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BSc in Material Engineering Sciences

# Additive Manufacturing and Characterization of Three-dimensional Anepeptic Structures

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## **Additive Manufacturing and Characterization of Three-dimensional Anepectic Structures**

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## ABSTRACT

In recent years there has been an increase of interest in 3D anepectic structures. An anepectic structure is a metamaterial with auxetic behaviour (Negative Poisson's Ratio (NPR)) and negative coefficient of thermal expansion (CTE). This work intends to manufacture and characterize a 3D anepectic structure using materials with positive CTE.

A known auxetic structure was used, in which a second material was incorporated to obtain a negative CTE. To achieve the design used in this work, mechanical and thermal finite element simulations were performed to preview the thermomechanical behaviour.

The 3D anepectic structures were produced by additive manufacturing using polyvinyl alcohol and Nylon. Two models with different designs were developed. Its characterization necessitated the development of new methodologies to determine the Poisson's ratio (PR) and CTE. One of the models obtained the intended anepectic behaviour. Furthermore, there was a correlation between PR values and the number of cells in the structure.

Until this day, this is the first published report of a manufactured and characterized 3D anepectic structure, making this work innovative in the area of metamaterials.

**Keywords:** Anepectic, Auxetic, Negative Coefficient of Thermal Expansion, Negative Poisson's Ratio, Additive Manufacturing, 3D Structures



## RESUMO

Nos últimos anos tem existido um aumento de interesse em estruturas anepéticas 3D, isto é, auxéticas (Coeficiente de Poisson negativo (NPR)) e com coeficiente de expansão térmica (CTE) negativo. Este trabalho pretende manufacturar e caracterizar uma estrutura anepética 3D, constituída por materiais com CTE positivos.

Foi utilizado uma estrutura auxética conhecida, no qual se incorporou um segundo material com o intuito de obter um CTE negativo. Para obter esse *design*, realizou-se simulação mecânica e térmica de elementos finitos.

As estruturas anepéticas 3D foram produzidas por fabricação aditiva utilizando PVA e Nylon. Foi desenvolvido dois modelos com diferentes *designs*. Para a sua caracterização foi necessário desenvolver metodologias novas para a determinação do PR e do CTE. Sendo que um dos modelos obteve o comportamento anepético pretendido. Para além disso, verificou-se uma correlação entre os valores de PR e o número de células na estrutura.

Até à data, esta é a primeira publicação de uma estrutura anepética 3D manufacturada e caracterizada. Tornando este trabalho inovador na área dos metamateriais.

**Palavas chave:** Anepético, Auxético, Coeficiente de expansão térmica negativo, Coeficiente de Poisson negativo, Fabricação aditiva, Estruturas 3D



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## ACRONYMS

<b>2D</b>	Two dimensional.
<b>3D</b>	Three dimensional.
<b>ABS</b>	Acrylonitrile butadiene styrene
<b>CLTE</b>	Coefficient linear of thermal expansion
<b>CTE</b>	Coefficient of thermal expansion
<b>DER</b>	Double-Elliptic Ring
<b>FFF</b>	Fused filament fabrication
<b>FE</b>	Finite element
<b>NPR</b>	Negative Poisson's ratio
<b>NTE</b>	Negative thermal expansion
<b>PPR</b>	Positive Poisson's ratio
<b>PR</b>	Poisson's ratio
<b>PVA</b>	Polyvinyl alcohol



## SYMBOLS

<b>a, b, c</b>	Inner radius of ellipse
<b>L</b>	Length
<b>m</b>	Form factor of the elliptic rings in the xy plane
<b>min</b>	Minute
<b>mm</b>	Milimeter
<b>mm/s</b>	Milimeter per second
<b>MPa</b>	Megapascal
<b>N</b>	Newton
<b>n</b>	Form factor of the elliptic rings in the yz plane
<b>q</b>	Thickness of the elliptic ring
<b>T</b>	Temperature
<b><math>T_g</math></b>	Glass transition temperature
<b><math>t_0</math></b>	Width of the elliptic rings
<b><math>t_1</math></b>	Width of the inner material
<b>°</b>	Degrees (angle)
<b>°C</b>	Degrees Celsius
<b><math>\alpha_L</math></b>	Coefficient linear of thermal expansion in L direction
<b><math>\varepsilon_L</math></b>	Strain along the load direction
<b><math>\varepsilon_T</math></b>	Strain along the transverse direction

$\Phi$	Angle between the center of the inner material and its edge
$\nu_{LT}$	Poisson's ratio

## MOTIVATION AND OBJECTIVES

Over the past few years, there have been an increased interest in creating three-dimensional (3D) structures with a negative Poisson's ratio and negative coefficient of thermal expansion. Creating a structure that combines these two properties, called anepectic, has been taking its first steps. These structures could be relevant in biomaterial applications, aerospace, sensors, and composite materials. So far, the anepectic behavior of 3D structures has only been proven through analytical and finite element analysis.

Thus, based on the auxetic elliptic double ring structure, this dissertation aims to design, manufacture, and characterize mechanically and thermally the first non-virtual 3D anepectic structure. The main points of innovation are the design of a new anepectic structure and the experimental characterization of an anepectic 3D structure, proceeding to validation of behaviour through mechanical and thermal tests. To design the structure, finite element analysis will be performed, and for manufacturing the anepectic structure, it will be necessary to use additive manufacturing.

It is also intended to start understanding how the design parameters influence the thermal and mechanical behaviour of the structures.

# INTRODUCTION

## 1.1 Metamaterial

The designation metamaterial, from Greek "μετα" (beyond) and the Latin "materia" (material), refers to a material whose properties come essentially from its structures and not its composition [1]-[3]. Due to their design, metamaterials can have properties that are unusual in conventional materials [2]-[5].

Bose developed the first metamaterial for optics applications, but since then, acoustic and mechanical metamaterials have also been developed [6], [7]. Mechanical metamaterials are a subclass that aims to achieve specific mechanical properties due to their structure geometry [2], [7].

The possibility of designing metamaterials with tailor made properties beyond the conventional material is desirable to optimize to the pretended application. These metamaterials are composed of conventional materials with typical values for these properties. Among the unusual ranges of properties obtained in metamaterials stand out: negative Poisson's ratio (NPR), negative thermal expansion (NTE), negative compressibility, among others [2]. Studies of combining two or more negative properties in the same metamaterial only emerge in the last decade [2].

## 1.2 Auxetics

The elastic constant Poisson's Ratio (PR,  $\nu_{L,T}$ ) is defined by:

$$\nu_{L,T} = -\varepsilon_T / \varepsilon_L \quad (1.1)$$

where  $\varepsilon_L$  and  $\varepsilon_T$  are the strains along the load direction and the transverse direction, respectively [1], [8]. Conventional materials have a positive PR, which implies that, under

uniaxial compression, the material will expand along the orthogonal directions of the applied force, Figure 1.1 - a) [1], [9].

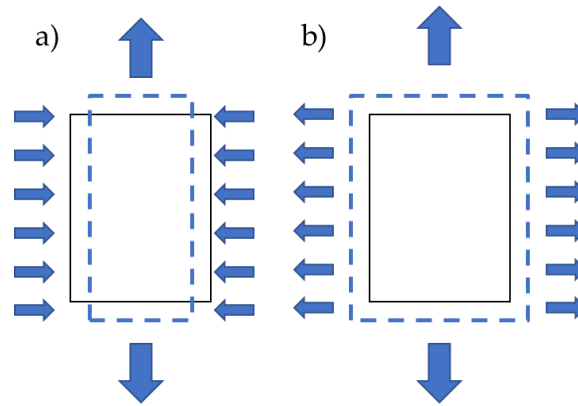


Figure 1.1 - a) Material with positive PR. b) Material with NPR.

A NPR structure is denominated auxetic and is characterized by shrinking (expanding) transversely when uniaxially compressed (tensioned), Figure 1.1 - b) [8], [10]. The geometry of the auxetic metamaterial is crucial to obtain this characteristic [11].

There are several naturally occurring structures that manage to reach a NPR; examples of this are some zeolites [12], [13], graphene monolayers [14], [15], and silicon dioxide [16], [17]. There is a high demand for artificial auxetic structures, both macro and micro scale. Among them, the following categories stands out: re-entrant models [18]–[20], double arrowhead [21]–[23], interconnected star models [3], [24], [25], models with rotating units [26]–[28] and ring-rod assembly [11], [29], [30]. The last structure will be studied in more detail throughout this dissertation.

Metamaterials with NPR are gaining interest in the biomedical industry. One of the most significant possibilities is their incorporation in stents [31], [32]. Dental floss [33] and smart bandages [34] are other applications in biomedicine. In addition, auxetic metamaterials have a wide range of applications: aerospace, military, sensors, filters, and textile industry [1], [10], [33], [35].

### 1.3 Coefficient of thermal expansion

Most solids have a positive coefficient of thermal expansion (CTE), i. e., expand upon raising the temperature [3], [9]. This phenomenon is due to the increase in the interatomic bond length as a result of the increased atomic and molecular vibrations [36], [37]. There are liquids and solids with NTE, usually associated with phase transformations, among them water [38], [39], some oxides [40]–[42], zeolites [43], [44] and some polymers [45], [46]. The mechanisms subjacent to this behaviour can be classified into three categories: flexible network, atomic radius contraction, and magnetovolume effect [47], [48].

It is possible to obtain metamaterials with NTE, although their constituents have positive CTE values. That is due to the geometry and the use of two materials with different positive CTE [49]–[51]. These structures can be classified as either bending-dominated structures whose walls bend and/or rotate due to concavities in the geometry, or to being bimaterial that will bend the walls or block; or, alternatively, as stretching-dominated structures, where the walls are made of two materials with different CTE in the form of triangles or tetrahedrons [4], [52], [53].

Materials may expand in isotropic or anisotropic form. In isotropic materials, the expansion occurs by the same extent in any direction. However, when the extent varies with the direction considered, the material is defined as anisotropic. For the last ones, the coefficient linear of thermal expansion (CLTE,  $\alpha_L$ ) is used and defined by:

$$\alpha_L = \frac{1}{L} \frac{\partial L}{\partial T} \quad (1.2)$$

where L is the length of the direction studied, and T is the temperature. [48], [54], [55]

The use of materials with NTE in composites allows them to act as thermal expansion compensators, making the composite a zero CTE. In addition they can reduce thermal stresses [47], [56], [57]. Metamaterials with NTE also have applications in the areas of electronics [48], aerospace [51], [55], civil engineering [55], and dental fillings [48], [56].

## 1.4 Anepectic

The anepectic designation, created by Raminhos et al. [58], applies to a material that simultaneously features NPR and NTE. Despite the scientific interest in recent years around this topic, the development of metamaterials with anepectic behavior is still beginning. [5]

Previous studies of anepectic structures focused primarily on two-dimensional (2D) structures, and most of these studies are finite element (FE) analysis and/or analytical analysis. The main 2D anepectic meshes studied until now are triangles connected [59], reentrant stars [3], [4], reentrant triangles [9], [60] and hybrid honeycomb [4], [61]. There are 2D meshes with experimental work: chiral [62] and stars reentrants [52], [58].

According to the literature, the first 3D metamaterial with anepectic properties was developed by Ai et al. [63]. The authors used FE method to demonstrate its anepectic properties. After Ai et al., between 2020 and 2021, more anepectic structures were designed and analyzed through modeling by FE and/or analytical analysis [50], [53], [64]–[69]. So far, none of these structures have carried out thermal and mechanical tests to validate their anepectic properties experimentally. Some of these structures are represented in Figure 1.2.

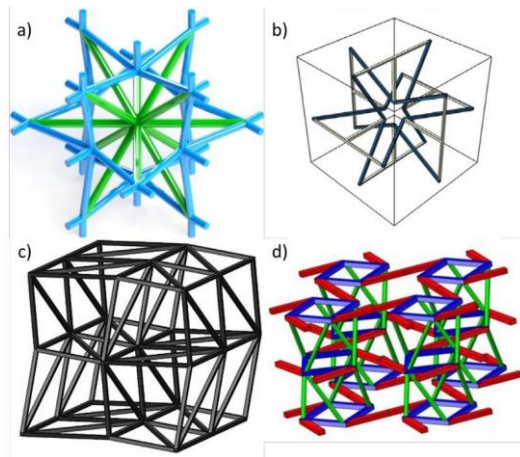


Figure 1.2 - Unit cell developed by Sarvestani et al. [67]; b) Unit cell re-entrant honeycomb by Peng et al. [66]; c) Structure design by Chen et al. [64]; d) Structure developed by Fu et al. [53].

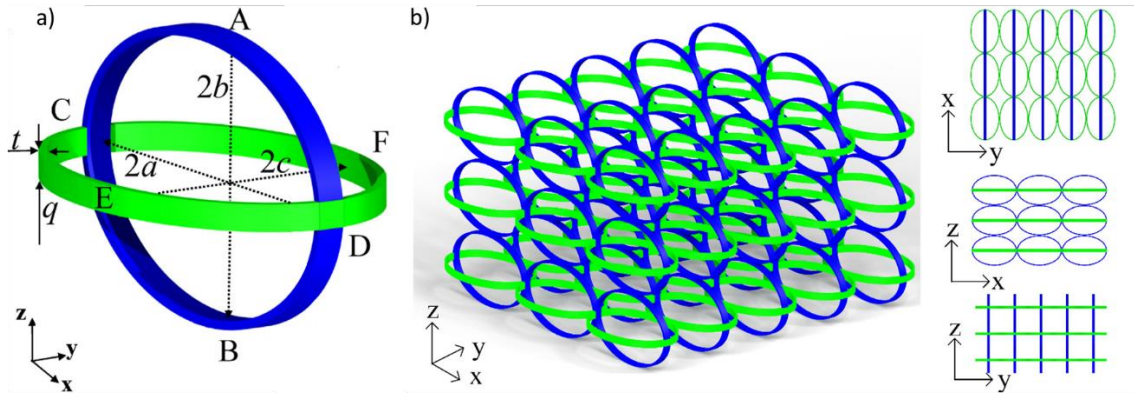
One of the most discussed applications is dental filling, because the materials used for this purpose undergo a sudden temperature variation due to food and drink and must withstand the forces of chewing [53], [63]. Another application is in satellites, as these suffer variations greater than 200°C between exposure and concealment from sunlight [9], [64]. Beyond that, it can be used in the aeronautic field [55]; antennas [53]; solar panels [53], [63], [64]; smart sensors [52], [60]; composite materials [52]; thermal and electrical components [52] and filters [63].

## 1.5 Double-Elliptic Ring (DER) structure

The design of an anegetic structure has been usually based on an auxetic structure, suitably modified to also acquire a NTE [3], [60]. The current work is based on a structure created by Wang et al., called latitude-and-longitude-inspired double-elliptic-ring structure (DER) [29]. The authors obtained a partially auxetic 3D metamaterial with the Poisson's ratio being positive in one direction and negative in the other.

The unit cell of the DER structure is composed of two orthogonally oriented ellipses, Figure 1.3 - a). When there is a compression in the z direction, the blue ellipse will expand in the x direction, i.e., points C and D will move apart. As a result, in the y direction, the green ring will contract, and points E and F will approach. This way, the unit cell has positive PR in the x direction and NPR in the y direction [29].

One of the structures studied by Wang et al. is represented in Figure 1.3 - b), obtained by replicating the unit cell on the three axes. The authors concluded that it exhibits a partially auxetic behavior through analytical analysis, FE analysis, and mechanical tests [29], [70].



## 1.6 Research methodology

In the literature, it is common to use computer simulation to study the behaviour of possible anepectic structures [52], [63], [65]. The use of this technique through the FE method allows understanding the behaviour of these structures when applying forces or varying the temperature, as was done by Sarvestani et al. [67]. In addition, computer simulation software allows the design of the structures to simulate their behaviour and optimize their parameters.

Many of the methods of producing metamaterials are based on techniques of additive manufacturing due to the advantage of manufacturing complex geometries in a fast and cheaper way [10], [71], [72]. In addition, it can produce structures with a high number of different materials [10], [72]. The techniques of 3D printing can be summarized in the following steps: design of the model in modelling software, dividing the model into layers (slicing), and finally printing [71].

There are several 3D printing techniques, but the most predominant are stereolithography, selective laser sintering, and fused filament fabrication (FFF or FDM) [71]. The FFF technique will be used to produce the structures studied in this work. It consists of the extrusion of a plastic filament through a heated nozzle. This deposition is layer by layer [71], [72]. Several parameters will influence the final properties of the structure, namely: the nozzle diameter, the extrusion flow rate, deposition speed, extrusion temperature, filament brand, among others [72], [73].

This work aims to produce an anepectic 3D structure inspired by the DER and validate its properties through mechanical and thermal experiences.



# MATERIALS AND METHODS

## 2.1 Structure design and printing equipment

The software used to model all the structures created in this work was SolidWorks 2020. A compromise between a reasonable printing time and the mitigation of boundary effects was achieved by selecting structures made from  $3 \times 3 \times 3$  unit cells for the majority of the experimental tests performed.

An Ultimaker 3<sup>TM</sup> 3D printer was used; this model uses a FFF technique and is capable of dual extrusion, with a nozzle diameter of 0.4 mm. All the prints had 100% infill to produce a solid structure, and the layer height was 0.15 mm. The slicer software used was Ultimaker Cura 4.8. After the print, all structures are stored in vacuum desiccators to prevent humidity absorption.

## 2.2 Finite element method

Thermal and mechanical behaviours were simulated through finite element method using SolidWorks 2020. A solid mesh type was used consisting of 8.925 mm tetrahedral mesh element size. For both mechanical and thermal simulations, a roller/slider in the yz, xz and xy plane, as shown in green in Figure 2.1, was used. For the mechanical simulation, a force of 10 N per item (pink arrows in Figure 2.1) was applied. For the thermal simulation, the temperature was raised from 0 °C to 200 °C.

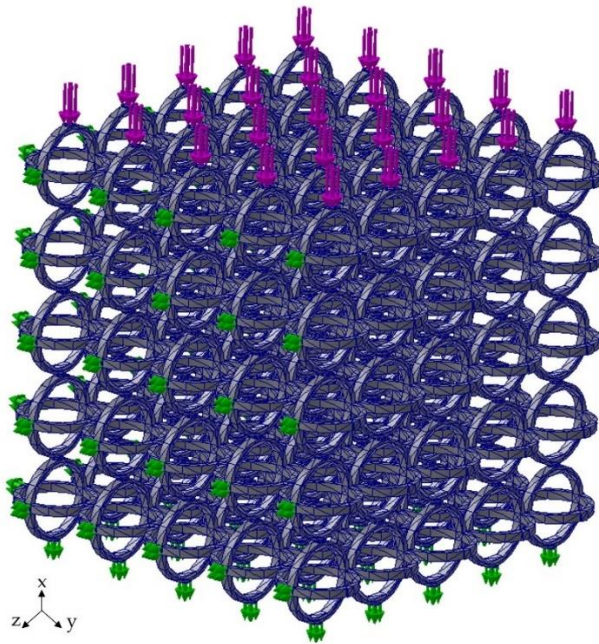


Figure 2.1 - Example of a mechanical simulation by finite element method.

## 2.3 Filament characterization

The filaments used to fabricate the different structures were polyamide (Nylon, PA6/66, Ultimaker) and polyvinyl alcohol (PVA, Ultimaker). For the mechanical characterization, both compression and tensile tests according to ASTM Standard D695-15 and ASTM Standard D638-14, respectively, were performed. All the test specimens were printed both vertically and horizontally, with no walls, 0.15 mm layer height and 100% infill. The compression tests were filmed with the intent of calculating the PR of the bulk materials from the recorded images.

## 2.4 Poisson's Ratio determination method

To determine the PR, the mechanical tests of the DER structures and the filament characterization were performed with a Shimadzu AG-50kNGT universal testing machine. All tests were performed at 1mm/min using a load cell of 50 kN. To determine  $\nu_{z,x}$  and  $\nu_{z,y}$  the experiments were filmed with two cameras simultaneously; one to film the xz plane (Galaxy S8 SM-G950F) and the other (Canon EOS 750D with 18-55mm lens) the yz plane. These cameras were set in parallel to the respective plane and centered to the middle cell. This was the best way to mitigate the parallax effect, although it was impossible to completely eliminate the effect due to the size of the structure.

