation density and grain size (static recrystallization; Figure 3.15). Thus, a concurrent effect occurred: hardness decreases due to the lower dislocation density and increases owing to the smaller grain size. However, among these mechanisms, the dislocation density is preponderant; thus, despite the finer grain size after the heat treatment, the hardness of HF-DED-1100 and HF-DED-980 conditions is lower concerning the as-built DED and HF-DED conditions.

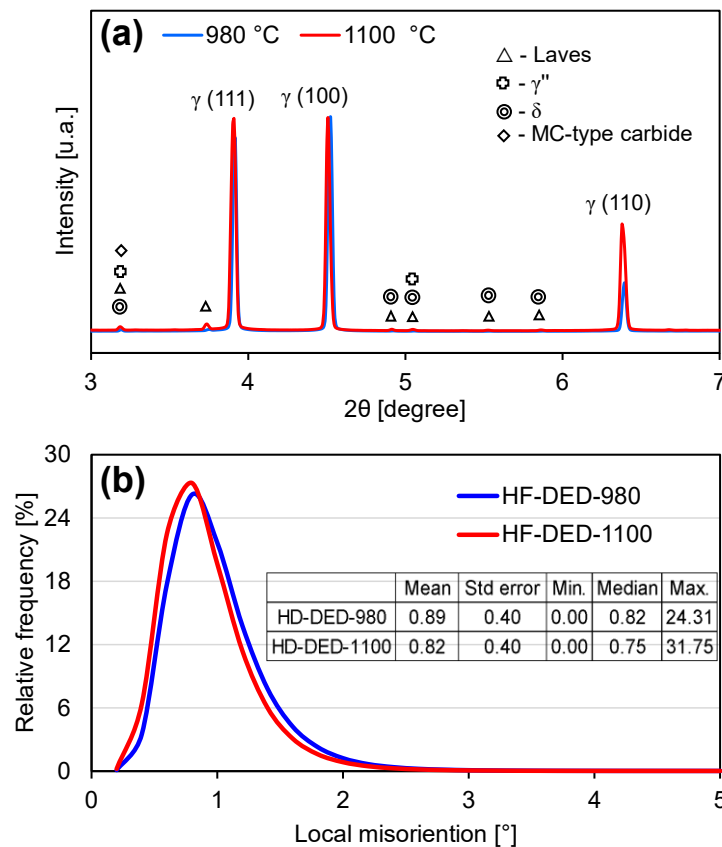


Figure 3.17 – (a) Synchrotron X-ray diffractogram and (b) Kernel average misorientation distribution calculated from Figure 3.15.

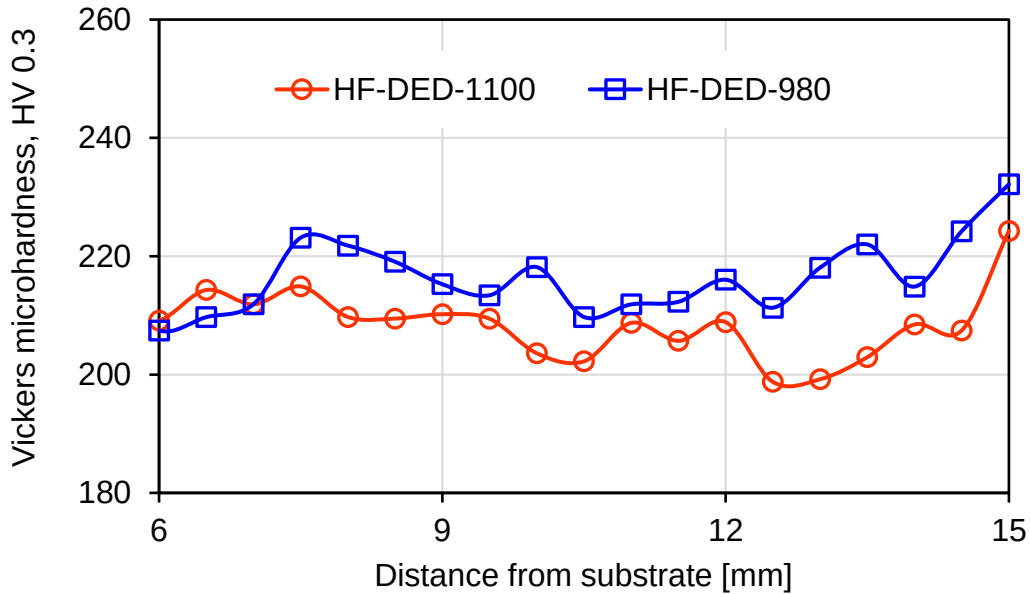


Figure 3.18 – Vickers microhardness (HV_{0.3}) profile for HF-DED-980 and HF-DED-1100 conditions.

3.2 Microstructure evolution during heat treatments

3.2.1 Post-deposition heat treatment (PDHT) – Ex situ EBSD

Figure 3.19 summarizes the effect of the PDHTs on Inconel[®] 625 fabricated via *in situ* interlayer hot forging arc plasma DED. Three distinct regions can be observed: (1) fine grain size region, which originated from the *in situ* dynamic recrystallization (hot forging effect); (2) static (*ex situ*) recrystallized region (PDHT effect); and (3) unchanged columnar grains, resulting from the primary solidification microstructure. As described by Duarte et al. [35], [36], the direct forging on the incandescent just deposited material (~ 900 – 1100 °C) induces dynamic recrystallization, leading to a fine and equiaxed grain structure at the layer top surface. The rest of the layer, where *in situ* dynamic recrystallization does not occur, still possesses remaining deformation due to the *in situ* interlayer hot forging. The remaining deformation can drive static recrystallization during PDHT (as observed in region (2)), characterizing a double grain size refinement mechanism (*in situ* and *ex situ*), as verified in our previous work [159] and also proposed by Wang and Shi [153] (Inconel[®] 718 fabricated via *in situ* interlayer ultrasonic impact peening laser-DED). However, due to the higher layer height (4 – 6 mm) of the arc plasma DED in relation to laser-DED [136], [153], the hot forging deformation does not affect the whole layer, i.e., a primary microstructure (region (3), refer to Figure 3.19) persists.

Region (1) had an almost similar extension and grain size in both PDHTs, 24.19 ± 6.1 and $39.11 \pm 11.4 \mu\text{m}$ for the solution and stabilization conditions, respectively. This was expected since region (1) rose from the hot forging process. In addition, static recrystallization (during PDHT) can also occur in the region (1), which explains the finer grain size of the solution PDHT condition. Region (2) diverged significantly between PDHTs, especially in extension (~ 950 vs. $440 \mu\text{m}$ for solution and stabilization, respectively). In addition, the average grain size of the solution PDHT was finer than the stabilization one (56.57 ± 32.1 vs. $74.03 \pm 25.9 \mu\text{m}$). Also, the solution PDHT (ASTM E112 grain size No. 5) accomplished the AMS 5662 and API 6A718 grain size requirements (ASTM E112 grain size No. ≥ 5 and ≥ 3 , respectively), showing that the *in situ* interlayer hot forging arc plasma DED industrial suitability. Due to the lower deformation induced during the deposition process in relation to laser-based additive manufacturing processes, Ni-based superalloys fabricated via arc plasma DED did not present evidence of static recrystallization during the typical PDHT [25], [107], [138], [346]–[348], [357]–[359], which prevents grain size refinement via static recrystallization (i.e., maintaining the coarse and oriented columnar grains) and, consequently, limits the use of Ni-based superalloys fabricated via arc plasma DED in mission-critical applications (not meeting the rigid materials requirements, e.g., oil & gas – API 6A718 – and aviation – AMS 5662 – industries). The present work shows that the coarse grain size challenge observed for Ni-based alloys fabricated via arc plasma DED can be overcome using the *in situ* interlayer hot deformation followed by an adequate PDHT (1100 °C/ 1 h).

The effect of PDHT peak temperature (980 and 1100 °C) can be related to the driving force for recrystallization since a higher temperature increases the transformation kinetics and induces deeper recrystallization through the layer height (the deformation induced by hot forging is higher closer to the layer top surface). This can be verified by the kernel average misorientation (KAM) maps, which demonstrate that the stabilization PDHT had higher KAM than the solution PDHT condition, i.e., the stabilization PDHT did not promote a complete recrystallization in all regions, failing to relieve the remaining deformation induced by the *in situ* interlayer hot forging. Despite the Inconel® 625 obtained by arc plasma DED does not have a mandatory PDHT, *in situ* grain size refinement (via dynamic recrystallization, i.e., interlayer hot forging effect) is insufficient to change the as-built microstructure noticeably, as previously reported by Farias et al. [159], Zhao et al. [128], and Li et al. [157] (solid-solution strengthened Ni-based superalloys fabricated via interlayer deformation DED), reinforcing the synergic effect of *in situ* interlayer hot forging (induce enough crystal defects) and selected PDHT (drive static recrystallizing) in promoting a significant grain size refinement.

As a consequence of the *ex situ* static recrystallization during the PDHT, the crystallographic texture (Figure 3.20, Figure 3.21, and Figure 3.22) also depends on the PDHT peak temperature. The pole figures (Figure 3.20) show that the stabilization PDHT did not alter the typical cube texture ($\{100\}\langle 100 \rangle$) aspect of the Ni-based superalloys obtained via arc plasma DED [340]–[343], [346], [348], [350], [352]. The finer grains (regions (1) and (2)) had an almost random-oriented microstructure (Figure 3.21 and Figure 3.22); thus, as the solution PDHT promoted a significant grain refinement, its general texture (regions (1), (2), and (3)) had a non-oriented aspect. Due to the minor extension of the region (2), the stabilization PDHT did not significantly alter the coarse and oriented microstructure, maintaining the cube texture. In addition, it is worth mentioning that the flat layer surface induced by the hot forging (Figure 3.1) can alter the melt pool morphology, i.e., the *in situ* interlayer hot forging can promote a melt pool with a flatter penetration aspect in relation to arc plasma DED (finger-like penetration with a concave aspect), tilting the grain growth direction in relation to melt pool center and promoting some rotated cube texture aspects (e.g., $\{100\}\langle 1\bar{1}0 \rangle$), as reported by Gustafsson et al. [377] (Inconel® 625 welded) and Moat et al. [383] (Waspaloy® fabricated via laser-DED).

Therefore, *in situ* interlayer hot forging was able to induce static recrystallization during the PDHT and alter the microstructure of the Inconel® 625 fabricated by arc plasma DED, changing the coarse and oriented columnar grains to fine equiaxed and non-oriented ones, eliminating the typical cube texture of fusion-based processed Ni-based superalloys (e.g., laser-DED, arc plasma-DED, PBF, welding, and casting).

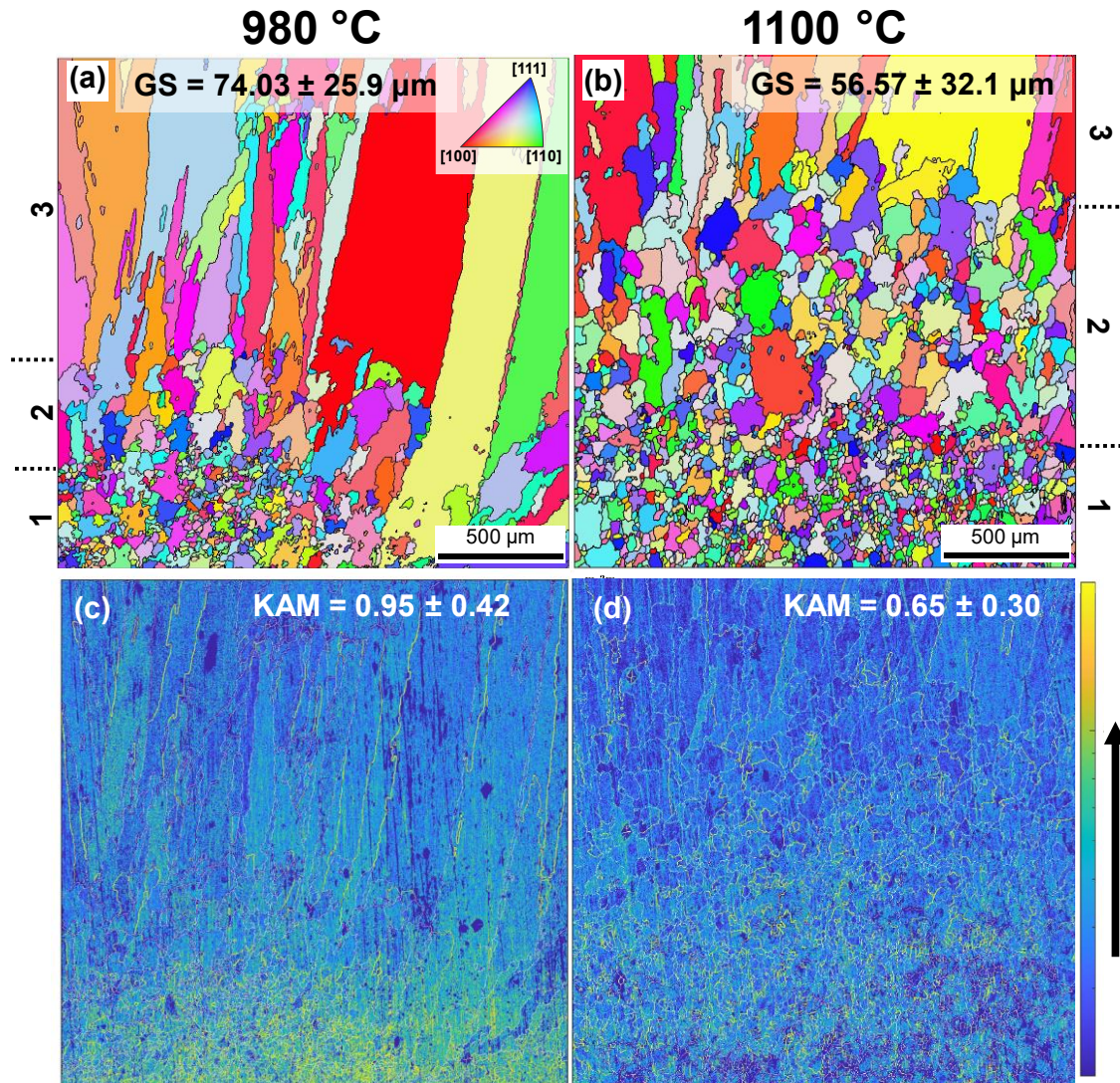
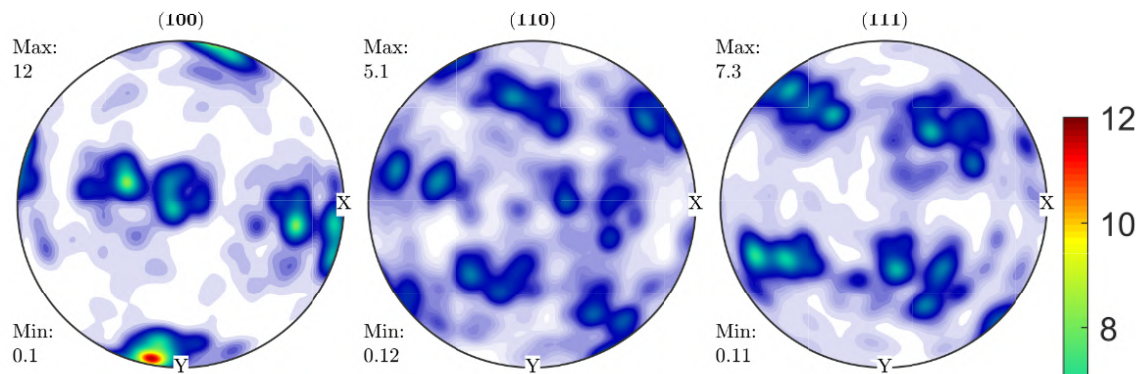


Figure 3.19 – *Ex situ* EBSD analysis of the Inconel 625 fabricated through in situ interlayer hot forging. Stabilization (980 °C/1 h) PDHT: (a) image orientation map (IOM) and (c) kernel average misorientation (KAM). Solution (1100 °C/1 h) PDHT: (b) IOM and (d) KAM. GS mean grain size.

Stabilization PDHT – 980 °C/ 1 h



Solution PDHT – 1100 °C/ 1 h

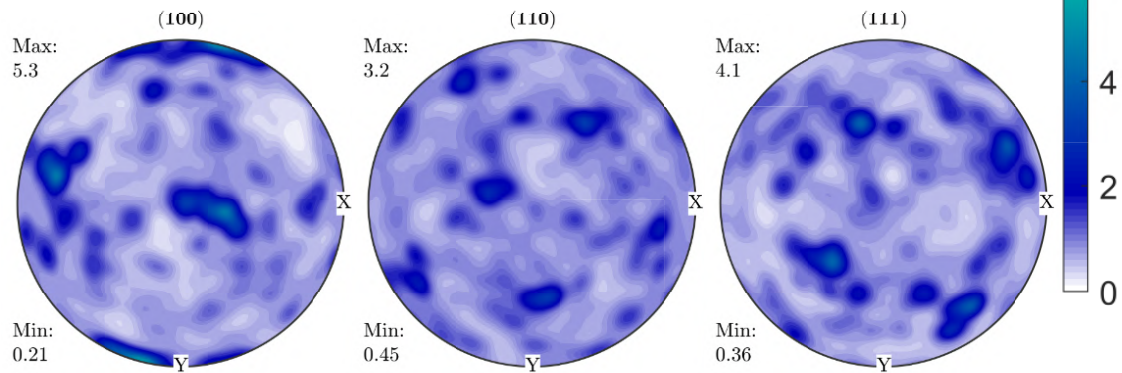


Figure 3.20 – Pole figure of the Inconel® 625 fabricated via *in situ* interlayer hot forging arc plasma directed energy deposition after stabilization (980 °C/ 1 h) and solution (1100 °C/ 1 h) PDHT.

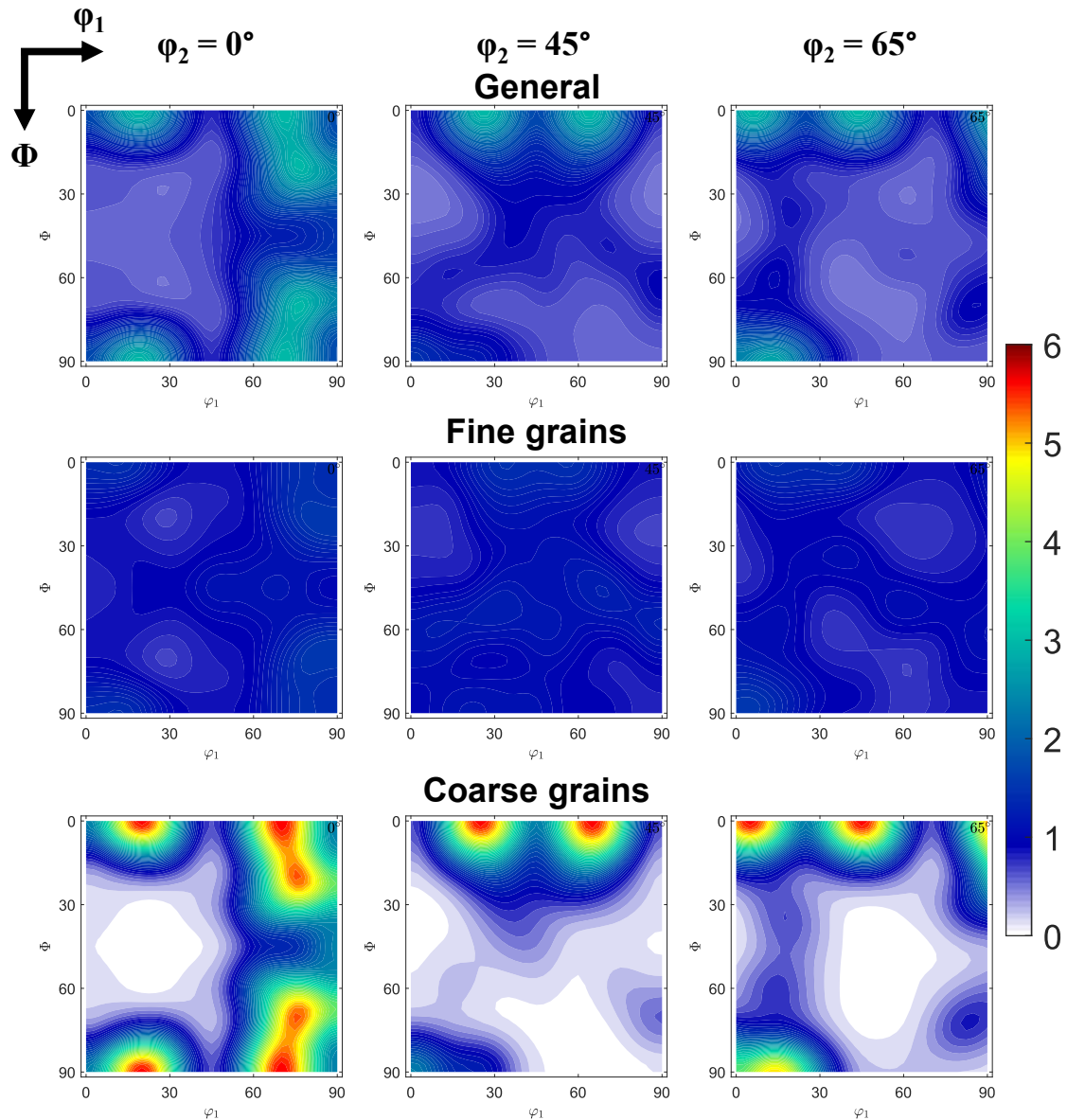


Figure 3.22 – Orientation distribution function (ODF) of the *in situ* interlayer hot forging arc plasma directed energy deposition of Inconel® 625 obtained by arc plasma DED after solution PDHT (1100 °C/ 1 h). Constant- ϕ_2 sections (0, 45, and 65°) through the Eulerian space (Bunge [384]). The general ODFs, refined grains ODFs, and coarse grains ODFs refer to regions (1, 2, and 3), regions (1 and 2), and region (3) in Figure 3.19b.

3.2.2 Time-solved SXR

3.2.2.1 In situ dislocation density estimation

The evolution of the full width at half maximum (FWHM) during the PDHT was tracked through the *in situ* SXR (Figure 3.23a). Peak broadening is affected by crystallite size (D), microstrain (ϵ , e.g., nonuniform strain, subgrains, and dislocations), and system configuration (e.g., instrument, material, and wavelength) [385], [386]. Given the similar deposition conditions

and SXRD configuration, the main difference between the solution and stabilization PDHTs can be related to microstrain evolution. In addition, subgrain formation and high-angle grain boundary migration decrease the crystal defects density [37], [387], [388], i.e., the diffraction peak broadening sources density should reduce along the PDHT's soaking time. Thus, the FWHM can qualitatively estimate the restoration phenomena during heat treatments [308]. In addition, the dislocation distribution and density of a deformed material are related to its SFE. A low/medium SFE material (e.g., Ni-based superalloys) possesses a low dislocation mobility due to the higher distance between partial dislocations, which hinder their constriction, i.e., the dislocation cross-slip and climb phenomena are harder to occur. Otherwise, high SFE materials have a low distance between partial dislocations, which promotes the recovery phenomenon (dislocation migration and annihilation) since the dislocation mobility is facilitated (dislocation cross-slip and climb) [243], [389]. Argon and Moffatt [390] proposed that the recovery rate is proportional to SFE^2 . Consequently, for low/medium SFE materials, slight dislocation recovery tends to occur before recrystallization. Thus, considering the low/medium SFE ($20 - 30 \text{ mJ}\cdot\text{m}^{-2}$) of Inconel® 625, recrystallization can be appointed as the dominant restoration phenomenon, i.e., the reduction of FWHM and dislocation density during PDHT can be associated with static recrystallization.

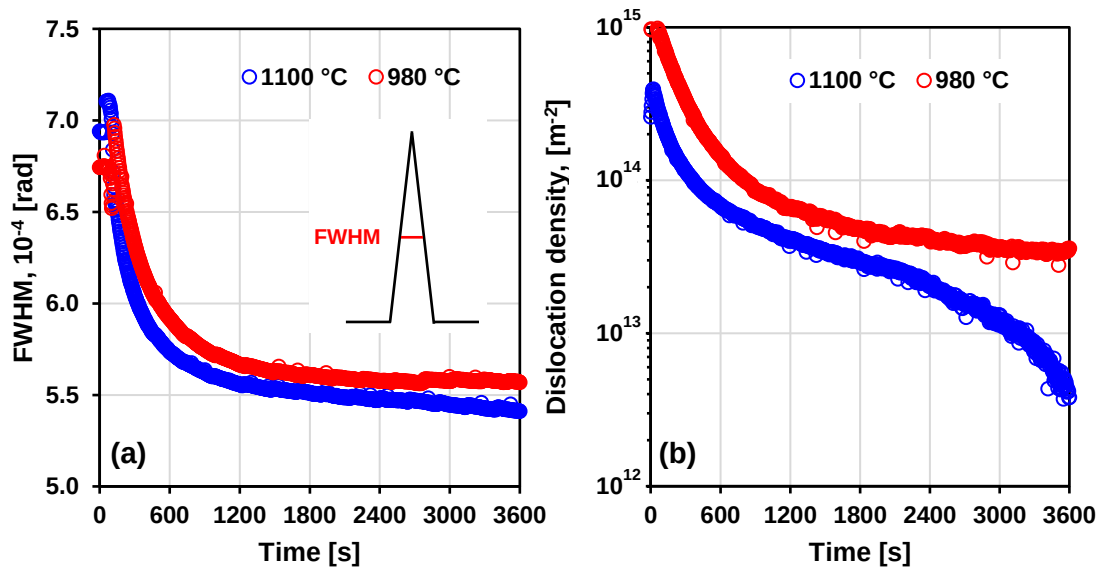


Figure 3.23 – Full width at half maximum (FWHM) and dislocation density evolution during solution (1100 °C) and stabilization (980 °C) PDHT.

From Williamson and Hall [391] (Equation 2.4) and Smallman and Westmacott [392] (Equation 2.5), the dislocation density (ρ_s , statistically stored dislocations – Figure 3.23b) can be related to the FWHM [393], [394]. In addition, given the lattice parameter (a) and diffraction

angle (θ ; Figure 3.24a) evolution during the PDHT (e.g., dissolution), the burgers vector (b ; Figure 3.24b) also changes over time. Thus, according to Bragg's law (Equation 2.6) and the correlation between planar distance (d_{hkl}) and lattice parameters for cubic materials (Equation 2.7), it is possible to correlate the diffraction data with the burgers vector (Equations 2.8 and 2.9). λ and α are the SXRD wavelength (0.14235 Å) and the Scherrer factor of a cube-shaped crystallite (0.9), respectively. Therefore, ρ_s evolution can be estimated (Figure 3.23b) from the Williamson-Hall plots (Figure 3.25a and Figure 3.25b), which were obtained in each 4 s during PDHTs.

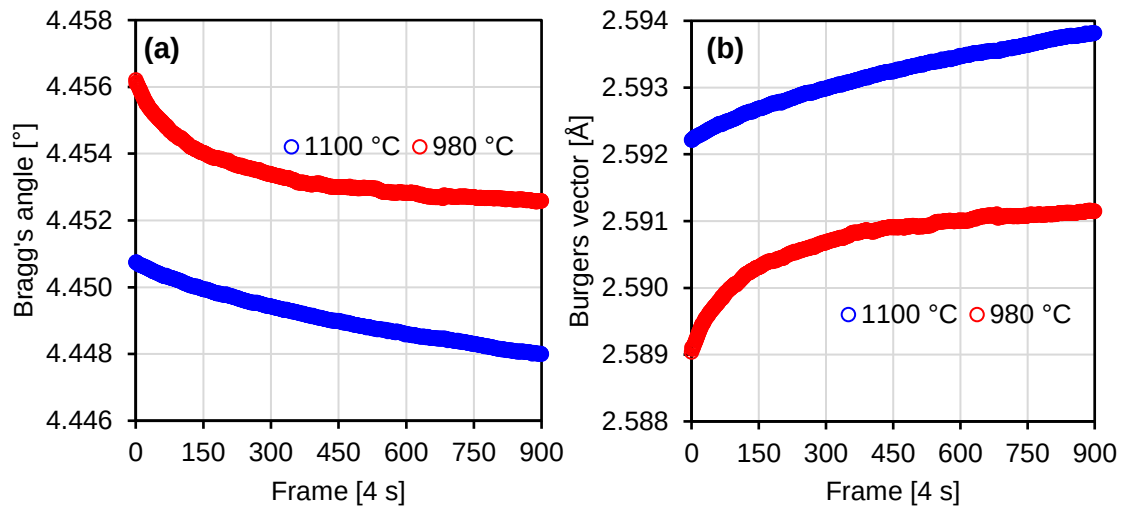


Figure 3.24 – Bragg's angle and Burgers' vector evolution during the stabilization (980 °C/1 h) and solution (1100 °C/1 h) post-deposition heat treatments.

$$FWHM \cdot \cos \theta = \frac{\alpha \cdot \lambda}{D} + 4 \cdot \sin \theta \cdot \varepsilon \quad (2.4)$$

$$\rho_s = \frac{16.1 \cdot \varepsilon^2}{b^2} \quad (2.5)$$

$$\lambda = 2d_{hkl} \sin \theta \quad (2.6)$$

$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \quad (2.7)$$

$$b = \frac{a}{\sqrt{2}}, FCC \text{ unit cell} \quad (2.8)$$

$$b = \frac{\lambda}{\sqrt{2} \cdot \sin \theta}, (200) \text{ plane} \quad (2.9)$$

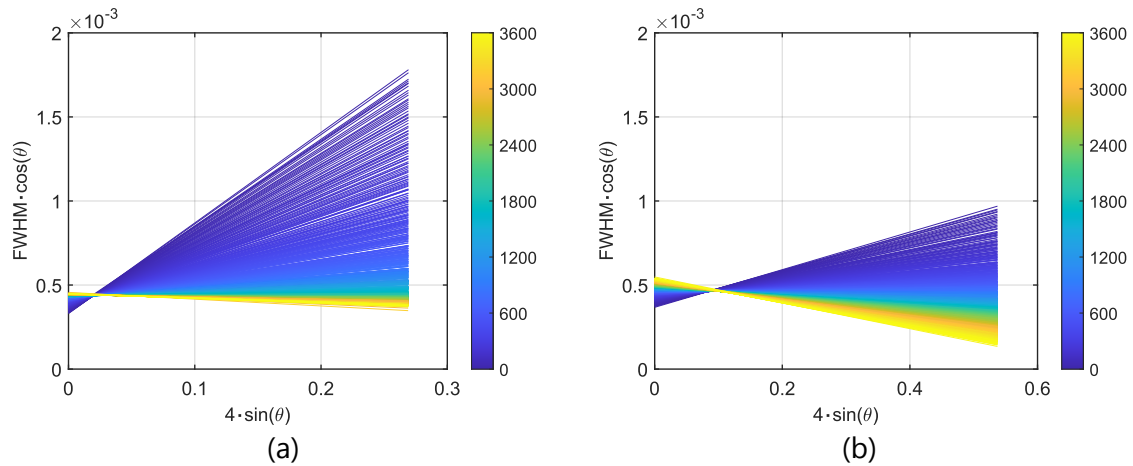


Figure 3.25 – Williamson-Hall plot for the (a) stabilization and (b) solution post-deposition heat treatments. The colorbar represents time evolution in seconds.

Both PDHTs had a fast FWHM and dislocation density reduction in the first ~ 1200 s (20 min), which indicates that static recrystallization quickly occurs in the *in situ* interlayer hot forged material, as reported by Li et al. [395] (Inconel[®] 718 fabricated by *in situ* hot rolling laser-DED). After 1200 s, the stabilization PDHT reached a plateau in the FWHM evolution, meaning that the diffraction peak broadening sources stopped reducing, which evidenced a static recrystallization interruption. The solution PDHT showed a continuous drop in the FWHM after 1200 s, which is related to some recrystallization in the lower deformed grains (region (2)) and grain growth [396] during the remaining soaking time (~ 2400 s). Besides, abnormal secondary grain growth [25] was not observed (Figure 3.19).

The recrystallization kinetics is associated with the deformation level (more deformed leading to faster kinetics) and temperature (higher temperature drives the recrystallization of less deformed regions) [165], [375]. Thus, the solution PDHT can induce recrystallization in a larger area than the stabilization PDHT [396], [397] since the localized and dynamic deformation aspects of hot forging cause a deformation gradient from the layer top surface [35], [36], [145]. In addition, the initial dislocation density ($\sim 4 \cdot 10^{14} \text{ m}^{-2}$ and $9 \cdot 10^{14} \text{ m}^{-2}$, refer to Figure 3.23b) was similar in magnitude to Ni-based superalloys fabricated via powder bed fusion (PBF) ($\sim 4.9 - 9 \cdot 10^{14} \text{ m}^{-2}$) [396], [398], which usually underwent static recrystallization during PDHT. Terris et al. [396] also verified a faster dislocation density reduction during the solution PDHT, which was also related to static recrystallization. Thus, the limited effect of PDHT on the grain size and crystallographic texture of the Inconel[®] 625 obtained by arc plasma DED [110], [136], [138], [200], [340]–[343], [346], [348], [350], [352] can be related to the low deformation level developed during the deposition (multiple thermal cycles), differing considerably from laser-based processes, which has higher deformation (driving force to

recrystallization) levels due to the focused heat source and steeper cooling rates [67], [397]. The *in situ* interlayer hot forging induced enough crystal defects and drove the static recrystallization of Inconel[®] 625 obtained by arc plasma DED during solution PDHT, which is typically not observed.

3.2.2.2 Secondary phase dissolution

Figure 3.26 depicts the SXR patterns and the evolution of the normalized peak intensity of the secondary phases (Laves and δ) identified in the present work. The diffraction patterns (Figure 3.26a) of the solution and stabilization PDHTs did not have a notorious difference between them, indicating that their final microstructure had similar secondary phases. It is possible to observe less difference in the matrix (γ) peak intensities for solution PDHT due to the lower texture index and finer grain size (Figure 3.19). In addition, the SXR results are per DuPont et al. [260] (experimentally-based solidification model for welding conditions) and Scheil–Gulliver model (Thermo-Calc[®], Figure 3.27a), which predicted the existence of Laves and MC-type carbides. σ and η phases (predicted by the Scheil–Gulliver model) are suppressed due to the higher ($\sim 10^2$ °C/s) [67], [399] cooling rate (arc plasma DED vs. local equilibrium). These phases (σ and η) were also not observed by Schmeiser et al. [397] and Oh et al. [266], which also used *in situ* SXR to characterize the Inconel[®] 625 fabricated via PBF. δ and γ'' phases originated from the multiple thermal cycles and PDHTs [292], [348], [397], [400]. They were not associated with the solidification sequence experienced by the material despite the Scheil–Gulliver model (Figure 3.27a) predicting δ phase formation during the solidification sequence, which was also not experimentally observed by Oh et al. [266] (melting pool solidification characterized by *in situ* SXR) and Cieslak [371], [401] (differential thermal analysis).

Stabilization PDHT increased the Laves and δ phases peak intensity (Figure 3.26b and Figure 3.26c). In opposition, the solution PDHT first causes a slight increase and then a drop in peak intensity. These results are corroborated by the time-transformation-temperature (TTT) diagram (obtained with JMatPro[®], Figure 3.27c), which was calculated from the Scheil–Gulliver model chemical composition distribution (Thermo-calc[®]; Figure 3.27b). During the stabilization PDHT, Laves and δ are stable in the interdendritic region, supporting the present *in situ* SXR and literature results [348], [399], [400], [402]–[407]. For the solution PDHT, Laves and δ are unstable, both in the interdendritic region and dendritic core, and should dissolve. However, their peak intensity increases initially, which can be related to local fast precipitation kinetics at

the interdendritic region due to the higher Nb segregation (up to 25% wt.), as experimentally verified by Antonsson and Fredriksson [268].

Given the theoretical peak intensity (Equation 2.10) and its correlation with phase volume in a multi-phase material diffractogram (Equation 2.11; phases observed in the manuscript – Figure 3.26a), the normalized data (Figure 3.26d and Figure 3.26e) of a single-phase diffraction peak (Equations 2.12, 2.13, and 2.14) can indicate the variation of phase content ($c(t)$) in relation the initial one (as-built condition, $c(0)$) [385]. I_{hkl} , I_0 , μ_0 , μ , M , e , A , and r are the integrated intensity, intensity of the incident beam, $4\pi \cdot 10^{-7} \text{ m} \cdot \text{kg} \cdot \text{C}^{-2}$, linear absorption coefficient, temperature factor (Debye-Waller), charge on electron, cross-sectional area of incident beam, and radius of diffractometer circle, respectively. Given that the sample-detector (2D Perkin Elmer) was kept constant (1561.85 mm), μ not affected the relative values of I_{hkl} (Equation 2.11) [408].

$$I_{hkl} = \overbrace{\left(\frac{I_0 A \lambda^3}{32 \pi r}\right) \left(\frac{\mu_0 e^2}{4 \pi m}\right)^2}^K \overbrace{\frac{1}{v^2} \left[F |hkl|^2 p \left(\frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta} \right) \right]}^{R(\theta)} \overbrace{\left(\frac{e^{-2M}}{2\mu} \right)}^{\text{Temperature}} \quad (2.10)$$

$$I_{hkl} = KR(\theta) \left(\frac{e^{-2M}}{2\mu} \right) (c_\gamma + c_{\gamma''} + c_\delta + c_{Laves} + c_{MC}) \quad (2.11)$$

$$I_{\text{single-phase}} = KR(\theta) \left(\frac{e^{-2M}}{2\mu} \right) c_{\text{single-phase}} \quad (2.12)$$

$$\frac{I_{\text{single-phase}}(t)}{I_{\text{single-phase}}(0)} = \frac{R(\theta(t)) e^{-2M(t)} c(t)}{R(\theta(0)) e^{-2M(0)} c(0)} \quad (2.13)$$

$$\frac{c(t)}{c(0)} = \frac{I_{\text{single-phase}}(t)}{I_{\text{single-phase}}(0)} \frac{R(\theta(0))}{R(\theta(t))} e^{-2(M(0)-M(t))} \quad (2.14)$$

At a specific temperature, all factors that affect the peak intensity (Lorentz–polarization, structure, multiplicity, absorption, and temperature factors) tend to be similar, independently of the exposure time at those temperatures. However, considering the phase transformation and precipitates dissolution during the PDHTs, slight changes in 2θ (affecting the Lorentz–polarization) can occur due to the phase evolution, change in composition, and morphology, as reported by Idell et al. [403] and Stoudt et al. [400] for Inconel® 625 fabricated via PBF. In addition, the multiplicity factor (p) is a function of the unit cell and does not change for a single-phase peak during the heat treatment. The structure factor ($F|hkl|^2$) is independent of the phase unit cell shape and size, implying that despite possible unit cell alteration (e.g., change in lattice parameters due to diffusion), atoms' positions (atomic scattering factor) will be similar, i.e., $F|hkl|^2$ can also be considered constant during the PDHT [385]. In relation to

v (unit cell volume), Lindwall et al. [399] and Zhang et al. [402] (both Inconel[®] 625 fabricated via PBF) considered this variable constant during the heat treatment. They [399], [402] obtained a good match between the secondary phase content estimated via image analysis and SXR, which indicated that v does not significantly change during the PDHT and can also be considered a constant parameter. The analysis described above indicated that $\frac{R(\theta_0)}{R(\theta)}$ (Equation 2.13) depends only on the Lorentz–polarization factor (i.e., 2θ). In addition, as exemplified for the δ phase (Figure 3.28a), the Lorentz–polarization factor was not significantly (almost constant, $\cong 1$) affected during the heat treatment. A similar analysis was also performed for the temperature factor (e^{-2M}), which also showed be independent of the time during the PDHT (Figure 3.28b). Thus, the relative phase content, $\frac{c(t)}{c(0)}$, can be expressed by Equation 2.15. For a better description of the SXR data analysis and temperature factor, see Supplementary material.

$$\frac{c(t)}{c(0)} = \frac{I_{single-phase}(t)}{I_{single-phase}(0)} \quad (2.15)$$

After about 1200 s of the solution PDHT soaking time, where chemical composition homogenization by diffusion is expected to be more notorious [400], secondary phases become unstable and start to dissolve, resulting in peak intensity reduction (Figure 3.26d and Figure 3.26e). However, the remaining time is insufficient to completely dissolve them, suggesting that longer soaking times or higher peak temperatures than that indicated in the material specification (ASTM B 446) are required for Inconel[®] 625 fabricated via arc plasma DED. Floreen et al. [407], wrought Inconel[®] 625, highlighted that the complete secondary phase dissolution could be achieved using long soaking times (24 h) at 1100 °C, reinforcing the present results. Idell et al. [403] and Stoudt et al. [400], Inconel[®] 718Plus and 625 fabricated via PBF, respectively, reported that δ phase could form during the entire isothermal annealing (1066 °C/1 h), attributing this to the high interdendritic microsegregation (especially Nb and Mo as also showed in Figure 3.27b), which increase both the stable temperature range and precipitation kinetics ($\sim 10^{1-2}$ times) in relation to the wrought material. Zhang et al. [404] and Shankar et al. [409], Inconel[®] 625 fabricated via PBF and hot rolling Inconel[®] 625, respectively, showed that a higher peak temperature (1150 °C/1 h) can homogenize the composition and dissolve the majority of secondary phases. In addition, it is worth mentioning the difference in dissolution/precipitation kinetics between the dendritic core and interdendritic regions expressed by the TTT diagram (Figure 3.27c). For interdendritic regions, the precipitation and partial dissolution of deleterious phases during the stabilization and

solution PDHTs, respectively, were correctly predicted and supported by the SXRD results and literature [400], [403], [404], [407], [409].

