MATERIALE PLASTICE
https://revmaterialeplastice.ro
https://doi.org/10.37358/Mat.Plast.1964

Effect of Process Parameters on the Hardness of 3D-printed Thermoplastic Polyurethane that Includes Foaming Agent

MARIANA CRISTIANA IACOB[1], DIANA POPESCU[1*], FLORIN BACIU[2]
[1] National University of Science and Technology Politehnica Bucharest, Department of Robots and Production Systems, 313 Splaiul Independentei, 060042, Bucharest, Romania
[2] National University of Science and Technology Politehnica Bucharest, Department of Strength of Materials, 313 Splaiul Independentei, 060042, Bucharest, Romania

Abstract: *Recent progress in Material Extrusion-based Additive Manufacturing (MEX) has introduced active foaming agents in filaments composition, thus allowing for the tuning, by various process parameters, the hardness and the mechanical behavior of 3D-printed parts. In case of thermoplastic polyurethane (TPU) filaments, these advances significantly broaden the range of applications, particularly in the domains of comfort and orthotics (wrist-hand orthoses, insoles), offering the dual benefits of design flexibility inherent in MEX and the comfort of lightweight and customizable structures. However, the field is still in its early stages, with only a limited number of research efforts dedicated to characterizing these novel materials. In this context, this study is focused on determining the influence of printing temperature (190°C, 220°C, 240°C), infill density (25%, 35%, 45%) and infill pattern (honeycomb, gyroid) over the hardness of cylindrical specimens made of Colorfabb varioShore TPU. A comprehensive methodology of calibration is also presented as mandatory for obtaining good quality and accurate products by establishing correlations between flow rate and printing temperatures. The findings showed that the printing temperature is the most relevant factor impacting the hardness of varioShore TPU prints. At a printing temperature of 190°C, which corresponds to less foamed prints, the honeycomb infill yielded higher hardness compared to the gyroid infill, but the difference was not significant. Also, at 220°C and 240°C, the mean values of hardness remain relatively consistent, regardless of infill density and pattern.*

Keywords*: 3D printing, varioShore, foaming agent, hardness, calibration, flow, process parameters*

1. Introduction

Recent advancements in the development of new filament materials for Material Extrusion-based Additive Manufacturing (MEX) [1] process have begun to explore the use of active foaming technology [2] allowing producing 3D prints with customizable mechanical properties by adjusting various process parameters that influence the density (foaming) within the part. Moreover, it is possible to achieve similar or different properties within the same part with only one deposition nozzle. Figure 1 illustrates several examples where spare parts, comfort items and orthotic prints were manufactured using MEX from thermoplastic polyurethane (Colorfabb variosShore TPU), as opposed to the more commonly known and used rigid polymers (ABS – acrylonitrile butadiene styrene, PETG - polyethylene terephthalate glycol, PLA–polylactic acid, etc.), showcasing the expanded range of potential applications in this context. If ABS is used for manufacturing customized surgical guides [3], PLA for wrist-hand splints [4], TPU is recommended for customized insoles for diabetic foot, for instance [5]. Currently, the filaments for MEX process can incorporate active foaming agents. These agents, triggered by factors such as printing temperature, induce bubble formation within the material, rendering it porous. Consequently, this alteration modifies 3D print's hardness and weight [6].

Among filaments that incorporate foaming technology, the commercially available options include Colorfabb LW-PLA (Lightweight polylactic acid) and Colorfabb varioShore TPU. Of these, the former has gained increased attention in current literature [7-9]. Therefore, in this study, specific emphasis is placed on the less scientifically explored varioShore TPU [10,11]. This material is suitable for appli-

**email: diana.popescu@upb.ro*

cations requiring flexible parts with variable properties, such as foot orthoses (insoles) and gaskets, shoes, as demonstrated in Figure 1, process parameters like printing temperature, printing speed, fan speed or flow ratio influencing the degree of foaming, hence the hardness and compressive strength [6]. According to producers' data (Colorfabb and Lubrizol Engineered Polymers, The Netherlands) [12], the hardness can be adjusted between shore 92A (not foamed, before 3D printing) and shore 55A (fully foamed) by varying the printing/nozzle temperature between 190-250°C (the nuances of green in Figure 1 correspond to different printing temperature, hence hardness). Therefore, the research question to answer in this study was how the combined effect of infill density, infill patterns and printing temperature, impact 3D print's hardness.