To obtain the distances between reference points a primary spray was applied (Fine Surface Primer (L) for Plastic & Metal (White) from TAMIYA), then red nail polish (from

MyLabel) and matte effect (Matte Effect Top Coat from KIKO MILANO). The reason behind the application of the matte effect was the removal of the light reflection from the nail polish. This light reflection appears as white points in the videos changing the center points throughout all the experiments.

For the calculation of PR, a MATLAB code was developed that processes all the raw data obtained from the mechanical tests (videos, stroke, and force data). The output given by the code is the stress-strain curve and the PR throughout the test. The developed algorithm is presented in Appendix A with the corresponding explanation.

An example of the raw data obtained by the developed MATLAB code is depicted in Figure 2.2. Due to camera resolution limitations, a vast variation is observed at the beginning of the experiment. For that reason, the initial data has been discarded. Furthermore, the data displays noise throughout the trial on the right side of the figure. To reduce the noise, a binomial fit (usually, polynomial order five) was performed using the OriginPro 2018 software, with the x axis being the relative displacement and the y axis the average distance between points of the pretended direction. Afterwards, using the fitting distance equations, it becomes possible to determine the respective strains and, lastly, the PR using the equation (1.1), obtaining the final data corresponding to the red line of Figure 2.2.

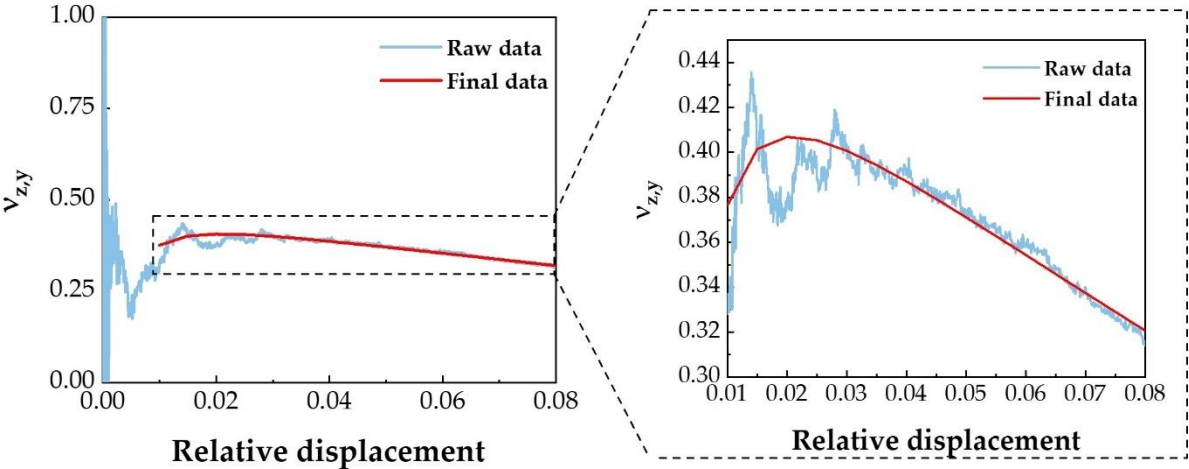


Figure 2.2 - Comparison between the PR of the raw data obtained by MATLAB code and applying a binomial fit of the distance data.

## 2.5 Coefficient of thermal expansion determination method

The set-up in Figure 2.3, with glycerol ( $\text{HOCH}_2\text{CH}(\text{OH})\text{CH}_2\text{OH}$ , LABCHEM, 100%) in a transparent container with flat walls (to reduce image distortion) was used to determine the CTE of the structures. To register the temperatures throughout the experiment, three type-k thermocouples (Figure 2.3 - 6) were used, connected to a data logger (Pico TC-08 and PicoLog 6 Software) (Figure 2.3 - 1). Two smartphones (Galaxy S8 SM-G950F and Galaxy ACE SM-G358FX) were used to take pictures every minute, one to register the xz plane and the other the yz plane (Figure 2.3 - 2). To minimize parallax effect, the cameras were aligned with the middle cell.

Since the samples floated in glycerol, supports were produced to immobilize the structure. These supports were made of acrylonitrile butadiene styrene (ABS) because its  $T_g$  is above  $90\text{ }^\circ\text{C}$  and the experience was limited to  $80^\circ\text{C}$  [74]. Stainless steel wires were placed to not allow movement or rotation in any of the axes while trying to restrict the structure as little as possible (Figure 2.3 - 5).

The temperature during the experiment was increased from ambient temperature up to  $80\text{ }^\circ\text{C}$ . A hot plate and a magnetic stirrer were used (Figure 2.3 - 3), to obtain the best temperature uniformity throughout the container during the test.

Before this experiment, similarly to the PR method, red dots were marked on strategic places, and an algorithm was created in MATLAB for the CTE calculations, which can be found in Appendix B.

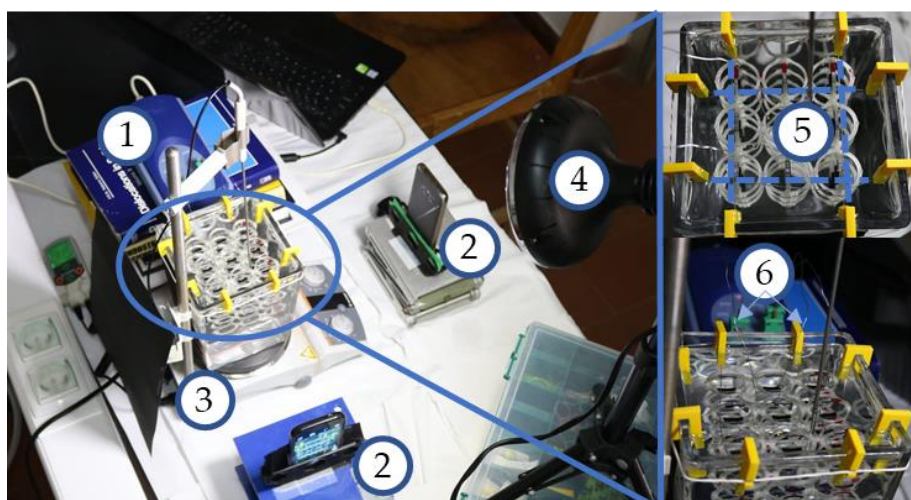


Figure 2.3 - Set-up of the thermal experience. 1: PicoLog 6; 2: Smartphones; 3: Heat plate; 4: Spotlight; 5: Stainless steel wires; 6: Thermocouples.

## RESULTS AND DISCUSSION

### 3.1 Filament Characterization

The two materials chosen for the current work are Nylon and PVA since it was reported in previous works that their combinations could obtain the anepectic behaviour pretended. This is due to having a similar but different stiffness, and a distinct CTE. [49], [58], [75]

Mechanical tests were performed to obtain the mechanical properties of the filaments used, Nylon and PVA. Appropriate specimens were printed according to two different printing orientations (horizontal and vertical) to understand the relevance of the printing orientation. The average stress-strain curves of the tensile and compression tests are represented in Figure 3.1. All the stress-strain curves are represented in Appendix C.

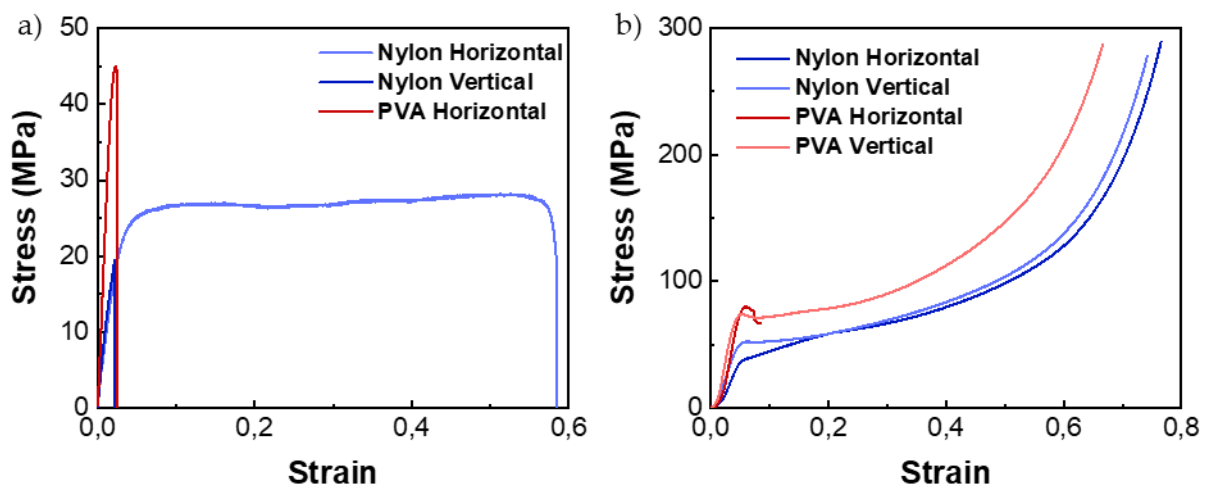


Figure 3.1 - Stress-strain curve of the sample average of Nylon and PVA in different orientations a) Tensile test b) Compressive test.

Table 3-1 shows the results of the Young's modulus, PR and fracture strain of the printed specimens. The three samples of PVA with vertical printing orientation fractured during installation on the testing machine, prior to the tensile test; this shows the fragile behaviour of these samples whenever printing vertically. That is due to a poor adhesion between layers when printing with the FFF technique. The Nylon samples present the same tendency, showing a ductile behaviour for the horizontal orientation and fragile behaviour for the vertical orientation prints.

Table 3-1 - Mechanical properties Nylon and PVA in horizontal and vertical printing directions.

		<b>Young's Modulus (MPa)</b>	<b>PR</b>	<b>Strain of fracture</b>
<b>Nylon</b>	Horizontal	784.49	0.34	0.555
	Vertical	855.82	0.30	0.022
<b>PVA</b>	Horizontal	2484.34	0.27	0.027
	Vertical	-	0.22	-

These results show the tremendous impact of the printing orientation on the mechanical behaviour of the samples due to the bad adhesion between layers.

The glass transition temperature ( $T_g$ ) and the CTE of the Ultimaker Nylon and PVA filament have been determined by Raminhos et al. [58]. These results are presented in Table 3-2. It is essential to note out that these values may not be exact for this work because another batch was used, although the supplier is the same. Furthermore, based on the literature, the filaments of the same brand have values similar to those considered in this work [76], [77].

Table 3-2 -  $T_g$  and CTE of Nylon and PVA (obtained from Raminhos et al.) [58].

	$T_g$ (°C)	CTE ( $\times 10^{-6} \text{ }^\circ\text{C}^{-1}$ )
<b>Nylon</b>	35	166.47
<b>PVA</b>	35	28.35

## 3.2 Structure design

### 3.2.1 Finite element analysis

To achieve the anepectic behaviour, some modifications to the DER structure were done. Parts of the structure were replaced with another material of higher CTE, with the intend of achieving a thermal expansion behaviour similar to the mechanical compression behaviour observed during mechanical testing by Wang et al. [29].

Four different cell arrangements were simulated through finite element (FE) method to predict the corresponding PR and CTE values; the characteristics of three of these arrangements are presented in Appendix D. The unit cell of Figure 3.2 was chosen as the object of study due to the exhibition of anepectic behaviour. It is observed that the unit cell of Figure 3.2 obtained a NTE in axis x and z due to the expansion of the nylon (in red).

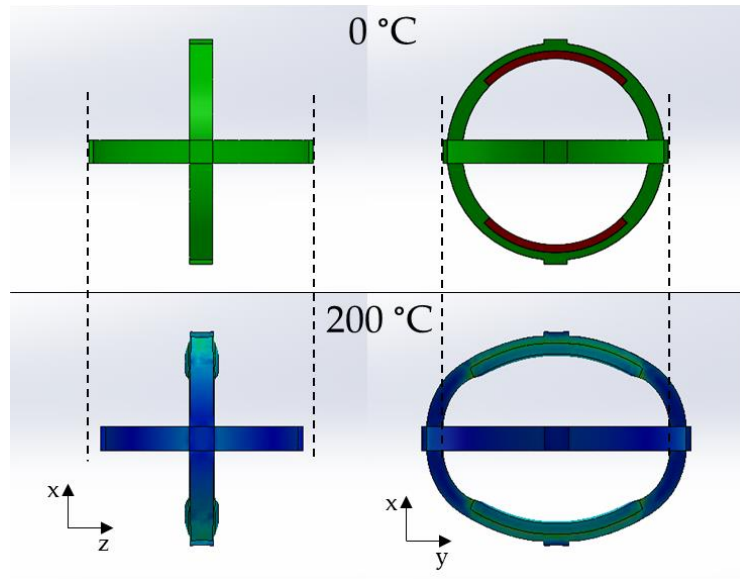


Figure 3.2 - Thermal expansion simulation of a cell, shows the NTE in x and z axis and positive CLTE in y axis.

The green material is PVA (lower CTE) while in red is the Nylon material. The design parameters used to obtain this cell was the same of the Wang et al [29] specimen S2, with the angle between the center of the inner material and its edge of  $45^\circ$  and the inner material width of 1 mm.

### 3.2.2 Design parameters

Due to the anepectic behaviour previously predicted by the FE analysis, the unit cell shown in Figure 3.3 was chosen as the basis for the remainder of this work. The  $a$ ,  $b$ , and  $c$  geometrical parameters correspond to the inner radius of both ellipses, as seen in Figure 3.3. Wang et al. defined  $m=a/b$  and  $n=a/c$  as the shape factor of the elliptic rings in the  $xy$  plane and the  $yz$  plane, respectively [29]. The thickness of each elliptic ring is defined as  $q_{ab}$  and  $q_{bc}$  for each elliptic ring. The width of the elliptic rings is defined as  $t_0$  and the width of the inner material (colour red in Figure 3.3) is  $t_1$ . The relation between these two parameters is defined as  $T (t_1/t_0)$ . The parameter  $\Phi$  is the angle between the middle of the inner material and its edge.

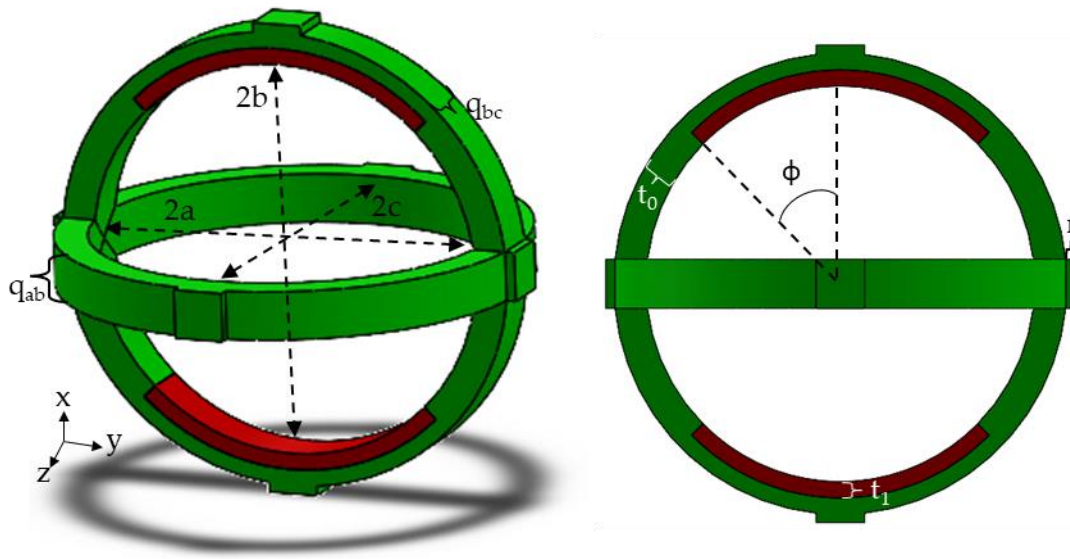


Figure 3.3 - Unit cell parameters.

### 3.2.3 Study of parameter $\Phi$

The influence of the angle  $\Phi$  in the anepectic behaviour was studied via FE simulations. The structure used was a  $5 \times 5 \times 5$  cell structure with the design parameters shown in Table 3-3.

Table 3-3 - Design parameter of the study of  $\Phi$ .

<b>a</b>	<b>m</b>	<b>n</b>	<b>q<sub>ab</sub></b>	<b>q<sub>bc</sub></b>	<b>t<sub>0</sub></b>	<b>T</b>	<b>r</b>
(mm)			(mm)	(mm)	(mm)		(mm)
12	1	1	3	3	2	0.5	0.5

The PR and the CTE of the simulated unit cell results are shown in Figure 3.4. The angle  $\Phi$  varied between  $5^\circ$  to  $75^\circ$  with a step of  $5^\circ$ . To determine the PR a 250 N of load was applied and for the CTE a variation of  $200^\circ\text{C}$  was used.

As shown in Figure 3.4 a), the parameter  $\Phi$  doesn't influence the PR obtaining an average value of -0.67 and 0.71 for  $v_{z,x}$  and  $v_{z,y}$ , respectively. This result was expected since PVA and Nylon have similar stiffnesses.

In Figure 3.4 b), the CLTE  $\alpha_x$  tends to achieve more negative values until it reaches a plateau between  $35^\circ$  and  $60^\circ$ , stabilizing at an average value of  $-4.8 \times 10^{-5} \text{ }^\circ\text{C}$ . From then on, it increases until the last value studied. It is also relevant to indicate that CLTE value for  $5^\circ$  is positive, revealing that it doesn't have enough Nylon to reach negative CLTE in the x direction.

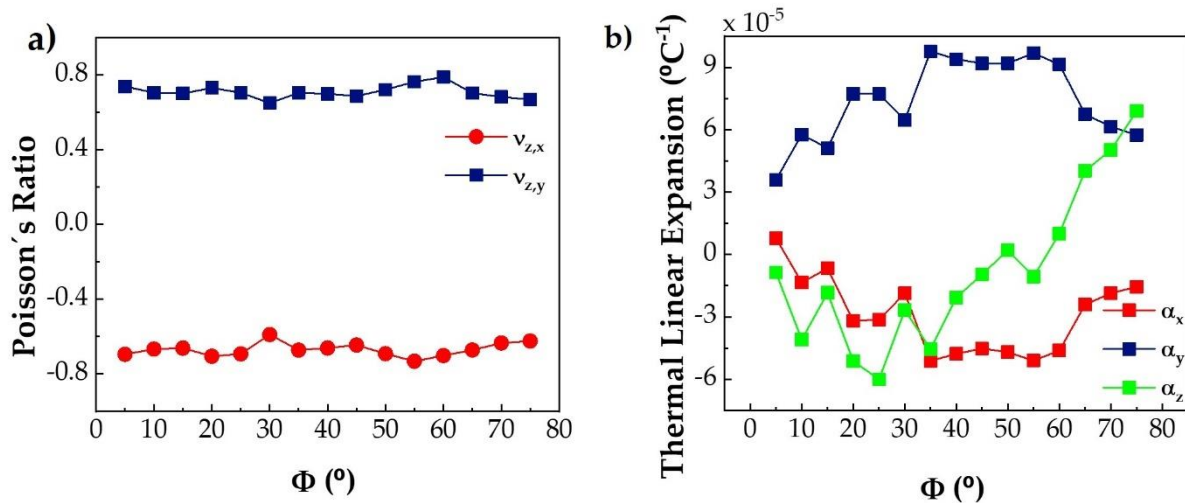


Figure 3.4 - Study of parameter  $\Phi$  a) PR; b) CTE.

Concerning  $\alpha_y$ ; it has the same tendency of  $\alpha_x$ ; with symmetric behaviour, although it only has positive values. This similar tendency shows an inverse relationship between the deformation of the diameter  $2b$  and  $2a$ .

For the third direction,  $\alpha_z$  achieved negative values until  $\Phi = 45^\circ$ , beyond which it shows increasing values until the end. These results indicate that the CTE of this structure using the parameter  $\Phi$  is tuneable, making structures with negative CLTE on x direction, z direction, or even on both directions simultaneously possible.