The δ phase is pointed out as a grain growth suppressor via the Zener pinning effect [247], [248], [405], which can be related to the FWHM plateau (stabilization PDHT – δ sub-solvus temperature; Figure 3.23), i.e., δ hidden the peak broadening source density reduction by pinning the high-angle mobile grain boundaries [410]. Additionally, despite the present work be focused on Inconel[®] 625 fabricated via *in situ* interlayer hot forging arc plasma DED, the presented Laves and δ phases evolution (Figure 3.26) can be extended to arc plasma DED, which is corroborated by previous literature results [25], [292], [346], [348], [399], [400], [402]–[405]. Thus, low peak temperature PDHT drives the secondary phase precipitation, while high peak temperature enables more homogenous chemical composition and lower content of secondary phases. The present SXRD results and those of Lass et al. [406] (Inconel[®] 625 fabricated via PBF) indicate that longer solution PDHT soaking times (> 1 h) could drive the complete dissolution of preexisting secondary phases. In addition, as highlighted by Xi et al. [107] and Luna et al. [310], the use of higher peak temperatures (1185 and 1177 °C/ 1 h, respectively) can almost completely dissolve Laves and δ phases. However, considering that the *in situ* interlayer hot forging grain size refinement primarily occurs by static recrystallization, longer soaking times or higher peak temperatures must be carefully analyzed to avoid abnormal grain growth, which can neutralize the *in situ* interlayer hot forging grain size refinement effect.

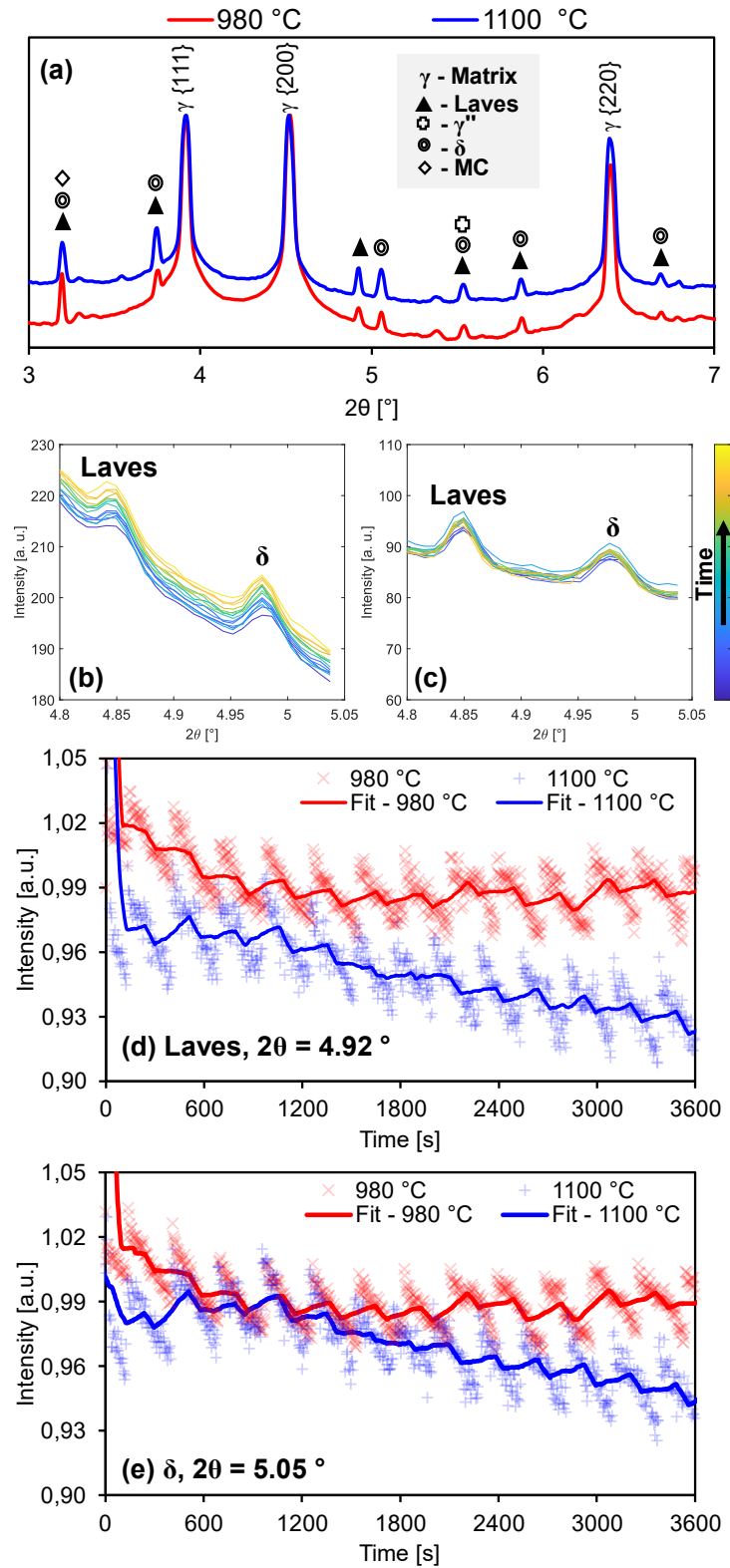


Figure 3.26 – SXR analysis. (a) SXR pattern, (b – c) Laves and δ phases isolated peaks evolution during the stabilization (980 °C) and solution (1100 °C) PDHTs, and (d – e) normalized peak intensity evolution. The fit function used in (d – e) is the moving mean.

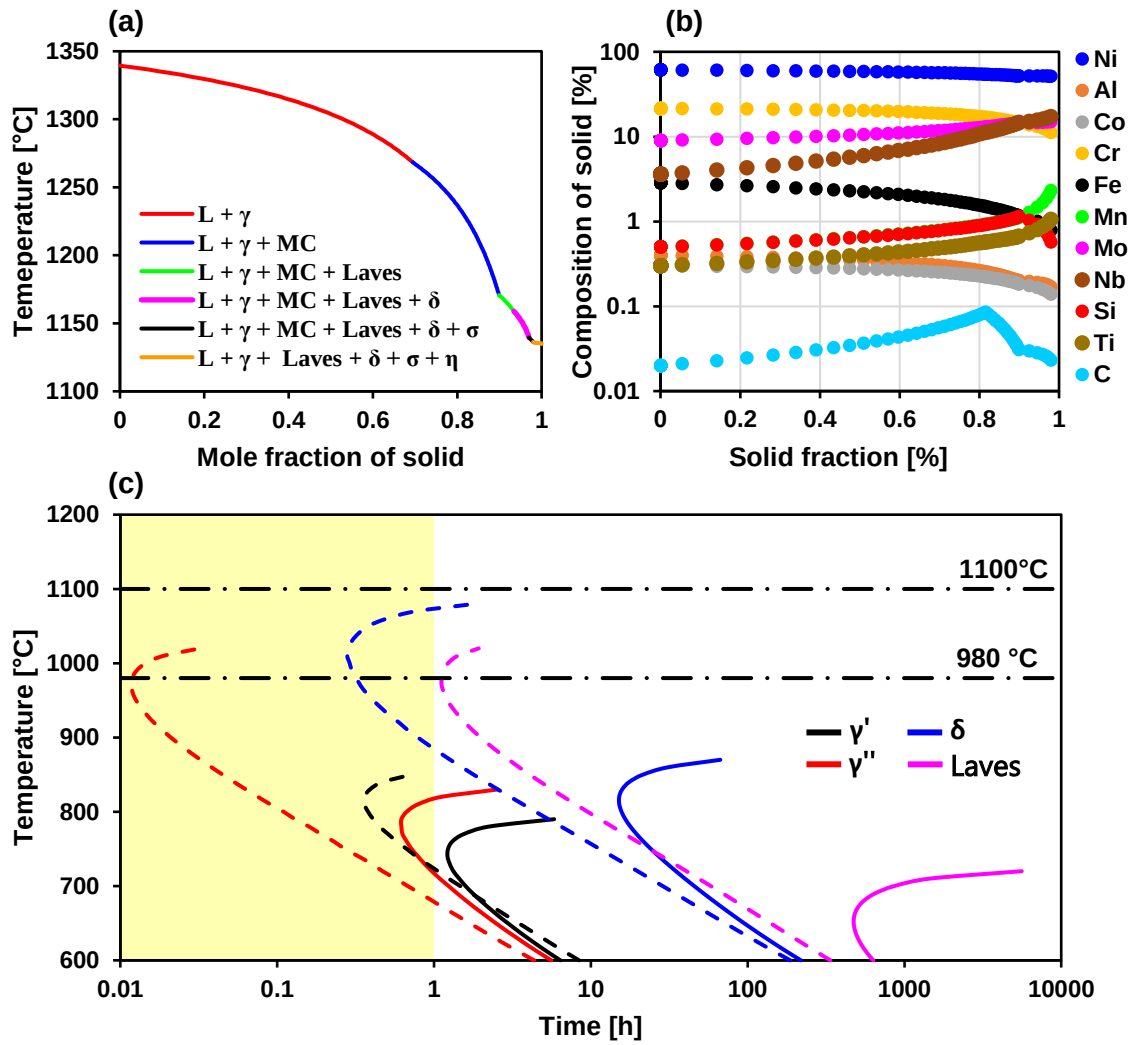


Figure 3.27 – (a) Inconel® 625 Scheil-Gulliver solidification model (Thermo-Calc®; TCNI11 database), (b) elemental distribution, and (c) transformation-time-temperature (TTT) diagram (JMatPro®) considering the dendritic core (solid line) and interdendritic region (dotted line) chemical compositions.

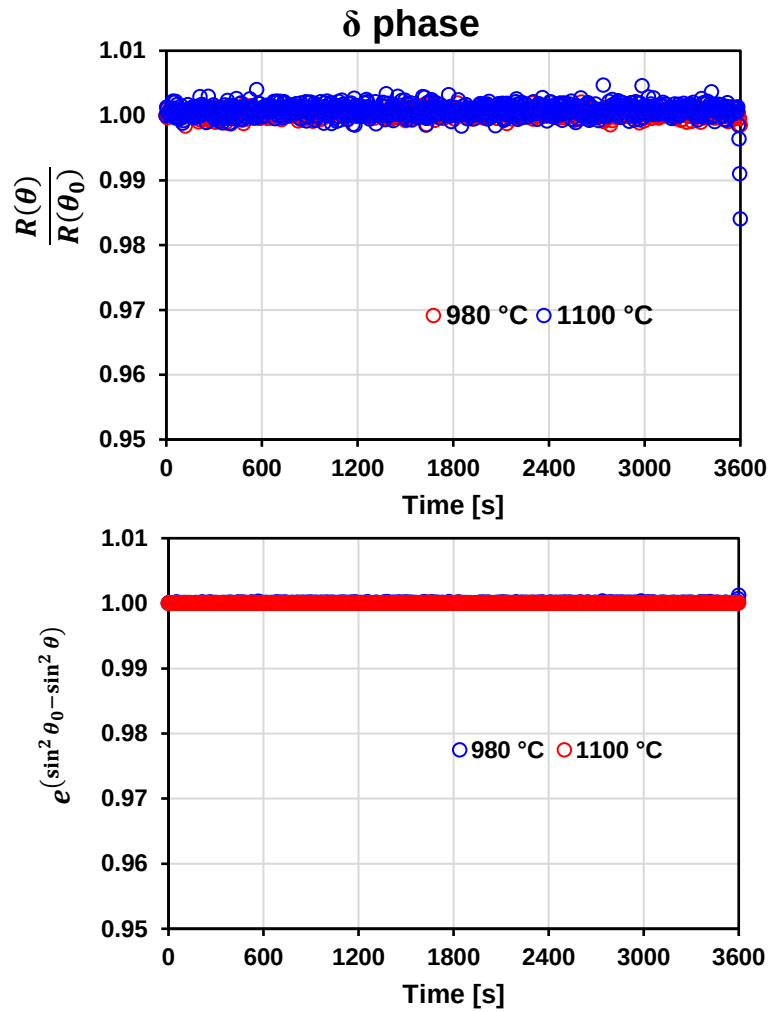


Figure 3.28 – Normalized Lorentz-polarization ($\frac{R(\theta)}{R(\theta_0)}$) and temperature ($e^{(\sin^2 \theta_0 - \sin^2 \theta)}$) factors variation during the post-deposition heat treatment.

3.3 Summary

The present work evaluated the effect of the in situ interlayer hot forging (HF) on the grain size, grain morphology, and crystallographic texture of the Inconel[®] 625 fabricated via gas metal arc direct energy deposition (DED). Initially, the deformation tool (a circular crown hammer) was designed based on a thermo-metallurgical experimentally validated FEM model. The as-built DED had a typical microstructure (coarse and cube-aligned grains); the HF-DED induced a thermo-mechanical-affected zone (due to dynamic recrystallization) and promoted finer and non-aligned dendrites and some equiaxed grains close to the interlayer fusion line. However, the as-built HF-DED had a low overall effect on the grain size and crystallographic

texture. Post-deposition heat treatments (980 °C / 1 h and 1100 °C/ 1 h, following the ASTM B 446 standard) drove static recrystallization on the thermo-mechanical-affected zone, which promoted a significant change in grain size (from 375 to 30 μm) and grain morphology (majority of equiaxed grain with aspect ratio < 2), eliminating the typical cube texture of Ni-based superalloys additively manufactured. The solution annealing heat treatment (1100 °C/ 1 h) had a more profound effect on the grain size refinement. Therefore, the DED + in situ interlayer hot forging grain size refinement was mainly related to the thermo-mechanical-affected zone static recrystallization during post-deposition heat treatment. In conclusion, DED + in situ interlayer hot forging proved to be a suitable alternative to overcome the coarse and oriented microstructure commonly observed in the Ni-based superalloys obtained by arc plasma-DED. Solution (1100 °C/1 h) PDHT had a better response, enabling a finer grain size and less oriented microstructure than stabilization (980 °C/1 h) PDHT. In situ SRXD, supported by the thermodynamic simulations, evidenced that the solution PDHT partially dissolved the secondary phases (Laves and δ). In contrast, stabilization PDHT induced their precipitation in the interdendritic regions. Also, the dislocation density evolution indicated that in situ interlayer hot forging generated enough crystal defects to promote grain size refinement via static recrystallization during PDHT, meeting the AMS 5662 grain size requirement.

3.4 Supplementary Material

3.4.1 Deposition Apparatus

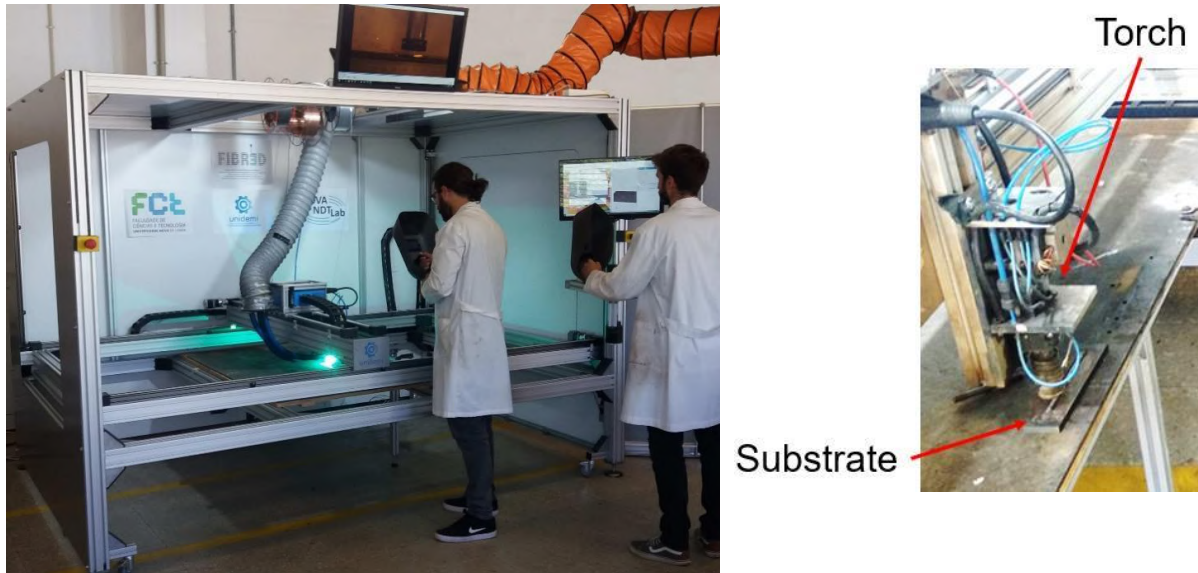


Figure 3.29 - 3 axis 3D printer and hot forging apparatus.

3.4.2 Deposition parameters development

First of all, trial and error bead-on-plate tests were performed to determine the operational window process (Figure 3.30). The criteria to classify a sound weld bead was based on visual inspection, morphological aspect, and deposition stability. Typical defects observed in the region out of the window process were humping and undercut. In sequence, inside the operational window region, a design of experiments (Box–Behnken response surface, Figure 3.31) was performed to statistically correlate the process parameters and bead-on-plate morphology (width, penetration, height, and contact angle). Considering that the in situ hot forging alter the bead surface, the most important parameters to be considered is the penetration since a deep penetration can completely remelted (erase) the effect of in situ hot forging. Figure 3.32 show the Box–Behnken response surface statistic data. Based on these results, the optimization condition was set, i.e., minimum penetration. The optimized parameters were adopted throughout the present work (Table 3.1).

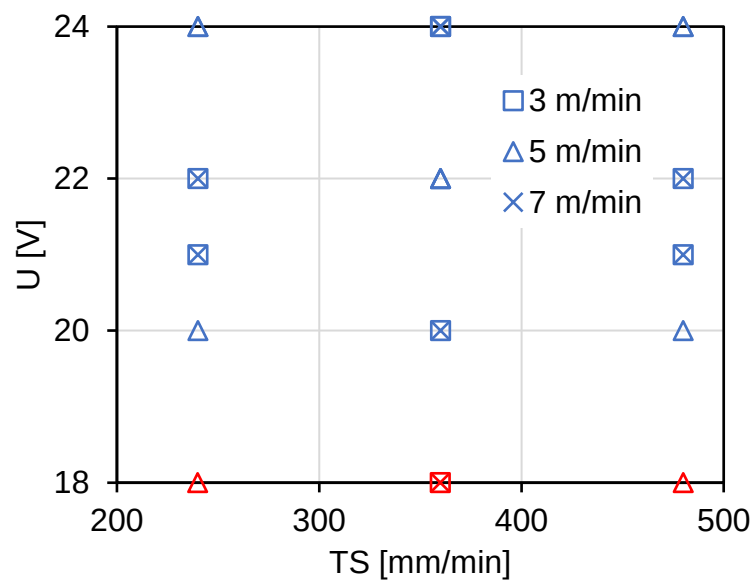
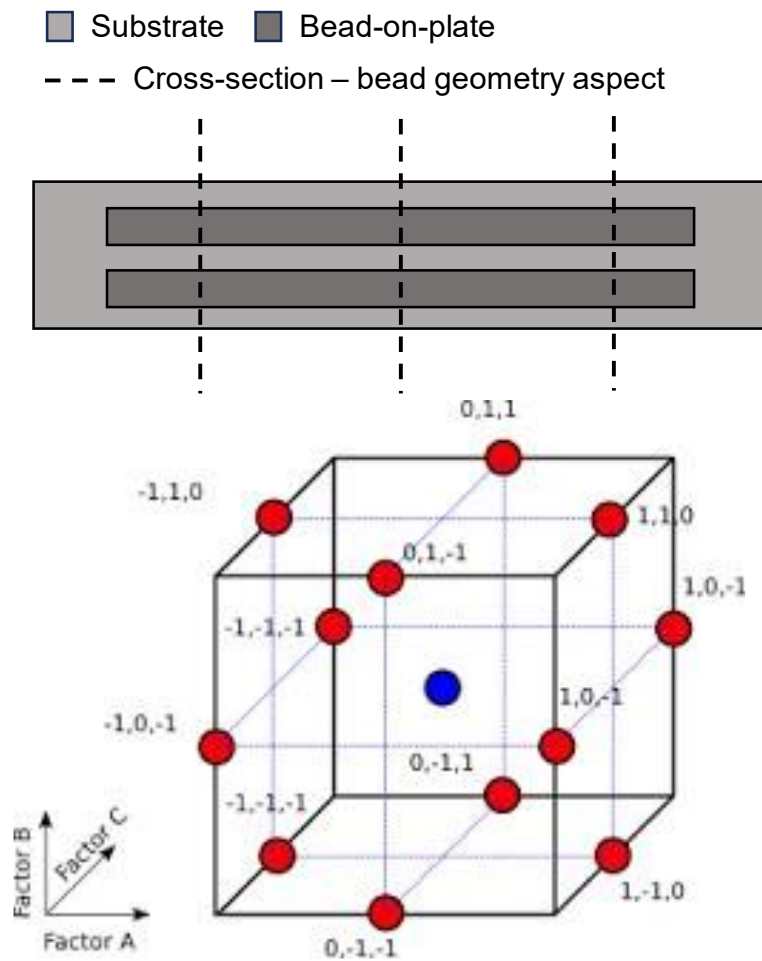


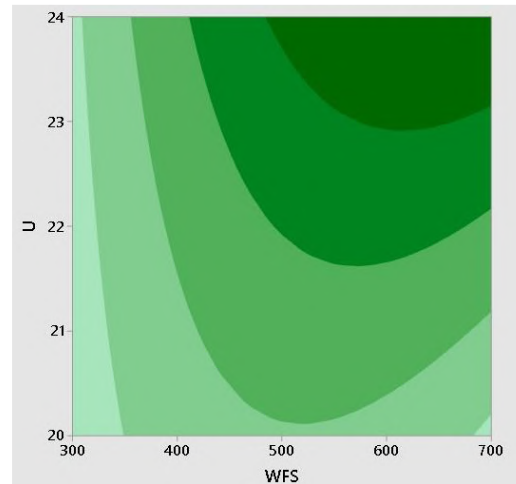
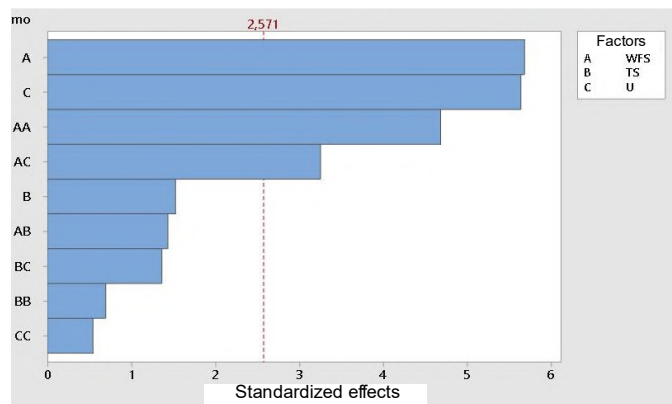
Figure 3.30 - Operational window for gas metal arc welding machine used in the present work. Blue and red colors represent the deposition parameter that promoted a defect-free and unsound bead-on-plate, respectively.



$$y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i < j}^k \beta_{ij} x_i x_j + \sum_{i=1}^k \beta_{ii} x_i^2 + \epsilon$$

Figure 3.31 - Methodology to analyze the bead-on-plates (superior) and Box–Behnken response surface (bottom).

Penetration



	GL	SQ (Aj.)	QM (Aj.)	Valor F	Valor-P
Model	4	1,21166	0,30292	20,05	0,000
Linear	2	0,80874	0,40437	26,76	0,000
WFS	1	0,40755	0,40755	26,97	0,000
U	1	0,40118	0,40118	26,55	0,000
Quadratic	1	0,26934	0,26934	17,83	0,002
WFS*WFS	1	0,26934	0,26934	17,83	0,002
2 Factors interaction	1	0,13359	0,13359	8,84	0,014
WFS*U	1	0,13359	0,13359	8,84	0,014
Erro	10	0,15109	0,01511		
Adjust fault	8	0,12380	0,01548	1,13	0,549
Pure erro	2	0,02729	0,01364	*	*
Total	14	1,36276			

Figure 3.32 - Box-Behnken response surface statistical analysis.

3.4.3 Physical properties of the filler metal and substrate

Table 3.2 - Physical properties of the filler metal and substrate.

Inconel® 625					
Temperature [°C]	Density [kg/m³]	Temperature [°C]	Specific heat [J/kg.K]	Temperature [°C]	Thermal conductivity [W/m.K]
23	8440	25	410	20	9,2
100	8410	100	430	100	10,1
200	8380	200	456	200	10,8
300	8340	300	480	400	12,5
400	8310	400	508	600	14,1
500	8260	538	536	800	15,7
600	8220	649	565	1000	17,5
700	8180	760	590	1200	19
800	8130	871	620	1400	20,8
900	8080	982	645	1600	22,8

1000	8040	1093	670		
1100	7990	1200	700		
1200	7910	1300	740		
1300	7850	1370	770		
1370	7800				
Substrate					
Temperature [°C]	Density [kg/m ³]	Temperature [°C]	Specific heat [J/kg.K]	Temperature [°C]	Thermal conductivity [W/m.K]
20	7815	0	430	0	40
1500	7290	100	500	200	39
		200	550	300	38
		300	580	500	33
		400	610	700	25
		500	650	900	25
		600	710	1500	33
		700	790		
		800	865		
		900	565		
		1440	630		
		2500	707		

3.4.4 Electrical deposition parameters

The electric current and arc plasma voltage oscillograms (Figure 3.33) were acquired with a data acquisition system and a LEM LA 200-P current probe (sampling rate of 5 kHz). The root mean square (RMS) of the current ($I_{RMS} = 72.1 \text{ A}$) and arc plasma ($V_{RMS} = 17.1 \text{ V}$) were calculated. For x values ($x_1, x_2, \dots, x_n$), the RMS can be calculated using Equation 2.16.

$$x_{RMS} = \sqrt{\frac{1}{n} \left(\sum_{i=1}^n x_i^2 \right)} \quad (2.16)$$

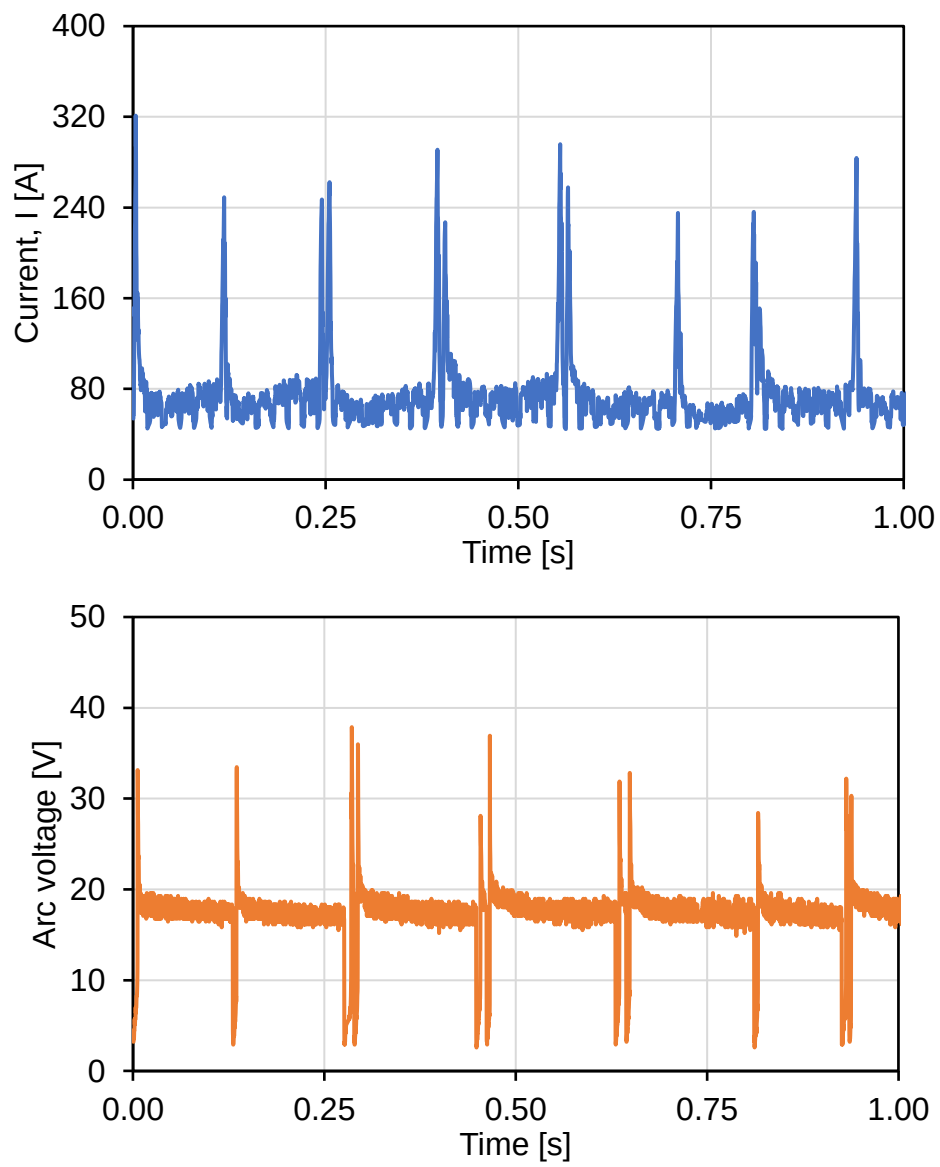


Figure 3.33 - Current and arc voltage oscillogram.

3.4.5 Goldak heat source parameters

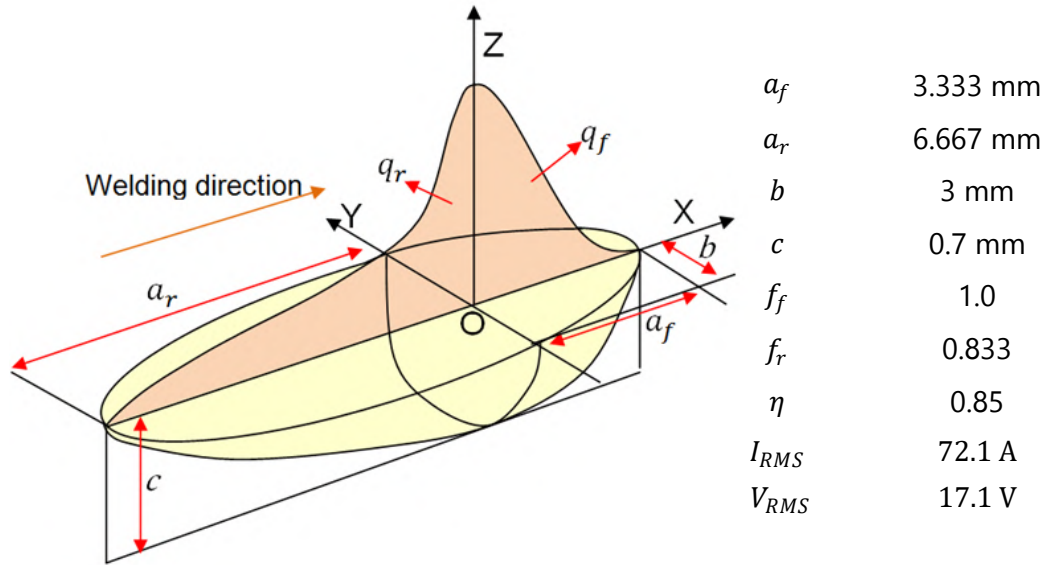


Figure 3.34 - Goldak's double ellipsoidal heat source model [411] and parameters.

3.4.6 Normalized in situ synchrotron X-ray diffraction (SXRD)

Given the theoretical peak intensity (Equation 2.17) and its correlation with phase volume in a multi-phase material diffractogram (Equation 2.18), the normalized data of a single-phase diffraction peak (Equations 2.19 and 2.20) can indicate the variation of phase content ($c(t)$) in relation the initial one (as-built condition) [385]. K is a constant, which depends on the X-ray diffractometer characteristics and X-ray wavelength (λ). All the variable names and symbols are presented in the glossary (the end of this section).

$$I_{hkl} = \overbrace{\left(\frac{I_0 A \lambda^3}{32 \pi r} \right) \left(\frac{\mu_0 e^2}{4 \pi m} \right)^2}^K \overbrace{\frac{1}{v^2} \left[F(hkl) \right]^2 p \left(\frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta} \right)}^{R(\theta)} \overbrace{\left(\frac{e^{-2M}}{2\mu} \right)}^{Temperature} \quad (2.17)$$

$$I_{hkl} = KR(\theta) \left(\frac{e^{-2M}}{2\mu} \right) (c_\gamma + c_{\gamma''} + c_\delta + c_{Laves} + c_{MC}) \quad (2.18)$$

$$I_{single-phase} = KR(\theta) \left(\frac{e^{-2M}}{2\mu} \right) c_{single-phase} \quad (2.19)$$

$$\frac{I_{single-phase}(t)}{I_{single-phase}(0)} = \frac{R(\theta) e^{-2M} c(t)}{R(\theta_0) e^{-2M_0} c(0)} \quad (2.20)$$

$$\frac{c(t)}{c(0)} = \frac{I_{single-phase}(t)}{I_{single-phase}(0)} \frac{R(\theta_0)}{R(\theta)} e^{-2(M_0 - M)} \quad (2.21)$$

At a specific temperature, all factors that affect the peak intensity (Lorentz–polarization, structure, multiplicity, absorption, and temperature factors) tend to be similar, independently of the exposure time at those temperatures. However, considering the phase transformation and precipitates dissolution during the PDHTs, slight changes in 2θ (affecting the Lorentz–polarization) can occur due to the phase evolution, change in composition, and morphology, as reported by Idell et al. [403] and Stoudt et al. [400] for Inconel[®] 625 fabricated via PBF. In addition, the multiplicity factor (p) is a function of the unit cell and does not change for a single-phase peak during the heat treatment. The structure factor ($F|(hkl)|^2$) is independent of the phase unit cell shape and size, implying that despite possible unit cell alteration (e.g., change in lattice parameters due to diffusion), atoms' positions (atomic scattering factor) will be similar, i.e., $F|(hkl)|^2$ can also be considered constant during the PDHT [385]. In relation to v (unit cell volume), Lindwall et al. [399] and Zhang et al. [402] (both Inconel[®] 625 fabricated via PBF) considered this variable constant during the heat treatment. They obtained a good match between the secondary phase content estimation via image analysis and SXRD, which indicated that v does not significantly change during the PDHT and can also be considered a constant parameter. The analysis described above indicated that $\frac{R(\theta_0)}{R(\theta)}$ (Equation 2.20) depends only on the Lorentz–polarization factor (i.e., 2θ). In addition, as exemplified for the δ phase (Figure 3.28a), the Lorentz–polarization factor was not significantly (almost constant, $\cong 1$) affected (less than 0.5 %) during the heat treatment.

The temperature factor, e^{-2M} , also depends on the diffraction angle [385]. In order to verify the effect of some diffraction angle variation during the heat treatment, a similar analysis to $R(\theta)$ was performed (from Equation 2.21-2.27; Figure 3.28b). The results indicate that e^{-2M} can also be considered constant during the PDHT. Thus, Equation 2.20 can be approximated by Equation 2.29 without a “compensation factor” or significant error. Therefore, the directed normalization of a single-phase diffraction peak (Figure 4 in the manuscript) can qualitatively express the phase content evolution along the time for a specific temperature (PDHT peak temperature).

$$\frac{e^{-2M}}{2\mu} = \frac{1}{2\mu} (e^M)^{-2} \quad (2.22)$$

$$M = \frac{6h^2T}{mk\theta^2} \left[\varphi(x) + \frac{x}{4} \right] \frac{\sin^2 \theta}{\lambda^2} \quad (2.23)$$

$$K_2 = \frac{6h^2T}{mk\theta^2\lambda^2} \left[\varphi(x) + \frac{x}{4} \right], x = \frac{\theta}{T} \quad (2.24)$$

$$M = K_2 \sin^2 \theta \quad (2.25)$$

$$\frac{e^{-2M_0}}{e^{-2M}} = e^{-2(M_0-M)} = e^{-2K_2(\sin^2 \theta_0 - \sin^2 \theta)} = \left(e^{(\sin^2 \theta_0 - \sin^2 \theta)} \right)^{-2K_2} \quad (2.26)$$

$$\overbrace{e^{(\sin^2 \theta_0 - \sin^2 \theta)}}^{\text{Figure S1b}} \cong 1 \quad (2.27)$$

$$\frac{e^{-2M_0}}{e^{-2M}} = \left(e^{(\sin^2 \theta_0 - \sin^2 \theta)} \right)^{-2K_2} \cong 1 \quad (2.28)$$

$$\frac{c(t)}{c(0)} = \frac{I_{\text{single-phase}}(t)}{I_{\text{single-phase}}(0)} \quad (2.29)$$

Glossary

I_0	Intensity of incident beam
A	Incident beam area
λ	0.14235 Å, Wavelength
r	Radius of diffractometer circle
μ_0	Permeability of free space
e	Electron charge
m	Electron mass
v	Unit cell volume
$F (hkl) ^2$	Structure factor
p	Multiplicity factor
θ	Bragg angle
t	Time instant
$c(t)$	Phase content at an instant t
$\left(\frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta} \right)$	Lorentz–polarization factor
e^{-2M}	Temperature factor
$\frac{1}{2\mu}$	Absorption factor
h	Planck's constant
T	Temperature
k	Boltzmann's constant
θ	Debye characteristic temperature
$\varphi(x)$	$\frac{1}{x} \int_0^x \frac{\epsilon}{e^\epsilon - 1} d\epsilon$, function tabulated see [385]
(hkl)	Miller indices
ρ_S	Dislocation density

<i>FWHM</i>	full width at half maximum
ε	Microstrain
<i>D</i>	Crystallite size
<i>b</i>	Burgers' vector
<i>a</i>	Unit cell parameter

4 HIGH-PERFORMANCE INCONEL 718 FABRICATED VIA ARC PLASMA DED: EFFECT OF POST-DEPOSITION HEAT TREATMENTS ON MICROSTRUCTURE AND MECHANICAL PROPERTIES

Ni-based superalloy 718 fabricated *via* arc plasma direct energy deposition (IN718 AP-DED) exhibit a limited response to heat treatment due to its coarse primary microstructure and interdendritic segregation, which may prevent its use in high-integrity applications. Thus, dedicated heat treatments for IN718 AP-DED must possess a homogenization temperature as high as possible to significantly dissolve the eutectics and increase the γ'' -former elements in solid-solution. The present work proposed heat treatments for IN718 AP-DED (homogenization – 1050, 1100, 1142, and 1185 °C / 2 h – followed by aging – 718 °C/ 8 h, cooling at 56 °C/ h, and 621 °C/ 8 h). The as-built IN718 AP-DED showed the typical coarse and oriented (cube texture) microstructure with eutectics (Laves and MC-typical carbides) in the interdendritic region, which were significantly dissolved during the homogenization, promoting a high-volume fraction of hardening phase (γ'' and γ') and outstanding quasi-static mechanical properties after the aging step. The present work showed that IN718 AP-DED mechanical properties can be optimized through dedicated heat treatments, meeting, for the first time, the AMS 5662 requirements (room temperature). Furthermore, the heat treatments did not alter the grain morphology and texture aspect, inducing a lower Young's modulus compared to the non-oriented material.

4.1 Room temperature tensile behavior and microstructure characterization

4.1.1 Introduction

Additive manufacturing (AM) can eliminate the permanent manufacturing setups and devoted tools (e.g., molds and dies), promoting supply chain decentralization and lead time reduction [2], [159], [160], [412], [413]. In relation to metal AM processes, arc plasma directed energy deposition (AP-DED) stands out due to its high deposition rate (1 – 12 kg/h) and relative density ($\sim 99.9\%$) [1], becoming suitable to medium/large parts fabrication. AP-DED is especially attractive for bad machinability, good weldability/printability, and high-cost

materials, such as Ti- and Ni-based alloys, due to its near-net-shape aspect (lower machining steps and post-processing time) [17], [337], [414], [415]. AP-DED uses an arc-based welding machine as the heat source to deposit the feedstock material, i.e., it is almost similar to welding, so that most welding metallurgy and operational fundamentals can be extended to AP-DED [14], [416]. Therefore, the typical coarse and highly oriented microstructure, inhomogeneous composition (dendritic core and interdendritic region) [417], poor post-weld heat treatment response, and inferior mechanical properties compared to thermomechanically processed materials were also expected for AP-DED. Moreover, in AP-DED, multi-layer multi-beads deposition strategy and directional and lower cooling rate typically promote coarser grain structures and significant microsegregation [20], [30], [418], which may prevent the use of parts fabricated by AP-DED in high-responsibility engineering applications (e.g., aeronautics and oil & gas industries).

Among high-performance alloys, the precipitation-strengthened Ni-based superalloy 718 (UNS N07718; commercially named Inconel® 718) is highlighted by its outstanding quasi-static mechanical properties and corrosion and creep resistance. It is used in critical applications, such as high pressure turbine disks and oil drilling equipment [59], [208]. However, Inconel® 718 fabricated *via* AP-DED (IN718 AP-DED) experiences printability challenges (hot cracks) [214] and exhibits inferior quasi-static mechanical properties and corrosion resistance compared to its wrought counterparts [419]–[422]. These limitations are mainly attributed to the as-built primary microstructure (coarse and aligned columnar grains) and high content of eutectics (Laves and MC-type carbides) [423], which compromise the IN718 AP-DED response to post-deposition heat treatments [333].