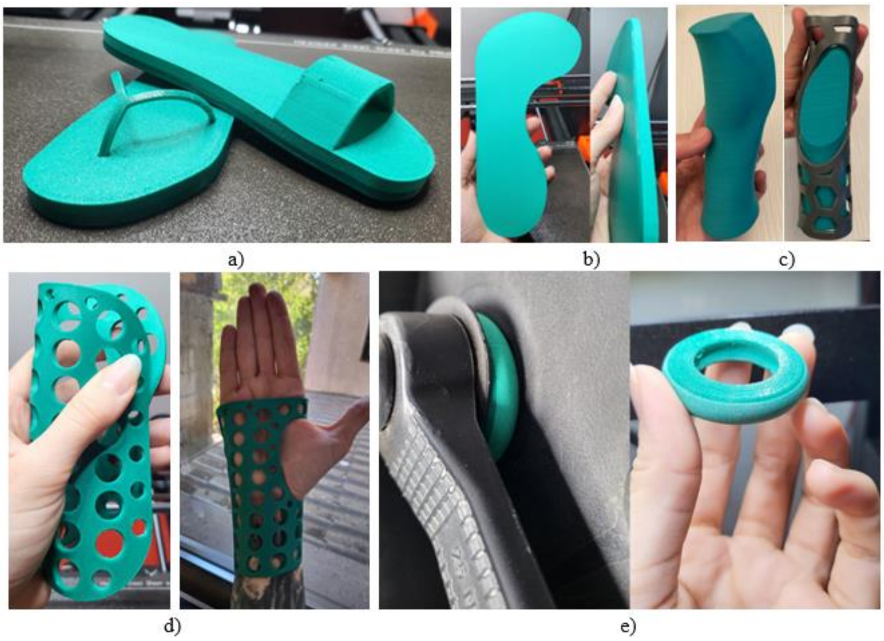

Figure 1. Examples of varioShore TPU parts with different harnesses: a) slippers, b) 3D-printed insole, c) hand dummy for orthoses testing, d) wrist-hand orthosis, e) gasket ring

A literature review on 3D printed TPU characteristics and process parameters was undertaken to document the existing knowledge in the field for different types of elastomers, often used for 3D printing flexible parts. The compressive behavior of 3D-printed TPU specimens with varying infill densities and patterns was investigated in a couple of studies. Research conducted by León-Calero et al. [13] investigated the damping capacity and energy absorption of specimens made of ten types of TPU filaments (among which FilaFlex, PolyFlex, FlexSmart) with different compositions and shore A hardness, and various infill patterns (honeycomb, grid, gyroid) and densities (10%, 20%, 50%, 100%). The results indicated that at larger infill densities, the pattern's effect on compressive strength is not significant. Additionally, the gyroid pattern exhibited lower compressive strength than honeycomb, and the filaments with higher shore A hardness demonstrated greater compressive resistance. The best candidate for compressive resistance was found to be Filaflex 95A with 20-50% infill. Nace et al. [14] studied the concentric (20%, 30%), cross (20%, 30%), cross 3D (20%, 30%) and gyroid (5%, 8%) patterns for comfort application using TPU-Ultimaker filament. The study found that the 2D patterns displayed elasto-plastic buckling of the cell walls, which make them unsuitable for insoles or other products designed for comfort. Yarwindran et al. [15] studied the tensile strength, flexural strength and hardness of Filaflex and NinjaFlex specimens with hexagonal infill pattern with ten densities ranging from 10-100% in increment of 10%. Filaflex specimens showed comparable hardness with the conventional materials used for insoles fabrication, for instance, recommending 3D-printed TPU for such applications. Bates et al. examined NinjaFlex TPU custom designed graded honeycomb structure in impact, quasi-static and cyclic compression tests [16], while in [17] were investigated the mechanical and viscoelastic characteristics of ultra-flexible TPU samples with shore 60A 3D printed at different temperatures. The findings revealed that the greatest tensile strength and elongation at break were obtained at the highest tested temperature of 230°C, whereas the highest storage modulus was observed at 220°C.

The literature review not only that supported collecting information on how infill-related process parameters affect compressive strength and hardness, but also revealed the scarcity of data on the calibration process for TPU [18], specifically concerning varioShore filament [12]. Consequently, this article also introduces, as another objective, a comprehensive methodology for calibrating 3D printers for TPU filaments, and investigates the relationship between printing temperature and flow ratio, as this initial step has an essential role in achieving flexible prints with the desired characteristics.