### 3.3 Manufacturing

To manufacture the structure with PVA and Nylon, optimizing printing parameters and changing some of the initial design parameters ( $q_{ab}$ ,  $q_{bc}$ , and  $t_0$ ) was necessary. Even after all the changes made, at least one of the rings frequently broke during the printing operation. This phenomenon happens due to the accumulation of material throughout the print in the zone of the overhang. At a particular time, the nozzle hits this accumulation and breaks the ring, even with different printing parameters (printing temperatures, velocities, z hop, and retraction) and design parameters. Appendix E summarizes most of the printing conditions attempted.

The solution found was to separate the structures into parts and assemble them after printing. This way, the printing stage went from 3D to 2D, mitigating all the previous problems caused by the overhang. The structure was broken down into three different basic building parts (design in Appendix F) in order to assemble the final structure (simple example in Figure 3.5). For this assembly, PVA-based white glue (Axton) was used, chosen to limit the influence on the thermomechanical properties of the structures.



Figure 3.5 - Example of an assembly.

Two structures, henceforth called model 1 (Figure 3.6 a) and model 2 (Figure 3.6 b), were manufactured; the difference between them consisting in the incorporation of Nylon in both rings in model 2, while in model 1 only the horizontal rings possessed Nylon inserts. Supports (in blue) were created to ensure the structure was as aligned as possible, while the PVA-based glue solidified. Table 3-4 shows the design parameters used in the two models produced with a  $3 \times 3 \times 3$  unit cell.

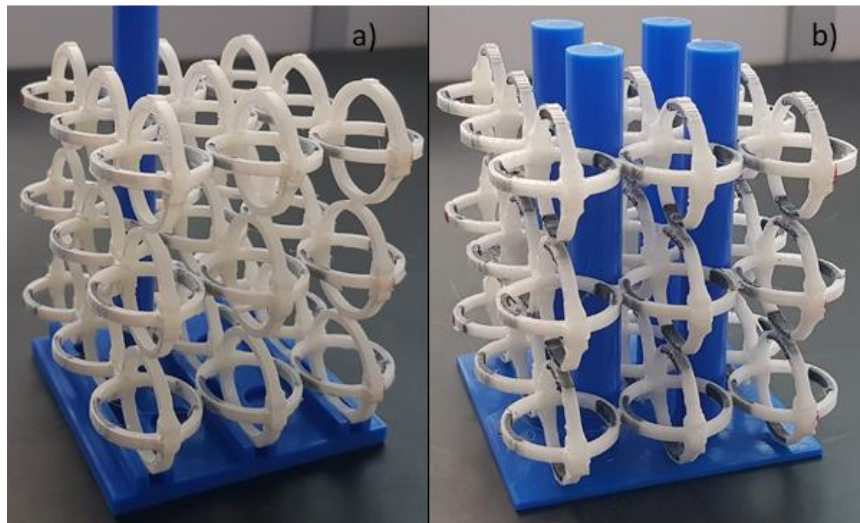


Figure 3.6 - a) Model 1; b) Model 2. PVA is the white material while Nylon is in black.

Table 3-4 - Design parameters of the models.

Parameters	<b>a</b>	<b>m</b>	<b>n</b>	<b>q<sub>ab</sub></b>	<b>q<sub>ac</sub></b>	<b>t<sub>0</sub></b>	<b>T</b>	<b>r</b>	<b>Φ</b>
Units	mm			mm	mm	mm		mm	°
<b>Model 1</b>	12	1	1	3	3	2	0.5	0.5	30
<b>Model 2</b>	12	1	1	3	3	2	0.5	0.5	30

### 3.4 Poisson's Ratio

#### 3.4.1 Printed unit cell vs assembled unit cell

A study was carried out to check that the use of PVA-based glue does not significantly affect the mechanical properties of the cell. Integral unit cells (without assembling) were printed with two different printing orientations and submitted to compression. The corresponding stress-strain curves are shown in Appendix G. In Figure 3.7 is possible to observe that fracture occurred between printing layers, irrespective of the printing orientations. This illustrates one of the disadvantages of the FFF technique, the introduction of undesirable anisotropic mechanical properties, in comparison to other techniques. In the sample depicted in Figure 3.7 b) fracture occurred for such a low value of strain that the determination of the PR was impossible.

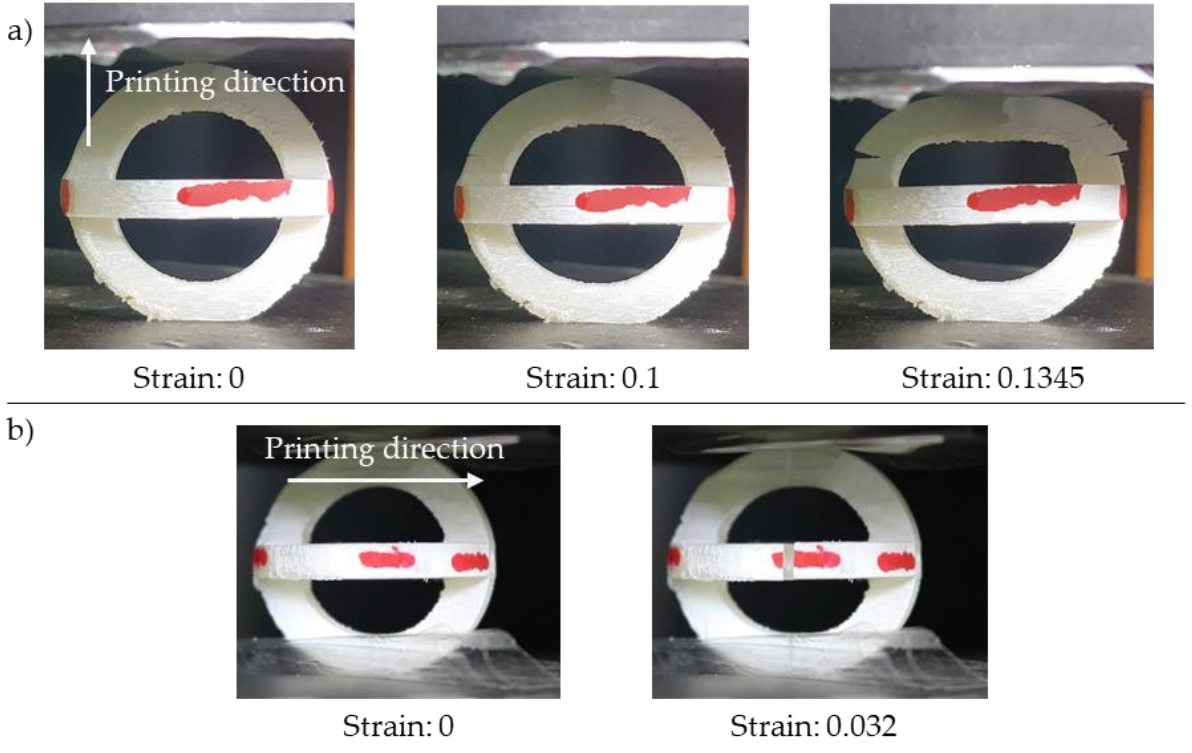


Figure 3.7 - Integral unit cells under compression until fracture.

Figure 3.8 presents an assembled unit cell that achieved the predicted PR value, denoting a partially auxetic behaviour. Because of the printing orientation of the assembled pieces, the unit cell supports compression better than the none assembled unit cells. Concluding that due to the printing orientation, printing in separate pieces allows the observation of deformation up to higher levels of strain (Appendix G).

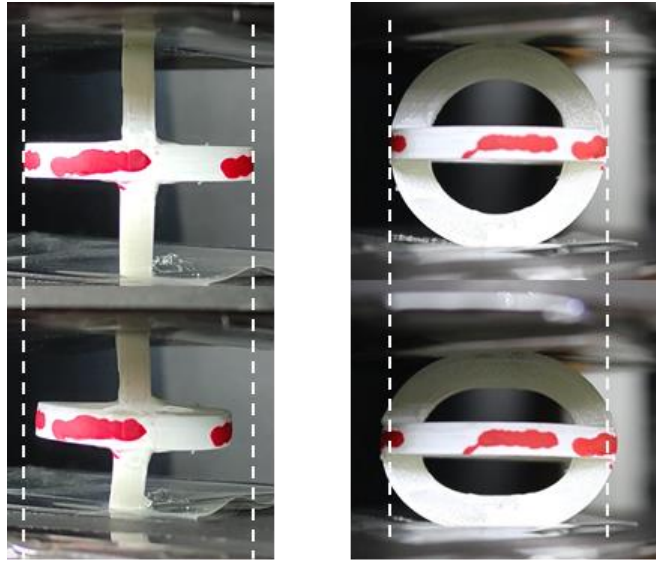


Figure 3.8 - Assembled unit cell under compression.

Nevertheless, some of the assembled samples failed at the junction zone in the beginning of the tests, as seen in Figure 3.9. This was probably due to the low amount of PVA glue, that did not allow a good joint between the assembled parts. So, it is possible to conclude that to obtain a properly assembled cell, it is necessary to ensure proper coverage of the junctions by the glue, so as to obtain the best adhesion possible.

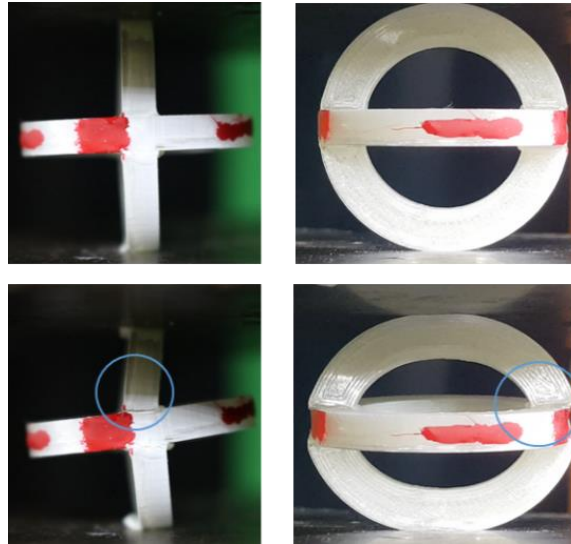


Figure 3.9 - Compressive test of an assembly cell unit with low amount of glue.

Figure 3.10 shows the average PR of the assembled cells. The explanation of the PR calculation and the PR curves of all the samples is displayed in Appendix H. The  $v_{z,y}$  exhibit positive values while  $v_{z,x}$  had values below zero. This was expected as this behaviour was observed previously in the FE analysis. Beyond that, similar results were obtained in the investigation conducted by Wang et al [29].

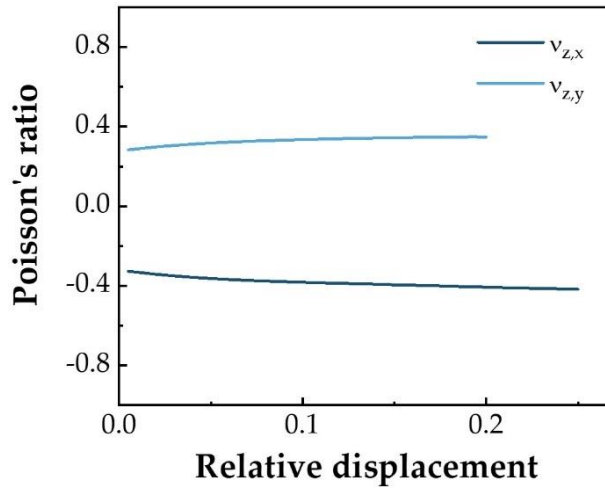


Figure 3.10 - Average PR of the assembled cells.

### 3.4.2 2 x 2 x 2 structure

Structures formed by 2 x 2 x 2 unit cells, based on model 1 were manufactured, in order to check if an assembled structure does not fracture in the junction zone under low strain. The corresponding stress-strain curves are shown in Appendix I.

Figure 3.11 shows the average values of the  $v_{z,x}$  and  $v_{z,y}$ . It is possible to conclude that the structure has a partially auxetic behaviour, as expected. Individually measured PR values, accompanied by an explanation of the PR calculation method are provided in Appendix J. Outliers, probably caused by the camera's resolution, were not considered for average calculation.

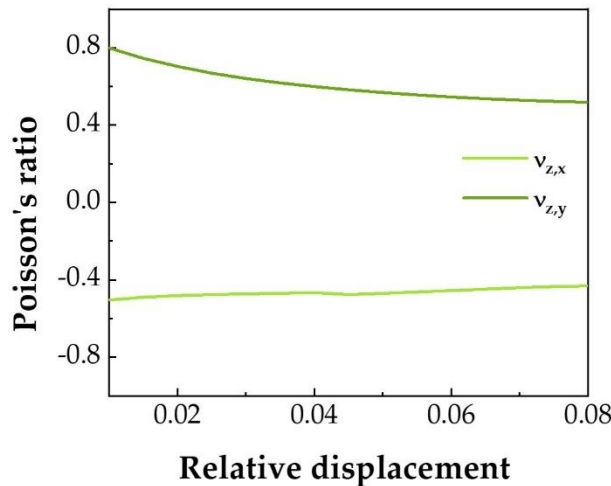


Figure 3.11 - PR of a 2 x 2 x 2 structure of model 1.

### 3.4.3 3 x 3 x 3 structure

Structures based on both models 1 and 2 were assembled as  $3 \times 3 \times 3$  arrays and tested under compression to confirm their auxetic behaviour on the  $xz$  plane and a positive PR on the  $yz$  plane (Appendix K). The average results are shown in Figure 3.12. Appendix L explains the PR calculation of these structures and presents all the samples PR curves. Some samples have unusual values between 0.01 and 0.025 of strain, most likely because of the camera's resolution (the higher the resolution, the larger the number of pixels, hence the more precise the data).

Figure 3.12 a) and b) describe the PR of the structure throughout the relative displacement range tested. As expected from the simulation predictions, the  $v_{z,x}$  and  $v_{z,y}$  obtained positive and negative values, respectively. The PR of the central cell (Figure 3.12 c) and d)) demonstrate a similar behaviour to the overall structure's PR.

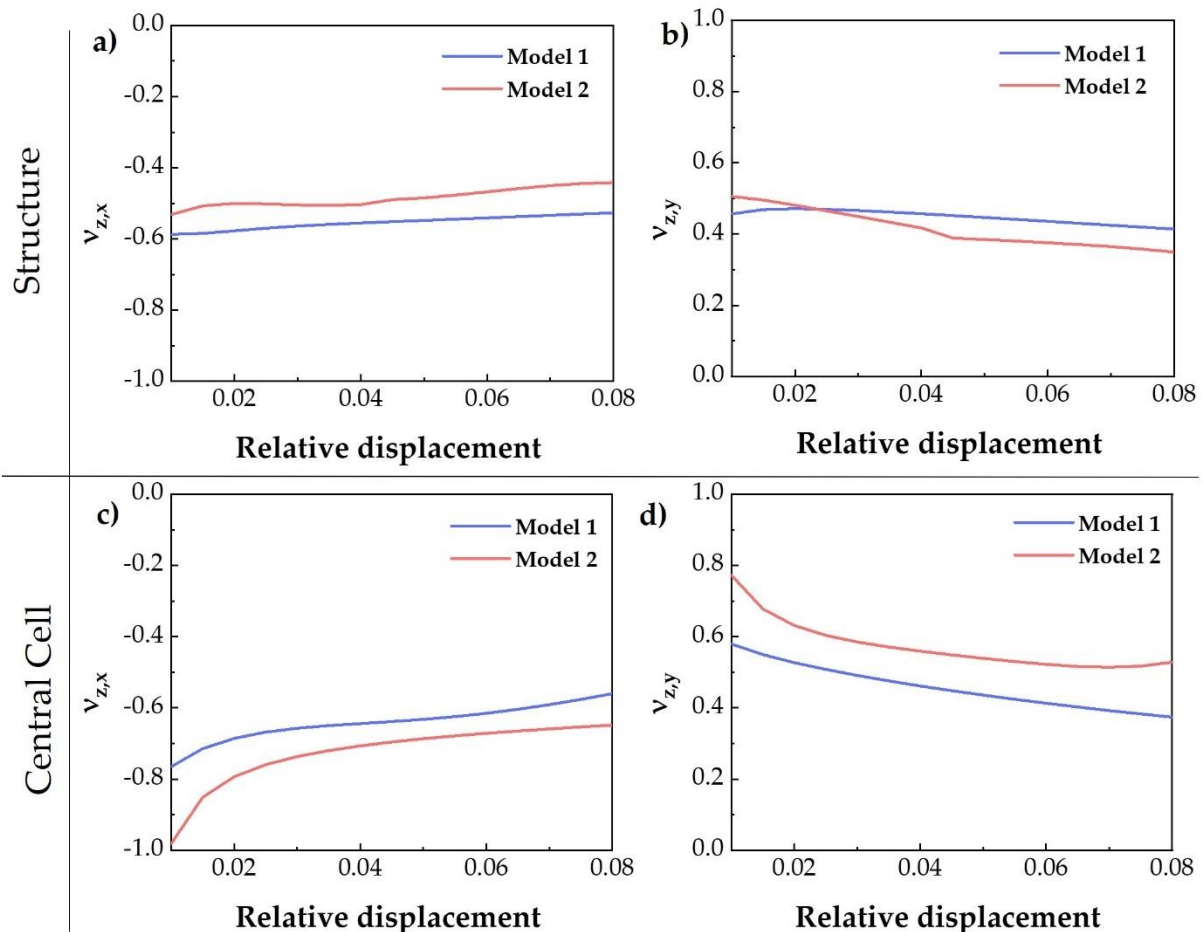


Figure 3.12 - Poisson's Ratio of model 1 and 2 throughout the experiment.

It is also possible to conclude that models 1 and 2 do not exhibit similar PR values. This could be because of the different amounts of Nylon, effecting the stiffness of the structure, and thus influencing the PR.

Beyond that, throughout the mechanical test, the PR in both directions is not constant, decreasing in absolute value; probably due to the progressive change in the shape of the rings (Figure 3.13). Alternatively, the observed effect could be due to edge effect and parallax errors.



Figure 3.13 - Example of the view yz of a structure (model 1) deformed.

### 3.4.4 Influence of the number of cells

In order to identify how the PR is influenced by the number of cells, a comparison of unit cell,  $2 \times 2 \times 2$  and  $3 \times 3 \times 3$  structures was performed. Figure 3.14 shows this study.

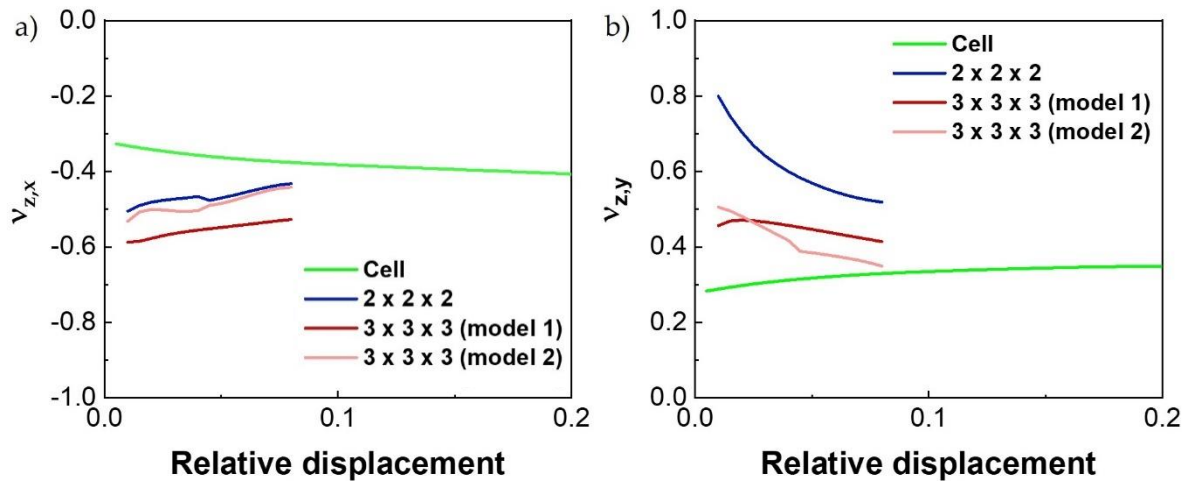


Figure 3.14-Influence of the number of cells in PR.