Each manufacturing route (e.g., casting, welding, and/or forging) results in a particular microstructure, leading to a different Inconel® 718 heat treatment response, which depends on the original microstructure [206], [253], [424]. A similar interpretation can be extended to metal AM processes due to their distinct essential variables, e.g., energy density and heat input (i.e., cooling rate ranging between 10^2 and 10^6 °C/s [67], [286]), travel speed, and feedstock material (e.g., wire and powder), which promotes different secondary dendrite arm spacing, deleterious phase content (especially Laves), and microsegregation patterns [269], [310], [343]. Thus, each metal AM processes requires a specific heat treatment [287], [298]–[300], [310], such as those developed for wrought (AMS 5662 and API 6ACRA), cast (AMS 5383), and powder bed fusion (PBF; ASTM F3055). Therefore, these standardized heat treatments applied to Inconel® 718 may have an inadequate or poor response to IN718 AP-DED due to its particular microstructure aspects.

Regarding IN718 AP-DED, Seow et al. [25] reported that the direct double-aging had a minor hardening effect (precipitation of γ'' and γ') due to its efficiency in dissolving the Laves phase, resulting in a heterogeneous distribution and low content of hardening phases. In addition, high-temperature heat treatments (AMS 5383 and a modified AMS 5383 – homogenization at 1185 °C/ 40 min) significantly reduced the interdendritic phase content; however, the wrought material requirements were not met (AMS 5662). Xu et al. [105] indicated that the precipitation strengthening effect is impaired by the heterogeneous composition (interdendritic solidification segregation) and low solubilized Nb (captured in Laves and MC-type carbides phases), which results in a lower mechanical properties (did not meet the AMS 5662). Kindermann et al. [21], [106] tested the aerospace (AMS 5662) and oil & gas industry (API 6ACRA) heat treatments, where API 6ACRA could almost completely dissolve the Laves phase and generate a low δ phase content due to the absence of the solution step. In contrast, the AMS 5662 heat treatment was inefficient in dissolving the Laves phase, resulting in a heterogeneous γ'' and γ' distribution and higher δ content in Nb-rich region (interdendritic). Xi et al. [107], [108], using the highest homogenization temperature (1185 °C/ 1 h – incipient melting temperature of Inconel® 718 arc-based welded [425]), partially met the AMS 5662 (yield strength requirement) using the straightforward manufacturing route (AP-DED + heat treatment), attributing this behavior to the almost complete Laves dissolution and higher γ'' and γ' phases content. In addition, Cormier et al. [426] (Inconel® 718 fabricated *via* laser wire DED) reported that the dissolution of Laves phases during the homogenization heat treatment (1000 – 1200 °C / 2 h) is sensitive to the primary microstructure (i.e., DED process dependent) and that achieving its complete dissolution can induce incipient melting (i.e., it must be avoided). These findings [21], [25], [105]–[108], [426] highlighted that typical heat treatments applied to IN718 AP-DED do not perform similarly to wrought material. Furthermore, the quasi-static mechanical properties requirements (AMS 5662) were not met using the standard heat treatments (e.g., AMS 5662, AMS 5383, and API 6ACRA). Compared to cast Inconel® 718, IN718 AP-DED typically possesses a finer dendritic microstructure and lower secondary phase content (faster cooling rate) [268], which results in a better heat treatment response and superior mechanical properties [21], [106]–[108]. Moreover, for applications at elevated temperatures, IN718 AP-DED must also have an equivalent behavior to wrought material, which has not yet been achieved. Seow et al. [25], Zhang et al. [109], and James et al. [319] reported that heat-treated IN718 AP-DED (modified AMS 5383 and AMS 5662) exhibited inferior performance compared to other AM processes [332], [427], [428] and non-compliance with AMS 5662, which emphasizes the

importance of developing PDHTs specifically tailored for IN718 AP-DED to allow its use in critical engineering applications.

When comparing, from the worst to the best condition, the direct aging [25], AMS 5662 [21], [105], [106], API 6ACRA [21], [106], AMS 5383 [25], and modified AMS 5383 (Xi et al. [107], [108], 1150 or 1185 °C / 1 h) heat treatments responses, it is possible to observed that the material strength increase with homogenization temperature. Therefore, considering the literature data [21], [25], [97], [98], [105]–[112], a recommended heat treatment for IN718 AP-DED would have a homogenization temperature as high as possible (≥ 1050 °C; eutectics dissolution), which should be followed by the typical AMS 5662 aging heat treatment. In addition, the solubilization step (e.g., 980 °C/ 1 h), usually performed after homogenization, should be avoided to prevent δ precipitation. In this regard, the present work studied the effect of novel dedicated heat treatments developed for IN718 AP-DED (1050, 1100, 1142, and 1185 °C / 2 h + AMS 5662 aging) on its microstructure and mechanical response. For this, *in situ* synchrotron X-ray diffraction (SXR) analyses were conducted during the homogenization to track the secondary phase dissolution. As-built and *post-mortem* (homogenization and homogenization + aging) conditions were microstructurally characterized through scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), electron backscatter diffraction (EBSD) and SXR. The response of the IN718 AP-DED to the heat treatments were evaluated using Vickers microhardness and uniaxial tensile testing (room temperature).

4.1.2 Materials and methods

4.1.2.1 Sample fabrication

4.1.2.1.1 Parts printing

Inconel[®] 718 single-bead multi-layer parts ($7 \times 55 \times 120$ mm³) were fabricated using an arc plasma DED 3D printer, which consists of a gas metal arc (GMA) welding machine (CITOWAVE III 520) coupled to an in-house developed 3-axis CNC [36]. The feedstock material (Voestalpine Böhler; 1.2 mm; Table 4.1) was deposited on a hot rolled C-Mn steel plate ($80 \times 10 \times 200$ mm³). The arc plasma DED current and voltage were measured using an open-loop LEM HTA 1000-S current transducer and a DAQ National Instruments 6008, respectively. Table 4.2 summarizes the deposition parameters.

Table 4.1– Feedstock material chemical composition (weight %).

	Ni	Cr	Fe	Nb	Mo	Ti	Al	C	Mn	Si	P	S
Measured	52.52	17.41	20.23	5.25	2.98	0.98	0.45	0.05	0.04	0.08	0.002	0.001
AWS A5.14	50-55	17-21	Bal.	4.75-5.5	2.8-3.3	0.65-1.15	0.2-0.8	0.08	0.35	0.35	0.015	0.015
ASTM F3055							0.4-0.8					
AMS 5383						0.80- 1.15	0.4-0.6					
API 6ACRA				4.87-5.2				0.045	0.35	0.35	0.01	0.01

Table 4.2 – Arc plasma directed energy deposition parameters.

Arc plasma current [†]	137 [A]
Arc plasma tension [†]	17 [V]
Wire feed speed	4 [m/min]
Travel speed	5 [mm/s]
Heat input [‡]	372.6 [J/mm]
Interlayer temperature	150 [°C]
Idle time	90 [s]
CTWD*	12 [mm]
Shielding gas	I3-ArHe-25
Shielding gas specification	AWS A5.32
Shielding gas flow	15 [l/min]
Substrate preparation	ISO 8501-1

[†] Root mean square (RMS)

[‡] Thermal efficiency ($\eta = 0.8$), BS EN 1011-1

* Contact-tip-to-work distance

4.1.2.1.2 Post-deposition heat treatments

After deposition, samples underwent the proposed post-deposition heat treatment (Figure 4.1). These heat treatments were designed based on the previous literature available for IN718 AP-DED [21], [25], [97], [98], [105]–[112], which indicated that optimized heat treatments for IN718 AP-DED must have an as high as possible homogenization temperature to ensure maximum dissolution of the deleterious phases (e.g., Laves), avoid the solution step to prevent δ precipitation, and follow the typical AMS 5662 aging heat treatment. The 1050 and 1100 °C homogenization temperatures were chosen based on the industrial heat treatment (API 6ACRA and AMS 5383, respectively). The maximum homogenization temperature (1185 °C) was selected based on γ /Laves eutectic incipient melting temperature [425]. Also, similar homogenization temperature was previously tested by Xi et al. [107] and Seow et al. [25], which showed a significant eutectic dissolution. However, differently from Xi et al. [107] (GMA-DED), Seow et al. [25] (plasma transferred arc DED – PTA-DED) reported that the 1185 °C homogenization temperature could drive static recrystallization and intense secondary grain growth. Thus, to prevent a possible secondary grain growth, an intermediate homogenization temperature (1142 °C) was also analyzed, exploring entirely the homogenization temperature range (from typical industrial heat treatments to recently developed ones). The

homogenization soaking time (2 h) was selected considering the upper limit established by AMS 5383 and API 6ACRA. After the homogenization step, the samples underwent the industrial aging heat treatment (AMS 5662). These heat treatments (Figure 4.1) were performed using a Nabertherm 1800 furnace with a temperature control of $\pm 2^\circ\text{C}$.

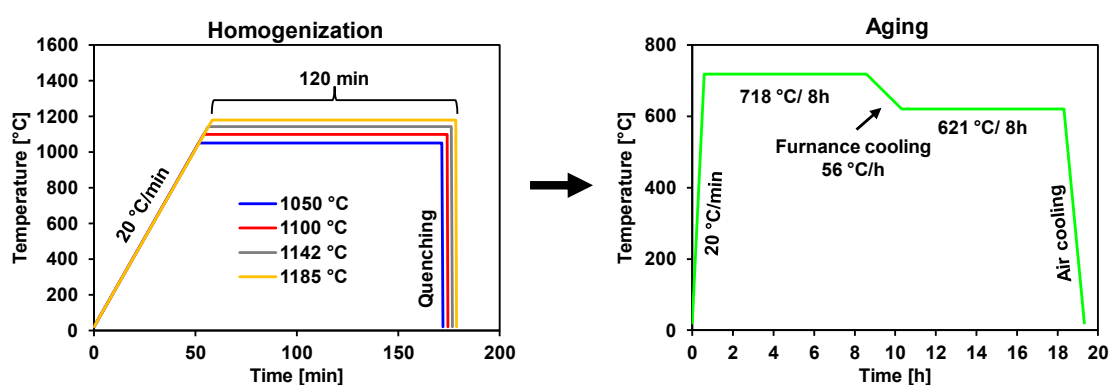


Figure 4.1 – Proposed post-deposition heat treatment schedules for Inconel® 718 fabricated *via* AP-DED.

4.1.2.2 Materials characterization

4.1.2.2.1 Microstructure

The microstructure was evaluated through SEM coupled with EDS and EBSD. Metallographic specimens (Figure 4.2) were, in sequence, ground, polished, and electrolytically etched (10 wt.% oxalic acid – $\text{HO}_2\text{C}-\text{CO}_2\text{H}$ – in water, 6 V for 25 s). The EBSD specimens were prepared following the same sequence, adding a final mechanical polishing (colloidal silica, $0.04\ \mu\text{m}$, 2 h). EBSD data post-processing (grain size, Taylor factor, pole figures, and Hill's model of the Young's modulus calculations) was performed using with the MTEX Toolbox.

4.1.2.2.2 In situ synchrotron X-ray diffraction (SXRD)

In situ SXRD in transmission mode (PETRA III/DESY) was performed during the homogenization step of the proposed heat treatment (Figure 4.1). Experiments were conducted using an acquisition rate, wavelength, and incidence area of 0.015 Hz, $0.14235\ \text{\AA}$, and $1 \times 1\ \text{mm}^2$, respectively. The X-ray beam was focused on the central region of the samples ($1.5 \times 10 \times 20\ \text{mm}^3$). *In situ* SXRD data (i.e., 2D Debye-Scherrer diffraction rings) were acquired in a 2D Perkin Elmer detector ($200 \times 200\ \mu\text{m}^2$) and post-processed using the Fit2D (integration along the full azimuthal angle) and an in-house developed python-based routines. This latter used the xrdfit package [429] and the pseudo-Voigt function to fit the diffraction peaks. For more details about the experimental setup, the reader is referred to our previous works [160], [413].

4.1.2.3 Mechanical tests

4.1.2.3.1 Uniaxial tensile test

To evaluate the heat treatment response (AMS 5662 and API 6ACRA), the tensile tests (room temperature and 650 °C) were carried out using specimens extracted along the deposition (horizontal) and building (vertical) directions (Figure 4.2). A tensile testing machine (Instron® 8562) equipped with a 100 kN load cell and a contact extensometer was used. Tensile tests were performed using a strain rate of $\sim 5.0 \cdot 10^{-4} \text{ s}^{-1}$. These specimens were machined out from heat-treated walls by electric discharge machining (EDM). Their final dimensions were achieved by turning and subsequent low-stress mechanical polishing of the gauge part to remove scratches from machining and to remove the deformed layer below the surface of tensile specimens inherited from machining and possibly affecting at the yield stress determination and tensile ductility. Two specimens were evaluated for each condition.

Cylindrical specimens with a 14 mm gauge length, a ~ 4 mm diameter and a 42 mm total length were used for these tests. These specimens were machined out from heat-treated walls by electric discharge machining. The as-built part had a thickness of ~ 7 mm. Therefore, for the fabrication of the specimens, the Electrical Discharge Machining (EDM) procedure must consider the wire diameter (~ 0.5 mm) and a minimum amount of material to be removed. Additionally, the arc plasma DED parts exhibit waviness, so the minimum thickness (~ 6 mm) must be considered. To prevent issues on the specimen surface (e.g., marks and unmachined regions), a diameter of 4 mm was selected. Furthermore, the gauge length (14 mm) was designed based on the minimum distance required by the water-cooled high-temperature extensometer (~ 10 mm), ensuring it remains outside the radius area in the specimen.

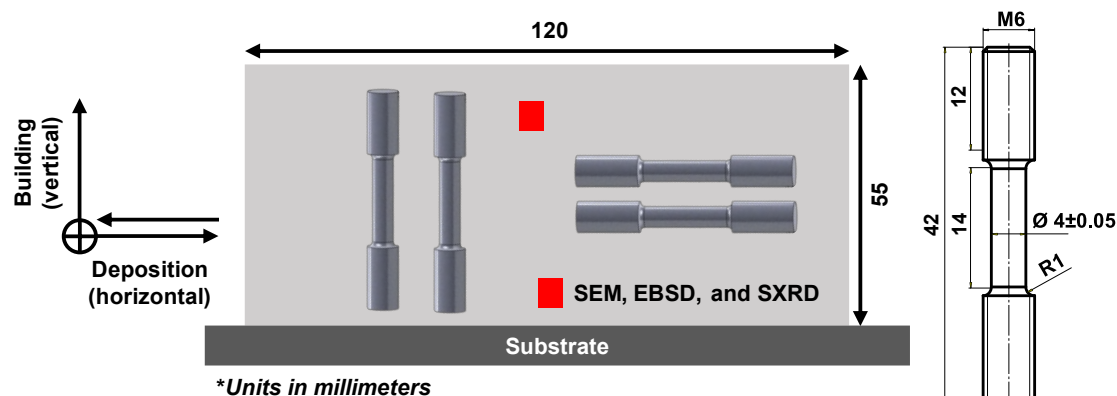


Figure 4.2 – Schematic representation of the tensile and microstructure specimens.

4.1.2.3.2 Vickers microhardness test

Vickers microhardness indentations (Mitutoyo HM-112) were performed (ISO 6507-1) using a load and dwell time of 0.5 kgf ($HV_{0.5}$) and 15 s, respectively. These indentations (nine in total) were distributed in a squared grid (1×1 mm).

4.1.3 Results and discussion

4.1.3.1 As-built microstructure

The microstructure of IN718 AP-DED in the as-built condition exhibited a columnar-dendritic grains with the presence of secondary phases (eutectics) in the interdendritic region (Figure 4.3), a typical microstructure of Ni-based superalloys fabricated *via* AP-DED, as also previously reported [21], [106], [160], [430]. Furthermore, based on the morphological characteristics (Figure 4.3b) and chemical composition (Figure 4.4), the interdendritic precipitates were identified as Laves and MC-type carbides. These findings are supported by the SXRD (Figure 4.3c), which showed characteristic diffraction peaks corresponding to these phases. Moreover, the SXRD results indicated the presence of δ and hardening phases (γ'' and γ'), which can be attributed to the AP-DED multiple thermal cycles and their solid-state precipitation [289]. Figure 4.5 depicts the δ phase needle morphology, which forms in Nb-rich regions (around the Laves phase), which is aligned with the Van et al. [292] (IN718 AP-DED).

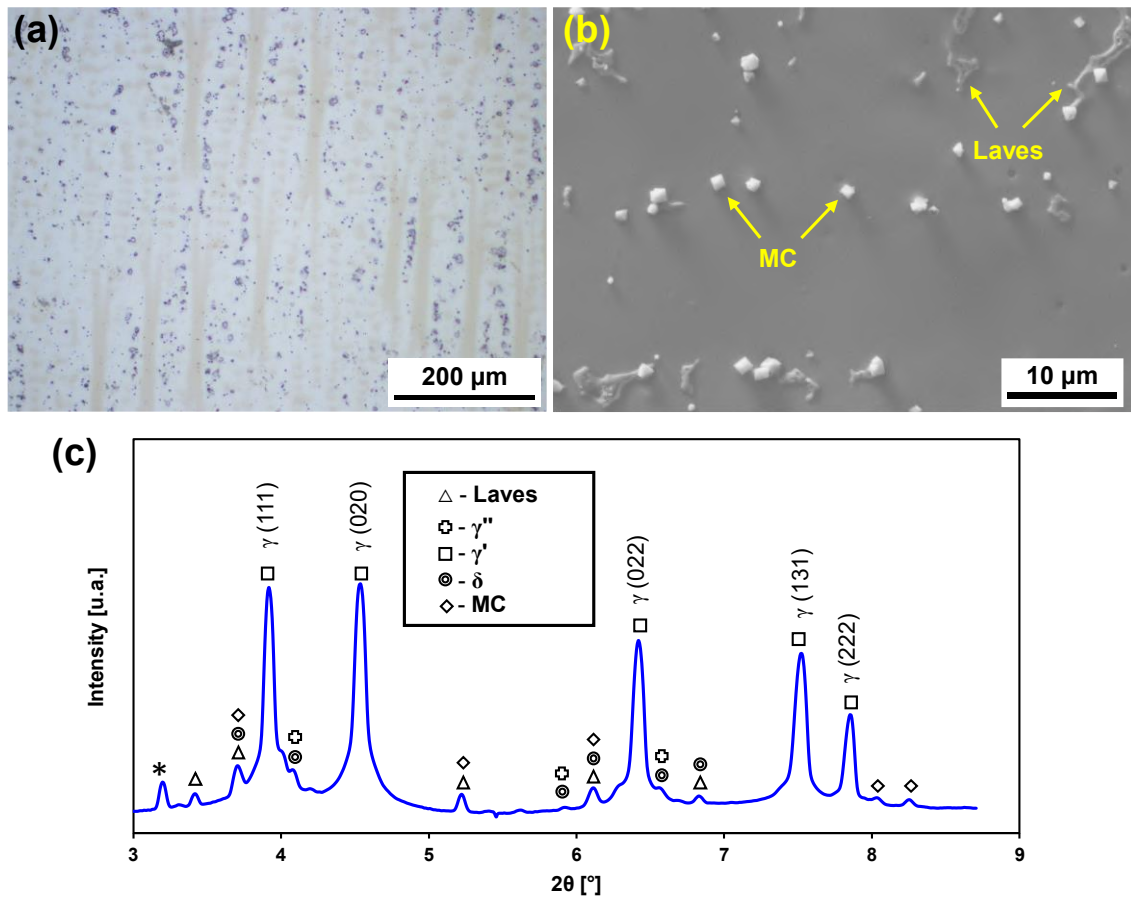


Figure 4.3 – (a) as-built microstructure of the IN718 AP-DED, (b) interdendritic eutectic, and (c) synchrotron X-ray diffraction. The symbol (*) represents a second harmonic peak, γ (022).

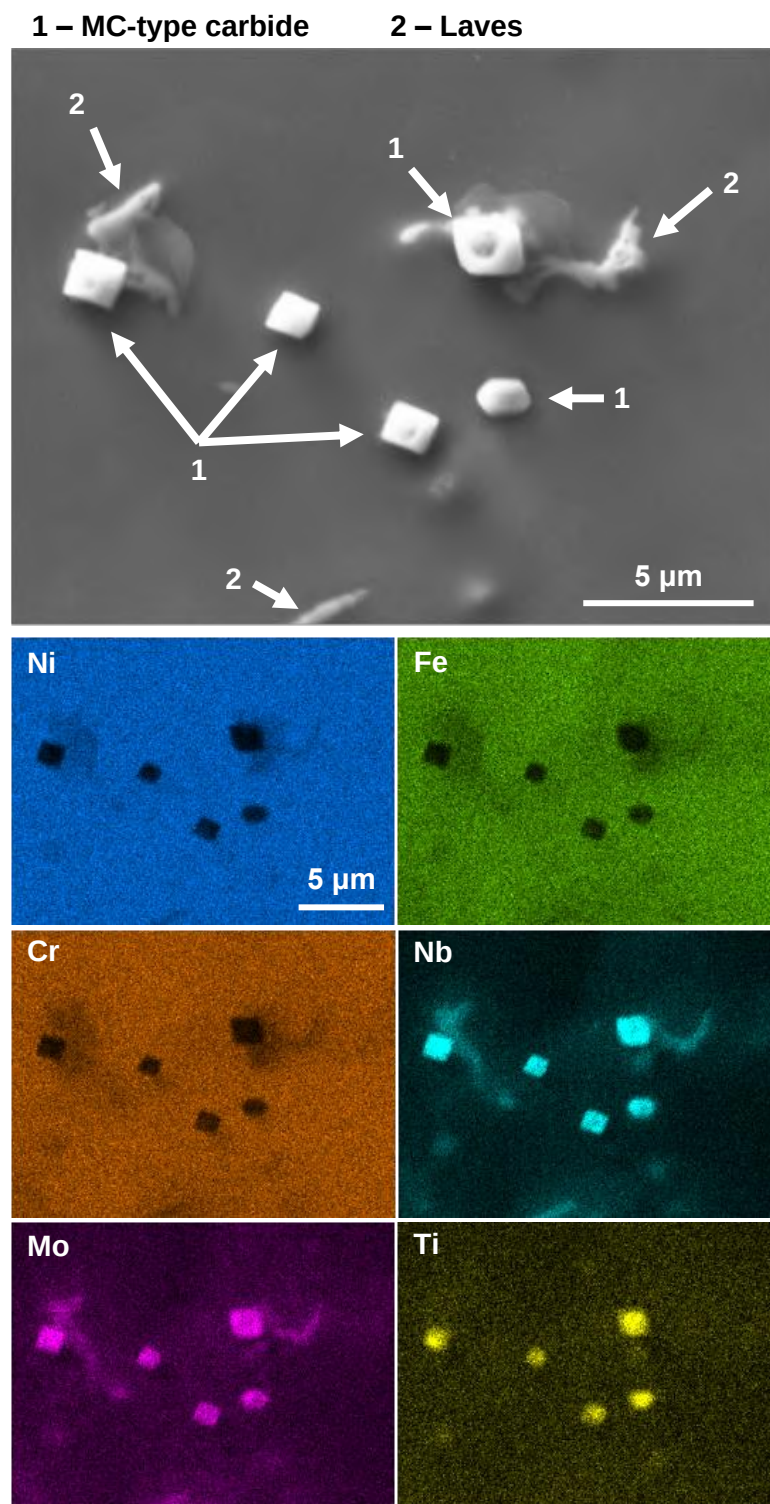


Figure 4.4 – EDS mapping of the interdendritic precipitates.

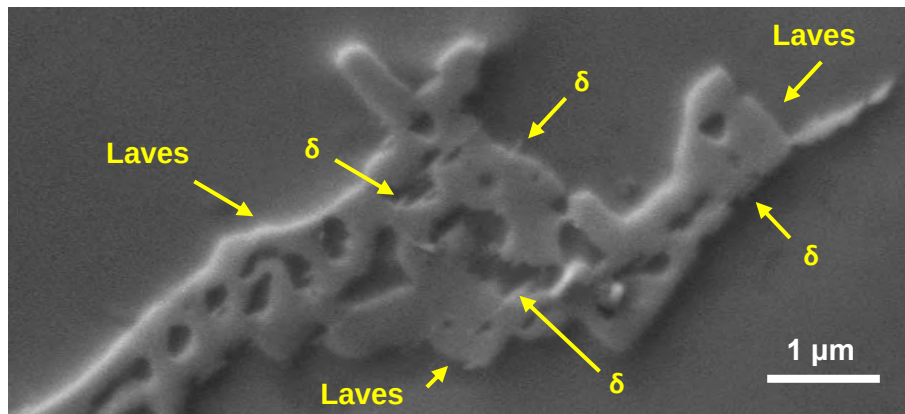


Figure 4.5 – δ phase formation in (Nb,Mo)-rich concentration regions (Laves).

The intense (200) diffraction peak of the matrix (γ) indicates a highly textured grain structure [431], which can also be further visualized *via* the 2D Debye-Scherrer diffraction pattern (Figure 4.6) that displays discontinuous rings with intense spotty features, characteristic of a coarse and highly oriented microstructure. These results are confirmed by EBSD, which revealed coarse columnar grains with a cube crystallographic texture aspect (Figure 4.6b). Gustafsson et al. [377] and Gäumann et al. [432] reported that, during solidification, the dendritic growth direction is almost perpendicular to the fusion line and follows the maximum thermal gradient. However, due to the changes in the thermal conditions during solidification, particularly the thermal gradient, the solidification mode can transition from columnar-dendritic to equiaxed-dendritic. In arc plasma DED, the region of equiaxed grains is typically remelted, while the partially oriented grains (columnar-dendritic) remain. Consequently, due to the epitaxial phenomena, each subsequent layer will have a oriented microstructure that is further intensifies upon successive layer-by-layer depositions [397], promoting the characteristic cube texture (Figure 4.6) observed for non-transformable solid-state alloys fabricated *via* fusion-based additive manufacturing [264], [426], [430], [433].

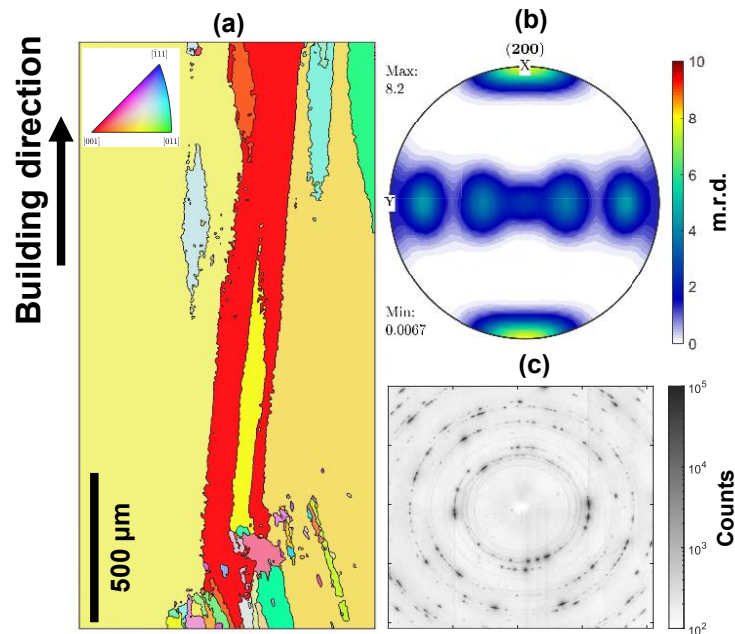


Figure 4.6 – Orientation image map (a), (200) pole figure (b), and (c) 2D Debye-Scherrer diffractions patterns of the Inconel® 718 fabricated *via* AP-DED. EBSD data post-processed using MTEX. Figure 4.6a shows the average grain misorientation.

The secondary phases identified (Laves and MC-type carbide) are in accordance with the Scheil-Gulliver solidification model (Figure 4.7). Similar results using the Scheil-Gulliver model were previously obtained by Yu et al. [265] and Jang et al. [289] for Inconel® 718 fabricated *via* laser-DED and AP-DED, respectively. However, based on the experimental solidification models of Knorovsky et al. [258] and DuPont et al. [260], the solidification sequence of Inconel® 718 should conclude with a divorced eutectic reaction. Due to the interdendritic segregation of Nb and C during solidification, the first eutectic reaction ($L \rightarrow \gamma + \text{MC-type carbide}$) occurs, consuming the rejected C (Figure 4.7). Subsequently, the second eutectic reaction ($L \rightarrow \gamma + \text{Laves}$) completes the solidification sequence. Moreover, Oh et al. [266] performed *in situ* SXRD in Inconel® 718 fabricated *via* laser-DED and did not observe the presence of δ phase in the solidification sequence. Therefore, despite the prediction of δ phase (Figure 4.7) by the Scheil-Gulliver model, its formation can be suppressed due to the fast cooling rate of DED processes ($\sim 10^2$ °C/s – AP-DED – and 10^{2-6} °C/s – laser-DED) [67], [399], [434]. Zhang et al. [402] (Inconel® 625 fabricated *via* PBF) showed that the activation energy for growth of δ , γ'' , and γ' (131.04 ± 0.69 kJ·mol⁻¹) is inferior to activation energy of Nb diffusion in Ni (202.6 ± 4.7 kJ·mol⁻¹), suggesting that δ , γ'' , and γ' nucleation and growth is not limited by long-range diffusion of heavy elements. In addition, δ formation is associated with the interdendritic region and Laves phase (Figure 4.5), i.e., Nb-rich regions;

thus, long-range Nb diffusion is not mandatory for δ precipitation. This fast kinetics at the interdendritic region allows the precipitation of γ'' , γ' , and δ during the multiple AP-DED thermal cycles [67], [292], [435], justifying their presence in as-built IN718 AP-DED diffractogram (Figure 4.3c).

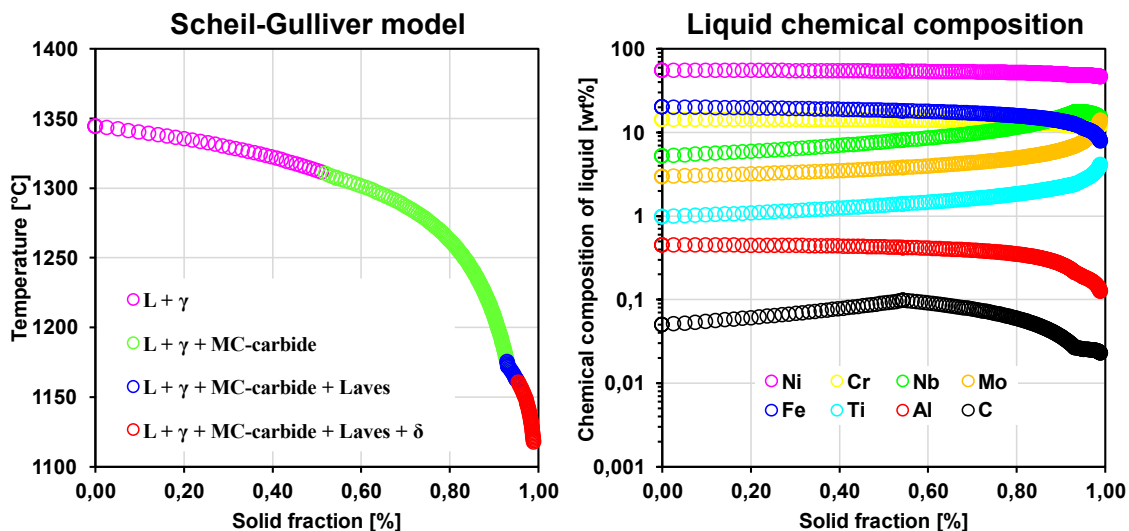


Figure 4.7 – Scheil-Gulliver solidification model (Thermo-Calc® 2023b) and liquid chemical composition evolution of the Inconel® 718 during the solidification.

In summary, the as-built IN718 AP-DED exhibits a typical coarse and aligned columnar-dendritic microstructure (cube texture aspect) with interdendritic eutectics (Laves and MC-type carbides), which result from the Inconel® 718 solidification sequence (Figure 4.7). Additionally, due to interdendritic segregation (enrichment of both Nb and Mo), in-process solid-state precipitation of γ'' , γ' , and δ can also occur. Therefore, considering that Nb is a primary γ'' former [222] and that Laves and MC-type carbides are rich in Nb (Figure 4.4), the homogenization heat treatment (Figure 4.1) must dissolve the secondary phases (especially Laves and δ) as much as possible, increasing the amount of Nb available in solid-solution during and enhancing the γ'' precipitation.

4.1.3.2 *In situ* SXRD during homogenization heat treatments

The effect of the homogenization heat treatment on IN718 AP-DED is shown in Figure 4.8. It can be observed that the proposed heat treatments eliminated all diffraction peaks related to γ'' , γ' , and δ , which restores the peak symmetry and eliminates the "shoulder" close to γ (111) and γ (220) peaks. Considering the equilibrium and time-transformation-temperature diagrams of Inconel® 718 (Figure 4.9), the homogenization temperatures are above the solvus temperature of γ'' , γ' , and δ (~ 1040 °C), which allows for their dissolution during the

homogenization soaking time [436]–[438]. In addition, standard-based homogenization heat treatments (1050 and 1100 °C / 2 h) do not significantly affect the peak intensity of the secondary phases (Laves and MC-type carbide). However, for higher homogenization temperature heat treatments (1142 and 1185 °C / 2 h), the intensity of the Laves and MC-type carbide peaks is reduced, with the 1185 °C / 2 h heat treatment showing a significant reduction in the MC-type carbide peak intensity and almost complete elimination of the Laves phase peak. These results are consistent with the findings of Seow et al. [25] and Xi et al. [107] (IN718 AP-DED), who reported the absence of Laves after the highest homogenization heat treatment (1185 °C for 40 min and 1 h, respectively).

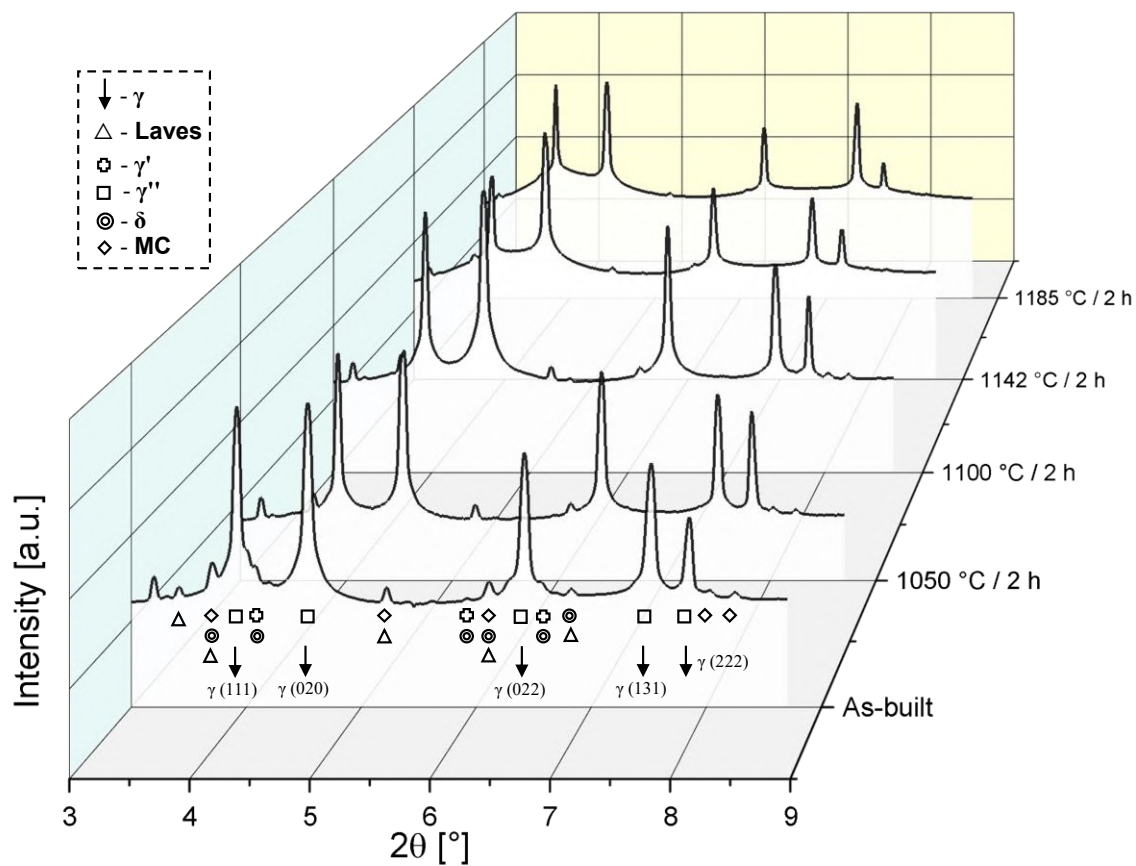


Figure 4.8 – Synchrotron X-ray diffraction pattern of the Inconel® 718 fabricated *via* AP-DED after homogenization heat treatment (1050, 1100, 1142, and 1185 °C/ 2 h).

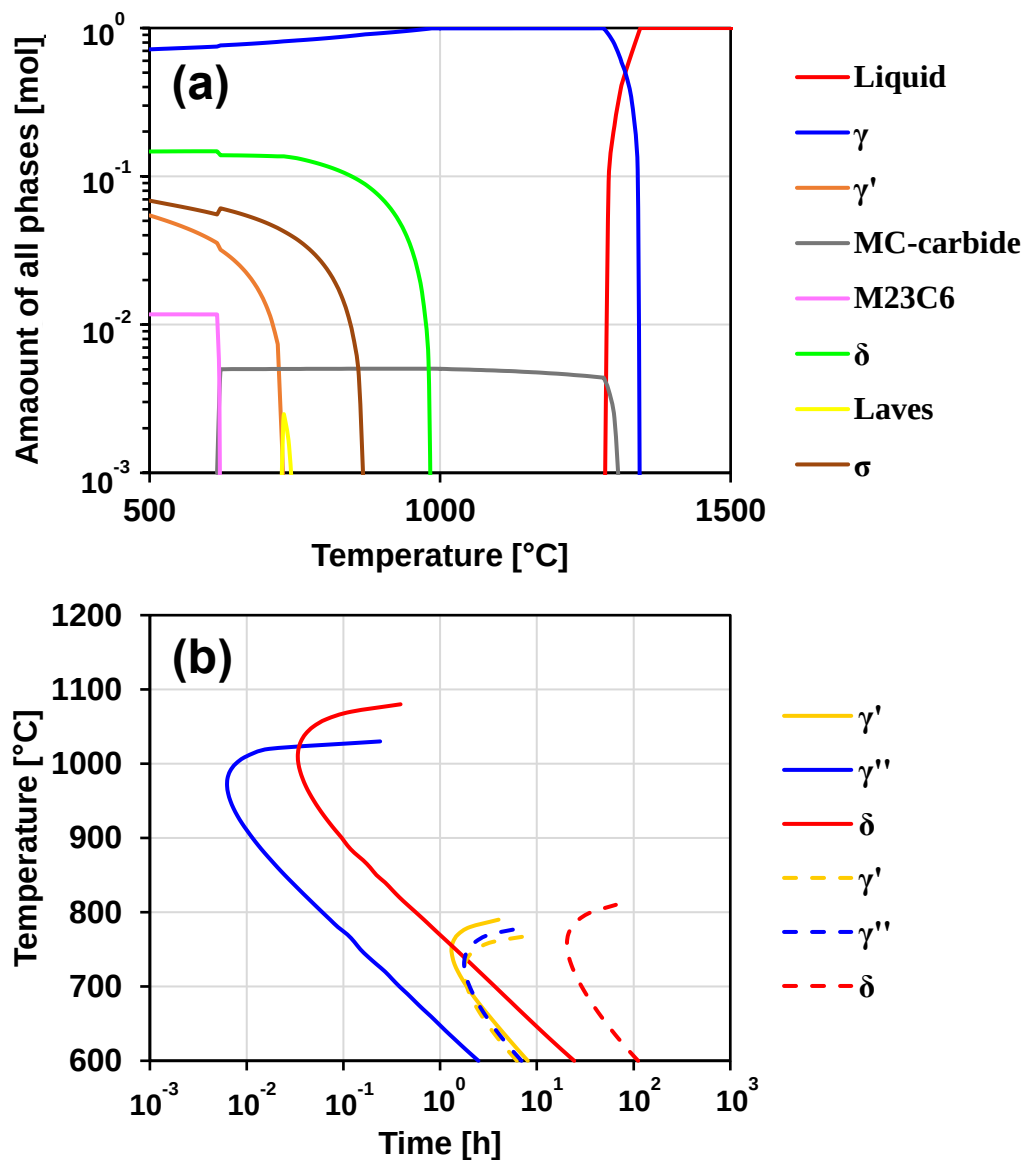


Figure 4.9 – (a) Equilibrium diagram of the Inconel® 718 (Thermo-Calc®) and (b) time-transformation-temperature (TTT) diagram (JMatPro® 13.3) of the dendrite core (dotted line) and interdendritic region (continuous line).

Tracking the peak intensity of Laves and MC-type carbides along the homogenization heat treatment (shown in Figure 4.10), it can be seen that the API 6ACRA-based heat treatment (1050 °C / 2 h) showcased an almost constant peak intensity. The AMS 5383-based heat treatment (1100 °C / 2 h) showed a better dissolution effect than API 6ACRA. The AMS 5383-based heat treatment had a constant peak intensity in the first half (1 h) and a posterior peak intensity drop in the second half of the soaking time, which can be attributed to (partial) phase dissolution. For a higher temperature heat treatment (1142 °C and 1185 °C), the peak intensity showed a continuous drop, indicating a more effective dissolution of the secondary phases compared to both API 6ACRA and AMS 5383 conditions. In addition, Figure 4.10 suggests that

the typical soaking time adopted in the literature (1 h) is insufficient to achieve significant secondary phase dissolution for IN718 AP-DED, which is in accordance with Cormier et al. [426]. These authors reported that even after the homogenization heat treatment (1225 °C/ 1 h), the Laves phase was not completely dissolved. The present results are per those of Kindermann et al. [21] and Seow et al. [25], who, *via* a microscopy analysis, verified the presence of δ , Laves, and MC-type carbide after the 1-hour homogenization heat treatments (API 6ACRA and AMS 5383, respectively). Furthermore, Seow et al. [25] and Xi et al. [107] did not verify remaining Laves after the 1185 °C heat treatment, supporting the present *in situ* SXRD results (notable peak intensity reduction during the heat treatment).