2. Materials and methods

2.1. Calibration process

The production of all samples was carried out using Prusa MK3s+ 3D printer with E3D Revo extruder, and green varioShore filament with a diameter of 1.75 mm. The tension screw on the extruder gears (Figure 2) was carefully adjusted to accommodate the flexible nature of the TPU filament. As different properties of varioShore TPU parts can be obtained based on changes in the printing temperature, calibrating flow rate vs. printing temperature was necessary for obtaining accurate and consistent dimensions. The calibration process followed the values referenced in [11]. Cubic and hollow rectangular specimens (10 mm x 10 mm x 10mm, 30 mm x 20 mm x 10 mm x 0.65 mm) were used, Table 1 presenting the set of 3D printing parameters used in the calibration process.

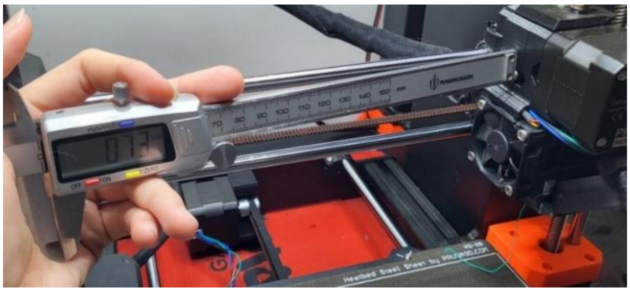

Figure 2. Adjusting the tension screw

In the first step, varioShore filament was 3D printed using the default profile settings provided by Prusa slicer. The extrusion temperature was 220°C and the flow rate was initially set at 85% (Figure 3a). Subsequently, a further test was performed, where the flow rate was adjusted to 58%, based on the values from reference [11] (Figure 3b). Since the values obtained closely matched those in [11], an incremental fine-tuning process was undertaken to achieve the desired dimensions of 0.65 mm for the walls and 10 mm for the cube (Figure 3c). The main objective was to minimize deviations of the flow rate, ensuring a consistent and precise extrusion across the entire 3D printing procedure.

Table 1. 3DP parameters settings for the calibration samples

Specimen	Variable 3DP Parameters	Fixed 3DP Parameters (10x10x10mm)	Fixed 3DP Parameters (30x20x10mm)
	Extrusion temperature [°C]		
1	190	Diameter of filament: 1.75 mm Bed temperature: 50°C Infill density: 30% Infill pattern: rectilinear Perimeters: 2 Top/bottom: 2 layers Line width: 0.65 mm Layer thickness: 0.2 mm Printing speed: 35 mm/s No adhesion	Diameter of filament: 1.75 mm Bed temperature: 50°C Infill density: 0% Infill pattern: none Perimeters: 1 Top/bottom: 0 layers Line width: 0.65 mm Layer thickness: 0.2 mm Printing speed: 35 mm/s No adhesion
2	200		
3	210		
4	220		
5	230		
6	240		
7	250		