With the exception of the  $v_{z,y}$  values of the  $2 \times 2 \times 2$  structure, it is possible to observe the existence of a strong correlation between the number of cells and the values of PR. As the number of cells increases, the  $v_{z,x}$  becomes more negative, while  $v_{z,y}$  turns more positive.

### 3.5 Coefficient of thermal expansion

To determine the CLTE, thermal tests were carried out on models 1 and 2. Beyond that, a single material structure was manufactured from PVA and tested as a control sample. Due to this methodology, it was possible to determine value of  $\alpha_z$  in two different planes. The explanation of the CLTE calculation for the  $3 \times 3 \times 3$  structure is presented in Appendix M. The thermal experiment was tested from room temperature (around 21 °C) up to 80 °C.

Using three thermocouples in different positions near the sample, it was possible to observe a variation in the glycerol temperature throughout the experiment. In all the performed experiments, the largest difference between the temperature of the thermocouples was below 5 °C. Due to this error, it was decided to calculate the CLTE every 10 °C, giving a variation of  $\pm 2.5$  °C. Figure 3.15 show an example of one of the experiments, where the average of the thermocouples is presented in green and the red band corresponds to the  $\pm 2.5$  °C margin around the average. Observation of the graph lead to the conclusion that none of the channels surpasses this variation.

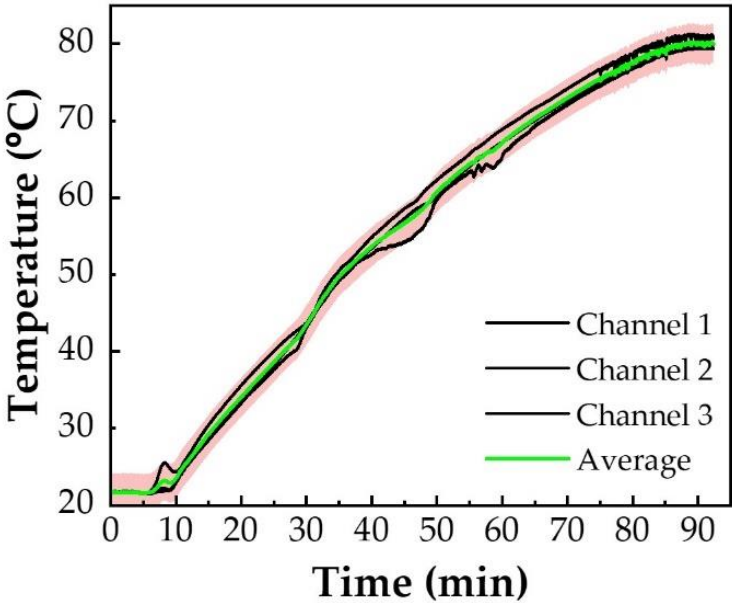


Figure 3.15 - Temperature registered of three thermocouples and their average throughout a thermal experiment. The red area represents the  $\pm 2.5$  °C variation of the average.

### 3.5.1 Study of model 1

Figure 3.16 shows the CLTE expansion in the three directions,  $x$ ,  $y$ , and  $z$ , of model 1. In all directions, the CLTE was positive throughout the experiment. This result was unexpected, since the FE simulation of a similar structure offers negative values for both  $\alpha_x$  and  $\alpha_z$ .

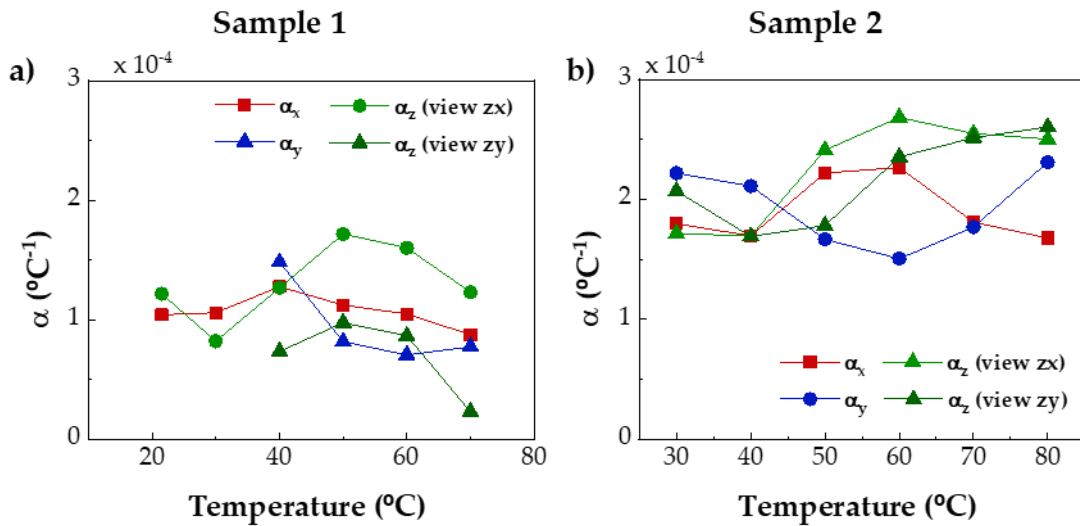


Figure 3.16 - CLTE of model 1 a) sample 1; b) sample 2.

### 3.5.2 Study of model 2

The CLTE of model 2 was determined along the  $x$ ,  $y$ , and  $z$  directions, as shown in Figure 3.17. The structure has a negative thermal expansion in two different directions,  $x$ , and  $z$ . This result was expected, as the expansion of nylon creates a deformation similar to that resulting from a compressive stress, resulting in  $\alpha_x$  and  $\alpha_z$  being negative and  $\alpha_y$  positive.

It is also observed that both  $\alpha_x$  and  $\alpha_z$  achieved positive values at 30  $^\circ\text{C}$ . After that, the values of the two CLTE decreased until the end of the experiment. This occurrence is, most likely, due to the structure only being capable of anepctic behaviour above the constituent materials'  $T_g$ , as already observed for 2D anepctic meshes by Raminhos et al. [58].

Due to the methodology used, it is possible to determine  $\alpha_z$  in two different views throughout the test. According to Figure 3.17, this parameter presents similar behaviour and values in both views, demonstrating that the results and the method created are reliable, slight variations notwithstanding.

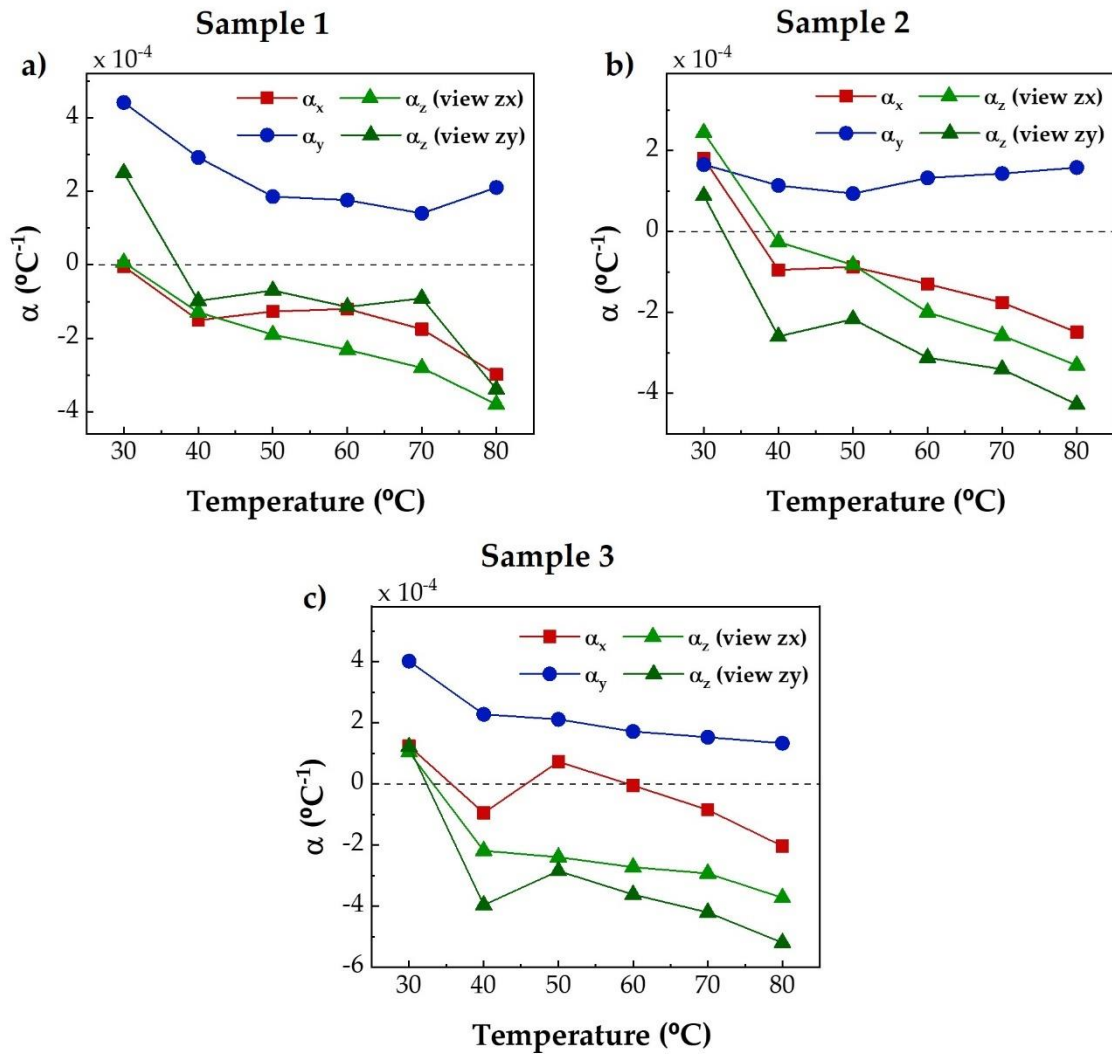


Figure 3.17 - CLTE of model 2 a) sample 1; b) sample 2; c) sample 3.

After these results, it is possible to conclude that the manufactured model 2 exhibited the intended anepctic behaviour.

### 3.5.3 Single material DER structure

The results of the thermal test of the single material DER structure, manufactured from PVA, are shown in Figure 3.18. It is possible to conclude that this structure, which is constituted of one material with positive CTE, obtained a negative CTE. This result is unexpected because it was not found in the literature a NTE metamaterial composed of one positive CTE material. It is relevant to point out that according to the literature, it was observed the opposite. Until now, a NTE metamaterial was achieved only when constituted of two materials, but the same metamaterial had a positive CTE when composed by one material [58], [78], [79].

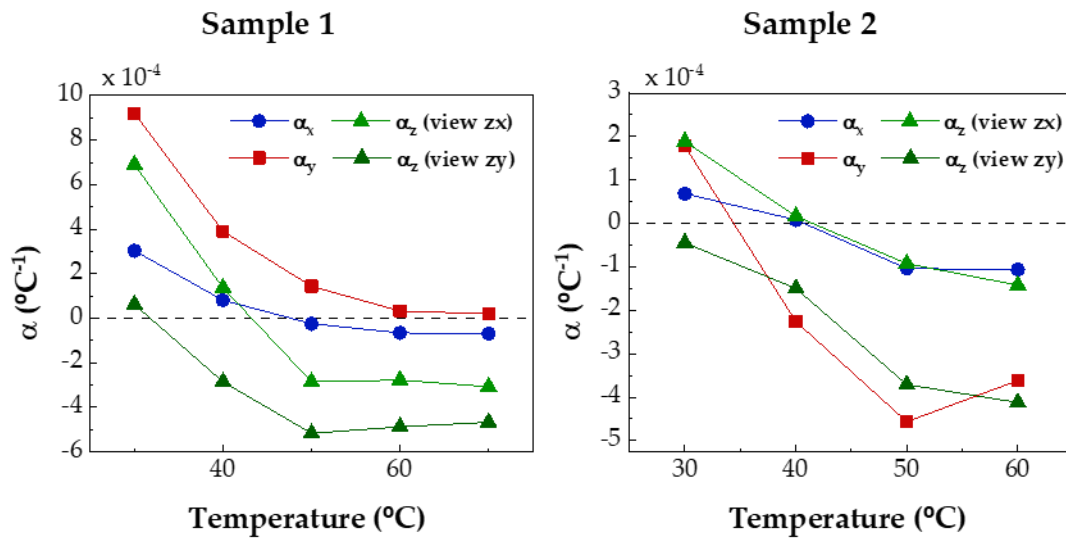


Figure 3.18 - CLTE of  $3 \times 3 \times 3$  DER structure of PVA a) sample 1; b) sample 2.

This NTE of a single material structure may be occurring due to the intrinsic geometry anisotropy. Despite that, as it is the first single material structure with this behaviour it is necessary to perform more tests to confirm these results.



## CONCLUSIONS AND FUTURE PERSPECTIVES

So far, the anepectic behaviour of 3D structures had only been demonstrated numerically by finite element (FE) or analytical analysis. Therefore, the breakthrough achieved with current work consisted in manufacturing and experimentally validating the anepectic behaviour of a 3D structure.

The influence of the printing orientation on the mechanical results were investigated. It was possible to conclude that it has a massive impact on the final properties of 3D printed material.

FE analysis was performed to obtain a 3D anepectic unit cell, in other words, with a negative Poisson's Ratio (PR) and a negative coefficient of thermal expansion (CTE). In order to achieve these properties, the incorporation of a second material in the unit cell developed by Wang et al. [29] was required. The chosen materials were PVA and Nylon due to the similar stiffness values but different CTE. All the design parameters of the unit cell studied were then defined.

Two 3D models were designed based on the unit cell developed. The difference between them is the amount of Nylon in each unit cell of the  $3 \times 3 \times 3$  structure. Both models were manufactured through a FFF technique, and Poisson's ratio and coefficient of thermal expansion were characterized. Model 2 (with Nylon in both rings) obtained an anepectic behaviour. A negative value was observed for  $\nu_{z,x}$ , while  $\nu_{z,y}$  was positive throughout the compressive test. The linear coefficient of thermal expansion in the x and z direction was negative, whereas the value was positive in the y direction.

A study of the influence of the number of cells on the PR values was made. As the number of cells increases, the PR tends to increase in module. However, it is relevant to deepen

this study since it was carried out only from the unit cell to the  $3 \times 3 \times 3$  structure due to the higher printing time. A similar study for CTE would be interesting to perform.

As a future perspective, it is relevant to study the influence of all the design parameters stated previously in this work. This parametric study allows to understand each parameter's influence and thus choose the appropriate parametric values for the application. The use of FE simulation will be a reliable tool to understand the behaviour of each parameter. As noted in chapter 3.2.3, a parameter change may have a high impact on the thermal and mechanical properties of the structure, being possible to change from negative to positive values. Beyond that, as a future perspective, the manufacture of the unit cell of Figure D.1 c) and characterization of the anepectic behaviour could be another way of transforming a DER structure into an anepectic metamaterial.

It might be relevant the study of different geometries beyond ellipses. A simulation study by FE method was made on a hexagonal and octagonal unit cell (Figure 4.1 a) and b)), both presented a NTE behaviour. This preview result not only shows the possibility of manufacturing an anepectic metamaterial similar to the DER structure with different geometry, as is another parameter to tailor the anepectic behaviour of these types of structures. Printing these geometries with a FFF printer is less challenging due to a less accumulation of material on the overhang as the printing angle of the overhang is higher. An example of a  $2 \times 2$  structure with a hexagonal geometry printed is shown in Appendix N.

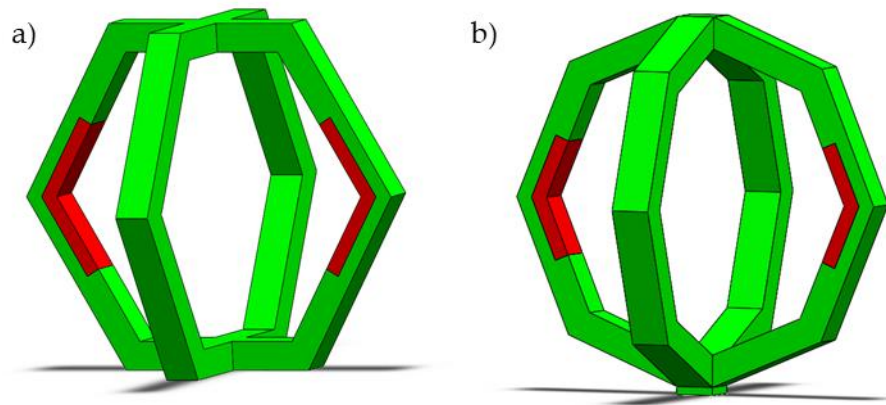


Figure 4.1 - Unit cell with different geometry. a) hexagonal unit cell; b) octagonal unit cell.

One of the difficulties of this work was the creation of a new methodology that would allow the determination of the linear thermal expansion coefficient for a 3D anepectic structure in the three directions. Since it was difficult to get the sample immobilized in a container with moving glycerol to obtain a homogeneous temperature and not restrict all its movement, as it will influence the thermal expansion of the structure. Thus, it would be necessary to optimize this methodology or create a better one.

Furthermore, Wang et al., based on a structure similar to DER, manufactured a structure in which the cells undergo a 90° rotation along the x and y axis (Figure 4.2) to obtain an auxetic behaviour in both directions [29]. Due to this behaviour, it would be interesting to replace part of the structure with a second material to obtain an anepectic behaviour, as performed in the present work. Then investigate if it would obtain negative values in all directions, both in PR and LCTE.

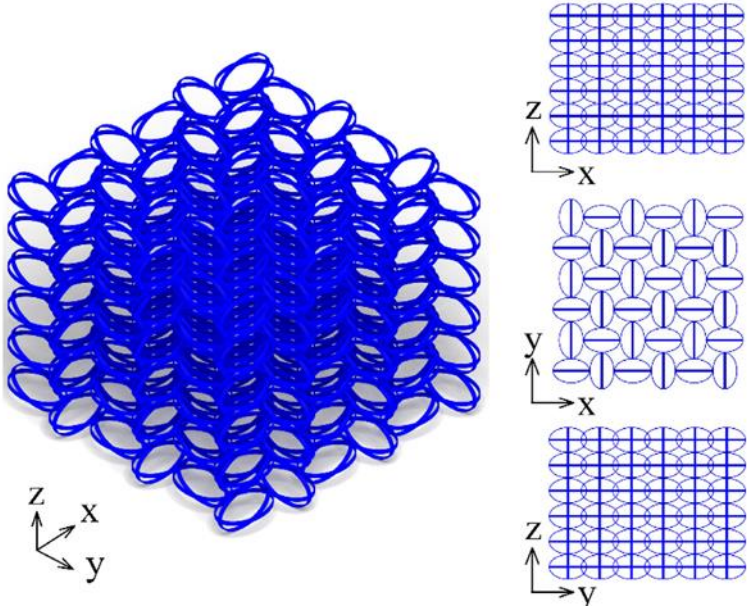


Figure 4.2 - Structure developed by Wang et al.[29]

From a future perspective, it would be interesting to realise thermal cycles during the thermal tests to observe if the anepectic behaviour changes throughout the cycles. Furthermore, it would be interesting to submerge the structures into a high temperature glycerol container to determine the reaction time.

Thermal tests were carried on a 3 x 3 x 3 structure of one material (PVA) and obtained a NTE. This result was unexpected since it is the first time that a metamaterial composed of one material with positive CTE obtained a NTE. For that reason, it is essential to reproduce these results to confirm it.



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## ALGORITHM TO DETERMINE PR

The first step of the algorithm is obtaining the data from the mechanical test, determining the area, calculating the strain and stress, and showing the graphs stress-strain and stress-stroke (until line 77 of algorithm). Then it obtains the video; asks what the maximum relative displacement and the step is; detect each red dot of the corresponded frame; determines the distance in pixel between the dots for each relative displacement and calculates the corresponding average strain (until line 236 of algorithm).