The dissolution of secondary phases expands the interplanar distance of the matrix (γ), i.e., reduces the Bragg's angle (θ) and increases the lattice parameter (a , Figure 4.11) [402], which is associated with the increase of lattice distortion due to the differences in atoms' radii (e.g., Ni – 0.1246 nm – and Nb – 0.143 nm) [439], [440]. Figure 4.11 corroborates the peak intensity tracking observations (Figure 4.10), indicating that the standard-based heat treatments (1050 and 1100 °C/ 2 h) had a lower dissolution effect in relation to others 1142 and 1185 °C / 2 h for IN718 AP-DED. Additionally, the 1100 °C homogenization heat treatment showed a slight increase in a , especially after the first hour of heat treatment (shown in Figure 4.10). The 1142 °C homogenization heat treatment showed a continuous increase in the lattice parameter, reaching a stable condition in the final stages of the heat treatment, suggesting that the adopted soaking time (2 h) was suitable for this temperature. Conversely, for the 1185 °C, a reached a plateau ($\approx$ 1h20min), indicating an overestimated soaking time.

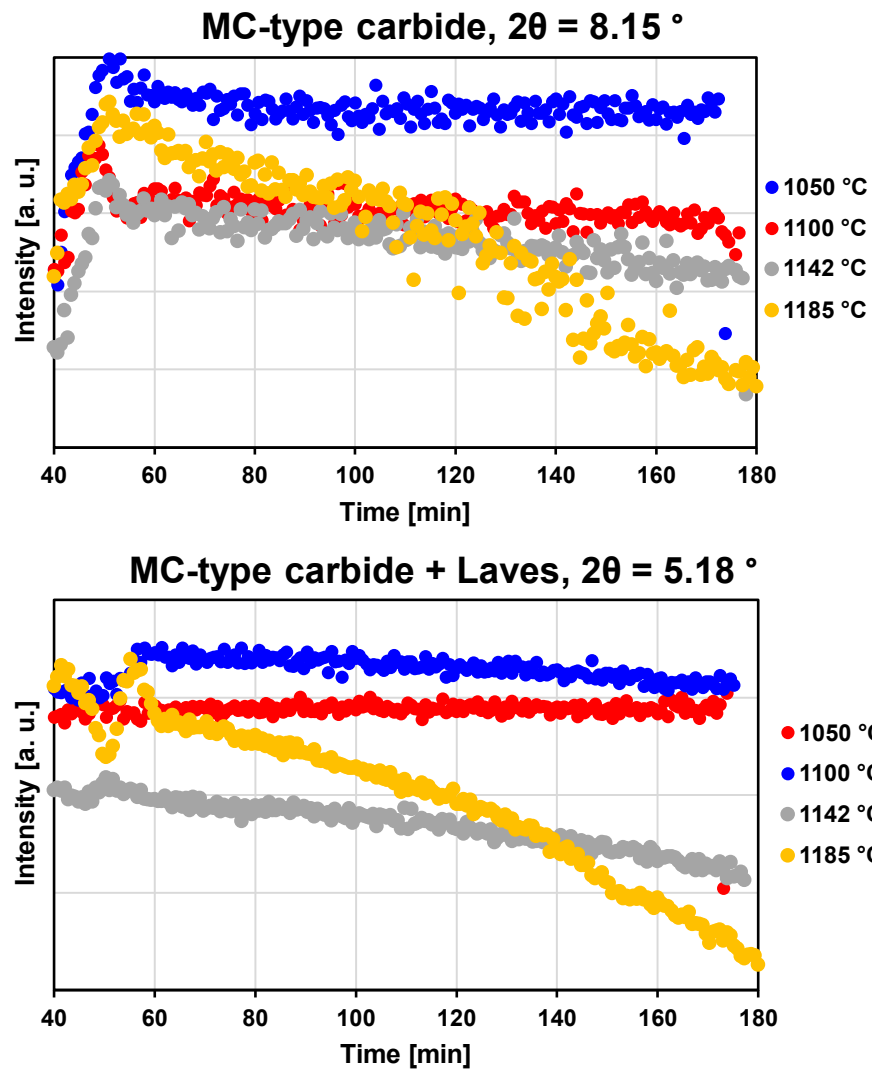


Figure 4.10 – Laves and MC-type carbide peak height intensity evolution over the homogenization heat treatment.

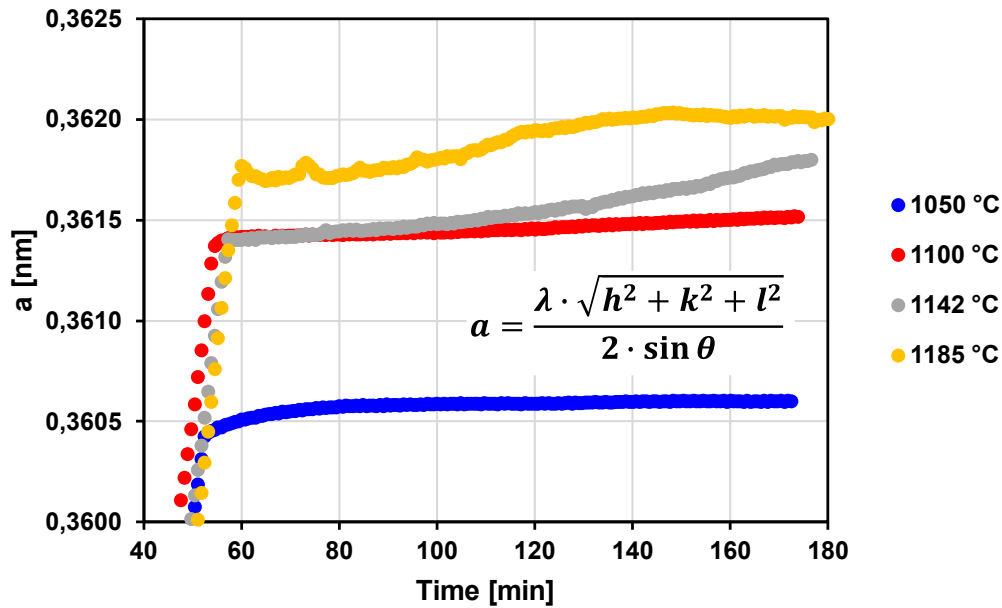


Figure 4.11 – Lattice parameter (a) evolution during the homogenization heat treatment.

4.1.3.3 Effects of the proposed heat treatments on microstructure

4.1.3.3.1 Homogenization

The *in situ* SXRD results were further reinforced by *post-mortem* SEM micrographs (Figure 4.12), which qualitatively showed the effect of the homogenization temperature on the secondary phase content, emphasizing that a higher homogenization peak temperature induced a lower secondary phase content, which was corroborated by the statistical particle analysis (Figure 4.13; ImageJ®). However, it is worth mentioning that all conditions had a significant dissolution of Laves phase in relation to the as-built condition. In addition, Figure 4.14 and Figure 4.15 demonstrate that the proposed homogenization heat treatments do not alter the typical coarse and oriented microstructure of the as-built material (Figure 4.6), maintaining the cube texture aspect. These results are in accordance with those of Xi et al. [43] (GMA-DED), who did not observe recrystallization during the heat treatment (1185°C/ 1 h). On the other hand, Zhong et al. [441] (laser-DED) and Seow et al. [25] (plasma transferred arc DED, PTA-DED) verified static recrystallization during the heat treatment (1100 °C/ 2 h and 1185°C/ 40 min, respectively). These results highlight the effect of the DED process and primary microstructure on the heat treatment response. The present authors believe that the main difference in the heat treatment response between PTA-DED, GMA-DED, and laser-DED arises from the focused heat source of the PTA (constrict arc) and laser (high energy density process), which promote a higher cooling rate and residual intragranular strain level [302]–[304], promoting static recrystallization (desirable) and posterior secondary grain growth

(undesirable) during a high-temperature and longer soaking times heat treatment. Similar behavior was observed for Inconel® 718 fabricated *via* PBF (stress relieved – ASTM F3055) [287]. Thus, high-density energy processes may have more restrictive limits on the homogenization heat treatment temperature and soaking time due to potential undesirable secondary grain growth. In contrast, GMA-DED had less restrictive limits [258], [260], [261], allowing for a homogenization temperature close to γ /Laves incipient melting temperature ($\sim 1185^\circ\text{C}$) and longer soaking times without altering the grain morphology and texture aspect (Figure 4.14 and Figure 4.15).

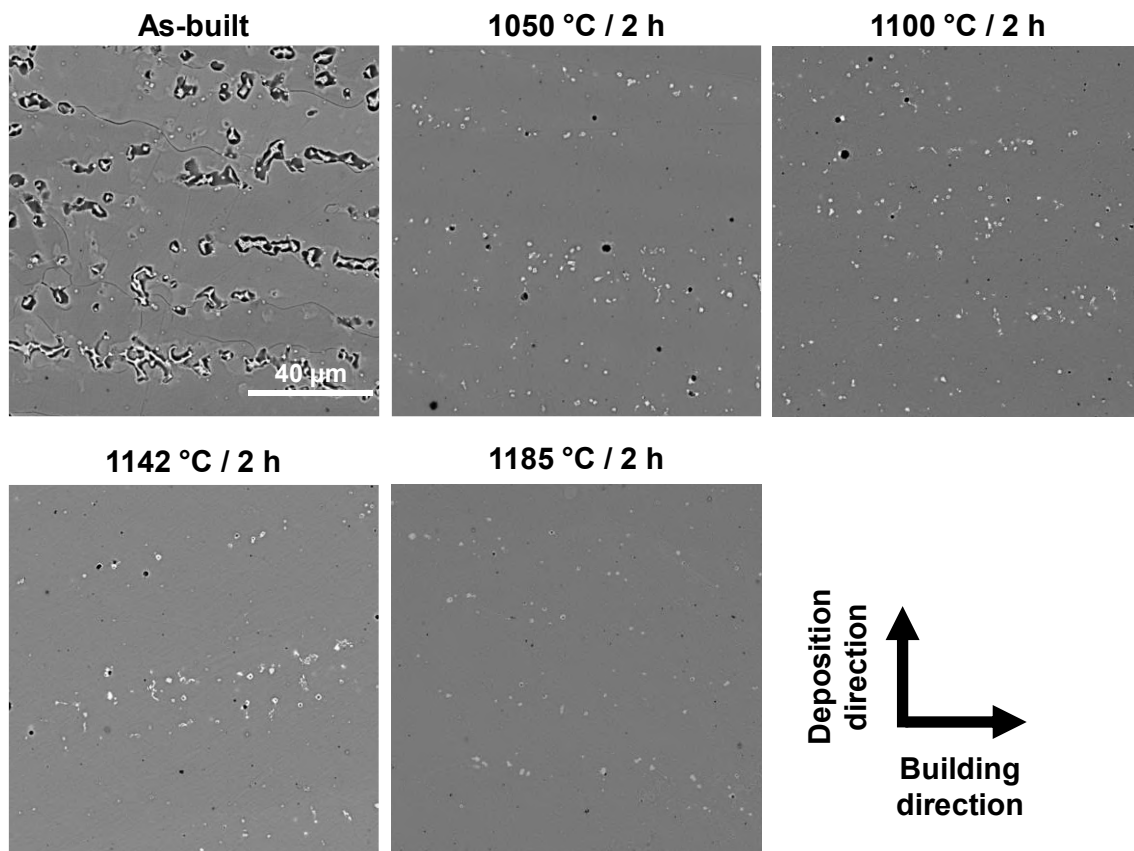


Figure 4.12 – SEM micrographs of the Inconel® 718 fabricated *via* AP- DED after homogenization heat treatments (Figure 4.1).

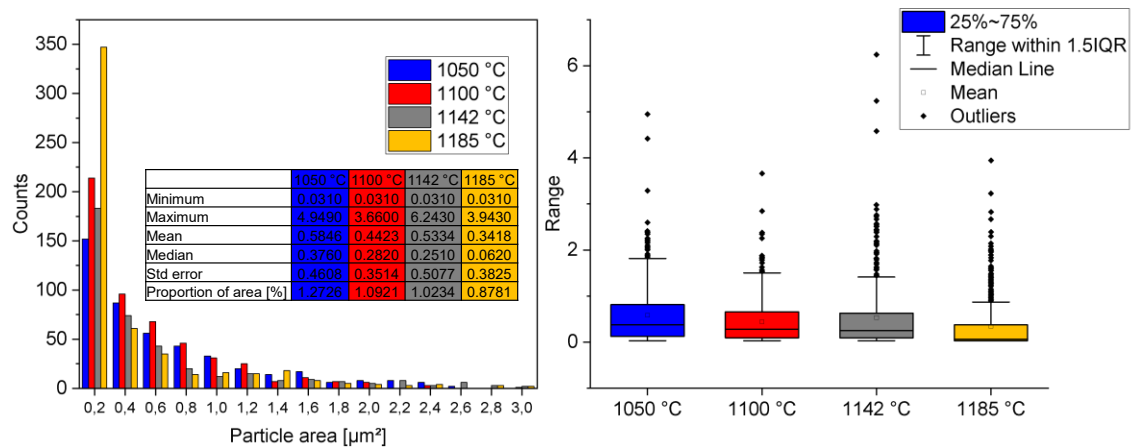


Figure 4.13 –Statistical distribution of particle size after proposed heat treatments. For details about the image segmentation and threshold setting, see Supplementary material.

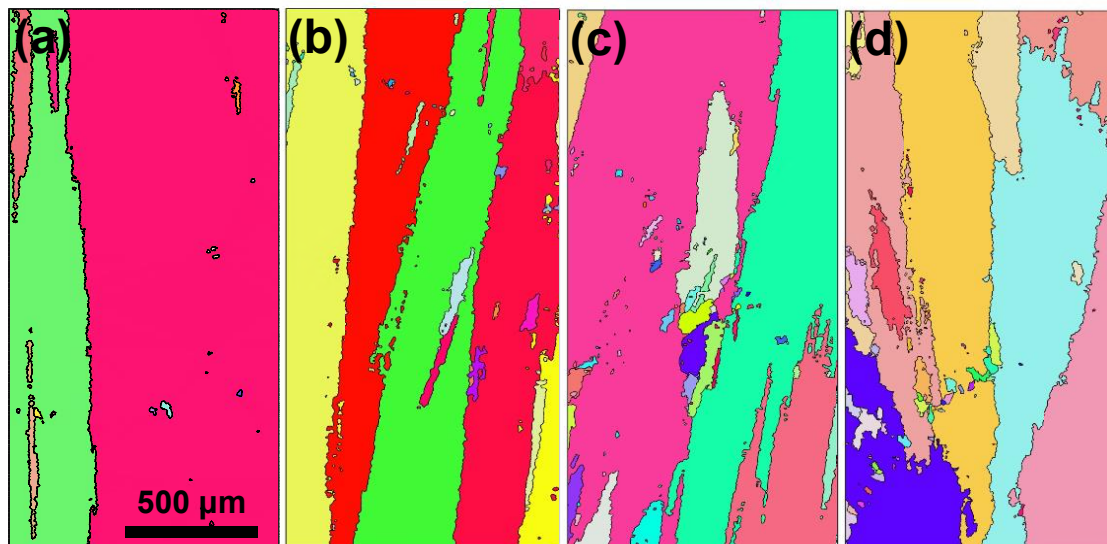


Figure 4.14 – Orientation image map obtained from EBSD analysis of Inconel® 718 fabricated *via* AP-DED after homogenization heat treatment: (a) 1050, (b) 1100, (c) 1042, and (d) 1185 °C / 2 h. For color key for crystal orientation maps and reference direction, see Figure 4.6.

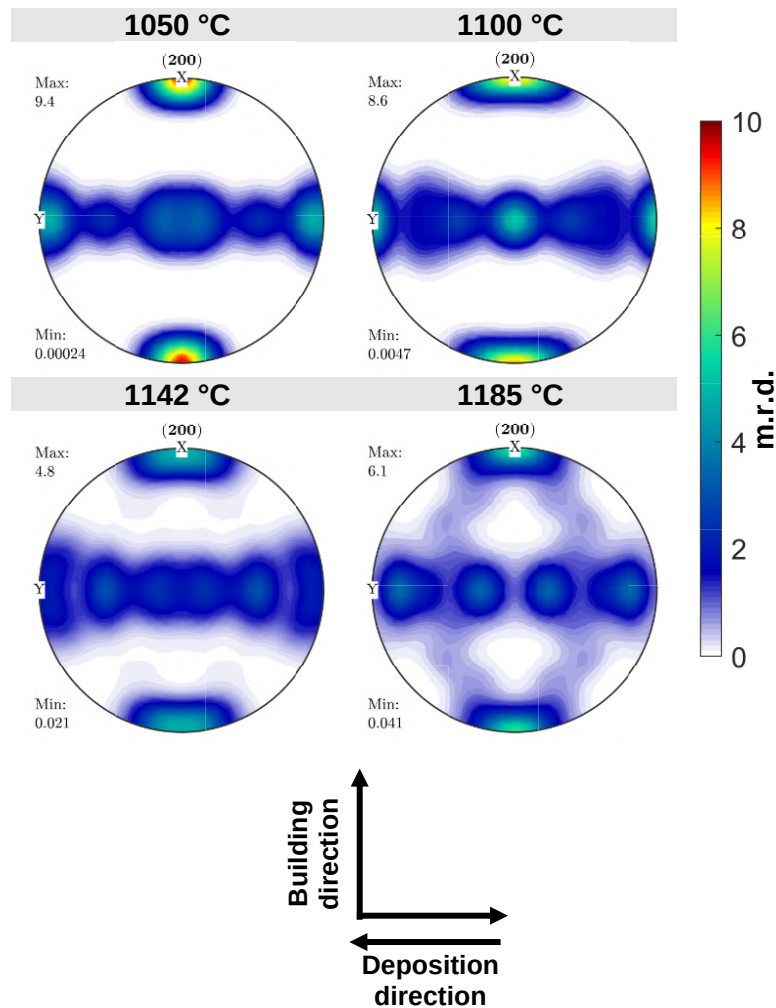


Figure 4.15 – Pole figures analysis of Inconel® 718 fabricated *via* AP-DED after homogenization heat treatment. These pole figures were calculated from the EBSD data of Figure 4.14.

4.1.3.3.2 Aging

After the homogenization, IN718 AP-DED underwent the mandatory aging step of heat treatment. Figure 4.16 showed that the homogenization temperatures and soaking time selected can promote a considerable volume of hardening phases (γ'' and γ' , Table 4.3). The volume fraction of γ'' and γ' was estimated using the Rietveld refinement [442], [443] of the SXR data (DIFFRAC.TOPAS® Bruker). The experimental and calculated SXR patterns for proposed heat treatments can be consulted in the Supplementary material. The estimated γ'' and γ' content is consistent with the available literature on Inconel® 718 fabricated *via* laser-based AM processes [222], [256], [307], [444]. In addition, the γ peak broadening was associated with the γ' superlattice reflections, which also reinforce the high volume fraction observed.

Ferreri et al. [307] measurements using neutron diffraction followed by the Rietveld method indicated that the volume fractions of γ' and γ'' for Inconel® 718 fabricated *via* PBF

(ASTM F3055) and wrought material (AMS 5662) were 5.4 and 6.5 and 17.7 and 17.8 %, respectively. Ferreri et al. [307] also estimated the γ , γ' , and γ'' lattice parameters, showing that, despite the peak superposition (e.g., γ and γ'), the subtle differences in phase lattice parameters are enough to accurately estimate the phase content *via* Rietveld refinement method applied to diffraction techniques in transmission mode (e.g., SXRD). In addition, Zhang et al. [222] (Inconel® 718 fabricated *via* PBF heat-treated – 1100 °C / 1.5 h + aging) estimated (15.52 and 5.09 %) the γ'' and γ' contents through the deconvolution of the main diffraction peak and the corresponding peak area fraction. Sui et al. [256] and Li et al. [444], using quantitative metallography, measured the γ'' (16.9 and 14.9 %) and γ' (5.4 and 4.9 %) contents for PBF Inconel® 718, respectively. Additionally, Ling et al. [309] (Inconel® 718 cast) reported that the AMS 5383 heat treatment promoted a higher content of γ'' (~ 17.1 %), which is almost similar to that estimated in the present work (17.39 %). The γ'' content developed for AP-DED using the proposed heat treatments are in same range than those of laser-based AM processes and wrought material.

Table 4.3 – Volume fraction (%) of the γ'' and γ' estimated *via* Rietveld refinement in IN718 AP-DED heat-treated (homogenization + aging).

	1050 °C	1100 °C	1142 °C	1185 °C
γ'	4.61	4.15	5.39	5.72
γ''	19.59	17.39	13.42	15.7

The γ' content (around 5 %) is less sensitive to the manufacturing route (PBF, laser-DED, , cast, and wrought) and heat treatment, which is also verified for IN718 AP-DED[222], [256], [307], [309], [444]. Thus can be related to the partition coefficient (~ 1) of Al (γ' former; $\text{Ni}_3(\text{Al}, \text{Ti})$) [207], which indicated that Al has an almost homogenous distribution in the dendritic core and interdendritic region (see Supplementary material). This results in approximately the same γ' precipitation kinetic in the dendritic core and interdendritic region (Figure 4.9b), regardless of the material process route, justifying the similar γ' content observed for AP-DED, cast, PBF, and wrought material. On the other hand, Ti had a partition coefficient of 0.69 [207], indicating a tendency to concentrate in the remaining liquid during solidification (Figure 4.7). Thus, given that the higher homogenization heat treatment temperatures (1142 and 1185 °C) are able to promote some (Nb,Ti)C (MC-type carbide) dissolution (peak intensity decreases – Figure 4.10), the Ti content in solid-solution raised and increased the γ' content (Table 4.3) in relation to lower homogenization (1050 and 1100 °C) heat treatment.

Furthermore, to the best of the authors' knowledge, this is the first time that the γ' and γ'' contents were estimated for IN718 AP-DED, demonstrating that, through dedicated

developed heat treatments, the IN718 AP-DED can have similar hardening phase content to that of traditional manufacturing routes despite its unfavorable coarse primary microstructure.

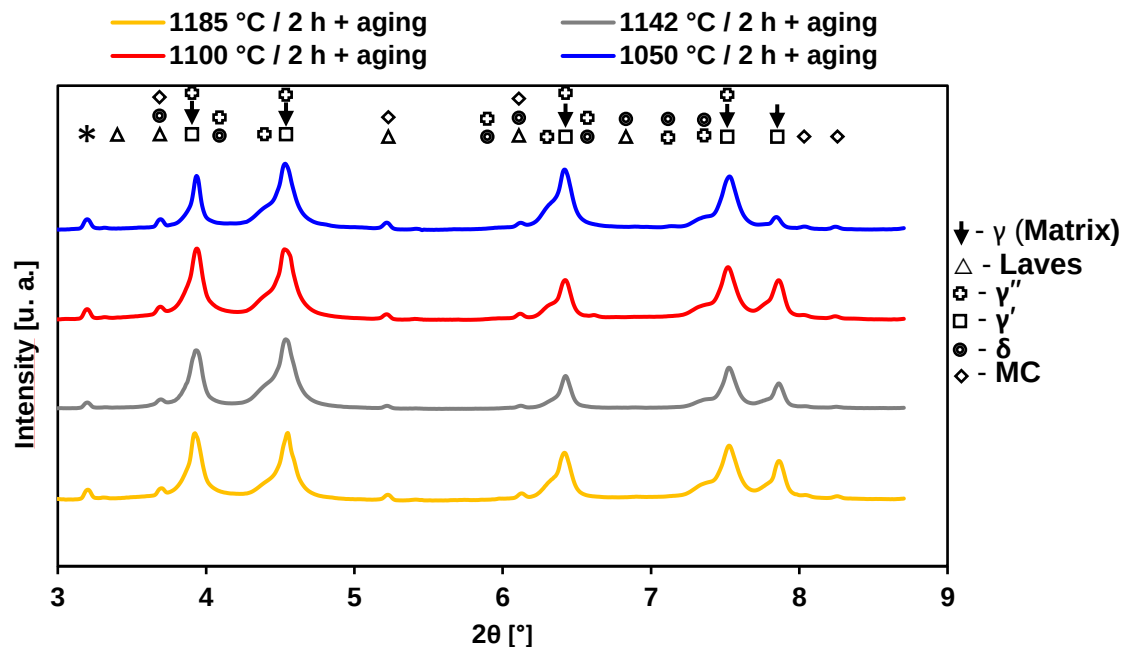


Figure 4.16 – Synchrotron X-ray diffractograms of the IN718 AP-DED heat-treated.

4.1.3.4 Mechanical properties

4.1.3.4.1 General aspects

The proposed heat treatments promoted higher Vickers microhardness ($\geq 400 \text{ HV}_{0.5}$) for all tested conditions (Figure 4.17), which are comparable to those of a wrought and superior to cast material. The IN718 AP-DED heat-treated met the AMS 5662 hardness requirements for all tested conditions. Furthermore, compared to AM processes, the present hardness results are similar to those of PBF and superior to most available data on IN718 AP-DED. The response of IN718 AP-DED to heat treatments was also evaluated through uniaxial tensile testing (Figure 4.18a). The results showed a typical strain-stress curve of a ductile material. The proposed heat treatments had a better response compared to IN718 AP-DED data available in the literature (Figure 4.18), where, for the first time, IN718 AP-DED (1050, 1100, and 1185 °C / 2 h + aging) can meet the strength and ductility requirements of AMS 5662 at room temperature using the typical process route (i.e., printing + heat treatment). This differs from the available literature [109]–[112], [138], [200], which reported that meeting the AMS 5662 requirements was possible only *via* the coupled AP-DED + interlayer mechanical deformation. Among the present results, only the 1142 °C / 2 h + aging in the horizontal direction did not meet the AMS 5662 requirements. Additionally, all tested heat treatments met the requirements of API 6ACRA and

ASTM F3055, which reinforces the applicability and scalability of the proposed heat treatments. Furthermore, due to the finer microstructure of AP-DED in relation to the cast material [67], all results (met the AMS 5383 requirements (Figure 4.18b and Figure 4.18c).

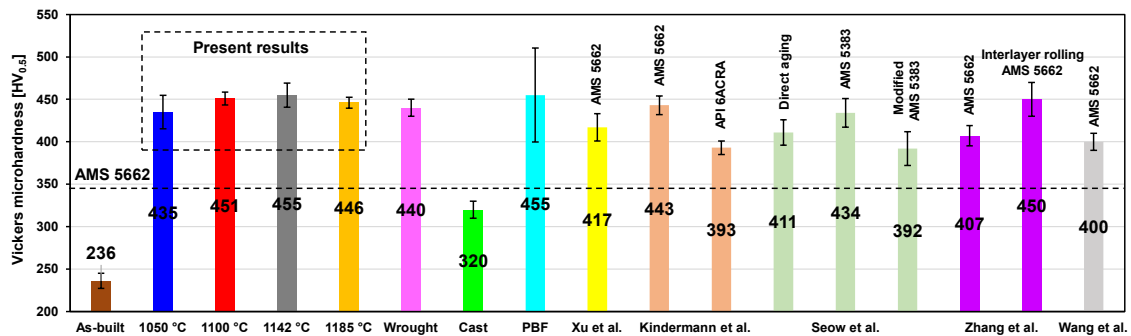


Figure 4.17 – Effect of the proposed heat treatments on Vickers microhardness (HV_{0.5}) of the IN718 AP-DED and their comparison with literature results. Wrought, cast, and PBF results were taken from [276]. The other references are Xu et al. [138], Kindermann et al. [21], [106], Seow et al. [25], Zhang et al. [109], and Wang et al. [301].

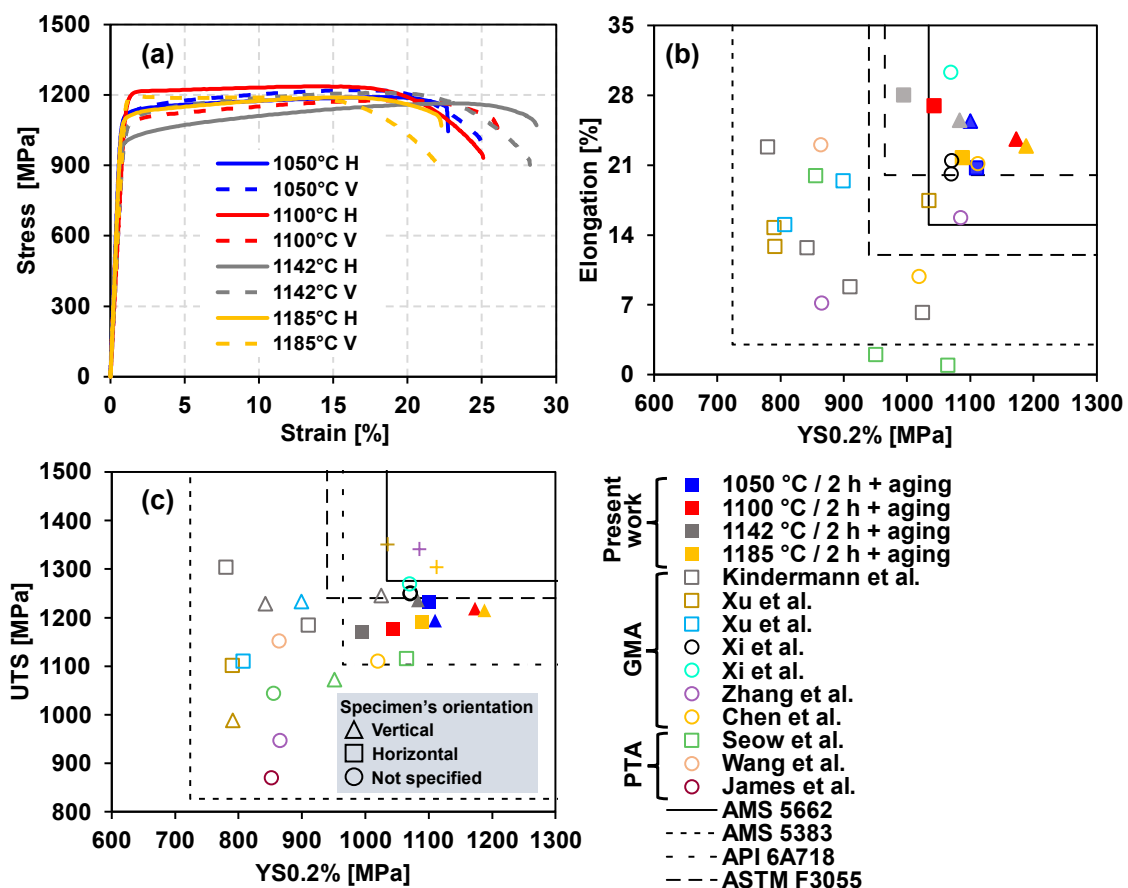


Figure 4.18 – (a) Uniaxial tensile test of the IN718 AP-DED heat-treated and (b – c) comparison of the present work results with the IN718 AP-DED ductility and strength at room temperature data survey. H and V represent the horizontal and vertical directions. The symbol + denotes the IN718 AP-DED + interlayer rolling.

4.1.3.4.2 Semi-quantitative analysis of the strengthening mechanisms

All studied conditions had the typical coarse and oriented microstructure (Figure 4.14) with an almost similar grain size (D ; Supplementary material). Thus, according to the Hall-Petch relation ($\sigma_{gb} = \frac{\alpha}{D^{0.5}}, \alpha = 750 \text{ MPa} \cdot \mu\text{m}^{-0.5}$) [240], [445], the observed differences in the yield strength (Figure 4.18) cannot be directly attributed to the grain boundary strengthening mechanism (σ_{gb}) [21]. Furthermore, due to the high temperature and long soaking time of heat treatments, the tested conditions showed a low and almost similar dislocation density (Bailey-Hirsh's relation, $\sigma_d = 0.2MbG\rho^{0.5}$) [25], [222], [244], which also not justified the material strength difference among the proposed heat treatments. M , b , G (65.9 GPa [446]), and ρ are the Taylor factor, Burgers vector, shear modulus, and dislocation density, respectively. M (2.56), b (0.251737 nm), and ρ ($4.065 \cdot 10^{13} \text{ m}^{-2}$) were computed from the EBSD (Figure 4.14) and SXRD (Figure 4.16) data, respectively. More details for these calculations are shown in the Supplementary material section. In addition, the intrinsic strength (Peierls–Nabarro stress, $\sigma_0 = M\tau_{crss}$) was constant in all conditions; τ_{crss} is the critical resolved shear stress (17.5 MPa [447]).

Sui et al. [256] (laser-DED) and Zhang et al. [222] (PBF) demonstrated for Inconel® 718 heat-treated in different conditions that the effect of solid-solution strengthening (σ_{ss} , Gypen and Deruyttere relation [448]) is insensitive to heat treatment condition. Thus, in the present work, it was assumed that σ_{ss} is similar in all tested heat treatments. In addition, it was adopted the σ_{ss} strengthening effect (181 MPa) estimated by Sui et al. [256] due to the similarity between the laser-DED and arc plasma DED, i.e., both processes are classified as DED (ASTM F3187) and exhibit almost similar solidification conditions ($\sim 2 \cdot 10^2$ and $5 \cdot 10^2$ °C/s, respectively) [67], [434] and microstructure aspects [287]. Therefore, only the precipitation strengthening mechanisms (including order, coherency, and/or Orowan's by-pass) can significantly vary between the proposed heat treatments.

According to Preuss et al. [449] and Goodfellow [450], Orowan's by-pass mechanism is a thermally activated phenomenon associated with the dislocation bowing/climb, which is not typically observed in Ni-based superalloys at room temperature. In addition, Laves and MC-type carbides (eutectics) have a low volume fraction (< 1 %, Figure 4.13), which results in an insignificant material strengthening [235]. Therefore, this effect of eutectics on material strength was not considered, as also indicated by Zhang et al. [138], [222]. Thus, both precipitation (Orowan's by-pass) and dispersion of secondary phase strengthening mechanisms can be considered negligible in the present work.

The order and coherency strengthening mechanisms of the hardening phases (γ' and γ'') depend on their respective particle size and volume fractions, which are determined by the heat treatment condition [451]. Chaturvedi and Han [241] highlighted that the combination of γ' order strengthening and γ'' coherency strengthening mechanisms showed the best agreement between the calculated and measured critical resolved shear stress. Zhang et al. [222] calculated the critical precipitate size for γ' (27 nm, ordering mechanism) and γ'' (17 nm, coherency mechanism) and Sui et al. [256] (Inconel[®] 718 DED) showed that the γ' order (17.4 nm) and γ'' coherency (15 nm) strengthening mechanisms significantly contribute to the material strength. Thus, in the present work, the γ' order ($\sigma_{o-\gamma'}$; Equation 4.1) and γ'' coherency ($\sigma_{c-\gamma''}$; Equation 4.2) strengthening mechanisms [452], [453] of the hardening phases were considered to compute the IN718 AP-DED heat-treated strength.

The combined contributions of the aforementioned strengthening mechanisms (σ_y), i.e., Peierls–Nabarro stress (σ_0), solid-solution (σ_{ss}), dislocation density (σ_d), grain boundary (σ_{gb}), order ($\sigma_{o-\gamma'}$), and coherency ($\sigma_{c-\gamma''}$), were numerically expressed by Equation 4.3 [454]. Table 4.5 shows the constants used to compute the material strength (Equations 4.1 – 4.3). The hardening phase volume fraction (f) was previously estimated Table 4.3. For details on the data and constants estimation methodology, see the Supplementary material. Table 4.4 depict the contribution of each strengthening mechanisms and compare the computed value with the yield strength experimentally measured in horizontal direction, showing a good agreement with a maximum relative error of 4.33%. The semi-quantitative analysis of the strengthening mechanisms supports the estimated γ' and γ'' volume fraction (Table 4.3) since, due to the main contribution of $\sigma_{o-\gamma'}$ and $\sigma_{c-\gamma''}$ for σ_y , the non-compliance in γ' and γ'' volume fraction would result in significant discrepancies between the estimated and measured values (Table 4.4).

$$\sigma_{o-\gamma'} = M \frac{\gamma'_{APB}}{2b} \left[\left(\frac{8\gamma'_{APB} f R_{\gamma'}}{\pi G b^2} \right)^{0.5} - f \right] \quad (4.1)$$

$$\sigma_{c-\gamma''} = 1.7 G M \delta_{\gamma''}^{1.5} \left(\frac{R_{\gamma''} f}{2b A^2} \right)^{0.5} \quad (4.2)$$

$$\sigma_y = \sigma_0 + \sqrt[k]{\sigma_{gb}^k + \sigma_d^k + \sigma_{ss}^k + \sigma_{o-\gamma'}^k + \sigma_{c-\gamma''}^k} \quad (4.3)$$

Table 4.4 – Strengthening mechanisms contributions for IN718 AP-DED strength.

	1050 °C	1100 °C	1142 °C	1185 °C
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γ' order, $\sigma_{o-\gamma'}$ [MPa]	134.60	127.84	145.29	149.57
γ'' Coherency, $\sigma_{c-\gamma''}$ [MPa]	770.34	725.79	637.59	689.63
Grain boundary, σ_{gb} [MPa]	35.71	62.43	66.56	69.21
Dislocation density, σ_d [MPa]	54.22	54.22	54.22	54.22
Solid-solution [256], σ_{ss} [MPa]	181.00	181.00	181.00	181.00
Intrinsic strength, σ_0 [MPa]	44.85	44.85	44.85	44.85
Estimated yield strength, σ_y [MPa]	1084.47	1054.21	986.40	1040.86
Measured yield strength [MPa]	1110 $\pm$ 7.1	1043 $\pm$ 10.6	995 $\pm$ 8.8	1088 $\pm$ 10.6
Relative error [%]	2.30	1.07	0.86	4.33

Table 4.5 – Physical constants used to estimate the γ'' and γ' strengthening effects. M and b , and $\delta_{\gamma''}$, were computed using EBSD and SXRD data, respectively. See Supplementary material sections for more details.

Taylor factor, M	2.56
Antiphase boundary energy, γ'_{APB}	12 mJ/m ² [452]
Burgers vector, b	0.252 nm
Average length of γ'' phase, $R_{\gamma''}$	17.4 nm [256]
Average length of γ' phase, $R_{\gamma'}$	15 nm [445]
Aspect ratio of γ'' phase, A	3.53 [256]
Shear modulus, G	65.9 GPa [446]
γ'' lattice misfit, $\delta_{\gamma''}$	0.02364
Exponent, k	1.14 [256], [455]

Considering the estimated content of hardening phases obtained *via* Rietveld refinement (Table 4.3) and the abovementioned semi-quantitative analysis (Equations 4.1 – 4.3), it is expected that the conditions with a higher hardening phases content (especially γ'') would have a higher yield strength. This trend can be observed when comparing, for instance, the yield strength in the horizontal direction and γ'' content (Table 4.3). However, this tendency fails for 1185 °C / 2 h + aging condition, which exhibited slightly higher strength than the 1100 °C / 2 h + aging condition. This can be explained by the higher γ' content, which counterbalances the lower γ'' content of 1185 °C / 2 h + aging in relation to 1100 °C / 2 h + aging. It is worth noting that γ' has a higher relative strengthening effect ($\sim$ 48 MPa) compared to γ'' (43 MPa) [222], [256].

For the 1185 °C / 2 h + aging, the discrepancy observed between the computed and measured σ_y can be related especially to the adopted average length of γ' phase ($R_{\gamma'}$, 15 nm) since this condition showed the higher γ' content (Table 4.3). The selected $R_{\gamma'}$ was based on the Slama et al. [445] results and was previously used by Sui et al. [256]. However, due to the different initial microstructure conditions (wrought vs. IN718 AP-DED), $R_{\gamma'}$ can show a subtle difference. In addition, given that $\sigma_{o-\gamma'}$ is proportional to $R_{\gamma'}^{0.5}$ (Equation 4.1), a small variation

of $R_{\gamma'}$, will significantly alter $\sigma_{o-\gamma'}$. From a literature survey of fusion-based AM processes [222], [256], [456], [457], $R_{\gamma'}$, can range from 10 to 24 nm, which can explain the observed discrepancies between the estimated σ_y and measured yield strength. Finally, despite this slight difference between the estimated and measured σ_y , the semi-quantitative analysis of the strengthening mechanisms for IN718 AP-DED proved to be accurate (maximum error – 4.33%) and representative, following the same tendency observed by experimental results. This analysis showed that the presented high strength results were related to the higher volume fraction of γ'' and γ' generated during the proposed heat treatments, which induced a hardening phases content similar to PBF, laser-DED and forging [222], [256], [307], [309], [444].