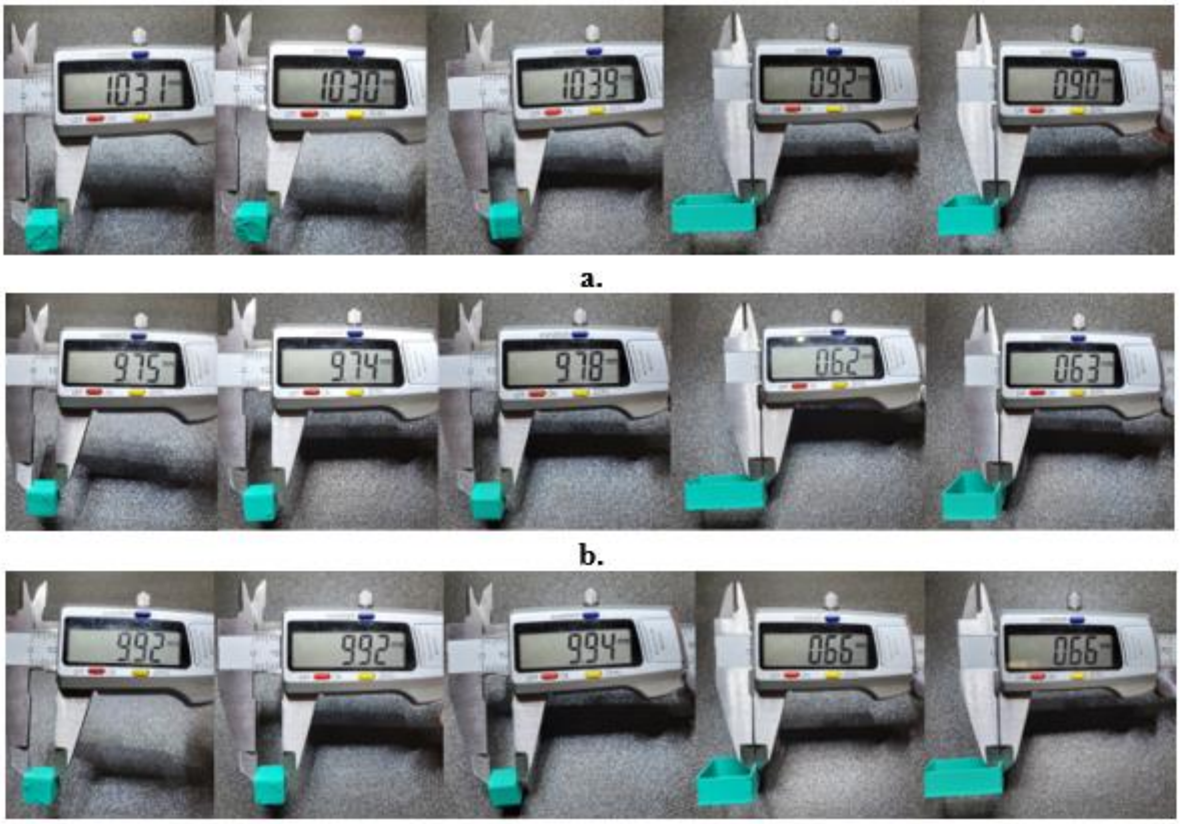

Figure 3. Calibration test - Overall dimensions (x, y, z) and line width: a. 85% flow rate; b. 58% flow rate; c. 60% flow rate (220°C)

Subsequently, the temperature calibration process was conducted by initially 3D printing test samples using the flow rate mentioned in the reference [11]. Employing a similar methodology as in Figure 3, the objective was to achieve precise layer thickness throughout the manufacturing process (Figure 4). The systematic calibration methodology adopted in this study aimed to guarantee consistent and accurate layer deposition for each temperature variation. Figure 5 is a graphical representation of the flow rate vs. the nozzle temperature.

Figure 4. Calibration parts for seven printing/nozzle temperatures

Table 2. Dimensions and weights of the calibration parts

Item	Variable 3DP Parameters		Specimen			
			Cube		Hollowed rectangle	
	Extrusion temperature [°C]	Flow (%)	Accuracy (X-Y-Z) [mm]	Weight [g]	Accuracy (X-Y) [mm]	Weight [g]
1	190	125	10.00 – 10.03 – 10.01	0.94	0.64 – 0.65	0.75
2	200	72	9.92 - 9.95 – 9.98	0.54	0.65 – 0.66	0.43
3	205	67	9.96 – 9.97 – 9.96	0.48	0.65 – 0.65	0.41
4	210	62	9.95 - 9.96 – 9.97	0.46	0.65 – 0.66	0.38
5	220	60	9.92 – 9.92 – 9.94	0.44	0.64 – 0.65	0.37
6	230	68	9.92 – 9.98 – 9.89	0.52	0.65 – 0.65	0.42
7	235	69	9.91 – 9.99 – 9.90	0.50	0.64 – 0.65	0.42
8	240	70	9.98 – 10.00 – 9.82	0.53	0.65 – 0.65	0.43
9	250	72	9.98 – 9.98 – 9.80	0.54	0.64 – 0.65	0.43