If the program detects more or fewer dots than expected, it will show the images in Figure A.1. The image on the left shows a black and white image of what the algorithm detects as red. The right image is the original frame, where a blue rectangle surrounds the red dots, and a green pixel is put in the considered centre of each red dot.

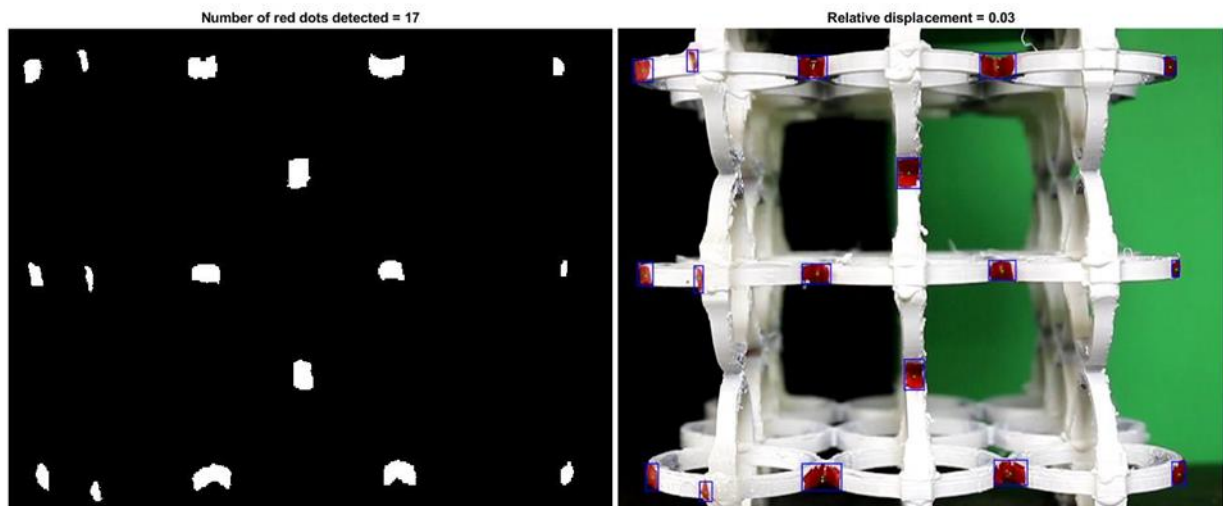


Figure A.1 - Figure shown by the algorithm so the user can see what the algorithm considers a red dot.

After that, calculate the PR of each relative displacement, shows the graphs distance vs relative displacement, strain vs relative displacement, and PR vs relative displacement. Lastly, it saves all data and graphics.

For the algorithm to be functional, it may be necessary to adjust the mask, that is, adjust the values of line 163. Another possible error is the dimension of the points intended to be discarded is not correct. For this case, line 174 is changed.

Below is the code and the detailed explanation (in green) of the algorithm used to determine the PR.

1	<code>clear all</code>
2	<code>close all</code>
3	
4	<code>fig = 1;</code>
5	<code>%% Open of document with mechanical results</code>
6	<code>fid = fopen('Document_name.txt');</code>
7	<code>tline = fgetl(fid);</code>
8	<code>tlines = cell(0,1);</code>
9	<code>num_line = 0;</code>
10	
11	<code>%% Asks for the volume of the structure</code>
12	<code>prompt_1 = {'Volume (mm^3):'};</code>
13	<code>dlgtitle_1 = 'Volume of structure';</code>
14	<code>dims = [1 35];</code>
15	<code>definput_1 = {'32991.9'};</code>
16	<code>answer_1 = inputdlg(prompt_1,dlgtitle_1,dims,definput_1);</code>
17	<code>volume = str2double(answer_1{1});</code>
18	
19	<code>time_data = [];</code>
20	<code>force_data = [];</code>
21	<code>stroke_data = [];</code>
22	<code>strain_z_data = [];</code>
23	<code>stress_data = [];</code>
24	
25	<code>%%Asks from what stroke does the test starts</code>
26	<code>prompt_2 = {'Compensated stroke (mm):'};</code>
27	<code>dlgtitle_2 = 'Shift data';</code>
28	<code>definput_2 = {'1.914'};</code>
29	<code>answer_2 = inputdlg(prompt_2,dlgtitle_2,dims,definput_2);</code>
30	<code>stroke_shift = str2double(answer_2{1});</code>
31	
32	<code>%%Get the data from the file and calculate stress and strain for each point</code>

33	<code>while ischar(tline)</code>
34	<code>    num_line = num_line + 1;</code>
35	<code>    if num_line == 11</code>
36	<code>        tlines{end+1,1} = tline;</code>
37	<code>        slipt_line = split(tlines{end});</code>
38	<code>        %% Distances of the structure, measured with a capiler</code>
39	<code>        dist_x = str2double(replace(slipt_line{5} ,",", ".")); %mm</code>
40	<code>        dist_y = str2double(replace(slipt_line{6} ,",", ".")); %mm</code>
41	<code>        dist_z = str2double(replace(slipt_line{7} ,",", ".")); %mm</code>
42	<code>        %% Method to determine the area for the calculous of the</code>
43	<code>stress</code>
44	<code>        volume_ap = dist_x * dist_y * dist_z; %mm^3</code>
45	<code>        area_ap = dist_x * dist_y; %mm^2</code>
46	<code>        area = (volume / volume_ap) * area_ap; %mm^2</code>
47	<code>    elseif num_line &gt; 21</code>
48	<code>        tlines{end+1,1} = tline;</code>
49	<code>        slipt_line = split(tlines{end});</code>
50	<code>        real_time = str2double(replace(slipt_line{1} ,",", "."));</code>
51	<code>        force = str2double(replace(slipt_line{2} ,",", "."));</code>
52	<code>        stroke = str2double(replace(slipt_line{3} ,",", ".")) -</code>
53	<code>stroke_shift;</code>
54	<code>        if stroke &gt;= 0</code>
55	<code>            time_data(end + 1) = real_time; %seconds</code>
56	<code>            force_data(end + 1) = force * 1000; %Newton</code>
57	<code>            stroke_data(end + 1) = stroke; %mm</code>
58	<code>            strain_z_data(end + 1) = stroke/dist_z;</code>
59	<code>            stress_data(end + 1) = (force * 10^3)/(area * 10^-6); %Pa</code>
60	<code>(N/m^2)</code>
61	<code>        end</code>
62	<code>    End</code>
63	<code>    tline = fgetl(fid);</code>
64	<code>End</code>
65	<code>fclose(fid);</code>
66	<code>63</code>
67	<code>64 %% Shows the graphs Stress vs Strain and Stress vs Stroke</code>
68	<code>figure(fig)</code>
69	<code>plot(strain_z_data, stress_data)</code>
70	<code>title('Stress vs Strain')</code>
71	<code>xlabel('Strain z')</code>
72	<code>ylabel('Stress (Pa)')</code>
73	<code>fig = fig + 1;</code>
74	<code>71</code>
75	<code>72 figure(fig)</code>
76	<code>73 plot(stroke_data, stress_data)</code>

74	title('Stress vs stroke')
75	xlabel('Stroke')
76	ylabel('Stress (Pa)')
77	fig = fig + 1;
78	
79	%%Obtain the video and save in vid
80	vid=VideoReader('Path_of_video\video_name.mp4');
81	
82	%% Ask what is the maximum strain and the step which it determine the PR
83	prompt_3 = {'Maximum:', 'Step:'};
84	dlgtitle_3 = 'Video strains';
85	definput_3 = {'0.08', '0.01'};
86	answer_3 = inputdlg(prompt_3,dlgtitle_3,dims,definput_3);
87	maximum_strain = str2double(answer_3{1});
88	step = str2double(answer_3{2});
89	
90	strain_video = 0 : step : maximum_strain;
91	
92	%% Makes vector with zeros to make the code more efficient
93	all_distance_y_central_cell = zeros(1,length(strain_video));
94	strain_y_central_cell = zeros(1,length(strain_video));
95	all_distance_z_central_cell = zeros(1,length(strain_video));
96	strain_z_central_cell = zeros(1,length(strain_video));
97	
98	
99	all_distance_y_structure = zeros(1,length(strain_video));
100	strain_y_structure = zeros(1,length(strain_video));
101	all_distance_z_structure = zeros(1,length(strain_video));
102	strain_z_structure = zeros(1,length(strain_video));
103	
104	%% Ask what time in the video starts the test to compensate with the time of the mechanical machine
105	prompt_4 = {'Extra time (s):'};
106	dlgtitle_4 = 'Extra time';
107	definput_4 = {'4'};
108	answer_4 = inputdlg(prompt_4,dlgtitle_4,dims,definput_4);
109	extra_time = str2double(answer_4{1});
110	
111	%% This "for" calculate the distance between points and the respectively strain
112	for s = 1:length(strain_video)
113	%% Find the index in vector strain_z_data (strain of the machine) that has the strain that we want to analyse
114	index_resolution = 1e-6;

115	index = find(abs(strain_z_data - strain_video(s)) <
116	index_resolution);
117	if isempty(index)
118	while isempty(index)
119	index_resolution = index_resolution + 1e-6;
120	index = find(abs(strain_z_data - strain_video(s)) <
121	index_resolution);
122	end
123	elseif length(index) > 3
124	while length(index) > 3
125	index_resolution = index_resolution - 1e-6;
126	index = find(abs(strain_z_data - strain_video(s)) <
127	index_resolution);
128	end
129	end
130	end
131	%% obtained the time of the video that has the strain of the
132	machine we want to analyse and obtained the respective frame
133	vid.CurrentTime = time_data(index(round(length(index)/2))) +
134	extra_time;
135	ImS = readFrame(vid);
136	
137	%% If is the initial of the experiment ask if knows all the
138	necessary data. If not, shows the first frame to obtained the
139	necessary data.
140	if s == 1
141	pergunta1 = questdlg('Do you know all the initial data?',
142	'Question 1');
143	if strcmp(pergunta1, 'No')
144	figure(fig)
145	imshow(ImS)
146	return
147	end
148	%% If knows the data, asks coordinates to crop the frame to
149	the necessary size to avoid errors
150	prompt_6 = {'Minimum x:', 'Maximum x:', 'Minimum y:', 'Maximum
151	y:'};
152	dlgtitle_6 = 'Crop image';
153	definput_6 = {'34' , '1026' , '731' , '1500'};
154	answer_6 = inputdlg(prompt_6,dlgtitle_6,dims,definput_6);
155	Minimum_x = str2double(answer_6{1});
156	Maximum_x = str2double(answer_6{2});
157	Minimum_y = str2double(answer_6{3});
158	Maximum_y = str2double(answer_6{4});
159	end
160	
161	%% Crops the frame
162	ImS = ImS(Minimum_y:Maximum_y, Minimum_x:Maximum_x, :);
163	

153	%% Divides the pixels in the red, green and blue part (numbers can be from 0 to 255)
154	I_Red = ImS(:,:,1);
155	I_Green = ImS(:,:,2);
156	I_Blue = ImS(:,:,3);
157	
158	%% Create an black and white image that put white only if shows the correspond conditions.
159	%% These conditions are to put in white only the red pixels.
160	%% There are two different ways to do it, as shown below.
161	%% Then fills holes in the mask.
162	%mask = ((I_Red >= 175 & I_Green <= 210 & I_Blue <= 220) & (abs(I_Red - I_Green) > 60) & (abs(I_Red - I_Blue) > 60))   (I_Red >= 120 & I_Green <= 100 & I_Blue <= 100)   (I_Red >= 95 & I_Green <= 40 & I_Blue <= 40)   (I_Red >= 59 & I_Green <= 40 & I_Blue <= 40);
163	mask = (I_Red - I_Green) > 40 & (I_Red - I_Blue) > 40 & (abs(I_Green - I_Blue) < 15);
164	mask = imfill(mask, 'holes');
165	
166	%% Get the central position of every dot
167	positions_centroid = regionprops(mask, 'centroid');
168	
169	%% Get the coordinations, and the height and with of the dots
170	positions = regionprops(mask, 'BoundingBox');
171	
172	%% Eliminate small dots. In other words, remove false dots
173	for i = length(positions):-1:1
174	if positions(i).BoundingBox(3) * positions(i).BoundingBox(4)
175	positions(i) = [];
176	positions_centroid(i) = [];
177	end
178	end
179	
180	num_shape = length(positions); % Number of dots
181	
182	%% If the number of dots is not the expectec shows the frame to visually try to understand the error.
183	if num_shape ~= 16
184	figure(fig)
185	subplot(1,2,1), imshow(mask);
186	title(num2str(num_shape));
187	subplot(1,2,2), imshow(ImS);
188	for k = 1 : length(positions)
189	all_positions = positions(k).BoundingBox;

190	<code>rectangle('Position', [all_positions(1), all_positions(2), all_positions(3), all_positions(4)], 'EdgeColor', 'b', 'LineWidth', 1 )</code>
191	<code>rectangle('Position', [round(positions_centroid(k).Centroid(1)), round(positions_centroid(k).Centroid(2)), 1, 1], 'EdgeColor', 'g', 'LineWidth', 1)</code>
192	<code>end</code>
193	<code>title(['strain = ', num2str(strain_video(s))]);</code>
194	<code>fig = fig + 1;</code>
195	<code>end</code>
196	
197	<code>T = struct2table(positions_centroid); % convert the struct array to a table</code>
198	<code>Table = T.Centroid;</code>
199	<code>Table = sortrows(Table, 2); % sort the table by second collum (by the height)</code>
200	<code>%% Divide and sort by width each line of dots</code>
201	<code>Table_Line_1 = sortrows(Table(1:5, :), 1);</code>
202	<code>Table_Line_2 = sortrows(Table(6, :), 1);</code>
203	<code>Table_Line_3 = sortrows(Table(7:11, :), 1);</code>
204	<code>Table_Line_4 = sortrows(Table(12, :), 1);</code>
205	<code>Table_Line_5 = sortrows(Table(13:end, :), 1);</code>
206	
207	<code>%%calculation of y strain of central cell</code>
208	<code>distance_y_central_cell = Table_Line_3(3,1) - Table_Line_3(2,1);</code>
209	<code>all_distance_y_central_cell(s) = distance_y_central_cell;</code>
210	<code>strain_y_central_cell(s) = (distance_y_central_cell- all_distance_y_central_cell(1))/all_distance_y_central_cell(1);</code>
211	
212	<code>%%calculation of z strain of central cell</code>
213	<code>distance_z_central_cell = Table_Line_4(1,2) - Table_Line_2(1,2);</code>
214	<code>all_distance_z_central_cell(s) = distance_z_central_cell;</code>
215	<code>strain_z_central_cell(s) = (distance_z_central_cell- all_distance_z_central_cell(1))/all_distance_z_central_cell(1);</code>
216	
217	<code>%%calculation of y strain of structure</code>
218	
219	<code>distance_y_structure_1 = Table_Line_1(5,1) - Table_Line_1(1,1);</code>
220	<code>distance_y_structure_2 = Table_Line_3(5,1) - Table_Line_3(1,1);</code>
221	<code>distance_y_structure_3 = Table_Line_5(5,1) - Table_Line_5(1,1);</code>
222	<code>average_distance_y_structure = mean([distance_y_structure_1 distance_y_structure_2 distance_y_structure_3]);</code>
223	<code>all_distance_y_structure(s) = average_distance_y_structure;</code>
224	<code>strain_y_structure(s) = (average_distance_y_structure- all_distance_y_structure(1))/all_distance_y_structure(1);</code>
225	
226	<code>%%calculation of z strain of structure</code>

227	
228	distance_z_structure_1 = Table_Line_5(1,2) - Table_Line_1(1,2);
229	distance_z_structure_2 = Table_Line_5(2,2) - Table_Line_1(2,2);
230	distance_z_structure_3 = Table_Line_5(3,2) - Table_Line_1(3,2);
231	distance_z_structure_4 = Table_Line_5(4,2) - Table_Line_1(4,2);
232	average_distance_z_structure = mean([distance_z_structure_1 distance_z_structure_2 distance_z_structure_3 distance_z_structure_4]);
233	all_distance_z_structure(s) = average_distance_z_structure;
234	strain_z_structure(s) = (average_distance_z_structure - all_distance_z_structure(1)) /all_distance_z_structure(1);
235	
236	end
237	
238	%%calculation of PR of central cell
239	Poisson_z_y_cell = - strain_y_central_cell(2:end) ./ strain_z_central_cell(2:end);
240	for i = 1:length(Poisson_z_y_cell)
241	if Poisson_z_y_cell(i) == -Inf    Poisson_z_y_cell(i) == 0    Poisson_z_y_cell(i) == Inf    isnan(Poisson_z_y_cell(i))
242	Poisson_z_y_cell(i) = 0;
243	end
244	end
245	
246	%%calculation of PR of structure
247	Poisson_z_y_structure = - strain_y_structure(2:end) ./ strain_z_structure(2:end);
248	for k = 1:length(Poisson_z_y_structure)
249	if Poisson_z_y_structure(k) == -Inf    Poisson_z_y_structure(k) == 0    Poisson_z_y_structure(k) == Inf    isnan(Poisson_z_y_structure(k))
250	Poisson_z_y_structure(k) = 0;
251	end
252	end
253	
254	%%Central cell grafs
255	
256	figure_1 = figure(fig);
257	plot(strain_video,all_distance_y_central_cell)
258	hold on
259	plot(strain_video,all_distance_z_central_cell)
260	hold off
261	title('Distance y e z between red dots in central cell vs strain')
262	xlabel('Strain')
263	ylabel('Distance (pixel)')
264	legend('y','z')

265	fig = fig + 1;
266	
267	figure_2 = figure(fig);
268	plot(strain_video, strain_y_central_cell)
269	hold on
270	plot(strain_video, strain_z_central_cell)
271	hold off
272	title('Strain y e z between red dots in central cell vs strain')
273	xlabel('Strain da maquina')
274	ylabel('Strain')
275	legend('y', 'z')
276	fig = fig + 1;
277	
278	figure_3 = figure(fig);
279	plot(strain_video(2:end), Poisson_z_y_cell)
280	title('Poisson_z,y of central cell vs strain')
281	xlabel('Strain')
282	ylabel('Poisson')
283	fig = fig + 1;
284	
285	figure_4 = figure(fig);
286	plot(strain_z_central_cell(2:end), strain_y_central_cell(2:end))
287	title('Strain z vs strain y')
288	xlabel('Strain z')
289	ylabel('Strain y')
290	fig = fig + 1;
291	
292	%% Structure grafs
293	
294	figure_5 = figure(fig);
295	plot(strain_video, all_distance_y_structure)
296	hold on
297	plot(strain_video, all_distance_z_structure)
298	hold off
299	title('Distance y e z between red dots in structure vs strain')
300	xlabel('Strain')
301	ylabel('Distance (pixeis)')
302	legend('y', 'z')
303	fig = fig + 1;
304	
305	figure_6 = figure(fig);
306	plot(strain_video, all_distance_y_structure)
307	hold on

308	plot(strain_video,all_distance_z_structure)
309	hold off
310	title('Strain y e z between red dots in structure vs strain')
311	xlabel('Strain da maquina')
312	ylabel('Strain')
313	legend('y','z')
314	fig = fig + 1;
315	
316	figure_7 = figure(fig);
317	plot(strain_video(2:end),Poisson_z_y_structure)
318	title('Poisson_z,y of structure vs strain')
319	xlabel('Strain')
320	ylabel('Poisson')
321	legend('line 1','line 2','line 3')
322	fig = fig + 1;
323	
324	figure_8 = figure(fig);
325	plot(strain_z_structure(2:end),strain_y_structure(2:end))
326	title('Strain y vs Straun z')
327	xlabel('Strain z')
328	ylabel('Strain y')
329	fig = fig + 1;
330	
331	%%Ask if want to save the data
332	question_save_1 = questdlg('Do you want record data?','Question 2');
333	if strcmp(question_save_1,'No')
334	return
335	end
336	
337	%%Save the data in an excel
338	Final_data_1 = num2cell([time_data.' force_data.' stroke_data.' strain_z_data.' stress_data.']);
339	header_1 = {'Time (s)','Force (N)','Stroke (mm)','Strain', 'Stress (Pa)'};
340	output_matrix_1=[header_1; Final_data_1];
341	
342	xlswrite('3por3_modelo2_amostral.xls',output_matrix_1, 'Compressao_y','A1');
343	
344	Final_data_2 = num2cell([dist_x dist_y dist_z area volume stroke_shift]);
345	header_2 = {'Distance x (mm)','Distance y (mm)','Distance z (mm)','Area', 'Sample volume', 'Stroke shift (mm)'};
346	output_matrix_2=[header_2; Final_data_2];
347	