Tensile tests performed in the vertical direction (almost parallel to dendritic growth directions) tend to have a higher yield strength than the horizontal ones (Figure 4.18). These results contradict the dependence on the measurement direction of the grain size, i.e., anisotropic grain size aspect. The “vertically measured” grains had a higher grain size than the “horizontally measured” grain size for IN718 AP-DED due to the columnar grain aspect (Supplementary material) [105], [138], [159]. Thus, it is expected that the “finer grain size” direction (horizontal), *via* the Hall-Petch relation, would have a higher strength, which is not experimentally verified. In addition, the intrinsic stress, precipitation, and dislocation density strengthening mechanisms are also direction-dependent due to the Taylor factor (M) variable in the model description [452], [458]. Therefore, the IN718 AP-DED strengthening mechanisms can be correlated to the crystallographic texture, where the cube texture, $\{100\}\langle 100 \rangle$, is appointed as a softer texture component (i.e., lower Taylor factor – 2.4; Supplementary material). Seow et al. [25], [316], Ni et al. [459], and Liang et al. [460] results demonstrated that the crystallographic texture aspects in cross-sections transversal to the building direction (i.e., vertical specimens loading area) is inferior to horizontal specimens, despite still having a cube texture aspect, reinforcing the dependence of the precipitation and dislocation density strengthening mechanisms in relation to specimen’s direction for texturized materials. Therefore, for the same heat treatment condition, the vertical specimens tend to have an equal or higher yield strength than the horizontal specimens due to the balance between the higher “grain size” (reduce the grain boundary strengthening) and higher Taylor factor (enhance the precipitation and dislocation density strengthening). Additionally, Hessel and Carstensen [461] and Gokcekaya et al. [462] reported that the maximum Schmid factor (0.408) for the $\{111\}\langle 110 \rangle$ slip system in Ni-based superalloys additively manufactured with cube texture aspects (i.e., $\langle 100 \rangle$ direction almost parallel to loading) will theoretically be similar between

the horizontal and vertical directions. This reinforces that the observed strength difference between the horizontal and vertical directions can be attributed to variations in the Taylor factor for each direction, as stated by Zhang et al. [222].

Given that the grain size and dislocation density are secondary strengthening mechanisms compared to precipitation one for IN718 AP-DED heat-treated (Table 4.4) [214], [276], and considering the low change in the crystallographic aspect between the horizontal and vertical direction observed for IN718 AP-DED [25], [316], the yield strength anisotropy is expected not to be expressive. This observation is in accordance with existing literature results for precipitation-strengthened Ni-based superalloys fabricated *via* DED (AP-DED [414] and laser-DED [426]). Additionally, when applying the Hosford analysis [463] (see the Supplementary Material) and anisotropic index (A_i) developed by Kok et al. [464] (Equation 4.4), where σ_H , σ_V , M_H , M_V , and n represent the yield strength (horizontal and vertical), Taylor factor (horizontal and vertical), and strain hardening exponent (Holloman's equation), respectively, a low difference between M_H and M_V (e.g., coarse columnar grains with cube texture) [460] results in a low A_i . This is further supported by the experimental A_i of 1100 °C / 2 h + aging (0.11), which demonstrated the maximum anisotropy among the tested conditions.

$$A_i = \frac{|\sigma_H - \sigma_V|}{\sigma_V} = \left| \left(\frac{M_H}{M_V} \right)^{n+1} - 1 \right| \approx \left| (n+1) \ln \left(\frac{M_H}{M_V} \right) \right| \quad (4.4)$$

4.1.3.4.3 Fractography

Fractography analysis evidence similarities for all conditions, as exemplified by the fracture surface of the 1100°C / 2 h + aging condition (Figure 4.19a), which display a typical ductile fracture aspect composed predominantly by dimples. According to Xu et al. [255], the volume fraction and morphology of the Laves phase had the main effect on the fracture behavior of the additively manufactured Inconel® 718. The long and aligned Laves phase, commonly observed for as-built IN718 AP-DED (Figure 4.12) [25], [138], [316], tends to fragment and debond at the interface (Laves/y) when loaded, promoting crack nucleation and growth. Otherwise, Laves phase with a granular morphology, as observed after the proposed heat treatments (Figure 4.12), tends not to fragment and promotes microvoids nucleation, growth, and coalescence (dimples generation) instead. This explains the high ductility (Figure 4.18b) observed for all heat treatments with high homogenization temperatures (present work and literature [25], [107], [108], which induces a sensible change in Laves phases content and morphology.

The fracture surface of the vertical specimen shows some shallow and larger dimples (dotted yellow circles, Figure 4.19b). For vertical specimens, the loading direction is aligned with the interdendritic eutectics, which can promote the fracture of the remaining high-sized secondary particles and generate these shallow dimples [256]. The horizontal specimen preserved some dendritic aspect on the fracture surface (Figure 4.19a), which is directly related to the position where secondary phases nucleated. During the horizontal specimen loading, the specimen cross-section is co-planar to the dendritic growth direction. Thus, as the microvoids nucleate from incoherent secondary phases, which are located at interdendritic region, a solidification pattern can be visualized at the fracture surface [21], [25], [109], [316].

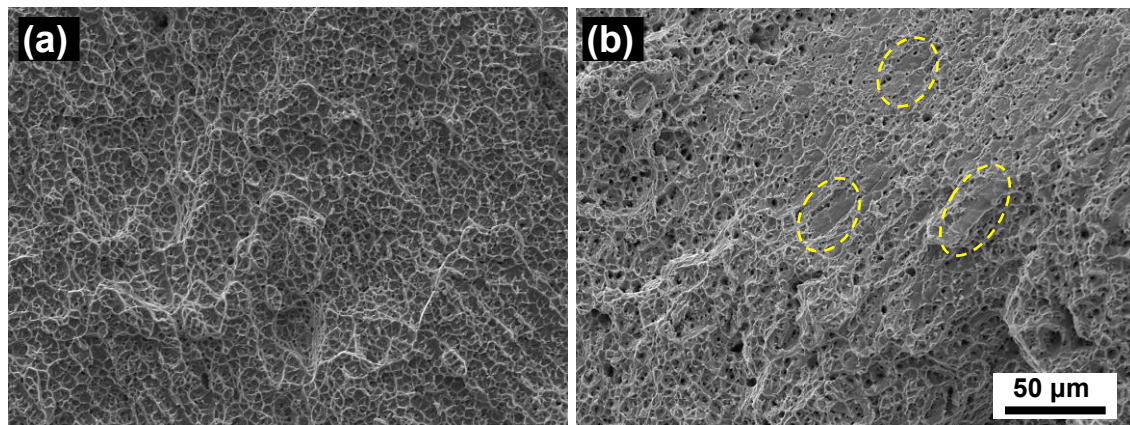


Figure 4.19 – Fractography analysis of the IN718 AP-DED (1100 °C / 2 h + aging): (a) horizontal and (b) vertical specimens.

4.1.3.4.4 Young's modulus

Table 4.7 shows that the present results and those of literature, independently of the heat treatment condition and AM, had Young's modulus significantly lower than wrought material, especially in the vertical direction. In addition, despite the high volume of hardening phases (Table 4.3) and remaining eutectics (Laves and MC-type carbides, Figure 4.12), these phases and the matrix have very similar elastic constants [465]. Thus, the Young's modulus of IN718 AP-DED heat-treated can be directly correlated with the matrix's elastic properties and crystallographic aspects [465]. Equation 4.5 related the Young's modulus (E_{hkl}) with the crystal orientation (hkl) for a cubic material and Equations 4.5 – 4,8 correlated the compliance (S_{ij}) and stiffness (C_{ij} , Table 4.6) material constants [463]. Using these data and Equations 4.5 – 4.8, E_{100} was computed (123 GPa), which showed a similar magnitude to the Young's modulus experimentally measured in the vertical direction for the present work and literature data (Table 4.7). These results reinforces the dependence of the IN718 AP-DED Young's modulus on the crystallography orientation [466]. Therefore, given that the proposed heat treatments did not

alter the typical cube texture aspect (Figure 4.14), it is expected that IN718 AP-DED heat-treated possesses a lower Young's modulus concerning to non-oriented material (Table 4.7).

$$\frac{1}{E_{hkl}} = S_{11} + \frac{(2S_{12} - 2S_{11} + S_{44})(k^2l^2 + k^2h^2 + h^2l^2)}{(h^2 + k^2 + l^2)^2} \quad (4.5)$$

$$S_{11} = \frac{C_{11} + C_{12}}{C_{11}^2 + C_{11}C_{12} - 2C_{12}^2} \quad (4.6)$$

$$S_{12} = \frac{-C_{12}}{C_{11}^2 + C_{11}C_{12} - 2C_{12}^2} \quad (4.7)$$

$$S_{44} = \frac{1}{C_{44}} \quad (4.8)$$

Table 4.6 – Average single crystal elastic stiffness (C_{ij}) constants of Inconel® 718 [467].

Single crystal stiffness constants [GPa]		
C_{11}	C_{12}	C_{44}
243.9 ± 14.4	154.9 ± 21	110.8 ± 5.8

Table 4.7 – Effect of heat treatments on the Young's modulus of IN718 AP-DED.

Reference	Process	Heat treatment	Orientation	E [GPa]
Present work	GMA-DED	1050 °C / 2 h + aging	Horizontal	158 ± 4
			Vertical	119 ± 3
		1100 °C / 2 h + aging	Horizontal	153 ± 6
			Vertical	120 ± 1
		1142 °C / 2 h + aging	Horizontal	158 ± 3
			Vertical	122 ± 2
		1185 °C / 2 h + aging	Horizontal	156 ± 1
			Vertical	122 ± 1
Hill's model			Horizontal	175
			Vertical	136
Kindermann et al. [21]	GMA-DED	AMS 5662	Horizontal	159 ± 2
			Vertical	124 ± 3
		API 6ACRA	Horizontal	152 ± 3
			Vertical	117 ± 2
James et al. [319]	PTA-DED	AMS 5662		178
Kumara et al. [467]	PBF	As-built	Horizontal	169
			Vertical	126
Wrought [21]				207 ± 5

The Young's modulus (Figure 4.20) was evaluated considering the real material crystallography data (Figure 4.14a and Figure 4.15) through Hill's model [468]. The results showed that the build (vertical) and deposition (horizontal) directions had the lowest Young's modulus (Table 4.7), which was directly correlated to the material crystallography texture. Thus, the typical cube texture observed in the Ni-based superalloys fabricated *via* fusion-based additive manufacturing induces a lower Young's modulus compared to the non-oriented microstructure material (e.g., wrought). In addition, the model indicated that the deposition

direction (horizontal specimens) had a higher Young's modulus in relation to the building direction (vertical specimens), which was also experimentally verified for all tested heat treatments. From Hill's stiffness matrix (Figure 4.21), IN718 AP-DED can be classified as a transversely isotropic elastic material, a similar behavior previously reported for non-transformable FCC materials fabricated *via* directional solidification and AM [466], [467], [469], [470], i.e., materials with aligned columnar grains. For a transversely isotropic elastic material loaded parallel to the isotropic plane (horizontal specimen), it is expected that Young's modulus (Table 4.7) ranging between E_{100} (123 GPa) and E_{110} (211 GPa) [466], [467] weighted by the crystallographic distribution, which justifies the anisotropic Young's modulus experimentally verified. Thus, the higher the cube texture component index, the closer to E_{100} the Young's modulus (horizontal specimens) will be.

Figure 4.20b reported the variation of Young's modulus with the build direction, highlighting that the maximum Young's modulus occurred at an orientation of 45 to 55 ° to the build direction. These results suggested that Young's modulus control and design can be performed by changing the build direction orientation or using a previously oriented substrate, allowing dedicated solutions for structural engineering, e.g., topological optimization to match the loading and specific printing orientation [470], since the post-deposition heat treatment did not significantly alter the grain morphology and texture aspect of the as-built material (GMA-DED).

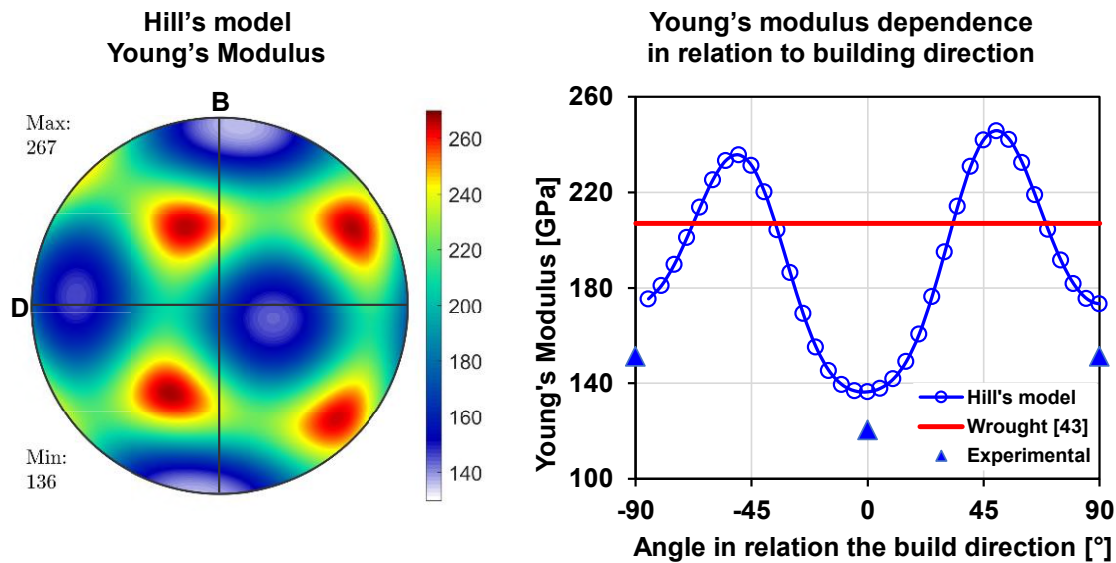


Figure 4.20 – Young's modulus calculation (Hill's model) in function of the material crystallographic orientation (left) and Young's Modulus in relation to the build direction (right). D and B means the deposition and build directions.

Ideal transversely isotropic material
Elastic stiffness matrix

$$C = \begin{pmatrix} C_{11} & C_{12} & C_{13} & 0 & 0 & 0 \\ & C_{22} & C_{23} & 0 & 0 & 0 \\ & & C_{33} & 0 & 0 & 0 \\ & & & C_{44} & 0 & 0 \\ Symm & & & & C_{44} & 0 \\ & & & & & \frac{C_{11} - C_{12}}{2} \end{pmatrix}$$

Inconel® 718 AP-DED
Hill's s elastic stiffness [GPa] matrix

$$C = \begin{pmatrix} 281.09 & 120.88 & 151.69 & 0 & 0 & 0 \\ & 282.95 & 149.83 & 0 & 0 & 0 \\ & & 252.15 & 0 & 0 & 0 \\ & & & 106.6 & 0 & 0 \\ Symm & & & & 105.74 & 0 \\ & & & & & 76.8 \end{pmatrix}$$

Figure 4.21 – Ideal elastic stiffness matrix for a transversely isotropic material and stiffness matrix for Inconel® 718 AP-DED.

4.2 Elevated temperature (650 °C) tensile behavior

The IN718 AP-DED exhibited an almost similar response to the proposed PDHTs, which can be correlated with similarities in microstructure and texture aspects and final effect on precipitation strengthening mechanism (Table 4.4). Additionally, for the first time, it has been demonstrated that IN718 AP-DED can meet the AMS 5662 requirements at elevated temperature (Figure 4.22b) using the typical process route (3D-printing + PDHT). The tested conditions (vertical) met the ultimate tensile strength (UTS), yield strength (YS_{0.2%}), and elongation requirements. Compared to Zhang et al. [109] (AMS 5662) and Seow et al. [25] (1185 °C/ 40 min + aging), the proposed PDHTs (Figure 4.22) showed a significant improvement in elongation, YS_{0.2%}, and UTS, which can be related to the insufficient eutectic dissolution (low-temperature PDHT [109]) and undesirable secondary grain growth [25]

observed by these authors, respectively. Relative to laser-DED [427] and PBF [428] (high-temperature PDHTs), the present results exhibited almost similar strength performance in vertical direction and lower in horizontal direction, which can be associated to the inefficiency of the heat treatments in alter the texture and grain morphology aspects of IN718 fabricated *via* AP-DED (gas metal arc-based – GMA-DED) [67], [107], [108], [223], [471]. However, for low temperature heat-treated PBF (AMS 5662) [329], [332], the present results were similar to them. This finding suggests that the homogenization PDHT soaking time and peak temperatures were correctly designed for IN718 fabricated *via* GMA-DED. According to the literature survey [21], [97], [98], [105]–[112], longer soaking time can be applied to IN718 fabricated *via* GMA-DED. This material did not show undesirable secondary grain growth, unlike what was observed for IN718 fabricated *via* AP-DED (plasma transferred arc) [25], laser-DED [441], and PBF [472], [473]. Thus, conditions recommended for hot isostatic pressing (1100 – 1185 °C / 6 h) and ingots homogenization (1150 – 1190 °C / 20 h) [474], [475] may also be adopted for IN718 fabricated *via* GMA-DED.

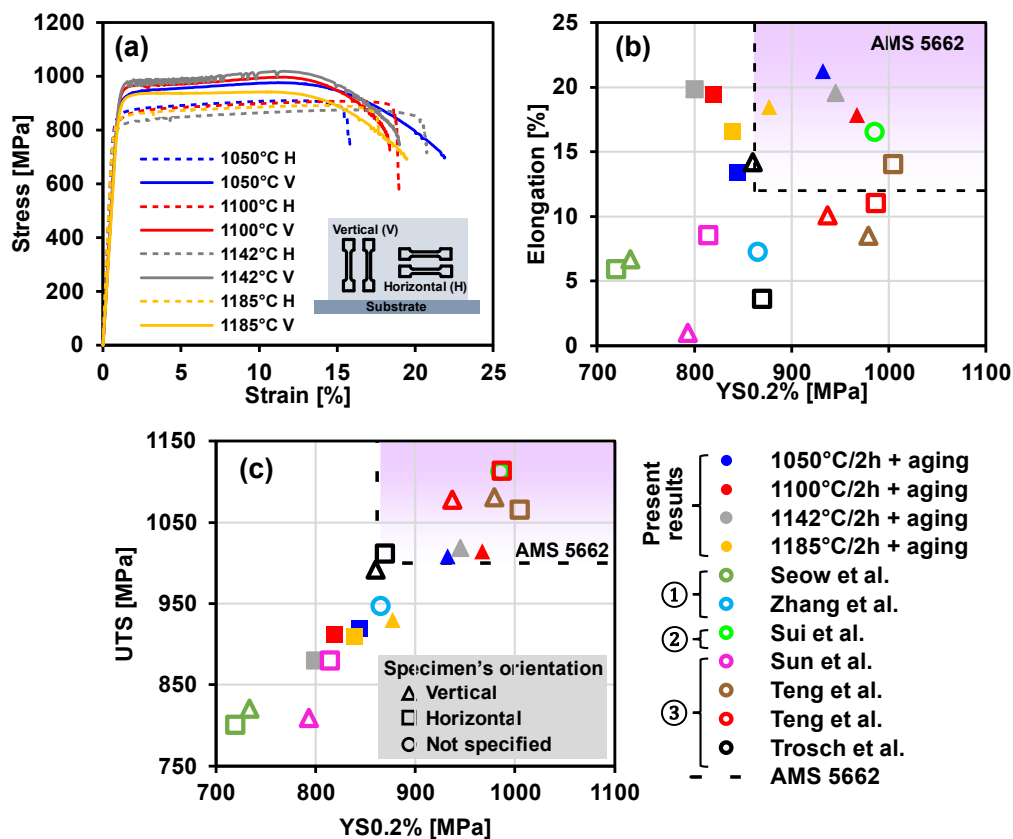


Figure 4.22 – IN718 AP-DED response to post-deposition heat treatments: (a) tensile test at elevated temperature and (b – c) comparison with the literature. The references are Seow et al. [25], Zhang et al. [109], Sui et al. [427], Sun et al. [332], Teng et al. [428], and Trosch et al. [329]. ①, ②, and ③ refer to AP-DED, laser-DED, and PBF, respectively.

The vertical specimens outperformed the horizontal ones for all tested conditions. At elevated temperatures, the grain boundaries become weaker in relation to the grain core [476]. Thus, considering that horizontal specimens were positioned transversely to columnar grains, they would have a higher grain boundary area compared to vertical specimens [159], [413], resulting in lower material strength. In addition, solid-solution [448], dislocation density [244], and precipitation [222] strengthening mechanisms are linearly correlated to the Taylor factor (M), *i.e.*, IN718 AP-DED strengthening mechanisms are direction-dependent [239], [256]. Consequently, they can be linked with the crystallographic texture (Figure 4.14), where cube texturized grains induce a lower M (~ 2.4 ; Supplemental material) in relation to a non-oriented microstructure (3.05 – 3.1) [239], [463], [477], [478]. Furthermore, based on previous works [25], [222], [316], [460], [467], [470], [479], the cross-section transverse to the building direction (vertical specimens) exhibit a slightly inferior cube texture index (slightly higher M) compared to cross-sections parallel to the building direction (horizontal specimens), which also promoted an anisotropic behavior. Additionally, Gokcekaya et al. [462] estimated, for a cube texturized material (IN718 fabricated *via* PBF), that the majority of the grains had an Schmid factor (0.408) similar in both horizontal and vertical directions, *i.e.*, macroscopically there is not a preferential direction that can easily active some slip system [463]. However, when the columnar grains were loaded along their principal axes (vertical direction), they experienced an almost isodeformation state from grain to grain. Consequently, due to the volumetric cube texture aspect, slip can occur simultaneously in most of the $\langle 001 \rangle$ oriented-grains. Otherwise, when columnar grains were loaded transversely to their main axes (horizontal direction), they underwent an isostress state, and the deformation could concentrate in a small number of grains with Schmid factor higher than that observed for cube-texturized columnar grains, resulting in lower stress for yielding [459], [480]. These findings support the correlation between observed anisotropy behavior of IN718 AP-DED and its dependence on M (texture aspects) and grain morphology (columnar) [464], [481]–[483], explaining the observed anisotropy at elevated temperature and the challenge to meet the wrought material (non-oriented) properties.

Figure 4.22a also shows the dynamic strain aging (DSA) phenomenon, which is characterized by the serrated aspect in the strain-stress curve [484]. According to Nalawade et al. [485], at 650 °C both interstitial and substitutional elements diffuse to generate Cottrell's atmospheres around dislocations in motion [486]–[488], which can cause consecutive dislocation locking and unlocking cycles, driving the serrated aspect. Beese et al. [489] did not observe DSA for Inconel® 625 fabricated *via* laser-DED. These authors described that the cube

texture and coarse microstructure resulted in positive strain-rate sensitivity ($\dot{\gamma}$) [490] and lower work-hardening rate ($\dot{n}$) so that the critical strain necessities to promote the DSA was not achieved. Otherwise, Banait et al. [491], [492] (IN718 lattices fabricated *via* PBF) observed a negative $\dot{\gamma}$ (occurrence of DSA) for all tested strain rates and temperatures, which was associated with the fine and almost non-oriented microstructure, reinforcing the correlation between the DSA and the crystallographic texture aspects in additively manufactured Ni-based superalloys. Additionally, Hayes [493] highlighted the interaction between the dislocation and interstitial elements (fast diffusion – C). If MC-type carbides remove C (promoting a sink effect) from the dislocation in motion faster than the Cottrell's atmosphere formation, DSA will not occur (positive $\dot{\gamma}$). Thus, given that the MC-type carbides are stable (Figure 4.9) and disperse intergranularly (Figure 4.12), the sink effect will be similar among the heat-treat conditions. Also, despite heavy elements (*e.g.*, Nb and Mo) can also forming Cottrell's atmosphere, its efficiency is secondary in relation to C due to the lower diffusion coefficient [485]. Therefore, the lower work-hardening (texture and coarse grain) and presence of high content of MC-type carbides (solidification sequence and inefficient of heat treatment to dissolve it) can explain the difficulty of observing DSA for IN718 AP-DED (GMA). Regarding the present work, DSA is observed (Figure 4.22a) only in the stronger and less oriented direction (vertical), particularly for higher strength conditions (1142 C / 2 h + aging). For the softer direction (horizontal), it is believed that the critical strain for a negative $\dot{\gamma}$ was not achieved due to the lower $\dot{n}$ typically observed in coarse and oriented microstructures [21], [106], [489], [494], [495].

The DSA occurrence in the present work differ from Seow et al. [25] (IN718 fabricated *via* plasma transferred arc DED). These authors observed expressive serration (DSA type C) in both directions, which can be related to the almost absence of cube texture (higher $\dot{n}$) after the heat treatment (recrystallization). Otherwise, Zhang et al.[109] did not verify DSA for IN718 AP-DED, which was associated with the insignificant effect of the AMS 5662 PDHT on primary microstructure (coarse and oriented microstructure) and limited PDHT response (low material strength). Therefore, the occurrence of DSA in IN718 AP-DED shows ambiguous results, which can be related to its process and PDHT dependence. Furthermore, the DSA was correlated with crystallographic texture and microstructure aspects, where a coarser and cube-textured microstructure showed less susceptibility to DSA [25], [109], [491], [492], as stated by Beese's et al. model.

Figure 4.23 exemplifies the fractography of the heat-treated IN718 AP-DED (1100 °C / 2 h + aging), which had a transgranular ductile aspect with fracture surface majority

composed of shallow and almost equiaxed dimples [257]. The horizontal specimen showed a dimple 'dendrite' pattern, attributed to the remaining interdendritic incoherent secondary phases that drove microvoids nucleation (Figure 4.23a.1). These microvoids subsequently grew and coalesce during loading, resulting in a dimple 'dendrite' pattern on fracture surface [25], [109], [332], [427], [496]. Furthermore, it is worth mentioning that, although DSA was associated with embrittlement phenomena [331], [487], [497]–[499], the present work did not evidence brittle fracture aspects. In summary, all conditions had a transgranular ductile fracture and met the elongation requirement (Figure 4.22b).

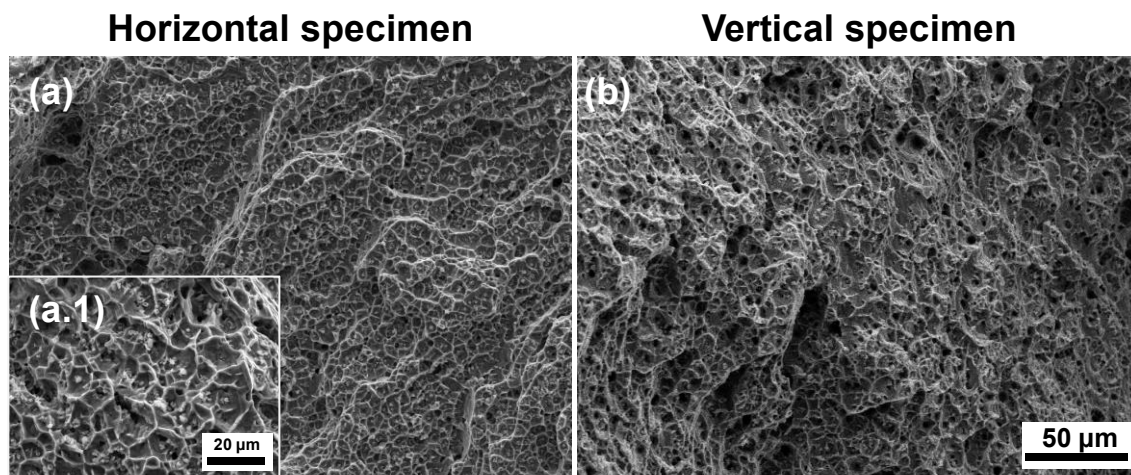


Figure 4.23 – Fractography analysis of IN718 AP-DED.

4.3 Summary

The present work comprehensively evaluated the effect of newly developed heat treatments on the microstructure and mechanical properties of the Inconel® 718 fabricated *via* arc plasma DED. The following conclusions can be draw:

- The proposed heat treatments resulted in superior mechanical response. For the first time, IN718 AP-DED met the AMS 5662 quasi-static mechanical properties requirements at room temperature through the typical manufacturing route, i.e., additive manufacturing + heat treatment.
- Independently of the homogenization temperature, IN718 AP-DED showed the typical coarse aligned-columnar grain morphology, i.e., the cubic texture showed by the IN718 AP-DED (gas metal arc-based) was not altered by the selected post-deposition heat treatments.

- The IN718 AP-DED Young's modulus showed a dependence in relation to the build direction and microstructure orientation. The horizontal and vertical specimens showed a lower Young's modulus in relation to wrought material.
- The proposed heat treatments can induce a high content of hardening phases (γ'' and γ'). 1050 and 1100 °C / 2 h + aging heat treatments showed the higher volume of γ'' (19.59 and 17.39 %), respectively.
- The proposed PDHTs enable the IN-718 AP-DED to meet, for the first time, the AMS 5662 requirements at elevated temperature (650 °C). The dynamic strain aging phenomena was observed, however without significant effect on the heat treatment response and fracture mode.
- The present work demonstrated that the heat treatments designed for IN-718 AP-DED may differ from those commonly employed for cast (AMS 5383), wrought (AMS 5662), and PBF (ASTM F3055) materials due to its unique microstructure.

4.4 Supplementary Material

4.4.1 Image segmentation for quantitative metallography

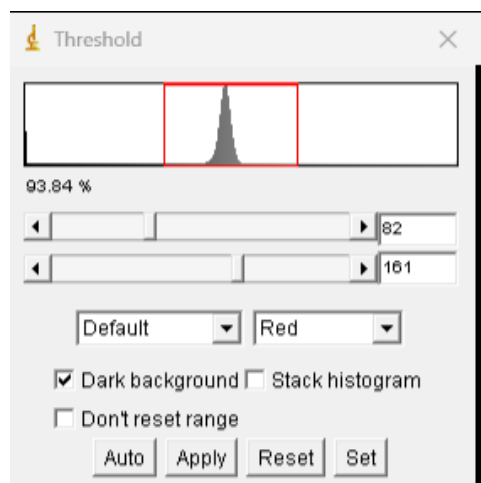
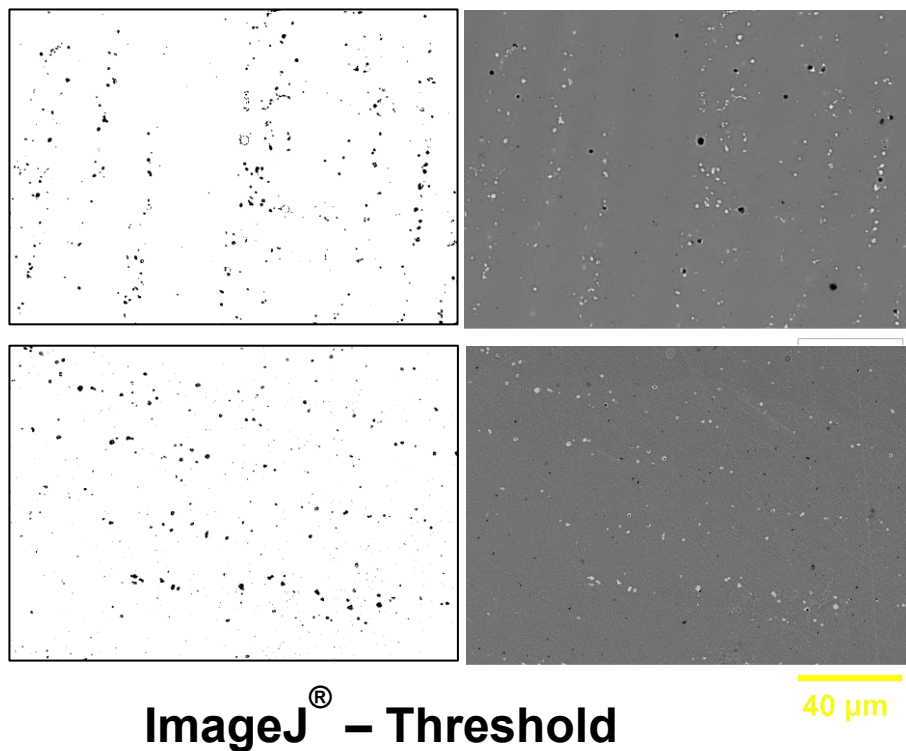


Figure 4.24 -SEM image segmentation using ImageJ[®] software.

4.4.2 As-built chemical composition

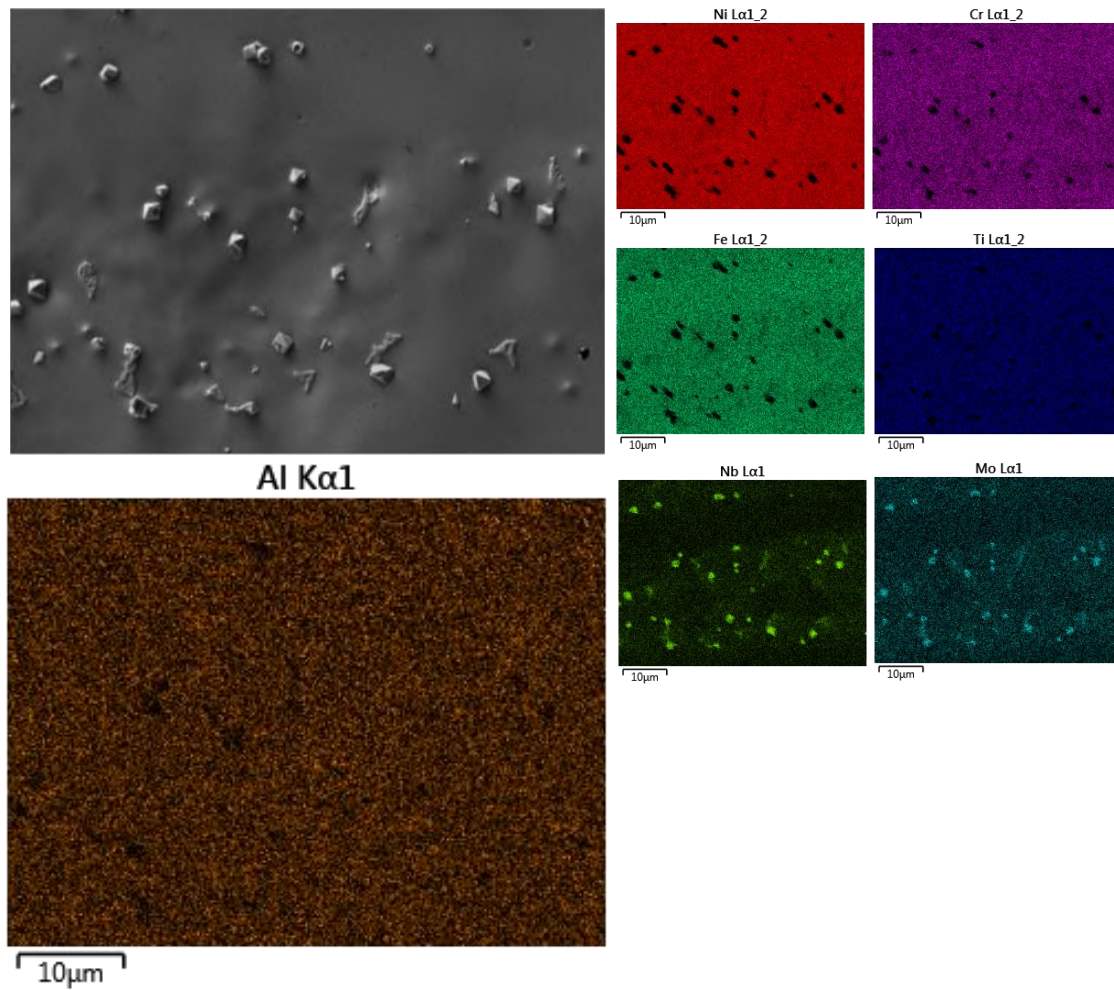


Figure 4.25 -EDS mapping.

4.4.3 Rietveld refinement and hardening phases volume fraction estimation

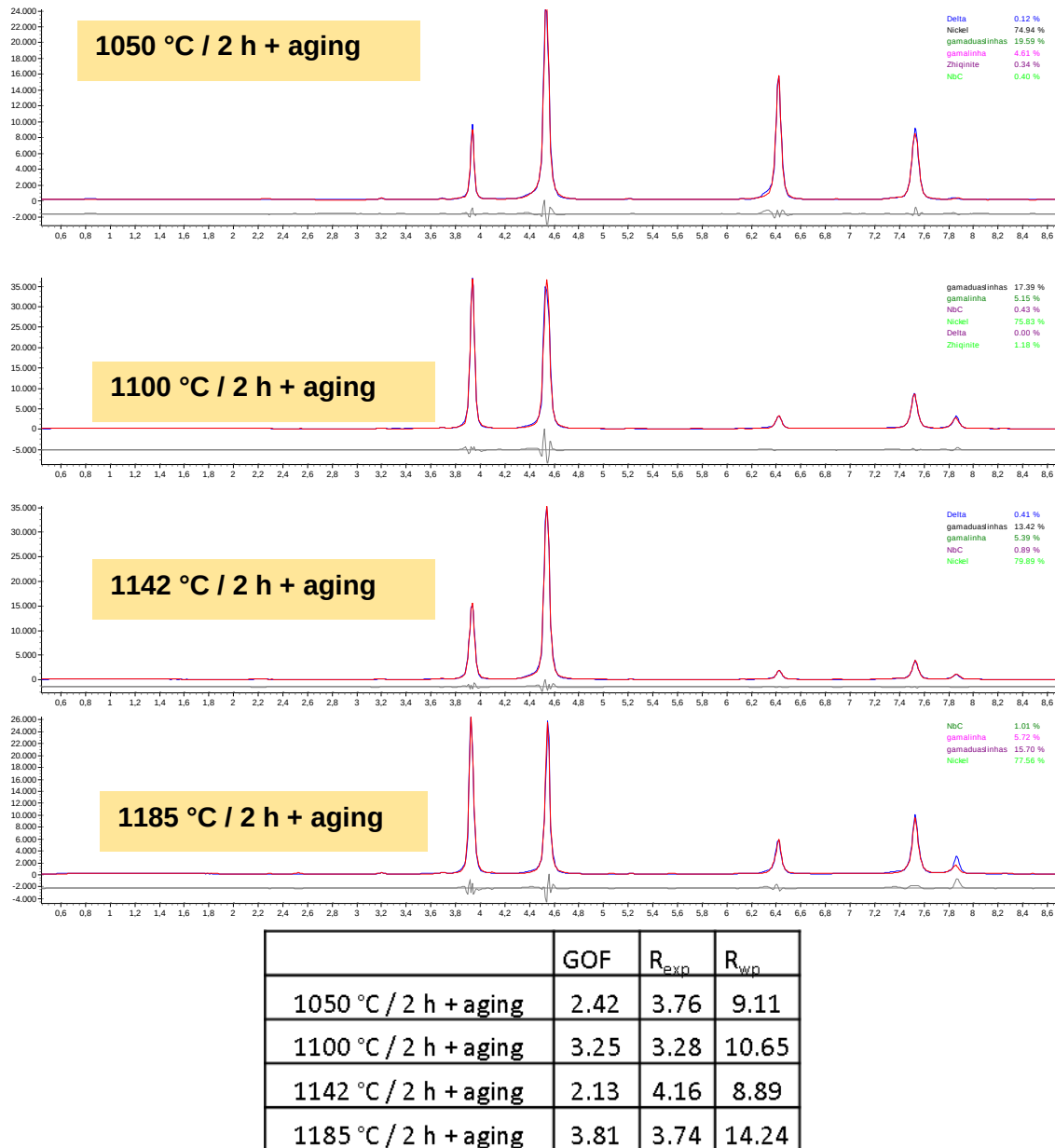


Figure 4.26 - Rietveld refinement and its statistical parameters. GOF, R_{exp}, and R_{wp} are the good-for-fitness, expected-profile quality, and weighted-profile quality, respectively.

4.4.4 Hosford [463] analysis for anisotropy in tensile behavior

Considering the typical Holloman's equation (power law) for a shear deformation ($\tau = K\gamma^n$), the Taylor factor definition ($M = \sum \frac{d\gamma}{d\varepsilon}$), and the relation between the shear stress and mean stress ($\sigma = M\tau$; uniaxial tensile test), the mean stress can be correlated with the Taylor

factor, such as indicated by Equation 4.5. In addition, adopting the anisotropic factor (A_i) described by Kok et al. [464] (Equation 4.4), A_i can be estimated as following the Equation 4.6. σ_H , σ_V , n , and K are the yield strength in the horizontal and vertical directions, the work hardening coefficient, and the proportionality constant, respectively.

$$\sigma = M^{n+1} K \varepsilon^n \quad (4.5)$$

$$A_i = \frac{|\sigma_H - \sigma_V|}{\sigma_V}$$

$$A_i = \frac{|M_H^{n+1} K \varepsilon^n - M_V^{n+1} K \varepsilon^n|}{M_V^{n+1} K \varepsilon^n} \quad (4.6)$$

$$A_i = \frac{|M_H^{n+1} - M_V^{n+1}|}{M_V^{n+1}}$$

$$A_i = \left| \left(\frac{M_H}{M_V} \right)^{n+1} - 1 \right| \approx \left| (1+n) \ln \left(\frac{M_H}{M_V} \right) \right|$$

4.4.5 Taylor Factor

The Taylor factor (Figure 4.27) was estimated through a weighted average, which account each grain size area (A_i) and its corresponding Taylor factor (M_i), according to Equation 4.7.