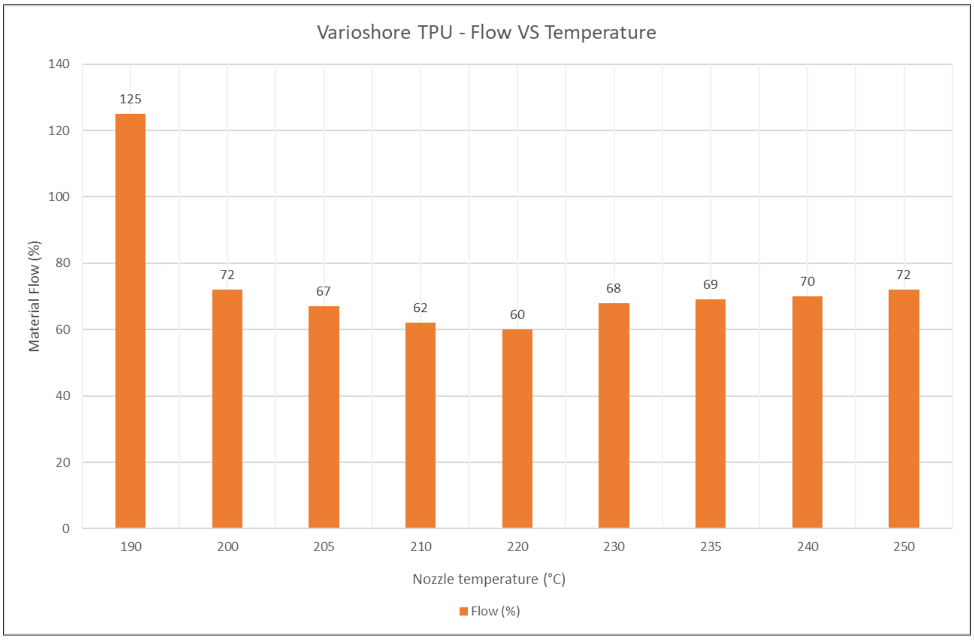

Figure 5. Flow rate vs. temperature

The relationship between nozzle temperature and material flow is critical. At 190°C, the material flow behaves normally, maintaining the expected pattern for a shore 92A TPU filament. However, at 220°C, the occurrence of foaming affects the volume, resulting in an increased porosity within the extruded material. This increased foaming translates to higher volume, thereby necessitating a reduction in the flow rate to restore the extrusion to the desired accuracy level.

2.2. 3D printing specimens

Table 3 displays the process parameters investigated in the study and their respective levels, with three specimens tested for each configuration. The flow rate values were determined through the calibration tests outlined in section 2.1.

Table 3. Specific parameters for cylindrical specimens

Specimen	Variable process parameters	Fix parameters
	*Printing temperature:* Levels: 190°C, 220°C, 240°C *Infill density:* Levels: 25%, 35%, 45% *Infill pattern:* Levels: gyroid, honeycomb	Bed temperature: 0 °C Flow rates: 110%, 60%, 72% Layer thickness: 0.2 mm Top/bottom layers: 4 Perimeters: 0 Printing speed: 35 mm/s Fan speed: 70% Disable for the first 4 layers, full speed at layer 6

2.3. Hardness measurements

The hardness of the specimens was evaluated using a shore A durometer (Digital Shore Durometer Sclerometer, China), which was applied to the compressive samples (Figure 6). Measurements were made in ten points on the surface, hardness variation being recorded as an effect of infill density. Mean values were computed for use in data analysis.

The results were compared with the 100% density parts used as reference (Figure 7).

Figure 6. Samples' hardness measurements (190°C, 220°C)

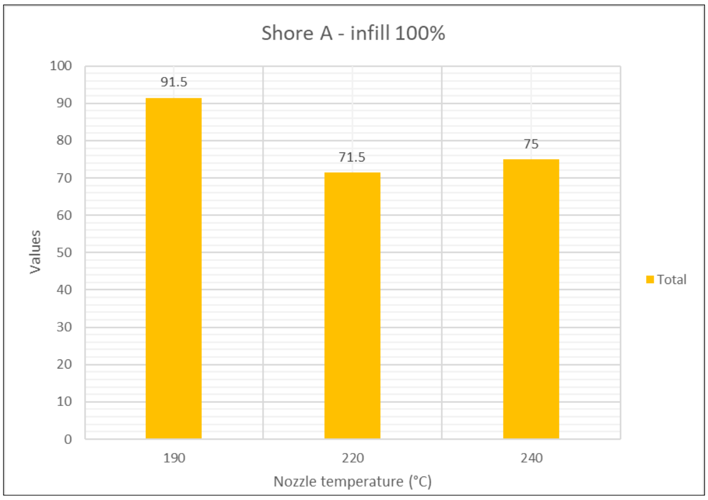

Figure 7. Shore A - compression samples 100% infill density

3. Results and discussions

3.1. Hardness measurements results

From the hardness measurements in ten points, the minimum, maximum, mean average, and deviation from the mean were computed to assess the variance within the set of numbers in relation to their mean (Figures 8-10).

For the samples with density infill of 25% it was observed that honeycomb pattern for 190°C and 220°C yields the largest standard deviation of 2.57 respectively 2.23. The infill density of 35% produced more stable hardness samples with a low standard deviation, about 0.34 for specimen 1, 240°C and 0.52 for specimen 3, 190°C, gyroid. The explanation is that the denser the infill, the less probable it is to measure the shore A hardness between the infill threads. 45% infill density produced the most stable samples with a 0.39 standard deviation for 190°C, gyroid.