348	<code>xlswrite('3por3_modelo2_amostr1.xls',output_matrix_2,</code>
349	<code>'Compressao_y','G1');</code>
350	<code>Final_data_vista_y = num2cell([strain_video.'</code> <code>all_distance_y_central_cell.'</code> <code>all_distance_z_central_cell.'</code> <code>strain_y_central_cell.'</code> <code>strain_z_central_cell.'</code> <code>all_distance_y_structure.'</code> <code>all_distance_z_structure.'</code> <code>strain_y_structure.'</code> <code>strain_z_structure.']);</code>
351	<code>header_poisson_y = {'Strain','Distance cell y (pixels)', 'Distance</code> <code>cell z (pixels)', 'Strain cell y', 'Strain cell z', 'Distance y</code> <code>stuture (pixel)', 'Distance z stuture (pixel)', 'Strain y stuture',</code> <code>'Strain z stuture'};</code>
352	<code>output_matrix_vista_y = [header_poisson_y; Final_data_vista_y];</code>
353	
354	<code>xlswrite('3por3_modelo2_amostr1.xls',output_matrix_vista_y,'vista_y'</code> <code>);</code>
355	
356	<code>Final_data_vista_y_2 = num2cell([Poisson_z_y_cell.'</code> <code>strain_video(2:end).'</code> <code>Poisson_z_y_structure.']);</code>
357	<code>header_3 = {'Poisson_z_y cell', 'Strain video', 'Poisson_z_y</code> <code>structure'};</code>
358	<code>output_matrix_vista_y_2=[header_3; Final_data_vista_y_2];</code>
359	
360	<code>xlswrite('3por3_modelo2_amostr1.xls',output_matrix_vista_y_2,'vista_</code> <code>y','L1');</code>
361	
362	<code>%% Ask if want to save graphs</code>
363	<code>question_save_2 = questdlg('Pretende guardar graficos?','Pergunta</code> <code>3');</code>
364	<code>if strcmp(question_save_2,'No')</code>
365	<code>    return</code>
366	<code>end</code>
367	
368	<code>%% Save the graphs</code>
369	<code>saveas(figure_1,'central_cell_distance_y_z.png')</code>
370	<code>saveas(figure_2,'central_cell_strain_y_z.png')</code>
371	<code>saveas(figure_3,'central_cell_poisson_z_y.png')</code>
372	<code>saveas(figure_4,'central_cell_declive_y_z.png')</code>
373	
374	<code>saveas(figure_5,'structure_distance_y_z.png')</code>
375	<code>saveas(figure_6,'structure_strain_y_z.png')</code>
376	<code>saveas(figure_7,'structure_poisson_z_y.png')</code>
377	<code>saveas(figure_8,'structure_declive_y_z.png')</code>



## ALGORITHM TO DETERMINE CTE

The first step of the algorithm is obtaining the images. After that, detects each red dot of the corresponded image; determines the distance in pixel between the dots center for each image and calculates the corresponding CLTE (until line 133).

For each image, it will show the images in Figure B.1. The image on the left shows a black and white image of what the algorithm detects as red. The right image is the original image, where a blue rectangle surrounds the red dots, and a blue pixel is put in the considered centre of each red dot that needs to determine all the distances.

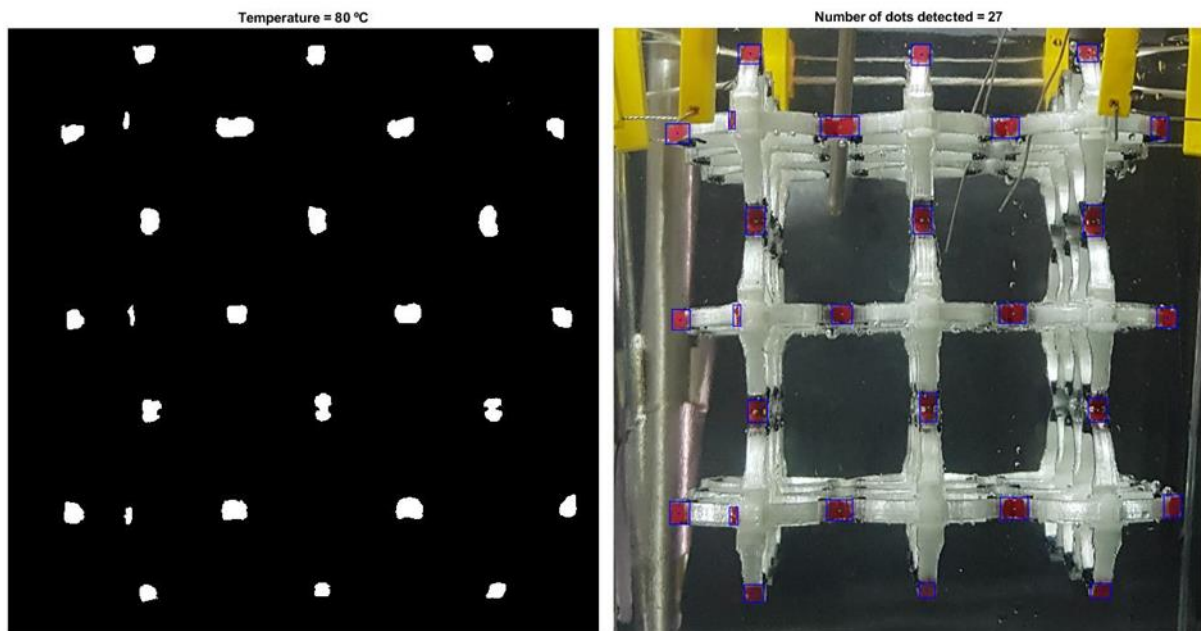


Figure B.1 - Figure shown by the algorithm so the user can see what the algorithm considers a red dot and what is the centre of each dot.

After that, shows the graphs distance vs temperature and CLTE vs temperature. Lastly, it saves all data and graphics.

Below is the code and the detailed explanation of the algorithm used to determine the CLTE.

1	<code>clear all</code>
2	<code>close all</code>
3	
4	<code>%Get the images</code>
5	<code>files = dir('*.jpg');</code>
6	
7	<code>%Vector of all the temperature study</code>
8	<code>Temperature = [21 30 40 50 60 70 80];</code>
9	
10	<code>% Makes vector with zeros to make the code more efficient</code>
11	<code>all_distance_x_estrutura = zeros(1,length(files));</code>
12	<code>all_distance_z_estrutura = zeros(1,length(files));</code>
13	<code>expansion_x = zeros(1,length(files));</code>
14	<code>expansion_z = zeros(1,length(files));</code>
15	<code>fig = 1;</code>
16	
17	<code>% This "for" calculate the distance between points and the respectively CLTE</code>
18	<code>for j = 1:length(files)</code>
19	<code>    I = imread(files(j).name);</code>
20	<code>    I = imrotate(I,-90);</code>
21	
22	<code>        % If is the initial of the experiment ask if knows all the necessary data.</code>
23	<code>        % If not, shows the first frame to obtained the necessary data.</code>
24	<code>        if j == 1</code>
25	<code>            pergunta1 = questdlg('Quer só a primeira imagem?','Pergunta 1');</code>
26	<code>            if strcmp(pergunta1,'Yes')</code>
27	<code>                figure(fig)</code>
28	<code>                imshow(I)</code>
29	<code>                Return</code>
30	<code>            End</code>
31	<code>        End</code>
32	
33	<code>        % If knows the data, asks coordinates to crop the frame to the necessary size to avoid errors</code>
34	<code>        prompt = {'Minimum x:', 'Maximum x:', 'Minimum y:', 'Maximum y:'};</code>
35	<code>        dlgtitle = 'Crop image';</code>

36	definput = {'34' , '1026' , '731' , '1500'};
37	answer = inputdlg(prompt,dlgtitle,dims,definput);
38	Minimum_x = str2double(answer{1});
39	Maximum_x = str2double(answer{2});
40	Minimum_y = str2double(answer{3});
41	Maximum_y = str2double(answer{4});
42	
43	% Crops the frame
44	I = I(Minimum_y:Maximum_y, Minimum_x:Maximum_x, :);
45	
46	% Divides the pixels in the red, green and blue part (numbers can be from 0 to 255)
47	I_Red = I(:, :, 1);
48	I_Green = I(:, :, 2);
49	I_Blue = I(:, :, 3);
50	
51	% Create a black and white image that put white only if shows the correspond conditions.
52	% These conditions are to put in white only the red pixels.
53	mask = (I_Red - I_Green) > 30 & (I_Red - I_Blue) > 30 & (abs(I_Green - I_Blue) < 20);
54	
55	% Then fills holes in the mask.
56	mask = imfill(mask, 'holes');
57	
58	% Get the central position of every dot
59	positions_centroid = regionprops(mask, 'centroid');
60	
61	% Get the coordinations, and the height and with of the dots
62	positions = regionprops(mask, 'BoundingBox');
63	
64	% Eliminate small dots. In other words, remove false dots
65	for i = length(positions):-1:1
66	if positions(i).BoundingBox(3) * positions(i).BoundingBox(4) < 250
67	positions(i) = [];
68	positions_centroid(i) = [];
69	End
70	end
71	
72	num_shape = length(positions); % Number of dots
73	
74	% Shows the image and the mask to visually understand if everything is well.
75	figure(fig)

76	subplot(1,2,1), imshow(mask);
77	title(num2str(j));
78	subplot(1,2,2), imshow(I);
79	title(num2str(num_shape));
80	for k = 1 : length(positions)
81	all_positions = positions(k).BoundingBox;
82	rectangle('Position', [all_positions(1), all_positions(2) ,all_positions(3) ,all_positions(4)], 'EdgeColor','b','LineWidth',1 )
83	rectangle('Position', [round(positions_centroid(k).Centroid(1)), round(positions_centroid(k).Centroid(2)),1,1], 'EdgeColor','g','Line Width',1)
84	End
85	
86	T = struct2table(positions_centroid); % convert the struct array to a table
87	Table = T.Centroid;
88	Table = sortrows(Table,2); % sort the table by second collum (z direction)
89	% Devide in line and sort in x diretion
90	Table_Line_1 = sortrows(Table(1:3,:),1);
91	Table_Line_2 = sortrows(Table(4:7,:),1);
92	Table_Line_3 = sortrows(Table(8:10,:),1);
93	Table_Line_4 = sortrows(Table(11:14,:),1);
94	Table_Line_5 = sortrows(Table(15:17,:),1);
95	Table_Line_6 = sortrows(Table(18:21,:),1);
96	Table_Line_7 = sortrows(Table(22:24,:),1);
97	% To visually observe where is the center of the dots
98	rectangle('Position', [round(Table_Line_2(1,1)), round(Table_Line_2(1,2)),1,1], 'EdgeColor','b','LineWidth',1)
99	rectangle('Position', [round(Table_Line_2(4,1)), round(Table_Line_2(4,2)),1,1], 'EdgeColor','b','LineWidth',1)
100	rectangle('Position', [round(Table_Line_4(1,1)), round(Table_Line_4(1,2)),1,1], 'EdgeColor','b','LineWidth',1)
101	rectangle('Position', [round(Table_Line_4(4,1)), round(Table_Line_4(4,2)),1,1], 'EdgeColor','b','LineWidth',1)
102	rectangle('Position', [round(Table_Line_6(1,1)), round(Table_Line_6(1,2)),1,1], 'EdgeColor','b','LineWidth',1)
103	rectangle('Position', [round(Table_Line_6(4,1)), round(Table_Line_6(4,2)),1,1], 'EdgeColor','b','LineWidth',1)
104	
105	rectangle('Position', [round(Table_Line_1(1,1)), round(Table_Line_1(1,2)),1,1], 'EdgeColor','b','LineWidth',1)
106	rectangle('Position', [round(Table_Line_1(2,1)), round(Table_Line_1(2,2)),1,1], 'EdgeColor','b','LineWidth',1)
107	rectangle('Position', [round(Table_Line_1(3,1)), round(Table_Line_1(3,2)),1,1], 'EdgeColor','b','LineWidth',1)
108	rectangle('Position', [round(Table_Line_7(1,1)), round(Table_Line_7(1,2)),1,1], 'EdgeColor','b','LineWidth',1)

109	rectangle('Position', [round(Table_Line_7(2,1)),
110	round(Table_Line_7(2,2)),1,1], 'EdgeColor', 'b', 'LineWidth',1)
111	rectangle('Position', [round(Table_Line_7(3,1)),
112	round(Table_Line_7(3,2)),1,1], 'EdgeColor', 'b', 'LineWidth',1)
113	
114	% calculation of the CLTE in x direction
115	
116	distance_x_estrutura_1 = Table_Line_2(4,1) - Table_Line_2(1,1);
117	distance_x_estrutura_2 = Table_Line_4(4,1) - Table_Line_4(1,1);
118	distance_x_estrutura_3 = Table_Line_6(4,1) - Table_Line_6(1,1);
119	average_distance_x_estrutura = mean([distance_x_estrutura_1
120	distance_x_estrutura_2 distance_x_estrutura_3]);
121	all_distance_x_estrutura(j)= average_distance_x_estrutura;
122	expansion_x(j) = ((average_distance_x_estrutura-
123	all_distance_x_estrutura(1))/all_distance_x_estrutura(1)) * (1/(Temperature(j)-Temperature(1)));
124	
125	% calculation of the CLTE in z direction
126	
127	distance_z_estrutura_1 = Table_Line_7(1,2) - Table_Line_1(1,2);
128	distance_z_estrutura_2 = Table_Line_7(2,2) - Table_Line_1(2,2);
129	distance_z_estrutura_3 = Table_Line_7(3,2) - Table_Line_1(3,2);
130	average_distance_z_estrutura = mean([distance_z_estrutura_1
131	distance_z_estrutura_2 distance_z_estrutura_3]);
132	all_distance_z_estrutura(j)= average_distance_z_estrutura;
133	expansion_z(j) = ((average_distance_z_estrutura-
134	all_distance_z_estrutura(1))/all_distance_z_estrutura(1)) * (1/(Temperature(j)-Temperature(1)));
135	
136	fig = fig + 1;
137	End
138	
139	%Grafos to verify the results
140	%Graf 1 : Temperature vs Distances in x and z directions
141	%Graf 2 : Temperature vs CLTE in x and z directions
142	
143	figure_1 = figure(fig);
144	plot(Temperature,all_distance_x_estrutura)
145	hold on
146	plot(Temperature,all_distance_z_estrutura)
	hold off
	title('Distance')
	xlabel('Temperature')
	ylabel('Distance (pixeis)')

147	legend('x','z')
148	
149	fig = fig + 1;
150	
151	figure_2 = figure(fig);
152	plot(Temperature(2:end),expansion_x(2:end))
153	hold on
154	plot(Temperature(2:end),expansion_z(2:end))
155	hold off
156	title('CTE')
157	xlabel('Temperature')
158	ylabel('Distance (pixeis)')
159	legend('x','z')
160	
161	%Ask if want to save the data
162	question_save_1 = questdlg('Pretende guardar dados?','Pergunta 2');
163	if strcmp(question_save_1,'No')
164	return
165	End
166	
167	Final_data_view_x_1 = num2cell([Temperature.' all_distance_x_estrutura.']);
168	header_1 = {'Temperature', 'Distance x (pixel)'};
169	output_matrix_view_x_1=[header_1; Final_data_view_x_1];
170	
171	xlswrite('3por3_modelo2_amostra2_view_zx.xls',output_matrix_view_x_1,'direcao_x');
172	
173	Final_data_view_x_2 = num2cell([Temperature(2:end). expansion_x(2:end).]);
174	header_2 = {'Temperature', 'Distance x (pixel)'};
175	output_matrix_view_x_2=[header_2; Final_data_view_x_2];
176	
177	xlswrite('3por3_modelo2_amostra2_view_zx.xls', output_matrix_view_x_2,'direcao_x','D1');
178	
179	Final_data_view_z_1 = num2cell([Temperature.' all_distance_z_estrutura.']);
180	header_3 = {'Temperature', 'Distance z (pixel)'};
181	output_matrix_view_z_1=[header_3; Final_data_view_z_1];
182	
183	xlswrite('3por3_modelo2_amostra2_view_zx.xls', output_matrix_view_z_1,'direcao_z');
184	
185	Final_data_view_z_2 = num2cell([Temperature(2:end). expansion_z(2:end).]);

186	header_4 = {'Temperature', 'Distance z (pixel)'};
187	output_matrix_view_z_2=[header_4; Final_data_view_z_2];
188	
189	xlswrite('3por3_modelo2_amostra2_view_zx.xls', output_matrix_view_z_2, 'direcao_z', 'D1');
190	
191	%Ask if want to save the graphs
192	question_save_2 = questdlg('Do you want to save the graphs?', 'Pergunta 3');
193	if strcmp(question_save_2, 'No')
194	Return
195	End
196	
197	%Save the graphs
198	saveas(figure_1, 'model_2_distance_view_xz.png')
199	saveas(figure_2, 'model_2_CTE_view_yz.png')



## STRESS-STRAIN CURVES OF FILAMENTS

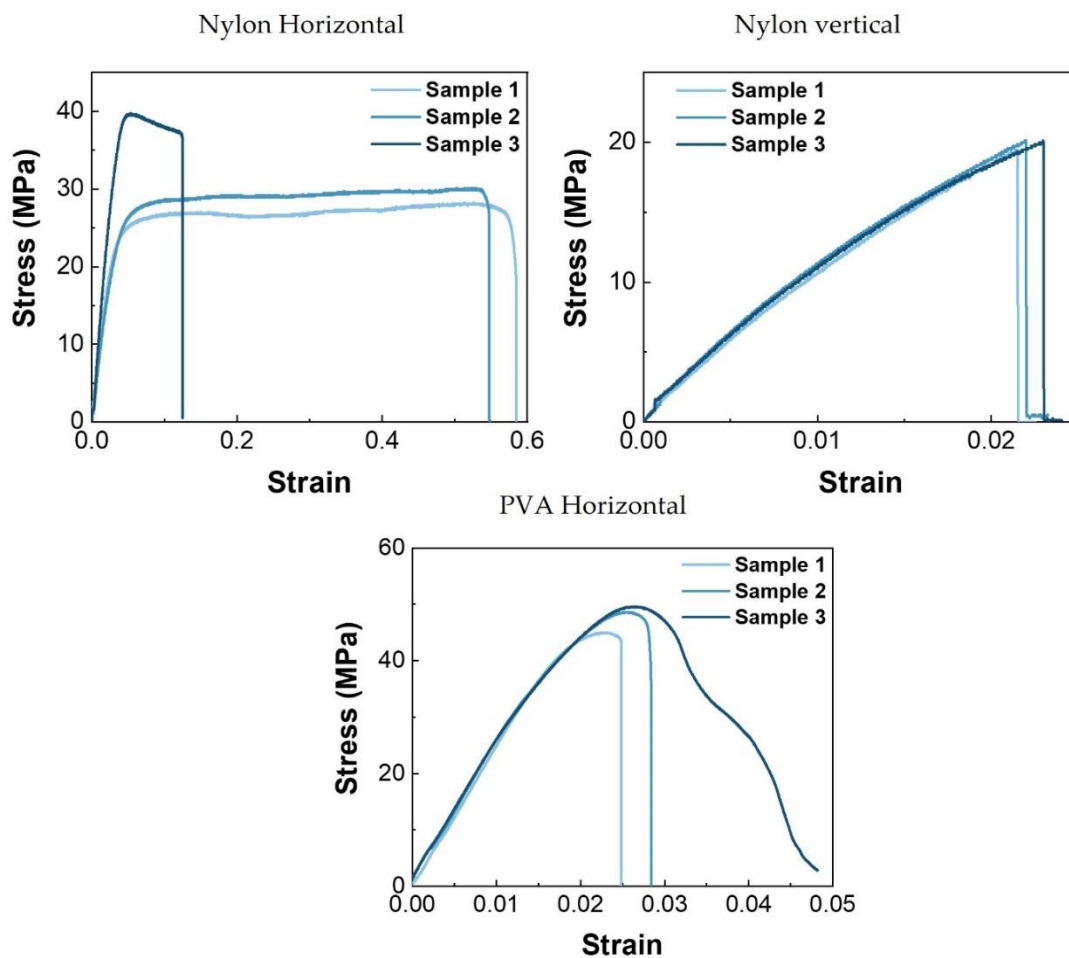


Figure C.1 - Tensile curve of Nylon (horizontal and vertical orientation) and PVA (horizontal orientation).