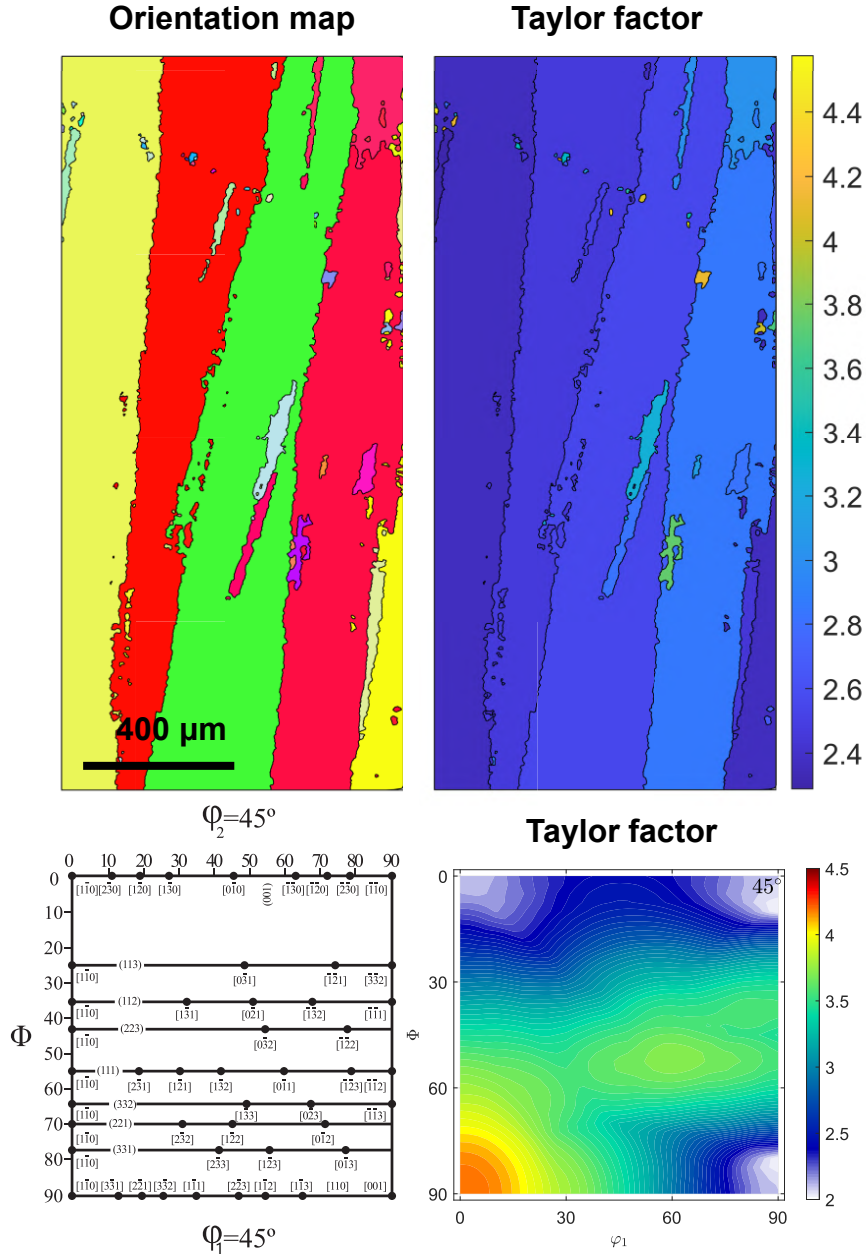


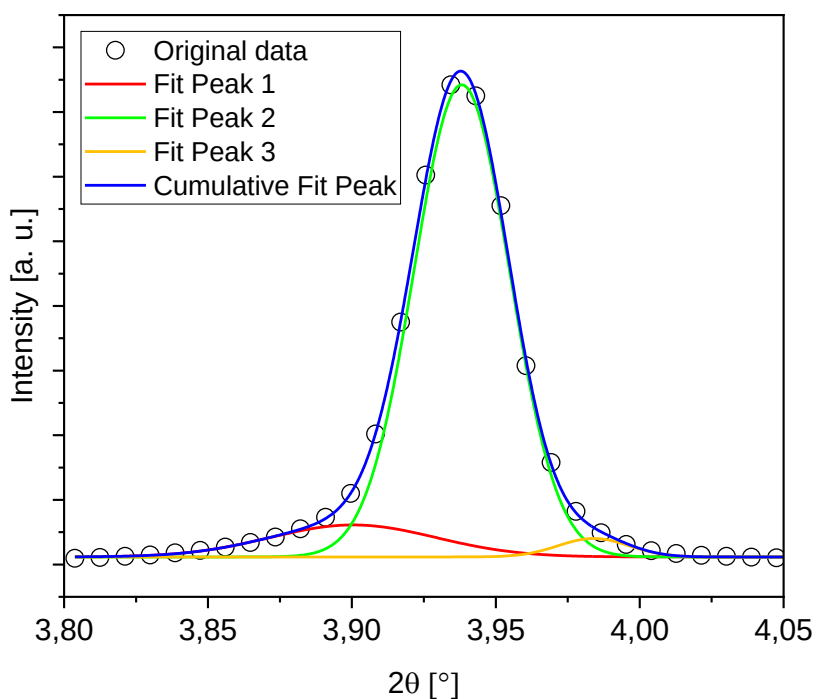
Figure 4.27 - Orientation map and its respective Taylor factor map. These data were based on the Figure 4.14.

$$M = \left(\sum_{i=1}^n M_i A_i \right) \cdot \left(\sum_{i=1}^n A_i \right)^{-1} \approx 2.56 \quad (4.7)$$

4.4.6 Burgers vector

The peak $\gamma(111)$ was fitted using a Gaussian function to calculate θ (Bragg's diffraction angle). Subsequently, Bragg's equation was applied to estimate the interplanar basal distance

(d_{111}). Additionally, a relationship was established between d_{111} and the lattice parameters (a), enabling the computation of the Burgers vector (b).

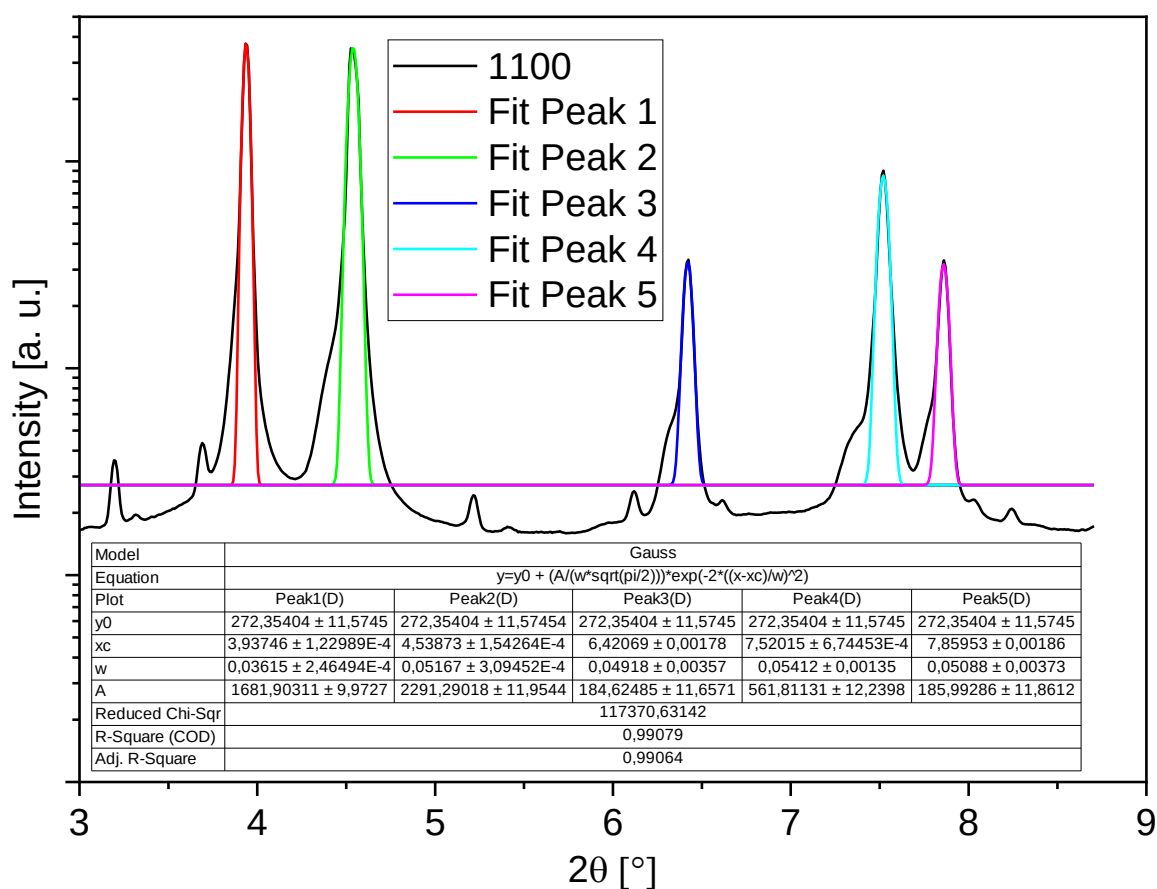


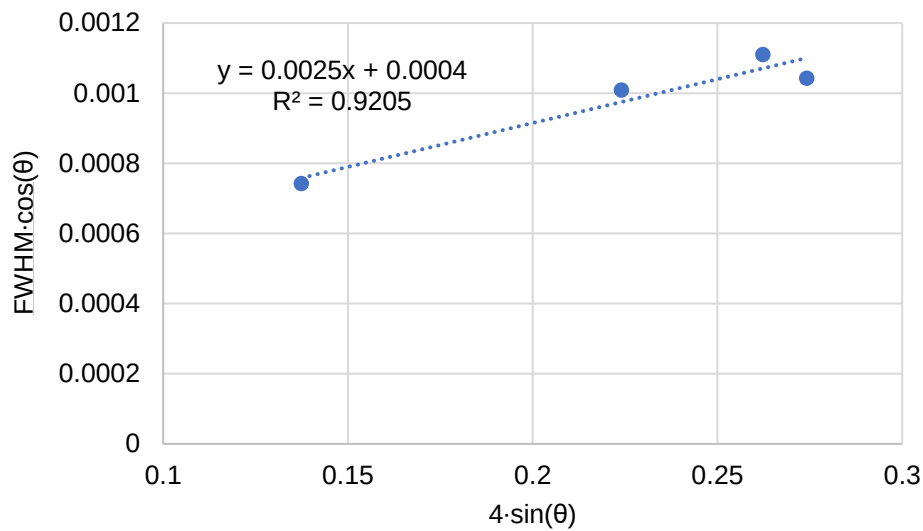
	$\lambda = 2d_{hkl} \cdot \sin \theta$	
	$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}}, (hkl) = (111)$	
	$b = \frac{a}{\sqrt{2}} \approx 0.251737 \text{ nm}$	

Model	Gauss		
Equation	$y=y0 + (A/(w*\sqrt{\pi/2}))*\exp(-2*((x-xc)/w)^2)$		
Plot	Peak1(B)	Peak2(B)	Peak3(B)
y0	$587,3027 \pm 79,6393$	$587,3027 \pm 79,63936$	$587,3027 \pm 79,63936$
xc	$3,90041 \pm 0,01444$	$3,93817 \pm 2,04927\text{E-}4$	$3,98399 \pm 0,00245$
w	$0,05761 \pm 0,01618$	$0,0332 \pm 6,01198\text{E-}4$	$0,02466 \pm 0,00494$
A	$178,64291 \pm 85,790$	$1519,57014 \pm 80,043$	$44,11407 \pm 9,16233$
Reduced Chi-Sqr	45956,9188		
R-square (COD)	0,99975		
Adj. R-Square	0,99963		

4.4.7 Dislocation density

The dislocation density was estimated using the Williamson and Hall and Smallman and Westmacott relations, which are expressed below. Initially, the peak positions of the γ matrix were fitted using a Gaussian function, as depicted in the Figure. Subsequently, based on the fitted data, the Full Width at Half Maximum (FWHM) and θ (Bragg's diffraction angle) were calculated, allowing the estimation of dislocation density for each condition. Following that, the dislocation density strengthening mechanism was determined using Bailey-Hirsh's relation ($BMbG\rho_s^{0.5}$).





$$\text{FWHM} \cdot \cos \theta = \frac{\alpha \lambda}{D_c} + 4 \cdot \sin \theta \cdot \varepsilon$$

$$\sigma_b = \text{BMbG}\rho_s^{0.5}$$

$$B = 0.2$$

$$G = \text{Shear modulus } (65.9 \cdot 10^{10} \text{ Pa})$$

$$M = \text{Taylor factor } (2.56)$$

$$\rho_s = \frac{16.1 \cdot \varepsilon^2}{b^2}$$

θ – diffraction angle

$$b = 0.251737 \text{ nm}$$

α – Scherrer factor (0.9)

$$\lambda = \text{Wavelength } (0.14235 \text{ \AA})$$

D_c – Cristalite size

$$\rho_s = \frac{16.1 \cdot \varepsilon^2}{b^2} = \frac{16.1 \cdot 0.0004^2}{0.251737^2} \approx 4.065 \cdot 10^{13} \text{ m}^{-2}$$

4.4.8 Figure 4.18 data

Reference	Process	Heat treatment	Orientation	E* [GPa]		YS _{0.2%} [MPa]		UTS [MPa]		D [%]	
				μ	σ_μ	μ	σ_μ	μ	σ_μ	μ	σ_μ
Present work	GMA-DED	1050 °C / 2 h + aging	Horizontal	158	3.5	1110	7.1	1404	32.5	21.4	1.9
			Vertical	119	2.8	1100	14.1	1460	20.5	26.2	1.8
		1100 °C / 2 h + aging	Horizontal	153	6.4	1043	10.6	1433	11.3	27.5	2.0
			Vertical	120	1.4	1173	24.7	1405	55.2	24.4	1.0
		1142 °C / 2 h + aging	Horizontal	158	3.1	995	8.8	1450	22.6	28.7	1.7
			Vertical	122	2.1	1083	38.9	1450	2.1	25.9	3.3
Xu et al. [138]	GMA-DED + cold rolling	AMS 5662	Horizontal			790	9	1102	78	14.7	1.3
			Vertical			791	14	988	6	12.8	1.2
						1035	20	1351	11	17.4	
Kindermann et al. [21]	GMA-DED	AMS 5662	Horizontal	159	2	910	2	1185	80	8.8	3.4
			Vertical	124	3	1025	9	1246	117	6.2	4.5
		API 6ACRA	Horizontal	152	3	780	6	1304	41	22.8	3.2
			Vertical	117	2	842	4	1229	136	12.7	5
Xu et al. [105]	GMA-DED	AMS 5662	Horizontal			807	1	1110	3	15	0.3
			Vertical			899	5	1233	16	19.4	2.8
Xi et al. [107]	GMA-DED	1185 °C / 1 h + aging	Horizontal			1070.14	9.3	1249.32	30	20.09	3
			Vertical			1071.17	11.3	1251.16	27	21.43	3.9
Xi et al. [108]	GMA-DED	1150 °C / 1 h + aging				1069.38	8.3	1269.62	1.11	30.27	1.38

Zhang et al. [109]– [112]	GMA-DED + cold rolling	AMS 5662			1085	23.3	1341.5	20.0	15.7	1.1
	GMA-DED				865	32	946.6	43.2	7.16	0.05
Chen et al. [200]	GMA-DED + hot rolling	AMS 5662			1112		1304		21.1	
	GMA-DED				1019.35		1110.74		9.8	
Seow et al. [25]	PTA-DED	AMS 5383	Horizontal		1065		1116		0.9	
			Vertical		951		1072		2	
		1185 °C / 40 min + aging			856		1044		19.9	
Wang et al. [301]	PTA-DED	AMS 5662			864	24	1152	28	23	2
James et al. [319]	PTA-DED	AMS 5662		178	852		870			
AMS 5662					1034		1276		12	
AMS 5383				207 ± 5 [21]	724		827		3	
API 6ACRA					965		1103		20	
ASTM F3055					940		1240		12	
*E, YS, UTS, D, μ , and σ_μ are the Young's modules, yield strength, ultimate tensile strength, ductility, average value, and error standard, respectively.										

4.4.9 Figure 4.22b data

		Heat treatment	Direction	YS _{0.2%} [MPa]		UTS [MPa]		Elongation [%]	
	Process			μ	σ_{μ}	μ	σ_{μ}	μ	σ_{μ}
Present results	Arc plasma-DED	1050°C/2h + aging	Horizontal	845	7.1	1018.5	21.9	14.00	2.6
			Vertical	932.5	24.7	1124	35.4	21.9	0.1
		1100°C/2h + aging	Horizontal	820	0.0	1061	2.8	20.0	1.4
			Vertical	967.5	24.7	1126.5	10.6	18.7	0.4
		1142°C/2h + aging	Horizontal	800	0.0	1016	22.6	20.4	0.4
			Vertical	945	35.4	1132.5	16.3	20.3	1.9
		1185°C/2h + aging	Horizontal	840	14.1	1036	8.5	17.2	0.9
			Vertical	877.5	31.8	1026.5	38.9	19.1	0.5
Seow et al.	Arc plasma-DED	1185°C/ 40 min + aging	Horizontal	734	18.0	821	2.0	5.9	3
			Vertical	720	21.0	800	51.0	6.7	3
Zhang et al	Arc plasma-DED	AMS 5662		865.7	32.0	946.6	43.0	7.2	0.05
Sun et al.	EB-PBF	AMS 5662	Horizontal	815	27.0	879	27.0	8.5	3.1
			Vertical	793	5.0	809	14.0	1	0.5
Sui et al	Laser-DED	1045 °C / 45 min h + aging	Vertical	986	15.0	1112	9.0	16.5	1
Teng et al.	Laser-PBF	AMS 5383	Horizontal	1005	16.0	1065	11.0	14	1
			Vertical	979	22.0	1081	14.0	8.5	3
		AMS 5662	Horizontal	987	10.0	1113	13.0	11	3
			Vertical	937	21.0	1078	15.0	10.1	2
Trosch et al.	Laser-PBF	AMS 5662	Horizontal	870		1011		3.6	
			Vertical	860		992		14.2	
AMS 5662 requirements				862		1000		12	
YS, UTS, μ , and σ_{μ} refer to yielding strength, ultimate tensile strength, average, and standard error, respectively.									

5 IN SITU INTERLAYER HOT FORGING ARC-BASED DIRECTED ENERGY DEPOSITION OF INCONEL 718

This study analyzes the effect of in situ interlayer hot forging (HF) on the grain size refinement of the Inconel 718 fabricated via arc-based directed energy deposition (IN718 DED). The grain size refinement mechanism was investigated using electron backscatter diffraction and synchrotron X-ray diffraction. HF induced a dynamically recrystallized zone (~ 1 mm) on the top layer surface and a deformed zone (~ 0.5 mm) immediately under the dynamically recrystallized zone, which is not completely remelted by the subsequent layer deposition, generating some recrystallized grains during the multiple thermal cycles (subsequently layer deposited). Additionally, during the homogenization heat treatment, the remaining deformed region recrystallized, generating a finer grain size and an almost non-oriented microstructure. Furthermore, in situ interlayer hot forging allowed meeting the Inconel 718 grain size requirement for the AMS 5662 standard, an achievement that is not possible by the typical process route (arc plasma DED + heat treatment). In relation to the microstructure aspect, both DED and HF-DED showed a similar primary microstructure, composed of oriented dendrites with interdendritic eutectics. In addition, the solidification sequence for Inconel 718 fabricated via DED was described, which showed a core-shell structure composed of oxides and MC-type carbides. Furthermore, in the aged condition, the γ'' and γ' morphology was characterized by transmission electron microscopy, and the volume fractions of γ'' and γ' estimated by TEM analysis and synchrotron X-ray diffraction (Rietveld refinement) were in good agreement. HF-DED did not show a significant improvement in tensile strength for Inconel 718 at both room and elevated temperatures. However, HF-DED was able to reduce the yield strength anisotropy and allow the Inconel 718 DED to meet the yield strength requirements at both room and elevated temperatures.

5.1 Grain size refinement mechanism

5.1.1 Introduction

Parts fabricated *via* fusion-based metal additive manufacturing (AM) processes, *e.g.*, directed energy deposition (DED), typically exhibit a coarse and oriented microstructure, a tendency especially notable for alloys that do not undergo solid-state allotropic transformations (*e.g.*, Ni-based superalloys) [21], [25], [106], [160], [413]. These microstructure aspects pose a barrier to the industrial scalability of DED processes (*e.g.*, blown powder and arc plasma) and their respective use in critical applications, as the grain size requirement is usually not met (*e.g.*, API 20S). Such undesirable microstructure arise from the thermal conditions during layer consolidation [377], [432]. The localized heat source induces an intense

thermal gradient in the melt pool, inhibiting the columnar-to-equiaxed transition [343], [374]. Additionally, due to the intrinsic layer-by-layer deposition fashion, the epitaxial grain growth is promoted, favoring grains with higher growth rate and ultimately leading to the typical oriented primary microstructure [264], [397], [430], [433]. In high energy density AM processes (*e.g.*, laser powder bed fusion), a significant residual strain level can be generated during the part printing [100]–[104]. This, in turn, can promote static recrystallization during the mandatory post-deposition heat treatments (PDHT, *e.g.*, ASTM F3055), partially mitigating the microstructure and coarse grain size issues [500]–[503]. Otherwise, this straightforward solution usually does not apply to lower density energy processes, *e.g.*, arc plasma DED (gas metal arc-based) and welding, which did not exhibit significant grain boundary migration during industrial PDHT (*e.g.*, AMS 5383 and API 6ACRA) [21], [25], [97]–[99], [105]–[112].

Technologies previously developed for welding and casting to refine the microstructure were also tested in arc plasma DED, which include, the addition of inoculants in melt pool [64], vibration [504], interlayer mechanical deformation (IMD) [110], [160], thermal management [67], and electromagnetic stirring [62]. IMD stands out because it does not alter the feedstock material composition (*i.e.*, not alter the material specification), can be applied to all weldable materials, achieves efficient grain size refinement (meeting material requirements), and promotes a non-oriented microstructure [34], [35], [120], [159]. IMD systems can be categorized based on temperature (hot or cold) and type of deformation (quasi-static – rolling – and dynamic –hammering/forging/peening) [110]. However, considering the complex part geometries, non-linear path planning, and constraints on deposition parameters (*e.g.*, preventing the complete melting of the deformed zone), IMD systems can diminish process control and flexibility. Moreover, the distinct deformation behavior (material), thermal distribution (DED process), and strain rate and deformation mode/intensity (IMD system) result in almost unique grain size refinement mechanisms for each specific material/IMD/DED combination [35], [37], [66], [124], [126], [127], [131], [138], [144], [145], [152], [153], [159], [160], [169], [170], [176], [505]–[508].

Dynamic IMD processes (hot and/or cold) demonstrate greater potential for industrial scalability due to their capability to be integrated with the welding torch on a robot arm, which enhances the path planning flexibility [35], [145]. In this regard, the present work describes the grain size refinement mechanism of *in situ* interlayer hot forging arc-based directed energy deposition (HF-DED). The subject of the present study was the precipitation-strengthened Ni-based superalloy 718 (Inconel 718). The grain size refinement mechanism was analyzed using electron backscatter diffraction (EBSD) and synchrotron X-ray diffraction (SRXD).

5.1.2 Materials and Methods

5.1.2.1 Materials and process description

The present work based on an in-house developed HF-DED prototype, which consists of a gas metal arc power source (CITOWAVE III) assembled in a 3-axis CNC machine (Figure 5.1). This is a latest version of the prototype described previously (Figure 3.2). The HF system comprises a moving part (shielding gas nozzle), which is driven by two pneumatics actuators and moves following a linear guideway/bearing system [160]. The forging tool (crown hammer) design was previously optimized (20 mm) by Farias et al. [159], which considered the forging temperature range of Ni-based superalloys (900 – 1100 °C) [52], [372]. Single-bead multi-layer parts, $7 \times 30 \times 120 \text{ mm}^3$, were fabricated using the HF-DED printer. The deposition parameters and feedstock material composition (Inconel 718; 1.2 mm; solid wire; 3Dprint AM 718 voestalpine Böhler) were summarized in Table 5.1 and Table 5.2). In sequence, the samples underwent a mandatory (AMS 5383) homogenization heat treatment (1100 °C/ 2 h). Furthermore, the literature [21], [25], [97]–[99], [105]–[112] and present results (Figure 5.20, Supplementary material) demonstrated that the PDHT had a negligible effect on the grain size and morphology of Inconel 718 fabricated *via* arc plasma DED (IN718 DED). Thus, to study the HF-DED grain size refinement mechanism, the present work compares the following conditions:

- i. IN718 DED;
- ii. Inconel 718 HF-DED (IN718 HF-DED);
- iii. IN718-HF-DED + homogenization heat treatment (IN718 HF-DED-HT).

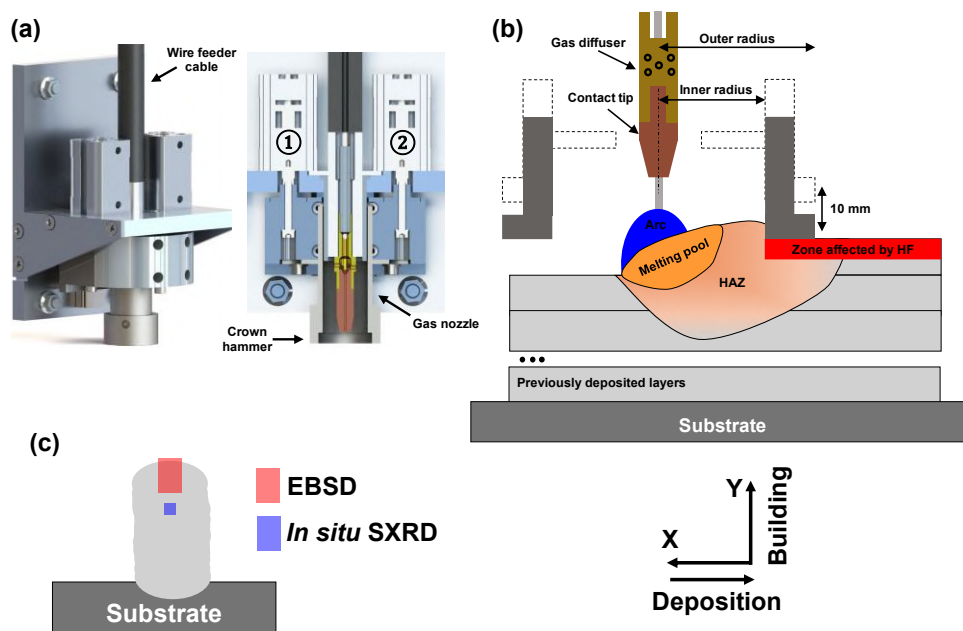


Figure 5.1 - (a) In situ interlayer hot forging (HF) system, (b) schematic representation of the effect of HF during part printing, (c) position where the ex situ EBSD and in situ SXR were performed. ① and ② are the pneumatics actuators.

Table 5.1 - *In situ* interlayer hot forging arc plasma DED parameters.

Arc plasma DED	
Arc plasma current [†]	137 [A]
Arc plasma tension [†]	17 [V]
Wire feed speed	4 [m/min]
Travel speed	5 [mm/s]
Heat input	372.6 [J/mm]
Interlayer temperature	150 [°C]
Contact-tip-to-work distance	12 [mm]
Shielding gas	I3-ArHe-25
Shielding gas specification	AWS A5.32
Shielding gas flow	15 [l/min]
Substrate preparation	ISO 8501-1
<i>In situ</i> interlayer hot forging	
Pneumatic pressure	5 [MPa]
Frequency	8 [Hz]

[†] Root mean square (RMS)

^{*} Thermal efficiency ($\eta = 0.8$), BS EN 1011-1

Table 5.2 - Feedstock material (filler metal) chemical composition (% weight).

	Ni	Cr	Fe	Nb	Mo	Ti	Al	C	Mn	Si	P	S
Measured	52.52	17.41	20.23	5.25	2.98	0.98	0.45	0.05	0.04	0.08	0.002	0.001
AWS A5.14	50-55	17-21	Bal.	4.75-5.5	2.8-3.3	0.65-1.15	0.2-0.8	0.08	0.35	0.35	0.015	0.015
AMS 5383				0.4-0.8								
API 6ACRA				4.87-5.2		0.80- 1.15	0.4-0.6	0.045	0.35	0.35	0.01	0.01

5.1.2.2 Microstructural characterization

SEM and EBSD samples were ground (SiC paper) and polished (diamond suspension). An additional polishing step was performed in the VibroMet[®] 2 (colloidal silica, 0.04 μm , 6 h). *Ex situ* SEM and EBSD were carried out (Figure 5.1c) using a Thermo Scientific Quattro Environmental Scanning Electron Microscope equipped with a high-velocity EBSD camera (EDAX[®] Velocity). The step size, voltage, and tilt angle were 1 μm , 20 kV, and 70 $^\circ$, respectively. Data post-processing was conducted in the MTEX-Toolbox [360].

In situ SXRD (Figure 5.1c) were conducted to assess the microstructure evolution during the PDHT. The SXRD (HEMS beamline of PETRA III/DESY) was performed in transmission mode with an acquisition rate, wavelength, and spot size of 0.25 Hz, 0.14235 $\text{\AA}$, and $1 \times 1 \text{ mm}^2$, respectively. The 2D Debye-Scherrer diffraction rings were captured through a Perkin Elmer detector. Data post-processing was carried out using FIT2D [509] and an in-house developed python-based routine. This routine integrates over the full azimuthal angle and employs a pseudo-Voigt function to fit the diffraction peaks [361], [429]. For experimental setup details, refer to our previous works [160], [413].

The transmission electron microscopy was carried out in a Titan3[™] G2 60-300 S/TEM (300 kV). The samples were prepared using a focused ion beam Scanning Electron Microscopes (Helios NanoLab 600 DualBeam, FEI).

5.1.3 Results and discussion

5.1.3.1 Microstructural effects of the interlayer hot forging – *ex situ* analysis

The IN718 DED showed (Figure 5.2) the typical microstructure of Ni-based superalloys fabricated *via* arc-based DED [21], [25], [97], [98], [105]–[112], characterized by aligned and coarse columnar grains. Additionally, in the last moments of solidification, it is possible to observe a change in the solidification mode from columnar to equiaxial [432]. According to Li et al. [343], as the solidification front advances (from the fusion line to layer top surface), a reduction of the thermal gradient is observed, which can promote the columnar-to-equiaxed transition [374]. However, Figure 5.3a shows that the subsequently deposited layer completely remelted the columnar-to-equiaxed zone, where the material is characterized by coarse columnar grains that cross several layers. Therefore, Inconel 718 fabricated *via* arc-based DED tends to exhibit a non-optimized microstructure, failing to meet the material grain size requirements (Figure 5.3c) for high-performance applications (*e.g.*, aeronautics – AMS 5662 – and Oil & Gas – API 6ACRA – industries).

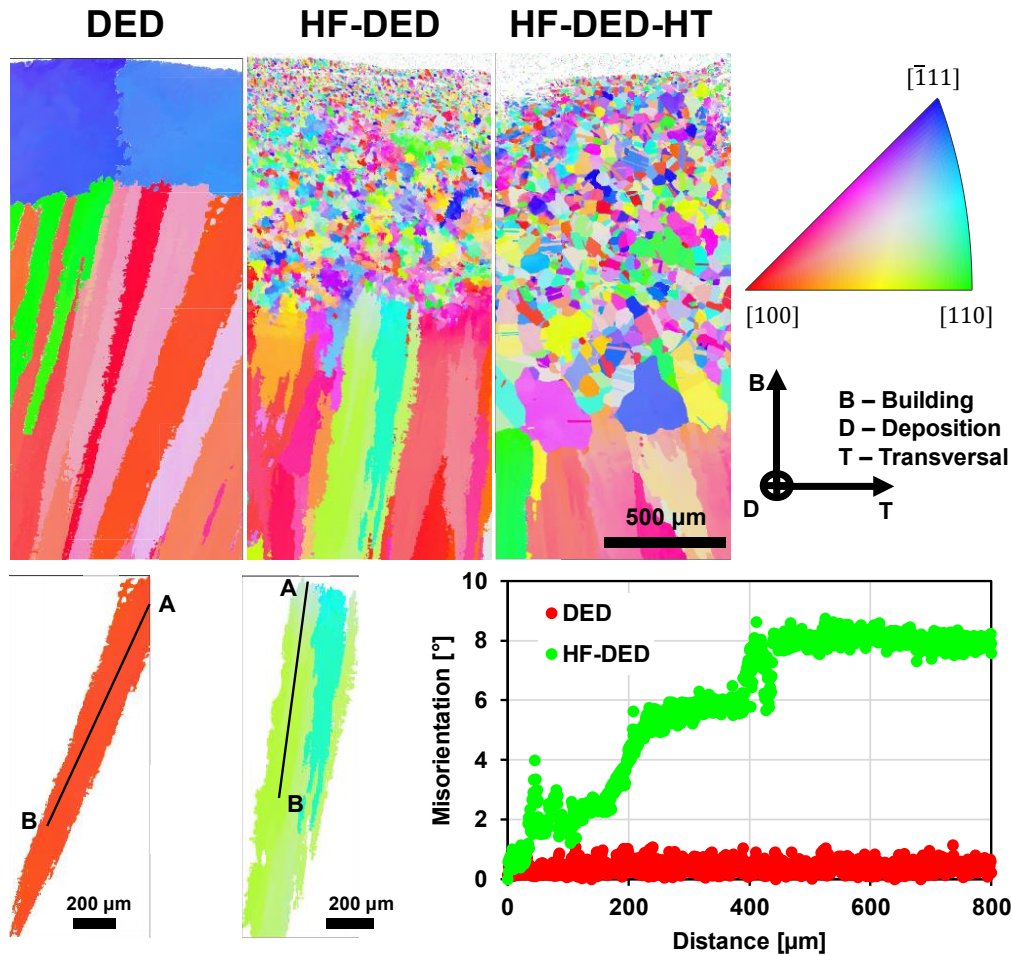


Figure 5.2 - Comparison of the effect of interlayer hot forging on the microstructure of the top layer surface of the Inconel 718 fabricated *via* arc-based DED. The points A and B indicated the start and end points, respectively. The distance was normalized in relation to the built direction.

When *in situ* interlayer hot forging is applied during the deposition (HF-DED), it is possible to observe the formation of a finer grain size zone on the top layer surface, which was larger than the columnar-to-equiaxed zone observed for conventionally fabricated DED parts (~ 0.5 vs. ~ 1 mm). This finer grain size zone is associated with a dynamic recrystallization effect induced by the imposed hot deformation [159]. Furthermore, after the heat treatment (HF-DED-HT), the refined grain size zone increases in length, which is attributed to post-dynamic recrystallization and further evidenced by the presence of multiple annealing twins [510]–[513], *i.e.*, there is a region that underwent deformation (storage energy) but did not experience dynamic recrystallization, which posteriorly recrystallized during the PDHT. This is also corroborated by comparing the intragranular misorientation observed in the DED and HF-DED conditions for a grain located immediately below the refined grain zones (columnar-to-equiaxed transition and dynamically recrystallized, respectively). DED exhibits the typical low

intragranular misorientation of as-solidified materials (maximum misorientation of approximately 1°), which is associated with the strain induced by the solidification shrinkage and thermal deformation [514]. In contrast, HF-DED induced a pronounced intragranular misorientation, which tends to stabilize as the depth in the sample increases, *i.e.*, HF induced an intragranular deformation gradient, as previously reported for interlayer rolling and peening [116], [153]. Additionally, the sum of the depth at which the misorientation stabilizes (~ 0.5 mm) and the length of the dynamically recrystallized zone (~ 1 mm) has a similar extension to the overall length of the recrystallized zone after the PDHT (~ 1.5 mm; HF-DED-HT). Therefore, the effect of *in situ* interlayer hot forging on the microstructure can be characterized by these two aforementioned regions, namely the dynamically recrystallized and remaining deformed (non-recrystallized) zones, with a total length of approximately 1.5 mm.

Analyzing the effect of the subsequently deposited layer on the HF-DED (Figure 5.3), it is possible to observe a small ribbon of recrystallized grains, which are almost equiaxed and have a grain spread orientation (GOS, Figure 5.3b) less than 2° [515]. These recrystallized grains either remained from the previously deposited layer (dynamically recrystallized) or recrystallized during the reheating thermal cycle. The differentiation between them can be evidenced by the presence of columnar grains with similar orientation both before and after the ribbon of recrystallized grains, *i.e.*, these new grains did not participate in the subsequent deposited layer solidification and were nucleated during the reheating thermal cycles (refer to the schematic representation – Figure 5.4). Additionally, away from the layer's center, a distinct orientation is observed before and after the ribbon of recrystallized grains, *i.e.*, these grains persist (they were not totally remelted) after the deposition of the subsequent layer, acting as an almost non-oriented substrate. The presence or absence of remaining dynamically recrystallized grains can be directly correlated with the finger-type penetration aspect of gas metal arc-based DED (Figure 5.4), which has the maximum penetration at the center of the layer [516], [517]. Thus, the region far away from the layer center (non-complete remelted) tends to have a finer microstructure than in the layer center, as reported by Gao et al. [161] (arc-based DED + interlayer incremental forming). The grain size refinement mechanism observed for IN718 HF-DED closely resembles that reported by Wang and Shi [153] (Inconel 718 laser-DED + interlayer cold ultrasonic peening). However, it differs by possessing incomplete melting of the deformed zone due to the lower penetration of laser-DED compared to arc-based DED. Furthermore, it is worth mentioning that the columnar grain width in the HF-DED was lower than DED, which can be attributed to the vibration induced by the hot

forging (dynamic loading) and the consequent restriction of lateral growth during solidification [35], [81], [157], [183], [504].

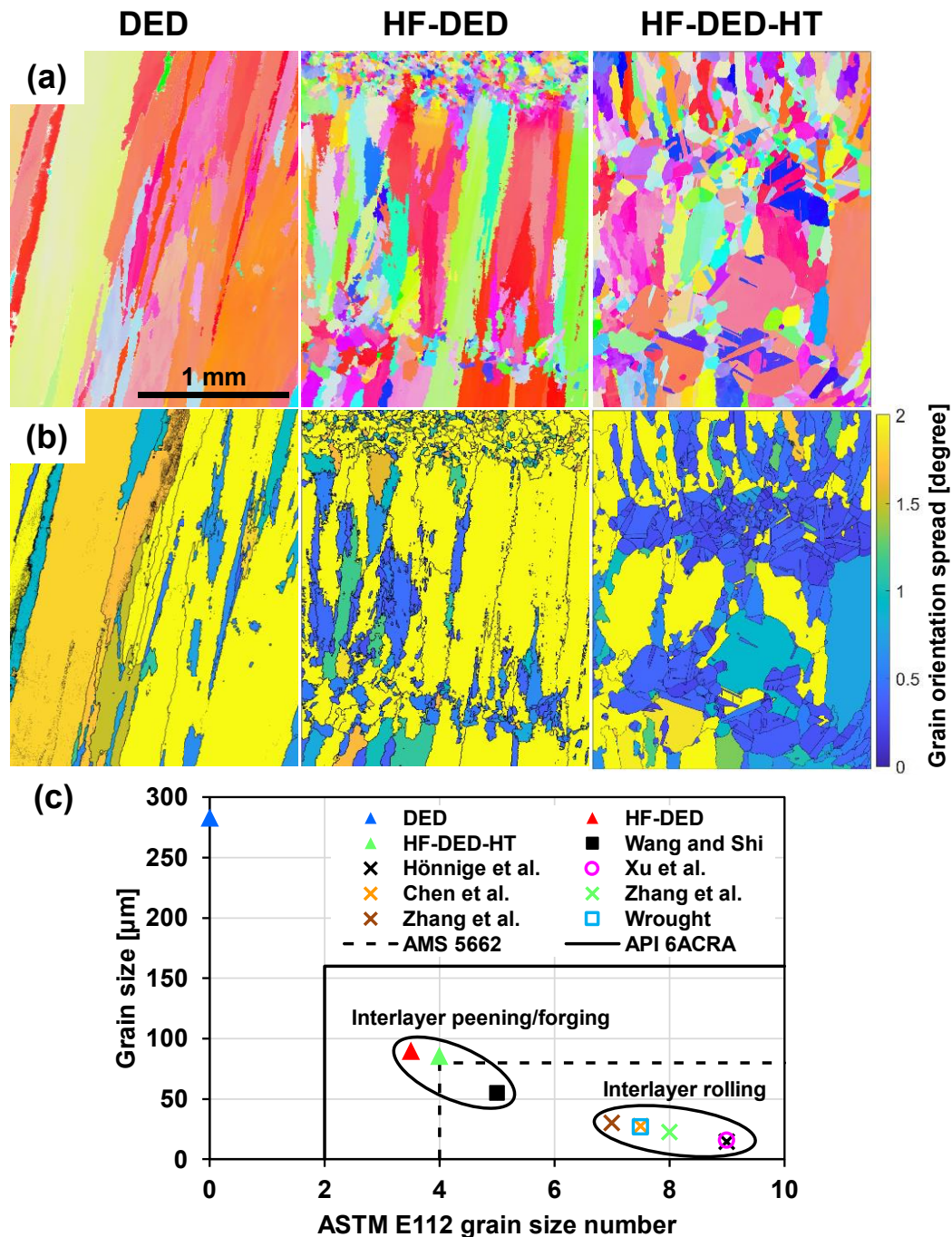


Figure 5.3 – (a) Effect of interlayer hot forging on the microstructure of Inconel 718 fabricated *via* arc-based DED (color code and axis references are the same as in Figure 5.2), (b) grain orientation spread distribution, and (c) the grain size measurement and its comparison with the literature. The cited references are: Wang and Shi [153], Hönnige et al. [37], Xu et al. [138], Chen et al. [200], Zhang et al. [110], and Zhang et al. [124].