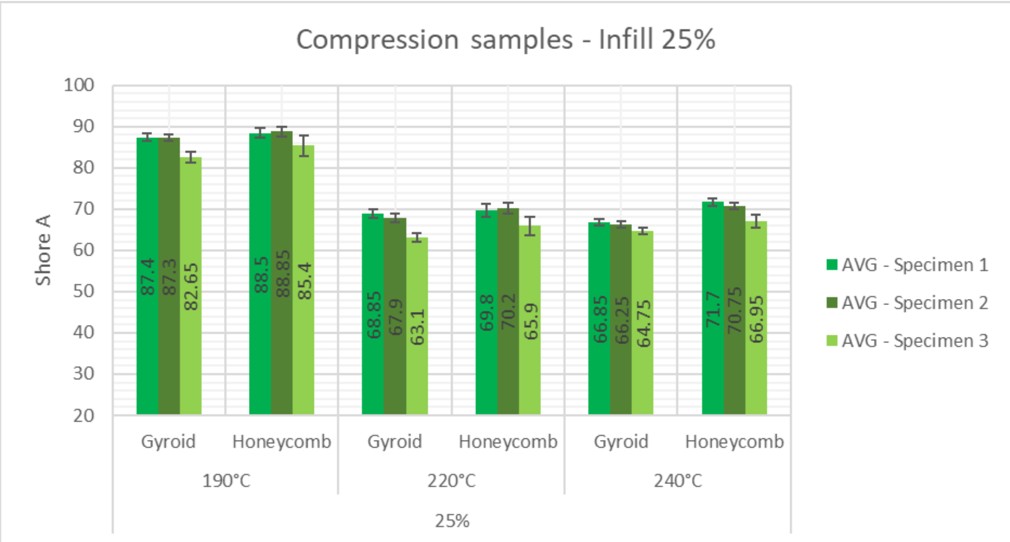

Figure 8. Hardness measurements for the 25% infill density samples (average values per sample)

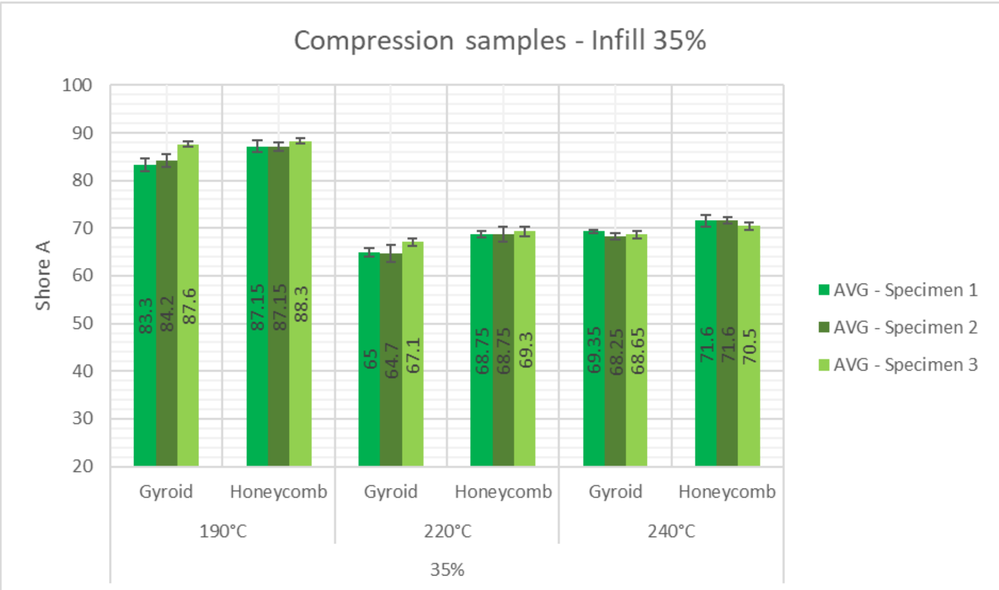

Figure 9. Hardness measurements for the 35% infill density samples (average values per sample)

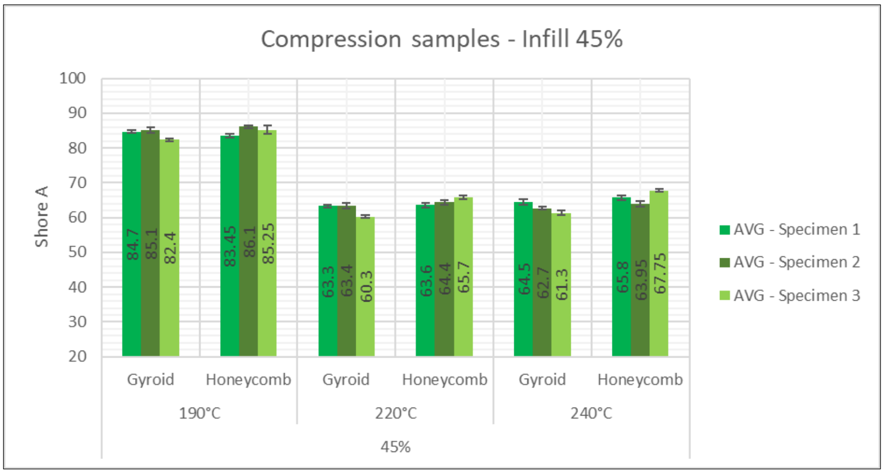

Figure 10. Hardness measurement for the 45% infill density samples (average values per sample)

Table 7 synthetically presents the results of hardness using the mean values for three specimens having the same process parameter configuration. The calculated p-value using one-way ANOVA produced a very low value for the data on printing temperature (significance level 0.05), presented in Table 7, showing there is statistically significant difference on hardness as function on printing/nozzle temperature.