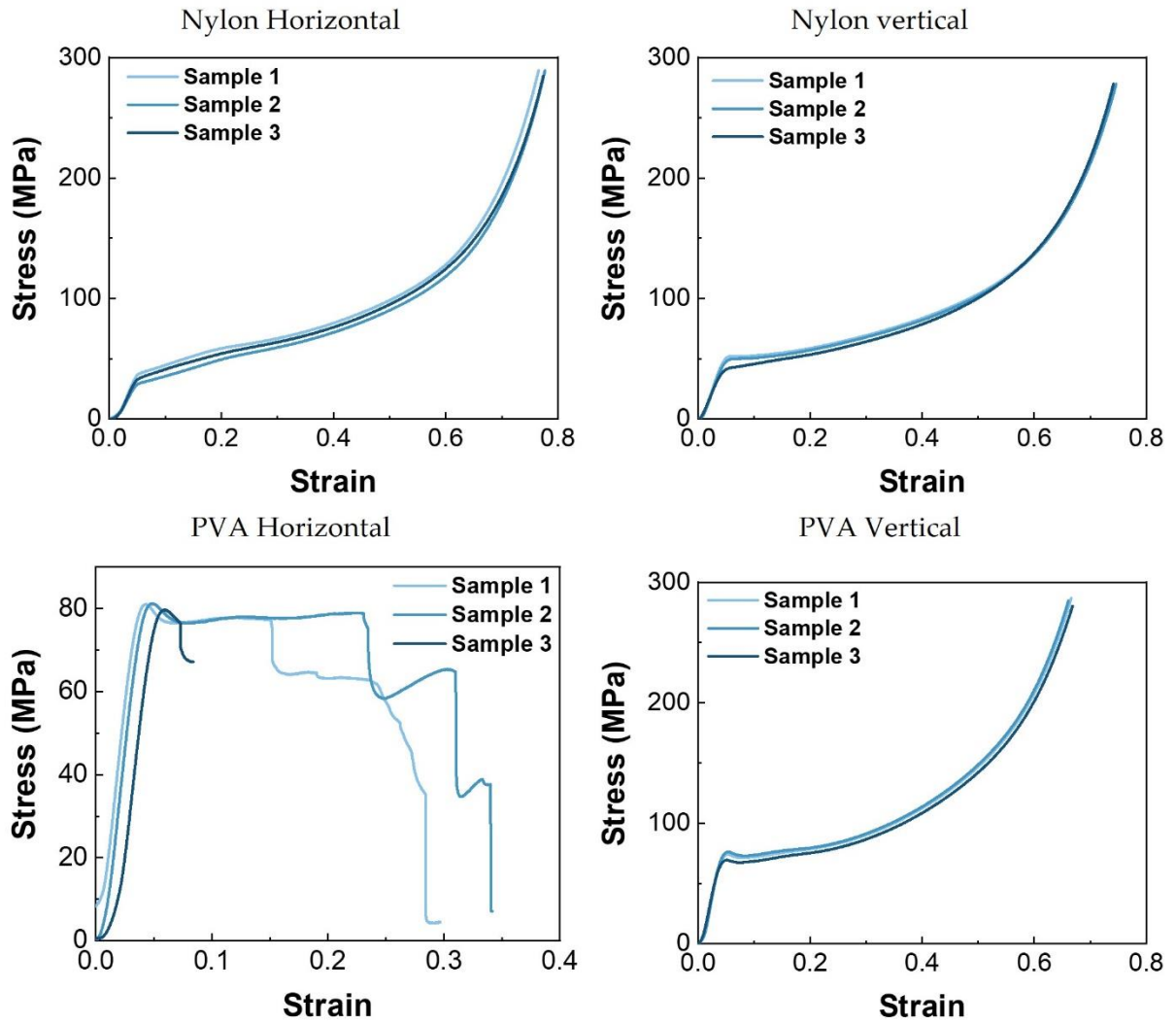


Figure C.2 - Compression curve of Nylon and PVA with the respective printing orientation.

## DIFFERENT CELL ARRANGEMENTS STUDIED

Figure D.1 displays three of the four cells studied using a FE analysis. After the thermal simulations it was observed that the arrangements a) and b) of Figure D.1 show a positive CTE, while the arrangement c) shows a negative CLTE in one of the directions. The main reason for not continuing the study of this arrangement is because it was observed a negative behaviour in only one direction while in the unit cell chosen (Figure 3.2) it has a negative CLTE in two directions.

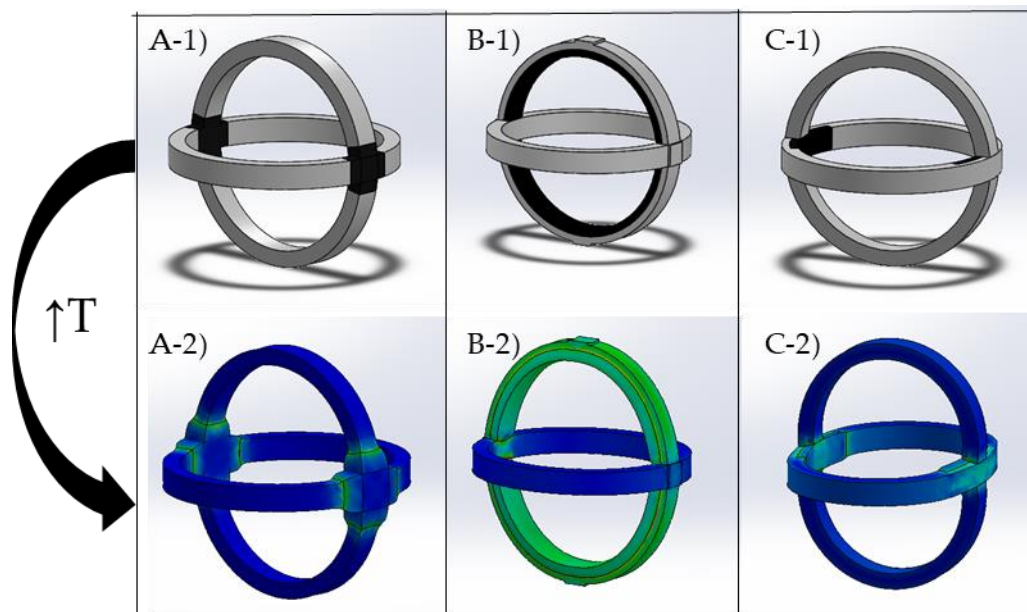


Figure D.1 - Thermal simulation of three different cell arrangements, in white is represented PVA, while in black is Nylon.



# PRINT ATTEMPTS

Table E-1 - Print attempts summary.

Test	q (mm)	t <sub>0</sub> (mm)	Temperature (°C)	Velocity (mm/s)	Support	Notes	Results
Unit cell (only PVA)	3	2	215	30	none		broke cell
Unit cell (only PVA)	3	2	215	25	tree support		broke support
Unit cell (only PVA)	3	2	215	25	none		broke cell
Unit cell (only PVA)	3	2	215	25	tree support		broke cell
Unit cell (only PVA)	3	2	215	25	Custom support		broke cell
Unit cell (only PVA)	3	2	220	20	tree support		broke one of rings
Unit cell (only PVA)	5	5	220	20	none		broke one of rings
Unit cell (only PVA)	5	5	220	20	custom support		printed
Unit cell (only PVA)	5	5	220	20	none		broke one of rings
Unit cell (only PVA)	5	5	205	20	none		Broke

<b>Unit cell (only PVA)</b>	5	5	190	20	none		broke one of rings
<b>Unit cell (only PVA)</b>	5	5	190	20	none	infill pattern: concentric	broke one of rings
<b>Ring (only PVA)</b>	10	5	190	20	none	z hop and retraction	printed
<b>Ring (only PVA)</b>	7,5	5	190	20	none	z hop and retraction	printed
<b>Unit cell (only PVA)</b>	10	5	190	20	none	z hop and retraction	broke
<b>Unit cell (only PVA)</b>	10	5	200	20	none	z hop and retraction	printed
<b>Unit cell (only PVA)</b>	7,5	5	200	20	none	z hop and retraction	printed
<b>Unit cell</b>	7,5	5	200 - 240	20 - 30	none	retraction	printed
<b>Unit cell</b>	5	5	200 - 240	20 - 30	none	retraction	broke
<b>Unit cell</b>	5	5	200 - 245	15 - 30	none	retraction	printed
<b>Unit cell</b>	3	2	200 - 245	15 - 30	none	retraction	broke
<b>Unit cell</b>	5	5	200 - 245	15 - 30	none	retraction	broke
<b>Unit cell</b>	5	3	200 - 245	15 - 30	none	retraction	broke
<b>Unit cell</b>	5	5	200 - 245	25 - 30	none	retraction	printed
<b>Unit cell</b>	5	5	200 - 245	30 - 30	none	retraction	printed
<b>3 x 3 x 3 structure</b>	5	5	200 - 245	30 - 30	Outside cell supports	retraction	broke in several cells
<b>3 x 3 x 3 structure</b>	5	5	200 - 245	30 - 30	support on the outside cells	retraction	broke in several cells

All print attempts were made with a 0.15 layer height and 100 % infill.

# DESIGN OF THE PRINTING PARTS

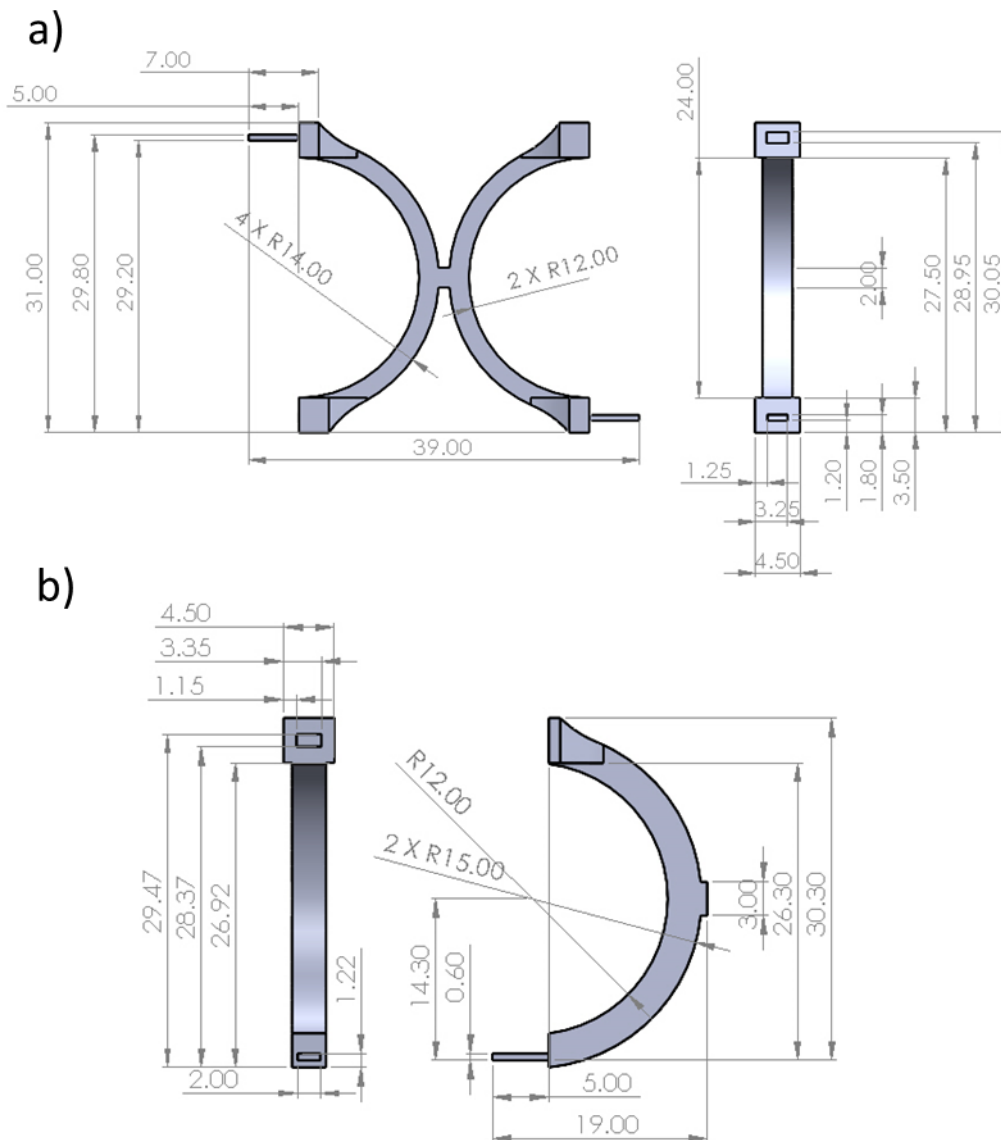


Figure F.1 - Design of the: a) inner connection piece; and b) exterior connection piece.

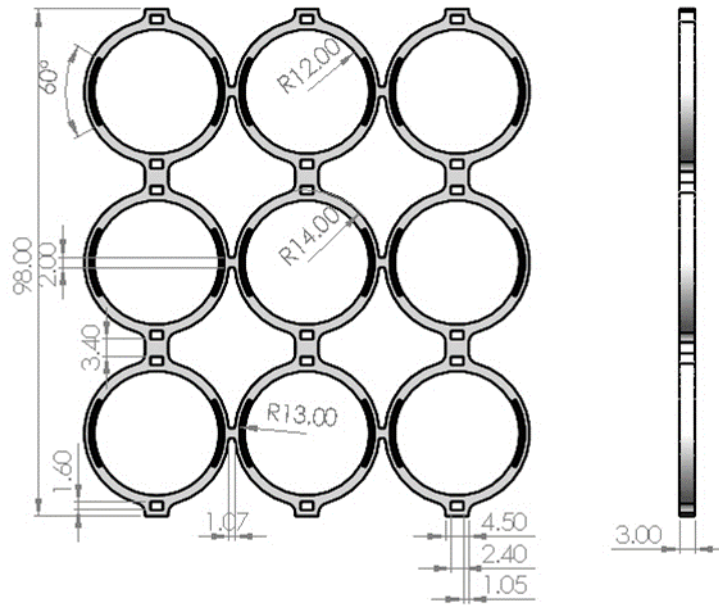


Figure F.2 - Design of the 3 x 3 ring mesh.

Table F-1 indicates the number of pieces necessary to manufacture all the structures used in this work.

Table F-1 - Number of pieces for each structure.

Structure type	Inner connection piece	Exterior connection piece	Ring mesh
Unit cell	0	2	1 (1 ring mesh)
2 x 2 x 2 structure	4	8	2 (2 x 2 ring mesh)
3 x 3 x 3 structure	18	18	3 (3 x 3 ring mesh)

# UNIT CELLS STRESS-STRAIN CURVES

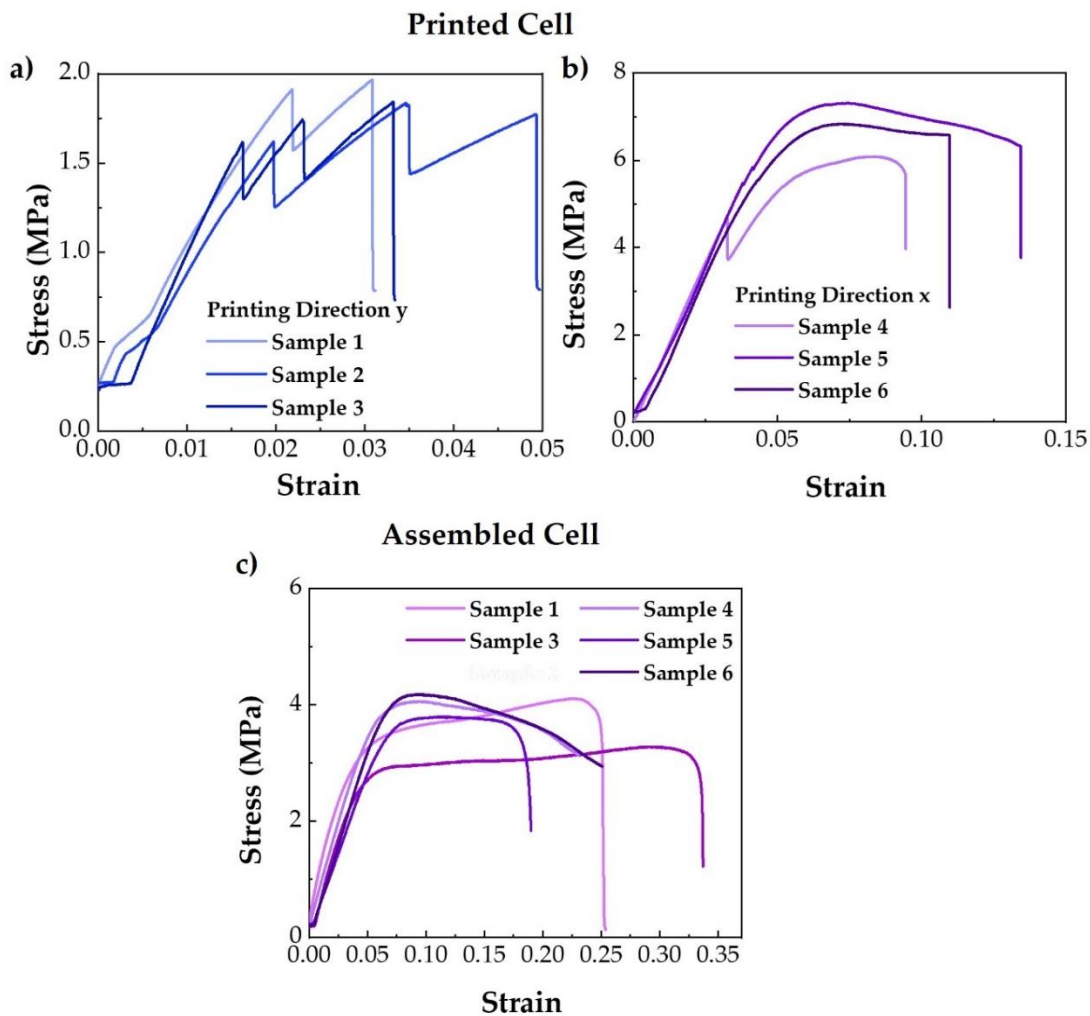


Figure G.1 - Stress-strain curves of unit cell: a) Printed in direction y; b) Printed in direction x; c) Assembled cell.



# UNIT CELL PR

Figure H.1 shows an example of a compressive test of a unit cell. The red circles indicate the dots used to determine the strain in x and y direction. The relative displacement of the compression cylinder was used as the strain on the z direction.

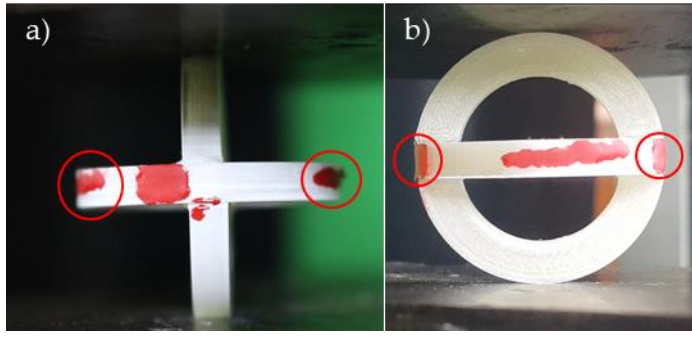


Figure H.1 - Example of a compressive test of a unit cell a) view of the xz plane; b) view of the yz plane.