HF-DED-HT exhibits intense static recrystallization, characterized by grains with lower GOS ($< 2^\circ$, Figure 5.3b) and the presence of annealing twins (Figure 5.3a). Moreover, as the

size of the deformed zone (~ 1.5 mm) is smaller than the layer thickness (~ 2 mm), achieving a fully recrystallized microstructure is not feasible, leading to the presence of some columnar grains, *i.e.*, the portion of the deposited layer that undergoes plastic deformation will recrystallize, whereas the remaining part (under elastic loading) will not undergo recrystallization [510]–[513]. However, these columnar grains were partially consumed by the grain growth of the static recrystallized grains. Furthermore, HF-DED-HT had a finer microstructure than DED and HF-DED, meeting the material grain size requirements (AMS 5662 and API 6ACRA), which demonstrates the HF's industrial scalability potential. When comparing HF with the interlayer rolling (cold and hot), HF has an inferior performance in terms of grain size refinement, which can be attributed to lower dynamic forces (~ 3 kN) [159] in relation to rolling (up to 160 kN) [114]. Consequently, HF exhibited a shallower deformed zone, leading to incomplete recrystallization of the deposited layer, while interlayer rolling can induce static recrystallization throughout the deposited layers [37], [136]. Otherwise, HF possesses a greater flexibility and almost unrestricted path planning (co-axial crown hammer), overcoming the process restrictions of interlayer rolling [162], [163]. Therefore, given its capability to refine grain size (Figure 5.3) and the nearly unrestricted path planning, *in situ* interlayer HF emerges as a promising technology to address the coarse microstructure issue typically observed in Ni-based superalloys fabricated *via* DED.

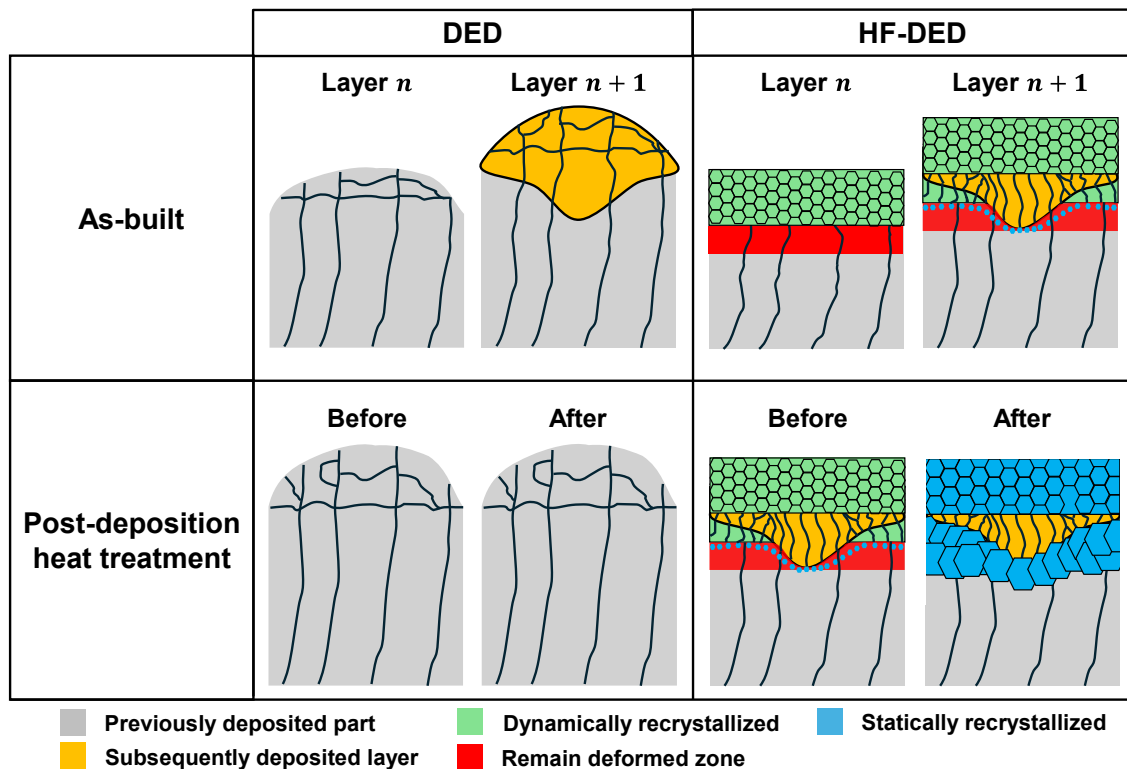


Figure 5.4 – Schematic representation of the grain size refinement of the *in situ* interlayer hot forging for alloys that did not undergo allotropic solid-state phase transformation.

Another critical microstructure issue related to Ni-based alloys fabricated *via* arc-based DED is the development of an oriented microstructure (crystallographic texture), which can compromise the material performance (*e.g.*, fatigue life and creep ductility) and induces a highly anisotropic behavior (*e.g.*, Young's modulus) [52], [72], [482]. The pole figures and 3D orientation distribution functions (Figure 5.5) show that the IN718 DED exhibited a strong cube texture ($\{200\}\langle 100 \rangle$), which is associated to the easy growth direction for face-centered cubic materials along $\langle 100 \rangle$ direction and the total melting of columnar-to-equiaxed transition zone [264], [343], [374], [397], [426], [430], [433]. HF-DED promoted some grain size refinement (remaining dynamically recrystallized grains and static recrystallized grains during reheating thermal cycles), which reduces its texture index in comparison to DED counterpart. However, due to the presence of coarse columnar grains, a cube texture aspect persists. The heat treatment led to significant static recrystallization throughout the microstructure (Figure 5.3), resulting in a non-oriented microstructure (HF-DED-HT). Therefore, HF can address two critical points related to additively manufactured Ni-based superalloys, namely, reducing the grain size through dynamic/static recrystallization and consequently promoting a non-oriented microstructure.

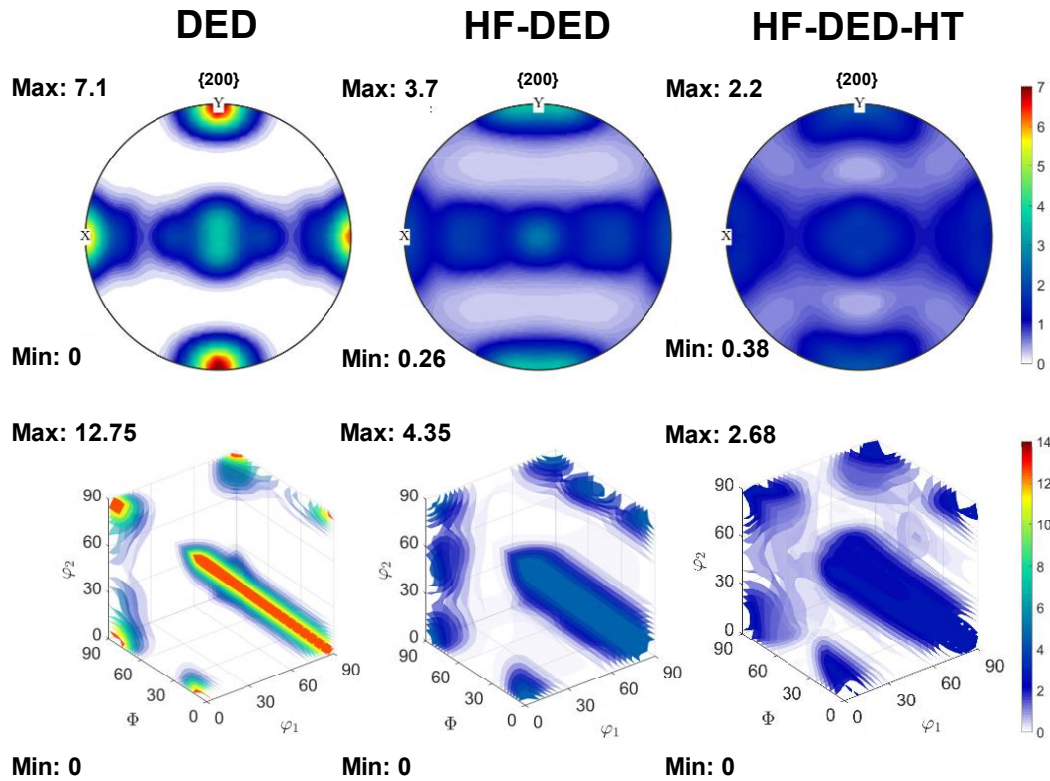


Figure 5.5 – Comparison of the effect of *in situ* interlayer hot forging on the texture aspect of the Inconel 718 fabricated *via* arc-based DED. The pole figures and 3D orientation distribution functions were estimated based on EBSD data (Figure 5.3).

5.1.3.2 Microstructural effects of the interlayer hot forging – *in situ* analysis

In situ SXRD during PDHT (Figure 5.6) reveals that the IN718 DED did not undergo significant changes in the diffractograms, as evidenced by the smooth alterations in the 2D Debye-Scherrer diffraction rings (Figure 5.6b) and full width at half maximum (FWHM, Figure 5.6c). Moreover, it is noteworthy that 2D Debye-Scherrer diffraction rings exhibited a dotted pattern [397], which serves as volumetric evidence that coarse and oriented microstructure of IN718 DED did not undergo significant changes during PDHT. These results are also corroborated by tracking the intensity of the {200} diffraction peak (Figure 5.6c), which showed no significant alteration, *i.e.*, the grain size and texture aspects of IN718 DED are almost insensitive to PDHT. These findings are in accordance with the previous literature results [21], [25], [97]–[99], [105]–[112] and are supported by the *ex situ* EBSD (Figure 5.3a and Figure 5.20).

The HF-DED material showed a notable decrease in FWHM during PDHT (Figure 5.6c), which is linked to restoration phenomena (recovery and recrystallization) [375]. Moreover, considering that Inconel 718 possesses a medium/low stacking fault energy [518], the main restoration phenomena can be attributed to recrystallization, such as evidenced by the *ex situ*

EBSD data (Figure 5.3a and Figure 5.20). At approximately 1500 s (Figure 5.6c), an intense reduction in FWHM is observed, which was correlated with a reduction in microstrain (*e.g.*, non-uniform strain and dislocations) due to recrystallization [393], [519], [520]. Subsequently (3200 – 4000 s), the slight increase in FWHM is associated with the increase in the number of grains (*i.e.*, lower grain size); finally (4000 – 7200 s), the slight reduction in FWHM can be related to grain growth (both reduction of microstrain and grain number). The intensity of the {200} diffraction peak continually decreased for HF-DED, indicating that static recrystallization and grain growth substantially modified the primary microstructure and texture aspects during PDHT (*i.e.*, from cube to non-oriented – Figure 5.5). These results are further supported by their respective evolution of 2D Debye-Scherrer diffraction rings, which exhibit significant changes during PDHT (from 0 s to 7200 s). Therefore, the significant changes in diffractograms, and consequently in {200} peak intensity and FWHM, of the HF-DED condition can be associated with the recrystallization and grain growth during PDHT, which corroborates the *ex situ* EBSD findings (Figure 5.2, Figure 5.3, and Figure 5.5).

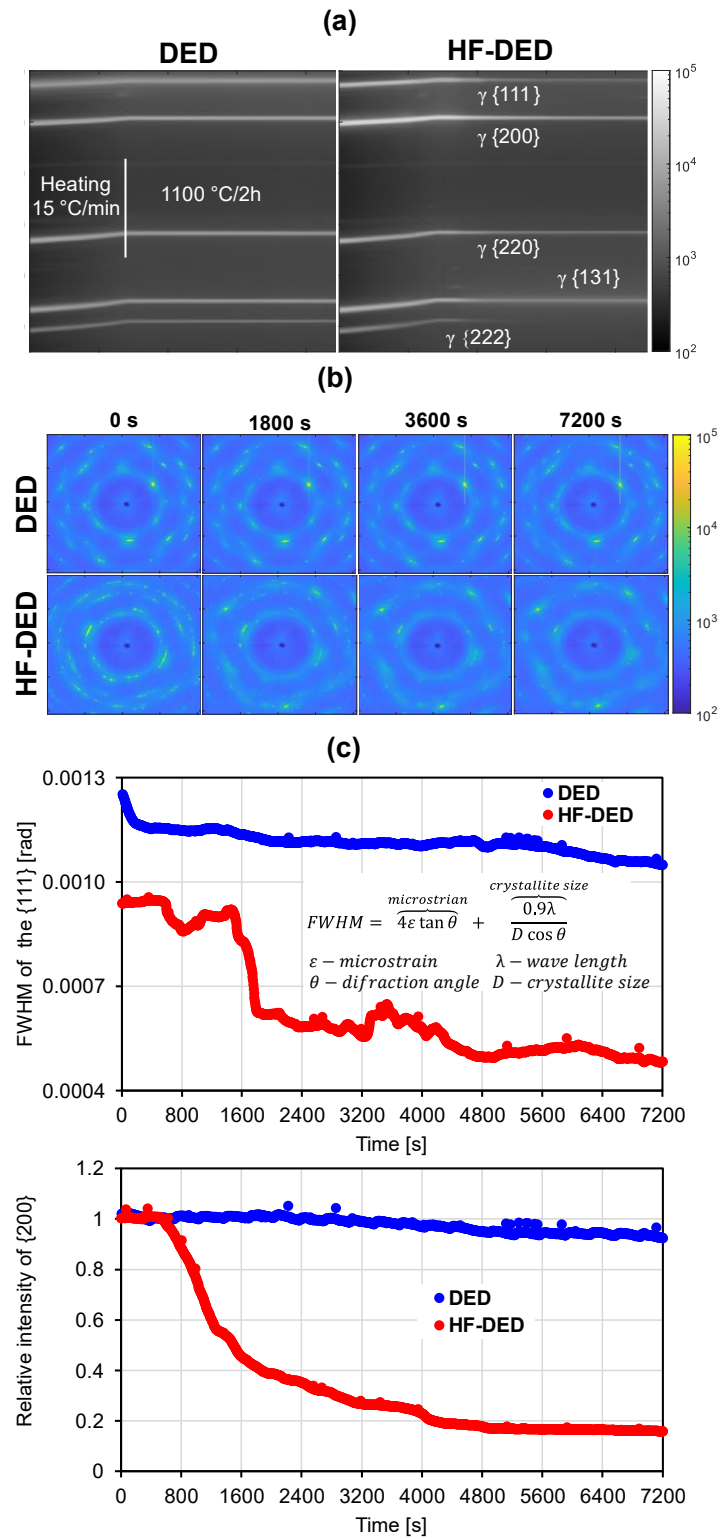


Figure 5.6 – *In situ* synchrotron X-ray diffraction data. (a) the diffractograms evolution with the time, (b) examples of 2D Debye-Scherrer diffraction rings alteration with the time, and (c) full width at half maximum (FWHM) and peak intensity evolution of the {111} and {200} diffraction peaks, respectively.

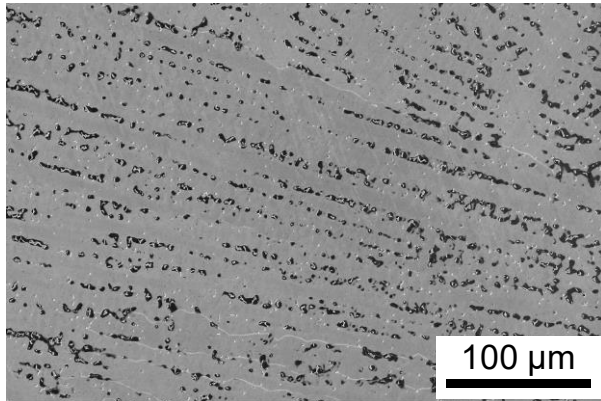
5.2 Microstructural characterization

Figure 5.7 compares the as-built microstructure of Inconel 718 fabricated via DED and HF-DED. In this figure it is possible to observe that the HF-DED induced non-oriented grains (almost equiaxed) in the interlayer region, which reinforces the EBSD data previously presented (Figure 5.3a). In addition, the columnar grains showed a thin aspect due to the vibration effect induced by the in situ interlayer hot forging (lateral growth restriction). On the other hand, DED showed a typical columnar and oriented microstructure observed in arc-based DED Ni-based superalloys [21], [25], [97]–[99], [105]–[112], which was also corroborated by EBSD data (Figure 5.3a). Furthermore, considering that both DED and HF-DED are fusion-based AM processes and that the thermal condition were almost similar between them [35], it is expected that a non-significant difference can be found in solidification sequence and interdendritic phases (Laves and MC-type carbides), as showed in Figure 5.7. Thus, the main difference between DED and HF-DED (as-built condition) lies in the grain size and grain morphology.

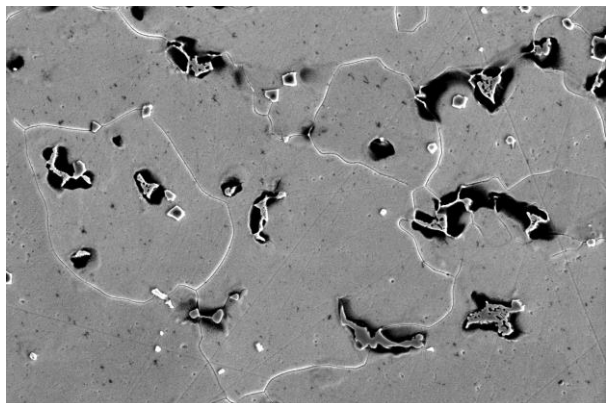
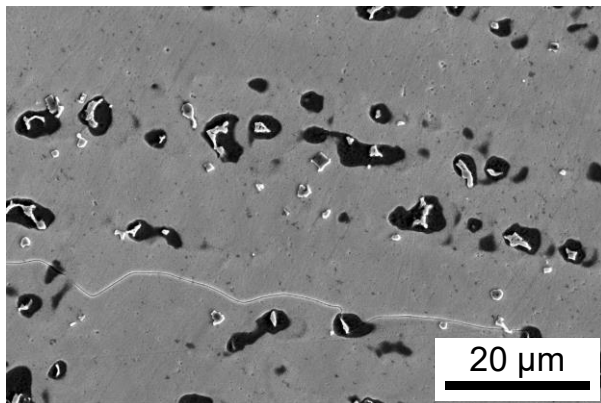
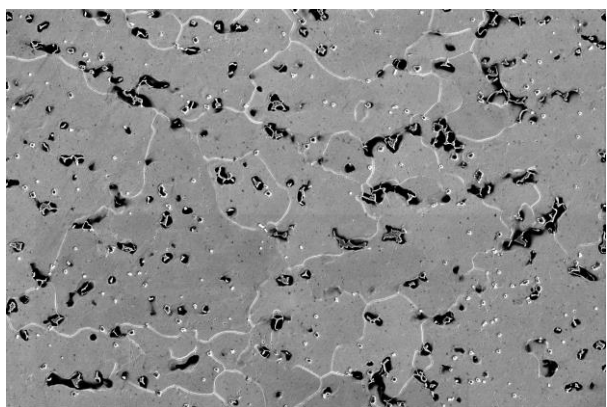
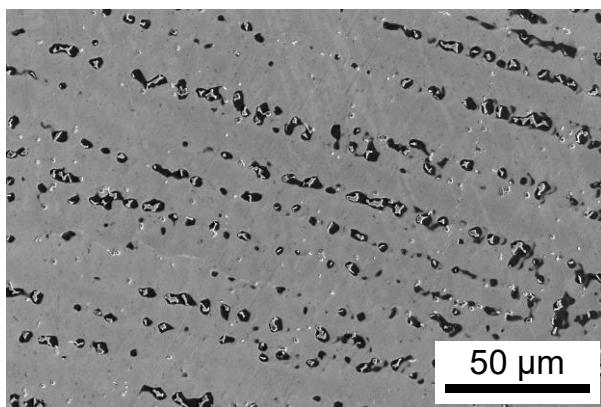
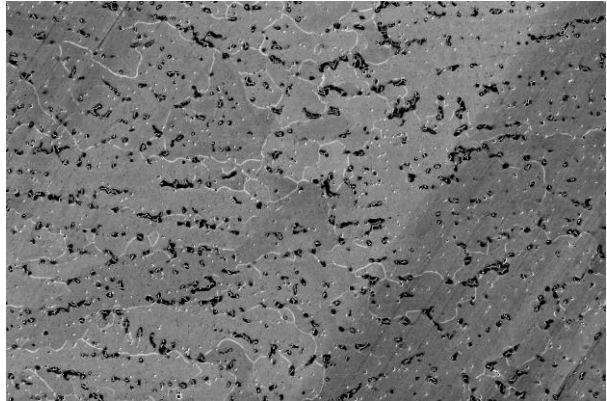
In this sense, the aspects of interdendritic precipitates, expressed by Figure 5.8 and Figure 5.9, can be used for both conditions. These figures reveals that the Inconel 718 (DED and HF-DED) followed the Dupont et al. [260] solidification sequence, i.e., the formation of MC-type carbides ($L \rightarrow \gamma + \text{MC-type}$) followed by the second eutectic reaction ($L \rightarrow \gamma + \text{Laves}$). In addition, in Nb-rich zones (close to Laves phases), the δ phase can precipitate due to the several thermal cycles, which reheated the material in the temperature range precipitation of δ phase (1020 - 650 °C) [289], [521]. Additionally, a core-shell structure was observed, which was initially classified as MC-type carbides (Figure 5.9); however, it is actually composed of three layers (from internal to external): (i) Al-based oxide, (ii) Ti-rich MC-type carbide, and (iii) Nb-rich MC-type carbide. Similar aspects for MC-type carbides and core-shell structure formation were previously reported for weld cladding by Silva et al. [259], Solecka et al. [263], and Farias et al. Additionally, to the best of the author's knowledge, this is the first time that this type of structure has been reported for arc-based DED. Therefore, the solidification sequence for Inconel 718 fabricated by DED and HF-DED can be expressed as follows:

- i. $L \rightarrow \gamma + \text{Al-rich oxide}$
- ii. $L \rightarrow \gamma + \text{Al-rich oxide} + \text{MC carbide (Ti-rich)}$
- iii. $L \rightarrow \gamma + \text{Al-rich oxide} + \text{MC carbide (Ti-rich)} + \text{MC carbide (Nb-rich)}$
- iv. $L \rightarrow \gamma + \text{Laves}$

DED



HF-DED



Building direction



Figure 5.7 - As-built microstructure of Inconel 718 fabricated via arc-based DED and HF-DED.

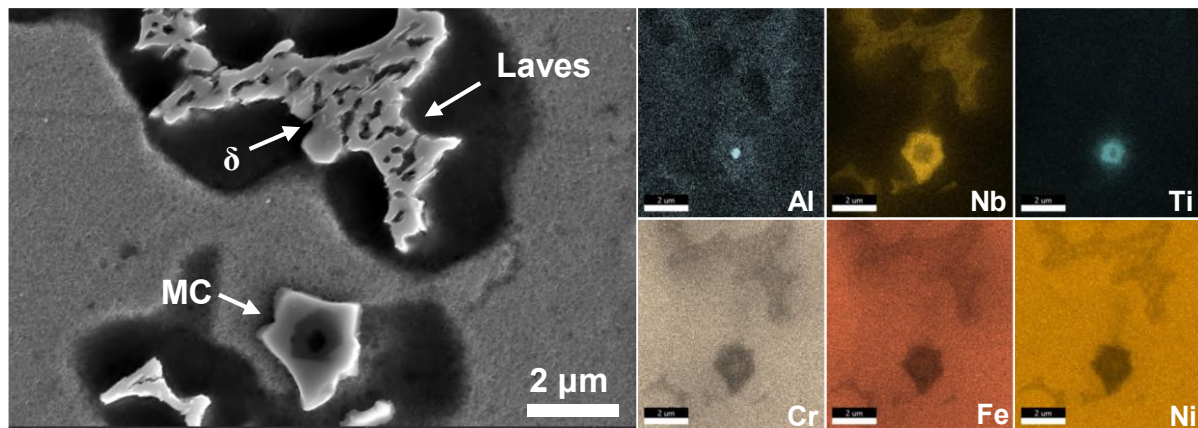


Figure 5.8 - Interdendritic microstructure of Inconel 718 fabricated via HF-DED (as-built condition)

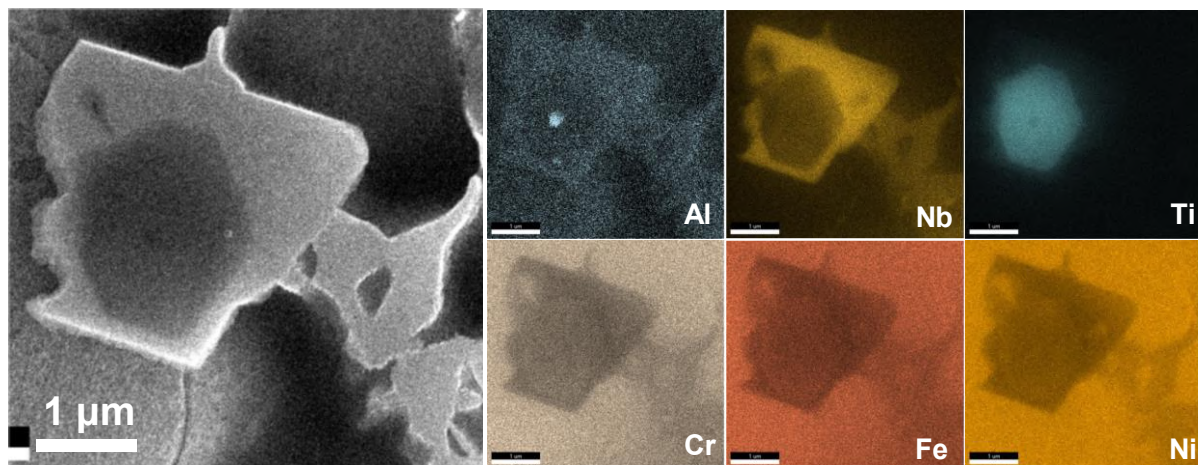


Figure 5.9 - Energy Dispersive Spectroscopy of MC-type carbide.

After the heat treatment (Figure 5.10), a significant difference in microstructure can be observed, particularly in the grain size and morphology aspects. The HF-DED condition resulted in an almost equiaxed grain structure, whereas the DED counterpart maintained a columnar and oriented grain structure, as previously shown by the EBSD data (Figure 5.3). In addition, the long-chain Laves and δ phase typically observed in as-built condition (Figure 5.11) had their morphology significantly altered (Laves - from long-chain to dotted aspect - and δ – total dissolution) during the heat treatment, which can be attributed to the Laves partial dissolution since the homogenization temperature (1100 °C) is superior to Laves phase stable temperature range (Figure 4.9) and complete dissolution of δ phase. The dissolution effect was also corroborated by the increase in Bragg's diffraction angle (increase in interplanar spacing, Figure 5.12). Specifically, the increase in solute content (especially Nb) in the solid-solution expands the interplanar spacing due to the difference in radii between the matrix (Ni) and the dissolved solute (Nb). Furthermore, according to Ruan et al. [522], some primary MC-type

carbides can pin the grain boundary during the grain boundary migration (grain growth), controlling the final grain size, as observed in Figure 5.10 (yellow circles).

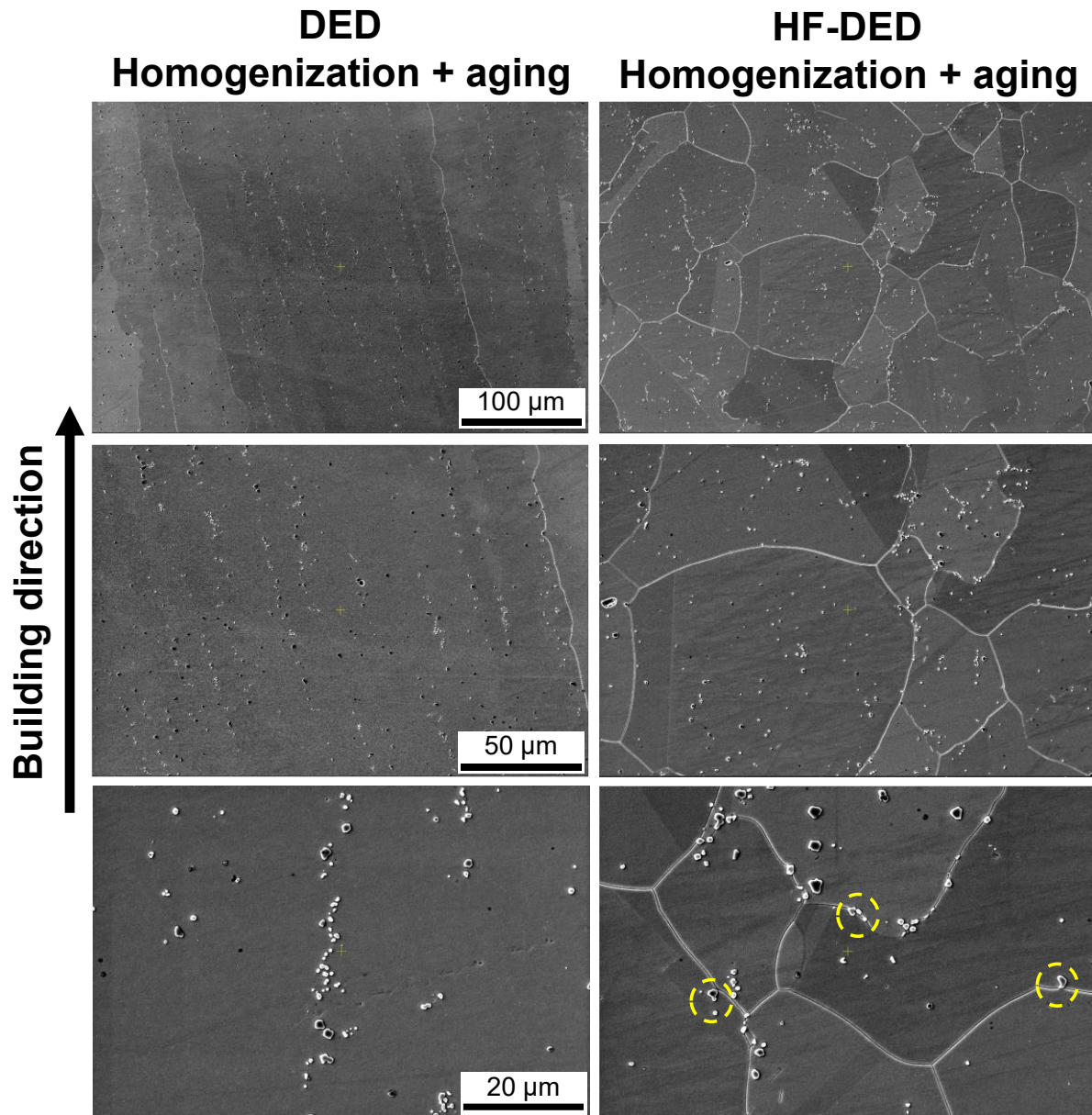


Figure 5.10 -Effect of post-deposition heat treatment (1100 °C / 2 h + aging) on the microstructure of Inconel 718 fabricated by HF-DED and DED.

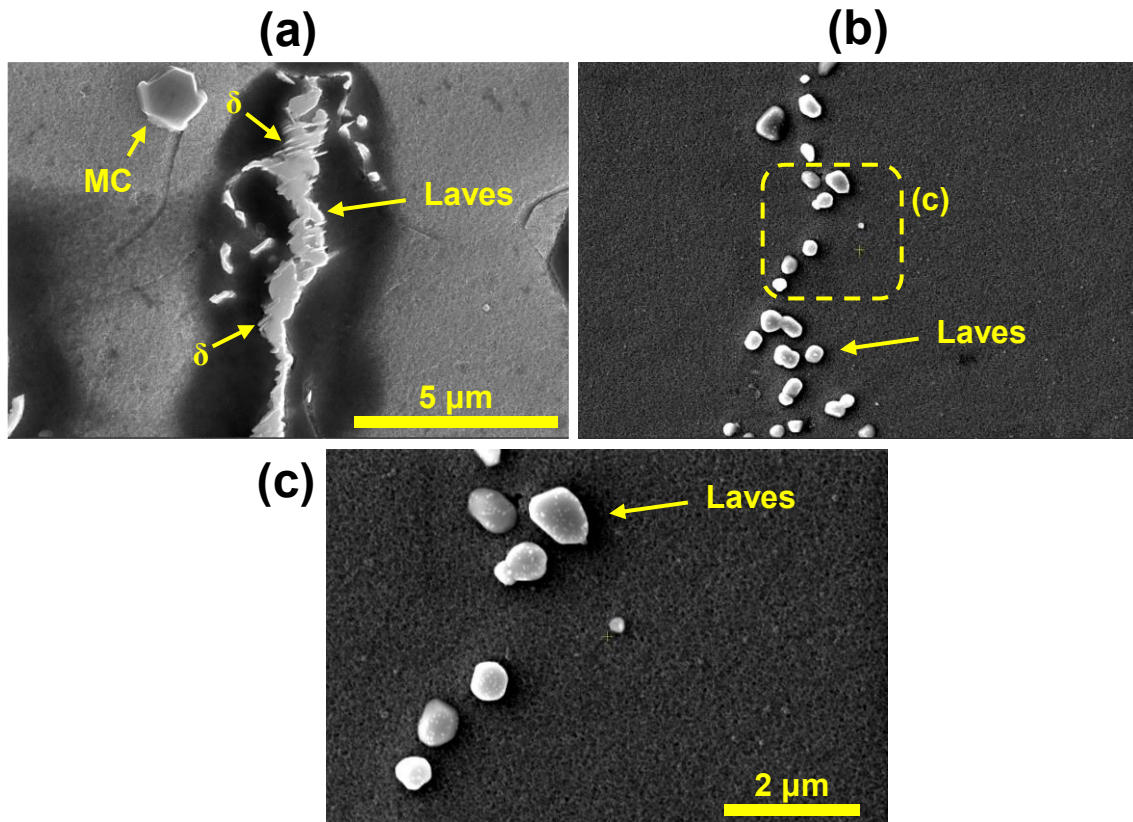


Figure 5.11 - Effect of heat treatment on Laves phase dissolution and morphology.

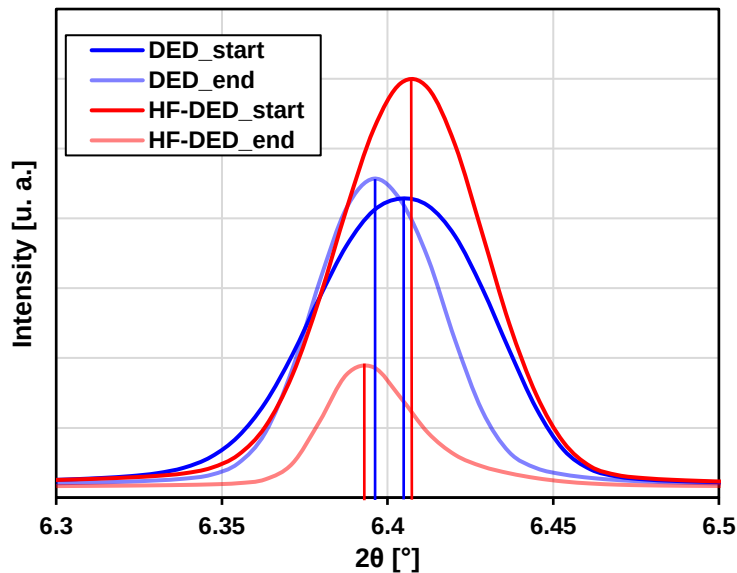


Figure 5.12 - Effect of post-deposition heat treatment on Bragg's angle diffraction.

Finally, in order to analyze the aged material, a transmission electron microscopy (TEM, Figure 5.13) analysis was performed on the Inconel 718 DED heat-treated condition. The γ' ($\text{Ni}_3(\text{Al}, \text{Ti})$) and γ'' (Ni_3Nb) phases can be distinguished by their composition and morphology (sphere/cubic vs. ellipse, respectively [523]; see Supplementary Material). According to

Theska et al. [524], in wrought Inconel 718 that was direct aged (i.e., without solution heat treatment, as in the present work), the typical precipitate morphologies observed were γ'' and γ' (monolith), $\gamma'-\gamma''$ duplets and $\gamma''-\gamma'-\gamma''$ triplets. These morphologies were also observed in the present work and confirmed by TEM analysis (Figure 5.14). In addition, the duplets and triplet suggest that γ'' precipitates first, followed by γ' . Shi et al. [525] reported that during the aging process, the precipitation of γ'' and γ' rejected Al and Nb for adjacent region, respectively. Rieli et al. [526] demonstrated that the enrichment of γ'' in Nb-rich zones occurred more easily for direct aging condition. Thus, as γ'' rejected Al, it can act as a heterogeneous nucleus γ' (pre-existed surface and Al-rich region), which can explain the formation of significant fraction of $\gamma'-\gamma''$ duplets and $\gamma''-\gamma'-\gamma''$ triplets, as observed in Figure 5.14. In addition, the γ' and γ'' particles size (Figure 5.15 and Supplementary Material) were estimated based on the chemical composition distribution (Figure 5.13). The γ' and γ'' particles sizes (~ 11 and 17 nm) were in accordance with the results estimated experimentally by Rieli et al. [526] (atom probe), Sui et al. [256] (TEM), and Slama et al. [445] (TEM).

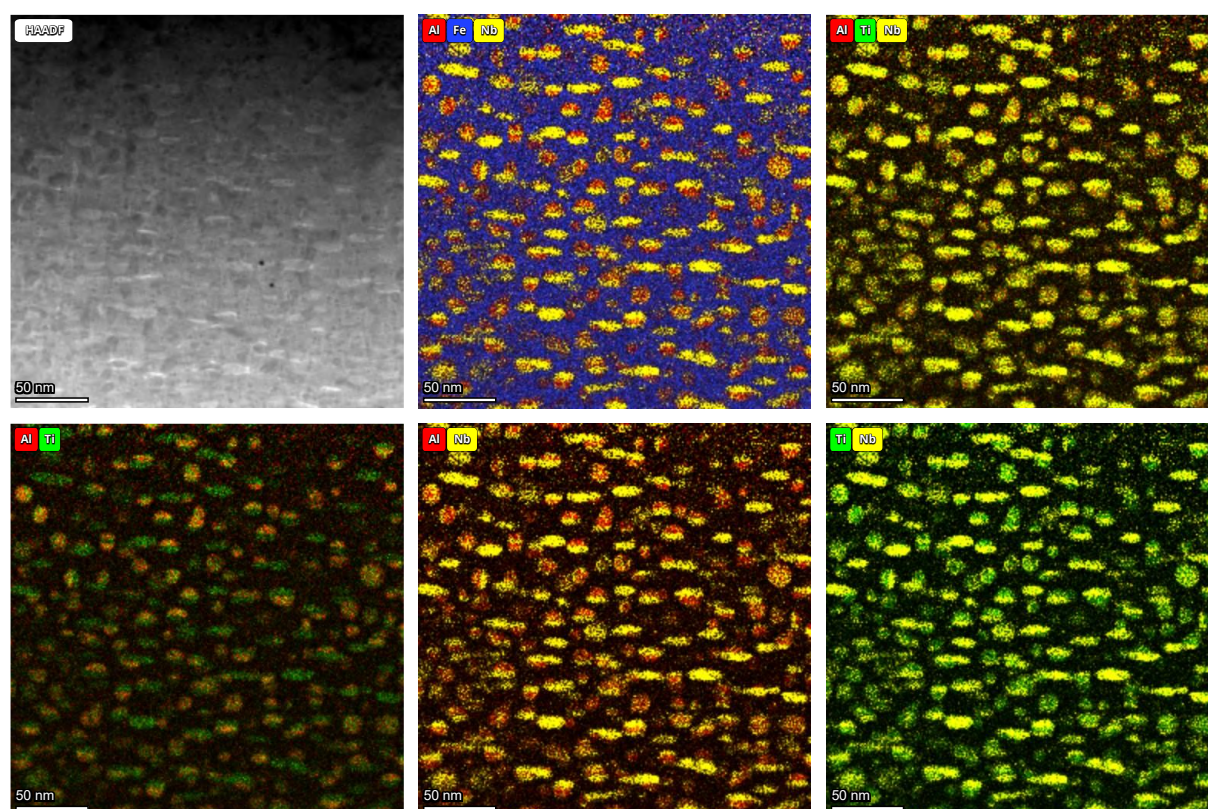


Figure 5.13 - Transmission electron microscopy coupled with Energy Dispersive Spectroscopy of MC-type carbide.

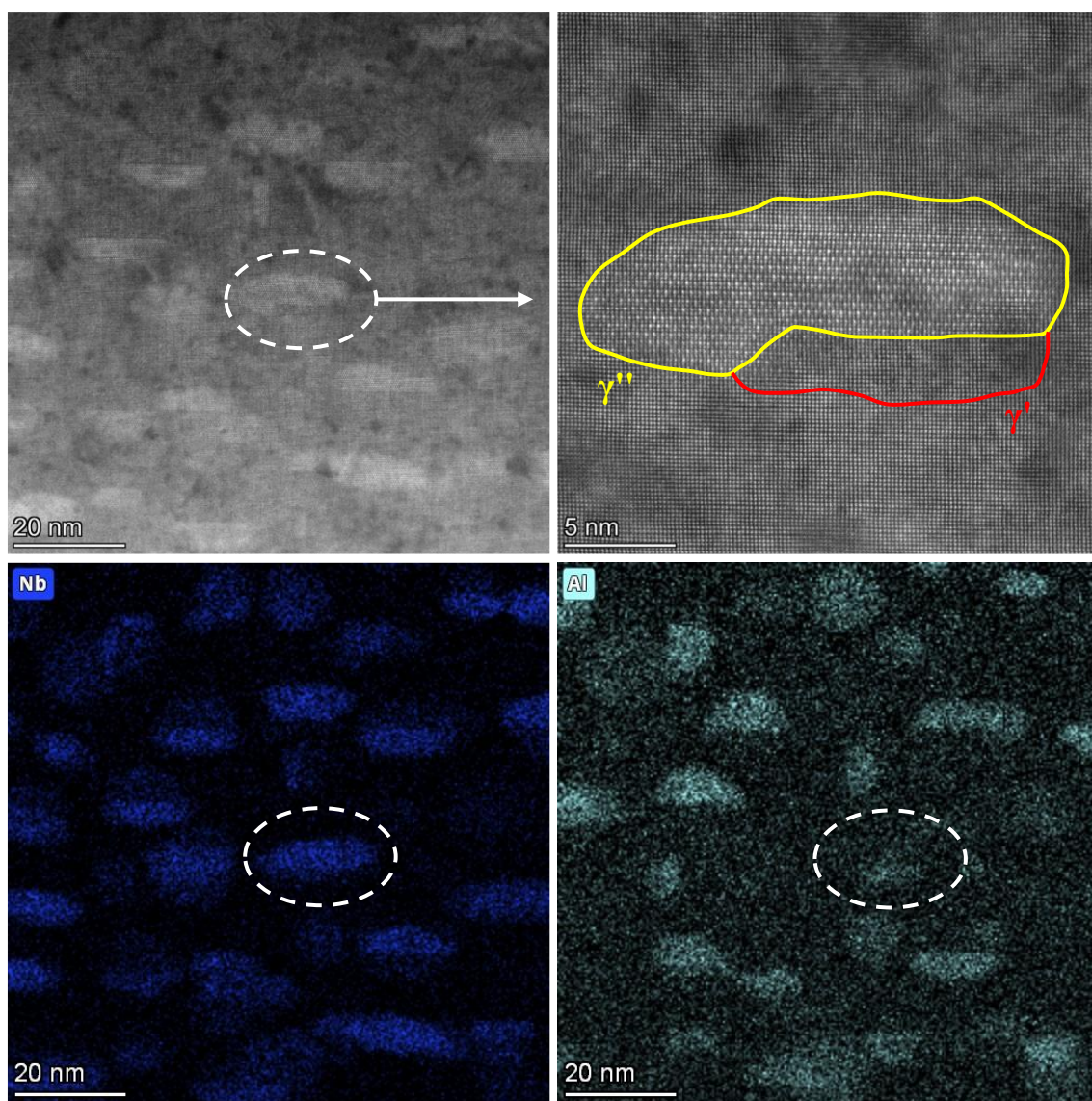


Figure 5.14 - High-magnification TEM, example of co-precipitation of γ' - γ'' duplets.