At 190°C, the hardness surpasses that of 220°C and 240°C due to the material's baseline hardness of Shore 92A, consistent with the filament's hardness on the spool. At 190°C, no foaming occurs, preserving its original hardness, as confirmed during the 3D printing process. As the temperature increases to 220°C, the filament exhibits foaming ability, reducing its hardness. However, beyond 220°C, the hardness shows a slight increase again until reaching 250°C.

Table 7. Mean values of hardness for different process parameters configurations

Printing temperature [°C]	Infill density [%]	Infill pattern	Mean hardness
190	25	Gyroid	85.78
		Honeycomb	87.58
	35	Gyroid	85.03
		Honeycomb	87.53
	45	Gyroid	84.06
		Honeycomb	84.93
220	25	Gyroid	66.62
		Honeycomb	68.63
	35	Gyroid	65.60
		Honeycomb	68.93
	45	Gyroid	62.33
		Honeycomb	64.56
220	25	Gyroid	65.95
		Honeycomb	69.80
	35	Gyroid	71.78
		Honeycomb	68.75
	45	Gyroid	62.83
		Honeycomb	65.83

In the absence of similar research on varioShore TPU, the results on hardness could not be straightforwardly compared with other data for literature. Nevertheless, a prior investigation [11] did provide insights into the relationship between compression strength and printing temperature for samples with 100% infill density. It indicated that higher densities corresponded to printing temperatures of

220°C, 205°C, 190°C, and 235°C. However, in [11] it was not included specific information regarding the number of top/bottom layers for the samples, as it primarily focused on compression testing. Another reference regarding varioShore TPU [12] confirmed our findings that specimens exhibited their highest shore A hardness at 190°C.

Figure 11 presents the main effects plots (Minitab, Minitab UK) for examining the influence of the three analyzed process parameters over the samples' hardness. A horizontal line or a line closer to the horizontal indicates that the parameter is not statistically significant. Thus, it can be observed that printing temperature is the most significant factor of influence followed by infill density, while the least important is the infill pattern. When considering the effect of infill density, passing from 35% density to 45% density produces more effect on hardness than density lower than 25%. In the case of printing temperature, the influence of passing from 190 °C to 220 °C is more significant than between 220°C and 240°C. The parallel lines from the interaction plots in Figure 12 showed there is no clear interaction effect between parameters.

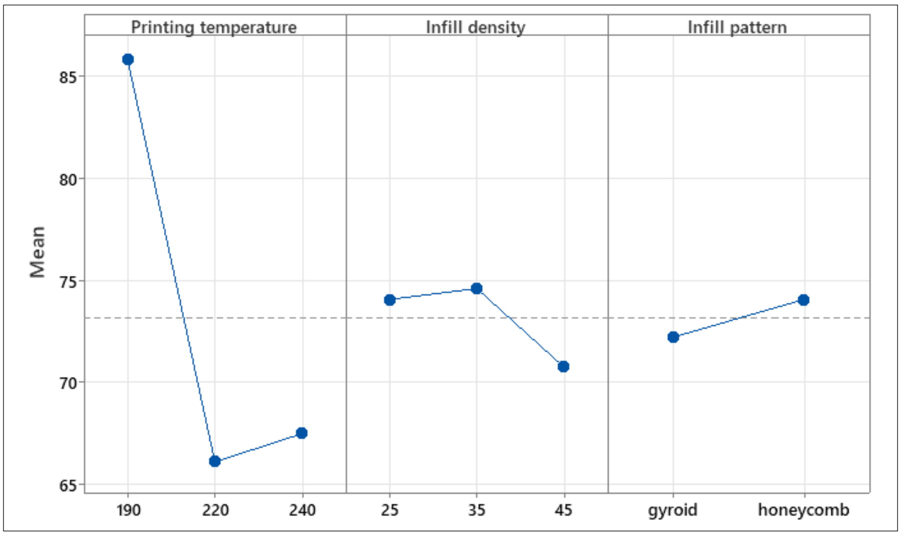

Figure 11. Main effects plot on hardness

The results in [12], for other types of thermoplastic elastomer filaments, showed that pattern does not have a significant influence on compressive strength and that the honeycomb pattern has more compressive strength than gyroid. These observations were confirmed in the current research also for varioShore TPU filament.