The figure below indicates the PR of the assembled cells.

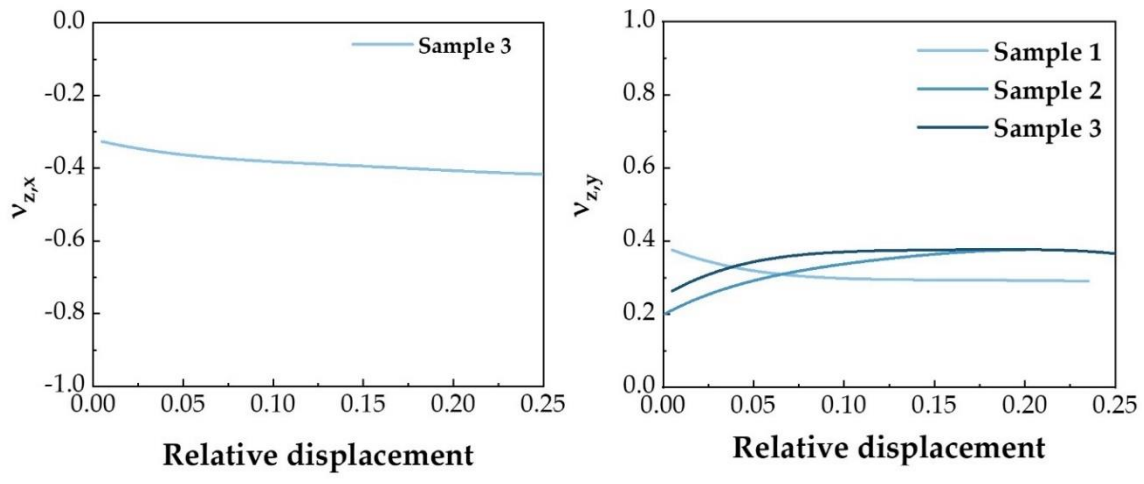


Figure H.2 - PR of the assembled cells.



## 2 x 2 x 2 STRESS-STRAIN CURVES

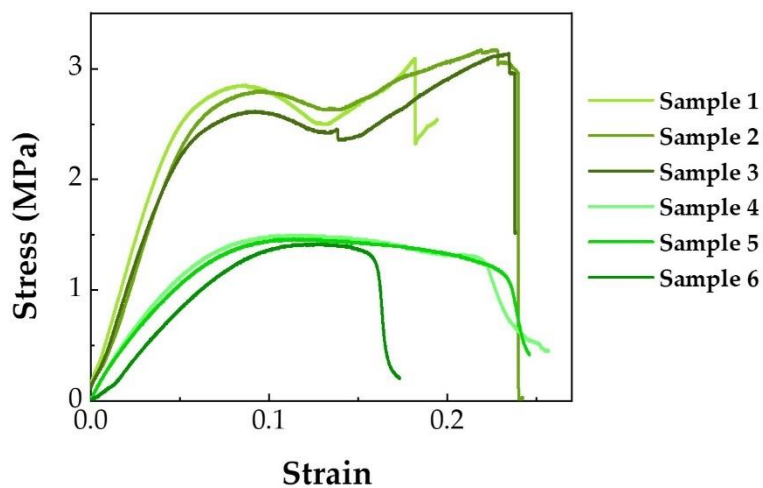


Figure I.1 - Stress-strain curves of 2 x 2 x 2 structure.



| J

## 2 x 2 x 2 PR

Figure J.1 shows an example of a compressive test of a 2 x 2 x 2 structure. The red circles indicate the dots used to determine the strain in x and y direction. The relative displacement of the compression cylinder was used as the strain on the z direction.

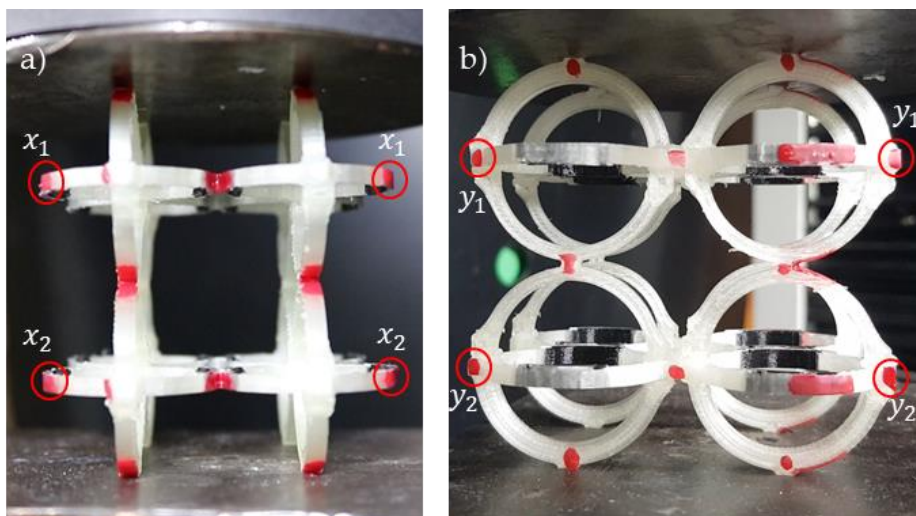


Figure J.1 - Example of a compressive test of a 2 x 2 x 2 structure a) view of the xz plane; b) view of the yz plane.

Figure J.2 shows the PR of the 2 x 2 x 2 structures. It is possible to observe that sample 3 is an outlier. For that reason, it was not accounted for the average calculation of PR.

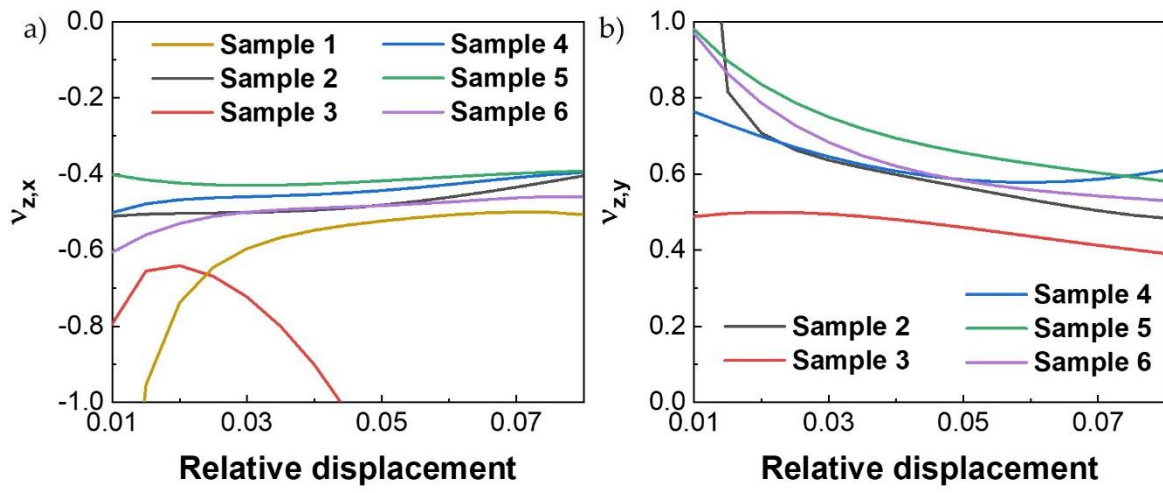


Figure J.2 - PR of the  $2 \times 2 \times 2$  structure.

### 3 x 3 x 3 STRESS-STRAIN CURVES

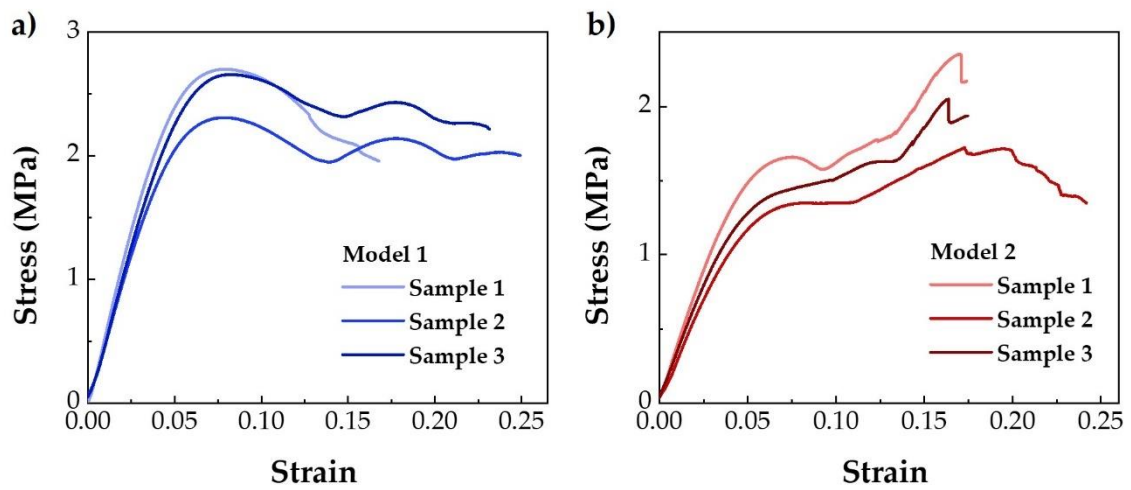


Figure K.1 - Stress-strain curves of 3 x 3 x 3 structure a) model 1; b) model 2.



| L

### 3 x 3 x 3 PR

Figure L.1 shows an example of a compressive test of a 3 x 3 x 3 structure. The blue circles indicate the dots used to determine the strains.

The exterior dots of the samples were used to determine the PR of the structure. For the strain in x and y directions, the distance between points 1 and 4, 6 and 9, and 11 and 14 of Figure L.1 a) and b), respectively, were used. In both views, the distance between dots 1 and 11, 2 and 12, 3 and 13, and 4 and 14 were used to calculate the strain in z direction.

To determine the PR of the central cell it was used the dots 5, 7, 8 and 10 of Figure L.1.

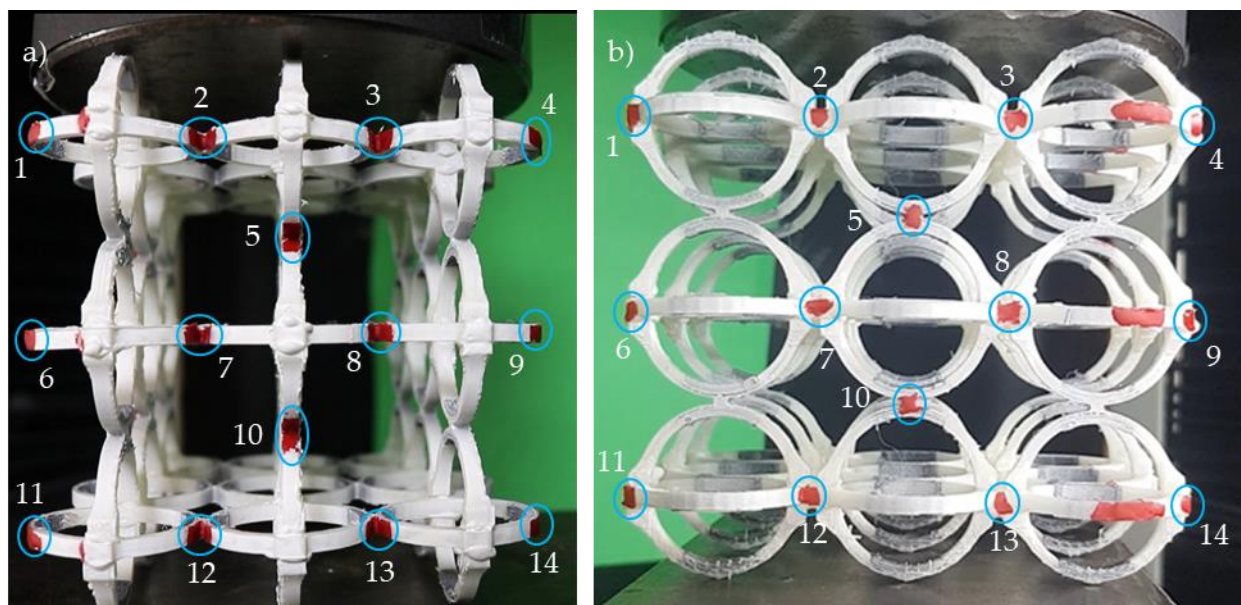


Figure L.1 - Example of a compressive test of a 3 x 3 x 3 structure a) view of the xz plane; b) view of the yz plane.

Figure L.2 shows the PR of the  $3 \times 3 \times 3$  structures and central cell of model 1 and model 2. For the calculation of the average PR some samples or zones of the sample were discarded for being outliers.

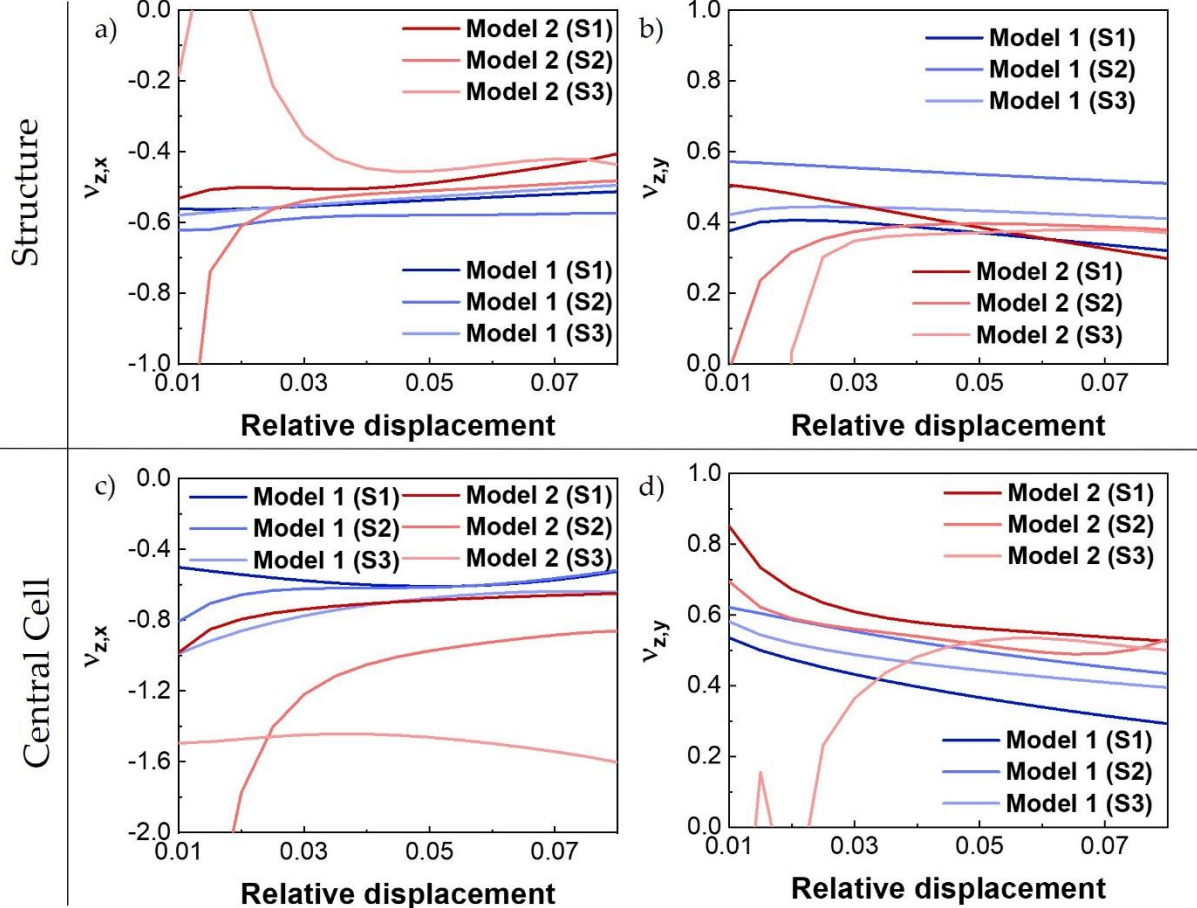


Figure L.2 - PR of the structure and central cell of model 1 and 2

## CLTE CALCULATION OF 3 x 3 x 3 STRUCTURE

Figure M.1 shows an example of a thermal test of a 3 x 3 x 3 structure. The blue circles indicate the dots used to determine the CLTE. For the determination of CLTE in x and y directions, the distance between points 4 and 5, 6 and 7, and 8 and 9 of Figure M.1 a) and b), respectively, were used. In both views, the distance between dots 1 and 10, 2 and 11, and 3 and 12 were used to calculate the CLTE in z direction.

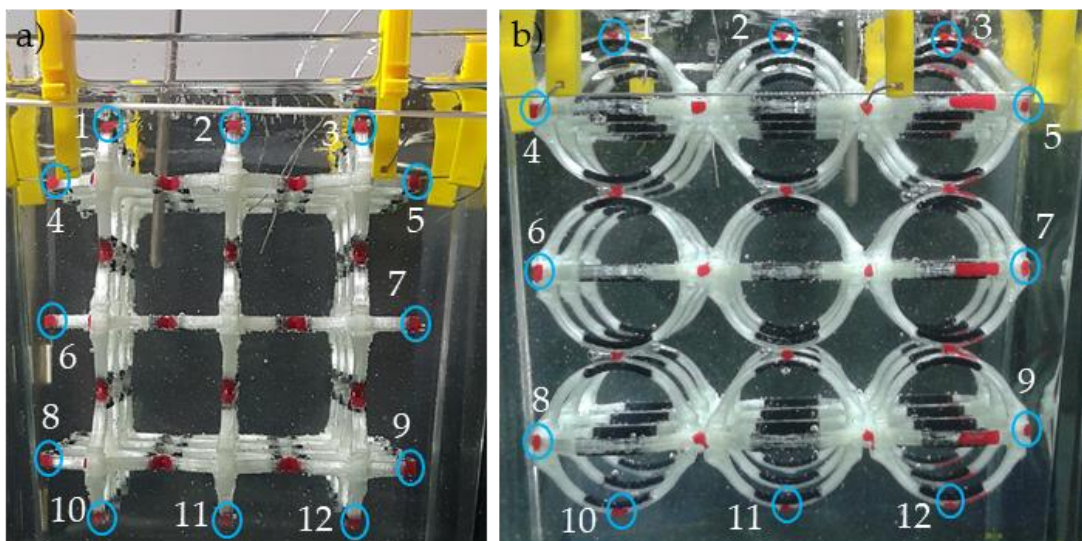


Figure M.1 - Example of a thermal test of a 3 x 3 x 3 structure a) view of the xz plane; b) view of the yz plane.



|N

# PRINTED HEXAGONAL GEOMETRY STRUCTURE

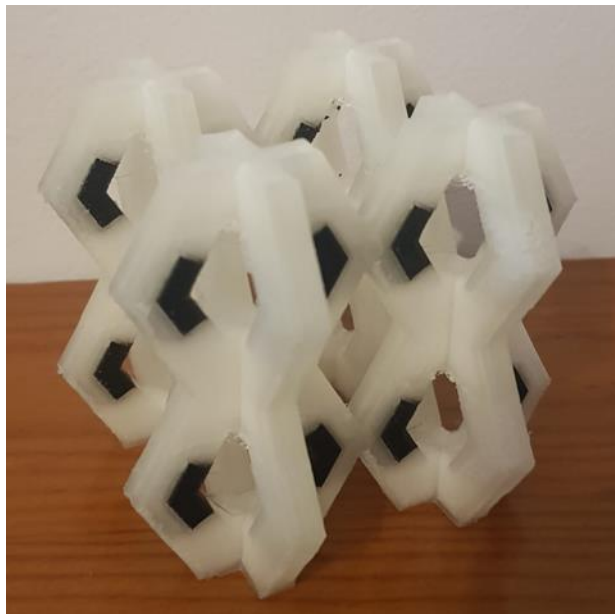


Figure N.1 - A 2 x 2 x 2 hexagonal geometry structure printed at once.





2021

Gonçalo Catatão

Additive Manufacturing and Characterization of Three-dimensional Anepectic