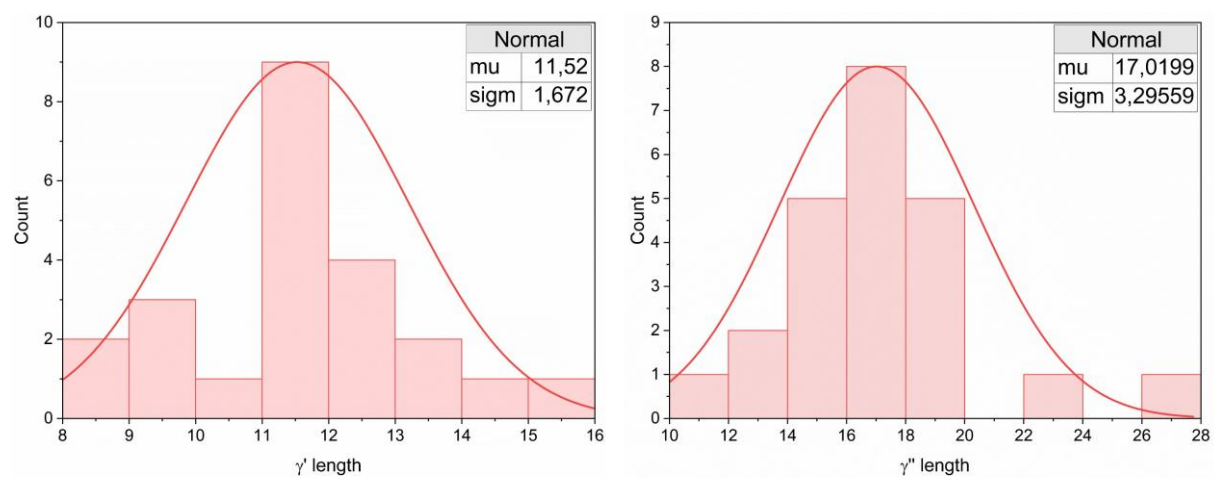


Figure 5.15 - Precipitates length (in nanometer, 10^{-9} m).

The hardening phase content was estimated using EDS-TEM analysis and compared with the Rietveld refinement (Table 4.3), which had a good agreement with almost similar values (γ'' – 17.39% vs. 17.02% – and γ' – 4.15% vs. 5.01%). This suggests that the Rietveld refinement applied to synchrotron X-ray diffraction can satisfactorily estimate the hardening phase content, as also demonstrated by Smith et al. [523] and Ferreri et al. [307]. Additionally, the volume fraction of the hardening phases estimated by EDS-TEM and SRXD is in accordance with the literature data for several process conditions (e.g., cast, forging, and powder bed fusion) [222], [256], [307], [309], [444]. These results corroborate that a long homogenization soaking time can induce significant dissolution of Nb-rich eutectic and improve the volume fraction of hardening phases.

5.3 Mechanical properties

To evaluate the effect of the in situ interlayer hot forging on mechanical behavior of the Inconel 718 fabricated via arc plasma DED, the Vickers microhardness profile ($HV_{0.3}$) was measured (Figure 5.17). For the as-built condition, HF-DED increased ($\sim 30 HV_{0.3}$) the material hardness in relation to DED. In addition, it is possible to verify that the peaks and valleys in the microhardness profile of HF-DED and DED are out of phase with the minimum of in situ interlayer hot forging condition equaling the maximum of DED. This indicates the remaining deformation in the material, even considering the deeper penetration of arc plasma DED processes. During part printing, regions near the fusion line exhibit finer dendrites, which coarsen as the solidification front progresses [527]. In practical terms, if hardness indentation occurs close to the fusion line, we expect higher hardness (associated with a finer microstructure) compared to measurements taken near the top surface of the layer. Refer to the schematic representation in Figure 5.16. Thus, throughout the hardness profile, hardness peaks and values occur due to distinct 'hard' and 'soft' regions in each layer. In contrast, for the HF-DED condition, the 'soft' region corresponds to the area near the fusion line, unaffected by mechanical deformation (finer dendrites), while the deformed region (top layer surface) becomes harder. As a result, an out-of-phase pattern emerges in the hardness profile for both DED and HF-DED conditions.

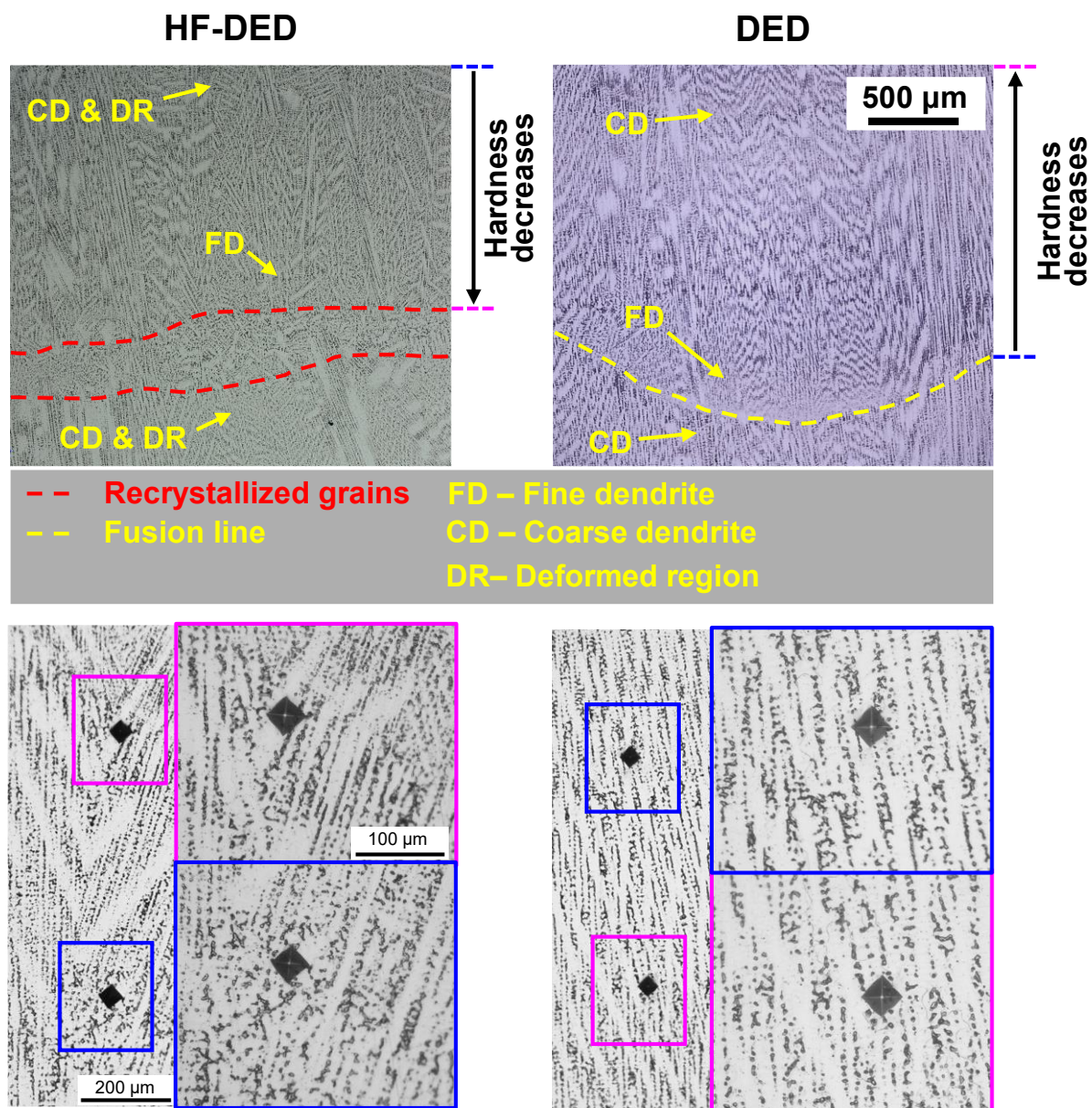


Figure 5.16 - Correlation between the microstructure and hardness profile of Inconel 718 fabricated via DED and HF-DED (as-built). The magenta and blue colors mean the hard and soft regions, respectively.

As previously demonstrated (Figure 5.3), the remaining deformation (increase of hardness) acts as driven force to static recrystallization during the heat treatment (homogenization step). However, in relation to aged condition (Figure 5.17), no significant difference was observed between DED and HF-DED. This result was expected, as the grain boundary strengthening mechanisms (Hall-Petch relation) has the least effective hardening mechanism for Inconel 718, as demonstrated in Table 4.4. Thus, it can be anticipated that the yield strength of the Inconel 718 HF-DED will be approximately those previously demonstrated by the Inconel 718 DED (Table 4.4). However, it is expected that the Young's modulus can

change since this property is directly related to crystallographic aspect [463]. In particular, the static recrystallization induced by the in situ hot forging potentially modified the crystallographic texture of the Inconel 718 DED (typically characterized by a cube texture, Figure 5.5), which can enhance the Young's modulus. Furthermore, due to the finer grain size, it is expected that Inconel 718 HF-DED will exhibit a higher ultimate tensile strength (UTS), attributed to a higher strain hardening resulting from the finer grain size.

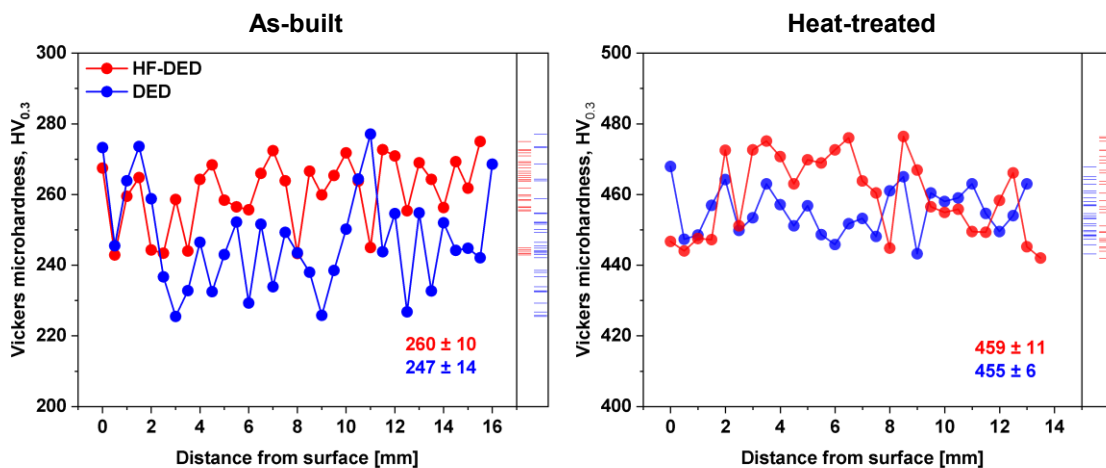


Figure 5.17 - Vicker microhardness profile (HV_{0.3}) of the Inconel 718 fabricated via arc plasma DED with and without Interlayer hot forging, as-built and heat-treated (1100 °C / 2 h + aging) conditions.

The above-mentioned hypotheses were confirmed by the tensile test (Figure 5.18), which demonstrated that HF-DED improved the UTS, i.e., the strain hardening effect during the tensile test. However, despite the grain size refinement induced by HF-DED, the grain size is still quite large (ASTM E 112 N° 4) compared to wrought material (ASTM E 112 N° 8 or higher) [528], which impairs both the DED and HF-DED to meet the UTS of the wrought material, i.e., the HF-DED continues to have lower strain hardening than wrought material. Furthermore, the anisotropy observed in yield strength (YS) was reduced (from 8.5 % to 0.1 %, i.e., a material almost isotropic), which can be related to the dependence of strengthening mechanisms on crystallographic texture (namely the Taylor factor). Thus, as HF-DED significantly reduced the texture index, a more homogeneous behavior is expected since solid-solution, dislocation density (strain hardening), and precipitation are direction-dependent [222], [239], [244], [256], [448]. Regarding elongation, both DED and HF-DED demonstrated the typical high-ductility of FCC materials, in especially those demonstrated by Inconel 718 fabricated via AM (Figure 4.18). It is worth noting that the low elongation observed for HF-DED in the horizontal direction was attributed to a printing defect (hot crack - Supplementary Material, Figure 5.22); however, its ductility still met the AMS 5662 requirement. Similar to UTS,

the Young's modulus (Table 5.3) was not significantly altered by HF-DED (increase from 120 to 150 GPa), despite its almost non-oriented microstructure; in relation to wrought material, the obtained values continue to be quite inferior (25 % less). Once again, this behavior can be associated with the coarse grain size, which consists of a primarily oriented microstructure (columnar) and coarse recrystallized grains (equiaxed). Even with an overall non-oriented microstructure, a small number of grains (Figure 5.3) bear the loading during the tensile test. Therefore, as these grains partially maintained their primary microstructure, orientation (cube texture), and stereology, a non-significant difference was observed between HF-DED and DED.

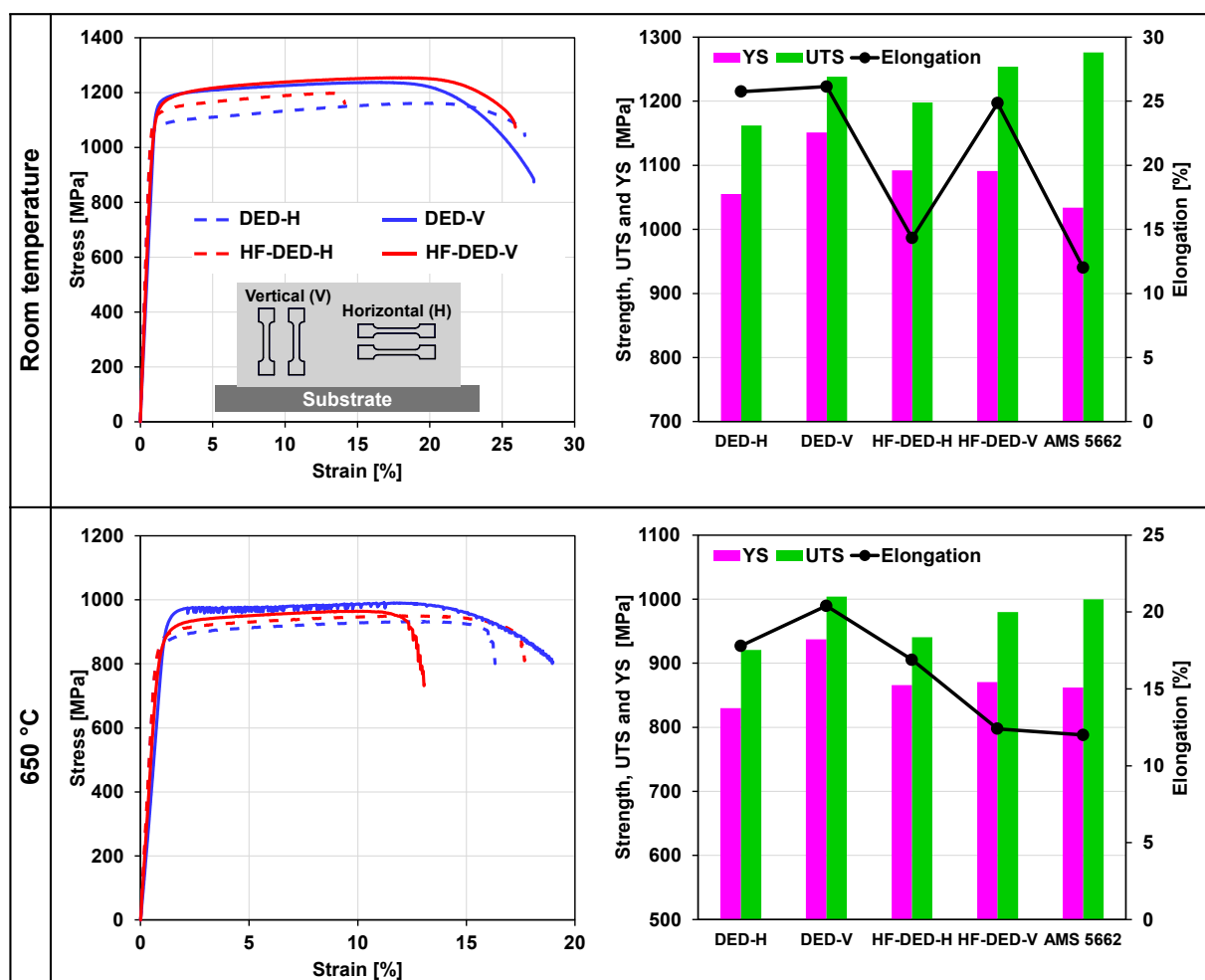


Figure 5.18 - Tensile test (room temperature and 650 °C) of Inconel 718 fabricated via DED and HF-DED.

Table 5.3 - Young's modulus of the Inconel 718 fabricated via DED and HF-DED.

Reference	Process	Heat treatment	Orientation	E
				[GPa]
Present work	GMA-DED	1100 °C / 2 h + aging	Horizontal	154
	GMA-DED + HF		Vertical	112
			Horizontal	155
			Vertical	130
Kindermann et al. [21]	GMA-DED	AMS 5662	Horizontal	159 ± 2
			Vertical	124 ± 3
		API 6ACRA	Horizontal	152 ± 3
			Vertical	117 ± 2
James et al. [319]	PTA-DED	AMS 5662		125
Baufeld [220]	GTA-DED	As-built		154 ± 1
Kumara et al. [467]	PBF	As-built	Horizontal	169
			Vertical	126
Wrought [21]				207 ± 5

Regarding the tensile test at elevated temperature (650 °C), the DED condition exhibited behavior similar to what was previously presented (Figure 4.22), i.e., the strain-stress curve showed a ductile aspect, and the quasi-static mechanical properties requirements were met only in the vertical direction. Additionally, the DED also displayed the dynamic strain aging phenomenon (serrated aspect in the strain-stress curve) [484], which was previously observed only for the DED heat-treated (1142 °C / 2 h + aging) condition. Once again, the dynamic strain aging was observed only for vertical specimens, reinforcing the dependence of this phenomenon on the material's crystallographic aspects and corroborating the Beese et al. [489] model. Conversely, the HF-DED did not exhibit this phenomenon, which is expected in conditions with a finer and less oriented microstructure (higher strain rate) [491], [492] in relation to condition without HF. This reinforces the hypothesis that, despite HF-DED promoting a finer grain size, the material still maintained some aspects of the primary microstructure and a coarse grain size, resulting in no significant difference in mechanical behavior.

Relative to material strength, HF-DED reduces the anisotropy in yield strength (from 11.4% to 5.2%), meaning that the dependence of strength mechanisms on the loading direction is almost resolved by HF-DED (at room and elevated temperatures). Additionally, the yield strength requirement was met for HF-DED conditions in both directions. However, UTS and ductility were reduced, which can be attributed to the increase in grain boundary length (finer grain size), as grain boundaries become significant in deformation mechanisms at elevated

temperatures. Currently, only the arc-based DED + warm interlayer rolling material [109]–[111] meets the AMS 5662 requirements, despite all DED + interlayer deformation processes (literature [136], [153], [200] and present work) also improving the material performance concerning DED at elevated temperature.

Figure 5.19 depicts the fractography of horizontal specimens, which showed a clear ductile aspect (both DED and HF-DED). DED conditions showed the typical dimple aspect of Ni-based superalloys fabricated by fusion-based additive manufacturing, where the dimples nucleated in the incoherent interface eutectic/matrix, creating the "solidification" (dendritic) pattern on the fracture surface [21], [25], [109], [316]. On the other hand, the grain size refinement induced by HF-DED significantly altered the fracture surface, which did not show a clear "solidification" pattern at room temperature. In addition, despite, the HF-DED (room temperature) exhibits a low ductility (Figure 5.18) due to the present of a printing defect (Figure 5.22), the remain tensile specimen cross-section showed a clear ductile aspect. At elevated temperature, the fracture surface of DED was not significantly altered, showing the typical dimple solidification pattern; HF-DED exhibited a mixed aspect, i.e., oriented (dendrites) and non-oriented (equiaxed grains) dimple patterns. Thus, both DED and HF-DED showed a typical ductile behavior of Ni-based superalloy at room and elevated temperatures.

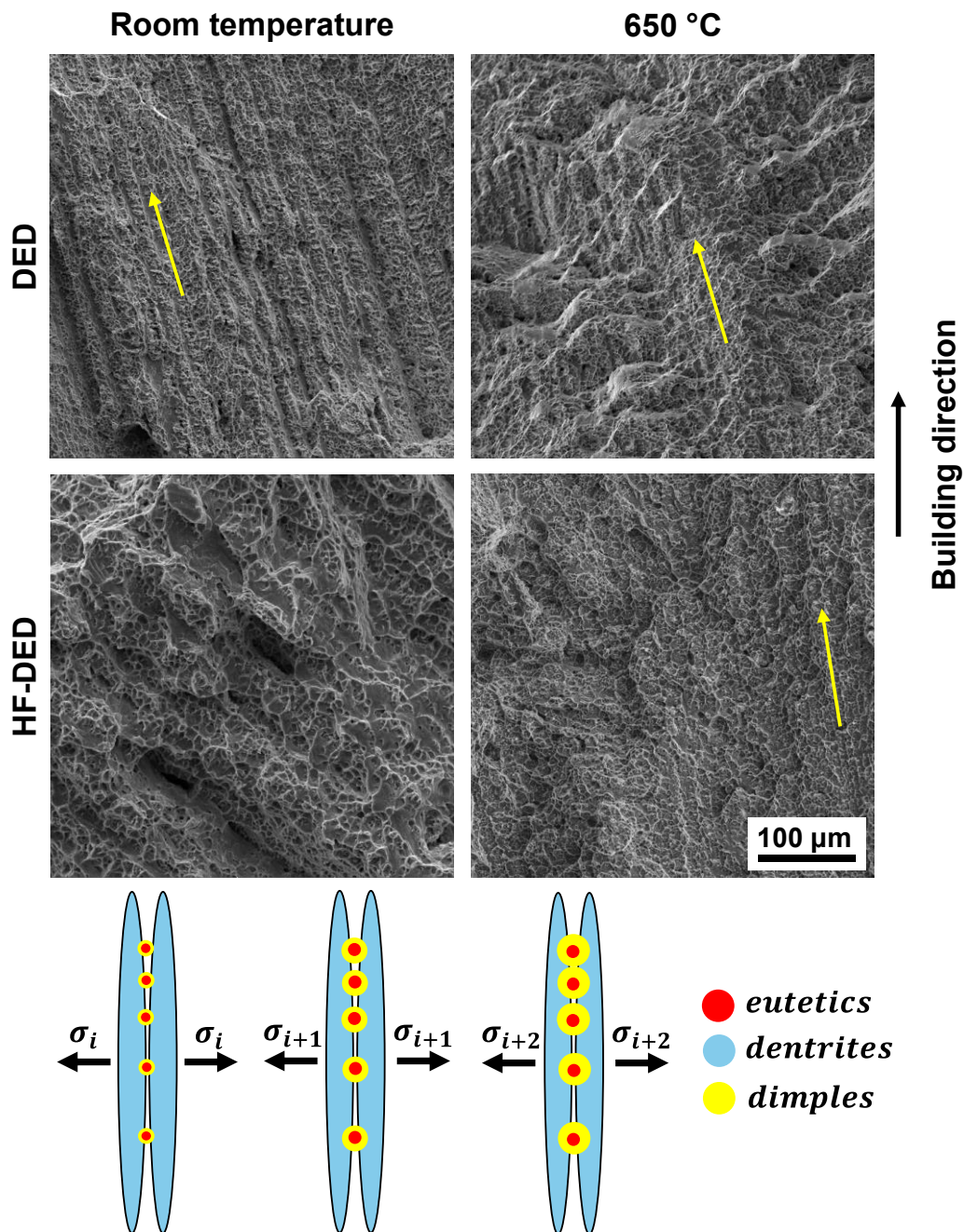


Figure 5.19 - Fractography of horizontal specimens tested at room temperature and 650 °C.

5.4 Summary

The grain size refinement of the in situ interlayer hot forging (HF) is attributed to the dynamic recrystallized zone generated due to localized deformation on the top surface of the just-deposited layer (i.e., material in elevated temperature). Additionally, this localized

deformation also induces a non-recrystallized zone (remaining deformation), which can subsequently experience static recrystallization during reheating thermal cycles and/or post-deposition heat treatments. The HF significantly altered the crystallographic texture of the IN718 AP-DED from a typical cubic texture ($\{100\}\langle 100\rangle$) to a nearly non-oriented microstructure. Ultimately, the HF-DED followed by the post-deposition heat treatment enables meeting the Inconel 718 grain size requirements (e.g., AMS 5662). This highlights the efficacy of dynamic interlayer deformation systems in refining the grain size of parts fabricated via AP-DED, underscoring the industrial scalability of the HF-DED.

In relation to the microstructure aspect, both DED and HF-DED showed a similar primary microstructure, composed of oriented dendrites with interdendritic eutectics. In addition, the solidification sequence for Inconel 718 fabricated via DED was described, which showed a core-shell structure composed of oxides and MC-type carbides. Furthermore, in the aged condition, the γ'' and γ' morphology was characterized and significant quantity of γ'' and γ' (monoliths), γ'' - γ' duplets, and γ'' - γ' - γ'' triplet were observed. The volume fraction of γ'' and γ' estimated by TEM analysis (quantitative metallography) agreed with those estimated by synchrotron X-ray diffraction (Rietveld refinement).

HF-DED did not show a significant improvement in tensile strength for Inconel 718 at both room and elevated temperatures. However, HF-DED was able to reduce the yield strength anisotropy and allow the Inconel 718 DED to meet the yield strength requirements at both room and elevated temperatures. Nevertheless, neither the ultimate tensile strength nor Young's modulus were significantly enhanced by HF-DED, which was related to the final coarse grain and their stereology.

Therefore, it can be concluded that the main advantage introduced by HF-DED is its ability to reduce the material grain size and promote an almost isotropic tensile behavior, as well as its capacity to be coupled with robotic arms or CNC machines (i.e., HF-DED does not require a dedicated facility).

5.5 Supplementary Material

5.5.1 Effect of the post-deposition heat treatment on the microstructure of the Inconel 718 fabricated via arc plasma direct energy deposition.

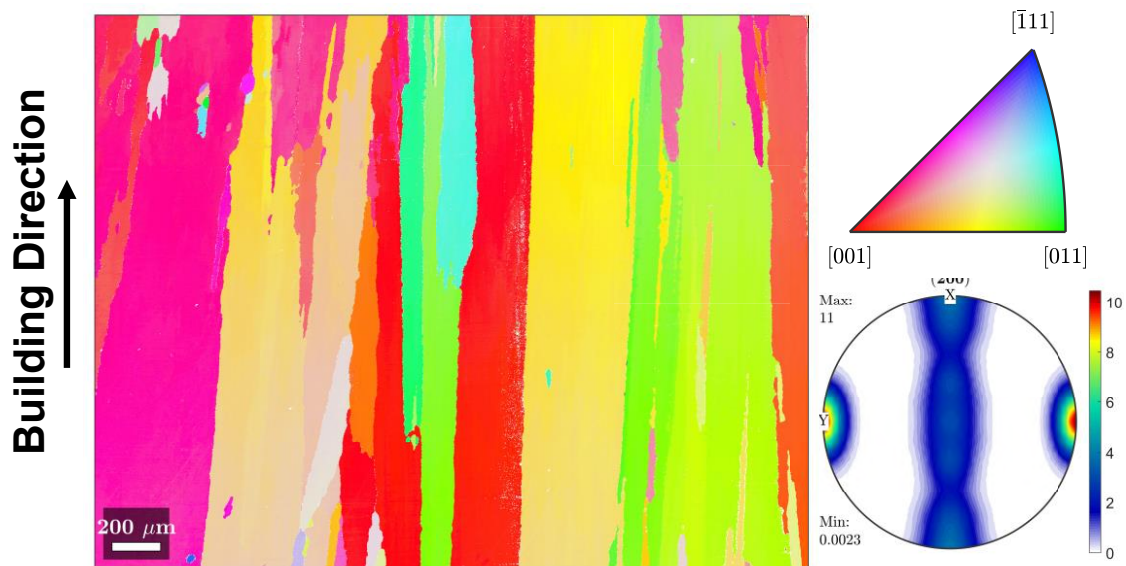
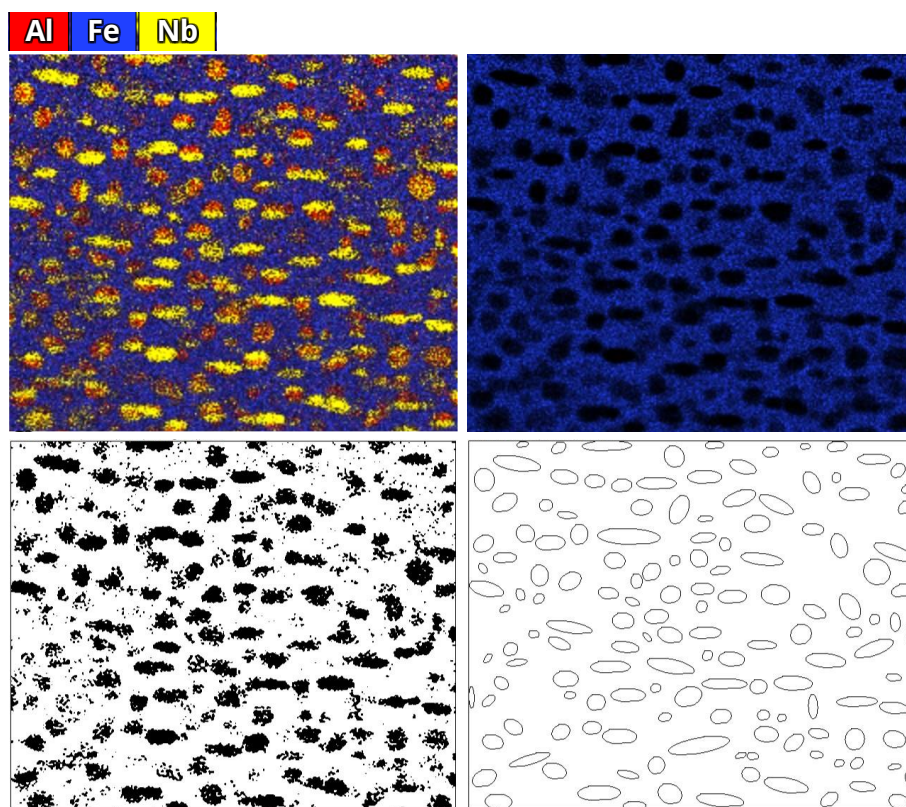


Figure 5.20 - Effect of post-deposition heat treatment on the microstructure of the Inconel 718 fabricated via arc plasma direct energy deposition.]



50 nm

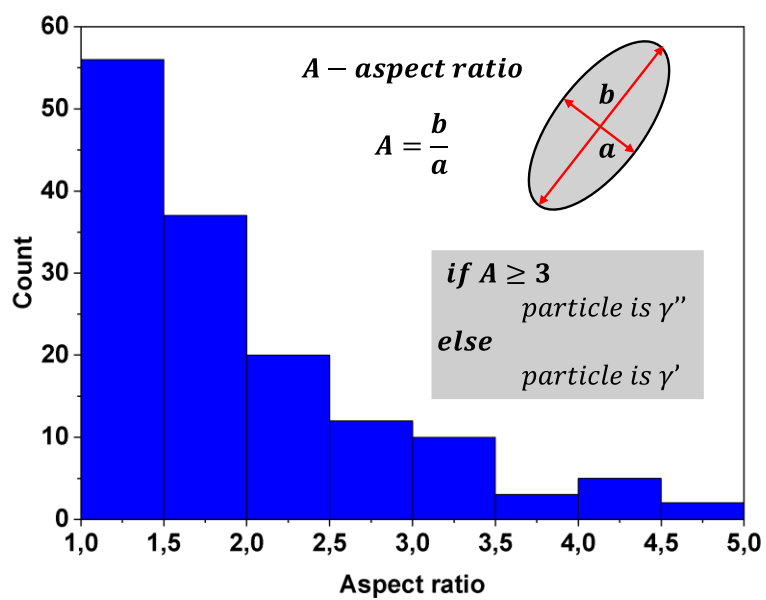


Figure 5.21 - Data-post processing of TEM analysis. An aspect ratio less than 3 was considered γ' .

5.5.2 Fractography

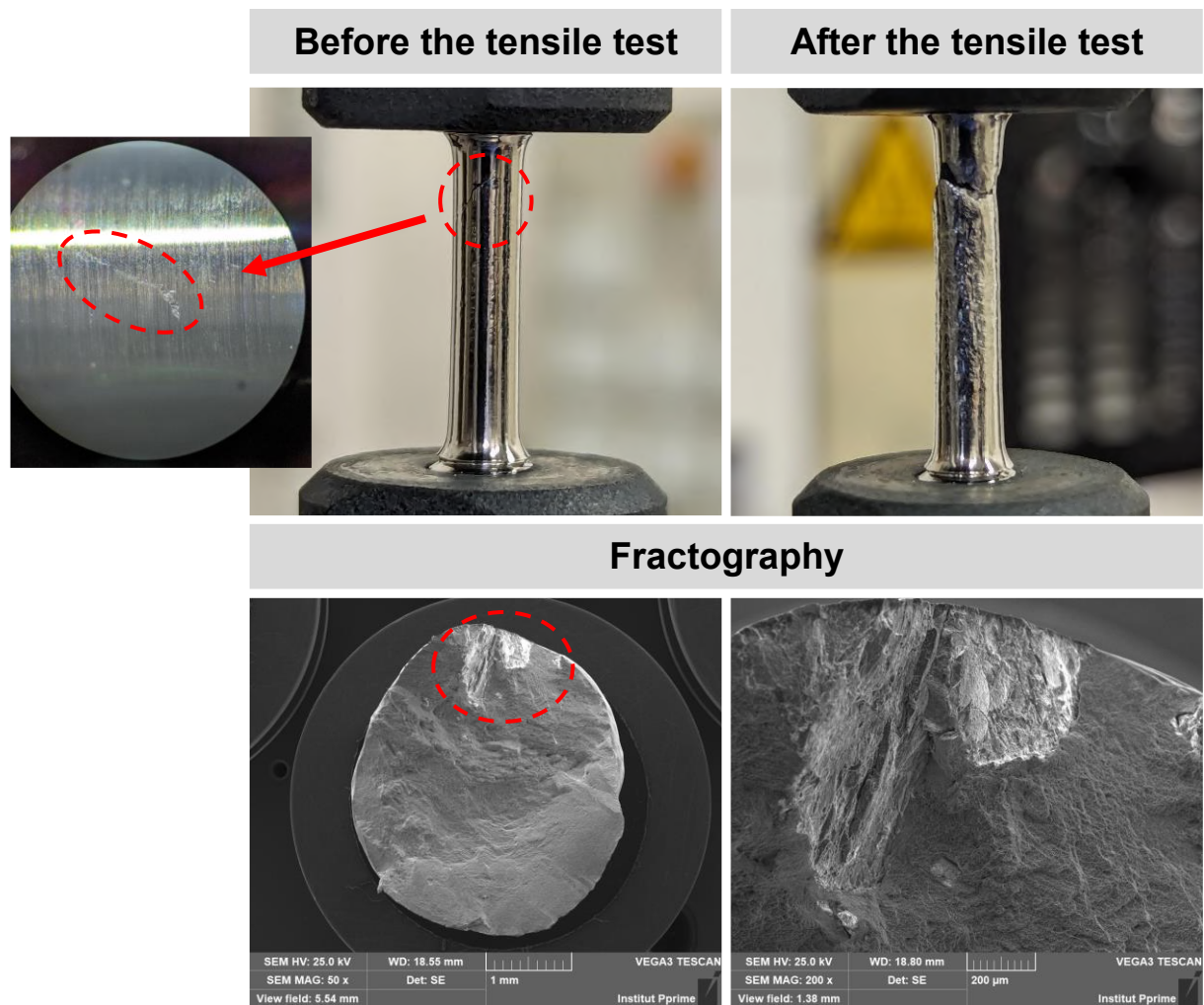


Figure 5.22 - 3D printing defect and its effect on material ductility and fracture aspects.

6 CONCLUSIONS

- The cold rolling system demands the highest loads, restricting the path planning flexibility; otherwise, hammering, peening, and forging (cold or hot) systems are more flexible and use less robust and more adjustable equipment.
- For the cold interlayer deformation, the grain size refinement occurred by the accumulation of strain deformation and static recrystallization during the subsequent deposition thermal cycle and/or post-deposition heat treatment. The interlayer hot deformation can drive in situ dynamic recrystallization and refining the top layer surface grains, which can also function as nucleation sites during the solidification of the next layer. In addition, the remaining deformed zone can also induce static recrystallizing during post-deposition heat treatment.
- Low to medium SFE alloys (e.g., AISI 316 stainless and Inconel 718) had the best response to interlayer (cold and hot) mechanical deformation.
- The use of FEM and a design of experiments to design the crown hammer radius and the process parameters, respectively, showed efficiency to obtain a significant effect on microstructure using HF.
- The main effect of the in situ interlayer hot forging results in the increase on the intergranular deformation (increase of dislocation density), which drives the static recrystallization during the post-deposition heat treatment.
- The proposed heat treatments resulted in superior mechanical response in relation to those previously present in literature. IN718 AP-DED partially met the AMS 5662 quasi-static mechanical properties requirements at room and elevated temperatures (25 and 650 °C).
- IN718 AP-DED microstructure was not changed by the proposed heat treatments (1050, 1100, 1142, and 1185 °C / 2 h), i.e., it is not possible to alter the crystallographic texture and grain size aspects of the IN718 AP-DED (gas metal arc) by typical printing + heat treatment routes.

- A long soaking time coupled with a higher homogenization temperature promotes to IN718 AP-DED a volume fraction of hardening phases similar to wrought material. In addition, the present work demonstrated that the heat treatments designed for IN718 AP-DED may differ from those commonly employed for cast (AMS 5383), wrought (AMS 5662), and PBF (ASTM F3055) materials due to its unique microstructure.
- In situ interlayer hot forging coupled with the proposed heat treatments are able to induce a fine (met the grain size requirement) and non-oriented microstructure for Ni-based superalloy fabricated by AP-DED.
- The grain size refinement of the in situ interlayer hot forging (HF) is attributed to the dynamic recrystallized zone generated due to localized deformation on the top surface of the just-deposited layer (i.e., material in elevated temperature). Additionally, this localized deformation also induces a non-recrystallized zone (remaining deformation), which can subsequently experience static recrystallization during reheating thermal cycles and/or post-deposition heat treatments.
- HF-DED was able to reduce the yield strength anisotropy and allow the Inconel 718 DED to meet the yield strength requirements at both room and elevated temperatures.
- The main advantage introduced by HF-DED is its ability to reduce the material grain size and promote an almost isotropic tensile behavior, as well as its capacity to be coupled with robotic arms or CNC machines.

7 FUTURE WORK

- Improve the in situ interlayer hot forging prototype by reducing its instability and enhancing its reproducibility.
- Develop similar HF system for non-axial deposition DED processes (e.g., PTA and Laser).
- Perform a phenomenological process parameters analysis, which addresses the changing in mechanical properties by temperature, the thermal profile during deposition, recrystallization maps, printability, and deposition stability.
- Analyze the effect of top-layer surface flattening induced by HF on susceptibility to lack of fusion.
- Compare the effect of several interlayer mechanical deformation systems on quasi-static mechanical properties of the IN718 AP-DED.
- Study the effect of banded microstructure developed by HF-DED (coarse and fine grains) on the fatigue and creep resistance of IN718 AP-DED.
- Develop a finite element model to simulate the deformation aspects during deposition and the effects of HF on residual stress.
- Evaluate the effects of HF and the proposed heat treatments on the corrosion resistance of IN625 and IN718 AP-DED.
- Extend the present study to different classes of alloys, particularly those with low to medium stacking fault energy (e.g., austenitic stainless steels).

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