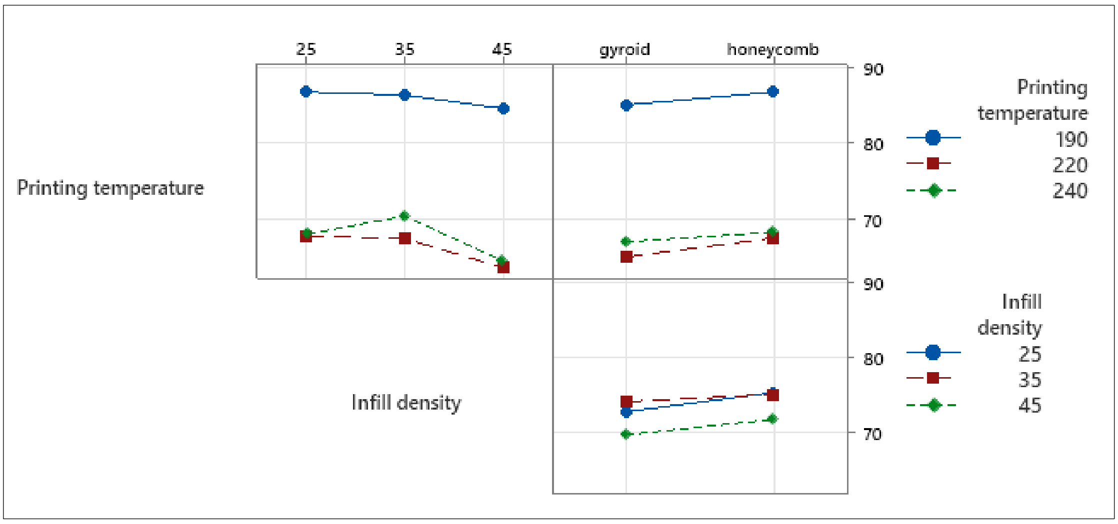

Figure 12. Interaction plots between analyzed process parameters

4. Conclusions

The primary objective of this study was to evaluate the impact of three process parameters, namely printing temperature, infill density and infill pattern, on the hardness of specimens manufactured by MEX from a novel filament, Colorfabb varioShore TPU. This filament, based on active foaming technology, expands the potential applications of 3D printing into the realms of comfort and orthotics by enabling the customization of hardness and compressive strength to meet specific requirements. In this sense, several examples of 3D prints were showcased to demonstrate the capability to produce products with varying hardness levels using a single printing nozzle on a cost-effective 3D printer, this being the practical rationale of the research.

Given the absence of similar research and the significant role of calibration for TPU filaments, considering their flexible nature, a comprehensive methodology for establishing correlations between flow rate and printing temperature was also introduced in this study, as another objective.

The results underscored that printing temperature exerts the most significant influence on hardness, while the infill pattern holds the least importance.

Further research will be focused on analyzing more levels of process parameters for a better approximation of compression strength dependence on these factors which affect the print foaming degree. The final purpose is to be able to identify the set of parameters that allow customization of the cushion factor for foot insoles, or the softness of the contact zone of the hand with the wrist orthosis. This customization aims to prevent pressure sores and enhance comfort without compromising the necessary strength for immobilization.

References

1.***ISO/ASTM 52900:2021 - Additive Manufacturing – General principles. Terminology
2.SUN, B., WU, L., Research progress of 3D printing combined with thermoplastic foaming, *Front. Mater.*, **9**, 2022, https://doi.org/10.3389/fmats.2022.1083931
3.POPESCU, D., LAPTOIU, D., MARINESCU, R., BOTEZATU, I. Design and 3D printing customized guides for orthopaedic surgery – lessons learned, *Rapid Prototyp. J.*, **24**(5), 2018, 901-913. https://doi.org/10.1108/RPJ-05-2017-0099
4.WANG, Z., DUBROWSKI, A. A Semi-Automatic Method to Create an Affordable Three-Dimensional Printed Splint Using Open-Source and Free Software. *Curēus*, **13**(3), 2021, e13934. https://doi.org/10.7759/cureus.13934
5.CHATZISTERGOS, P.E., GATT, A., FORMOSA, C., FARRUGIA, K., CHOCKALINGAM, N,. Optimised cushioning in diabetic footwear can significantly enhance their capacity to reduce plantar pressure. *Gait Posture*. **79**, 2020, 244-250. https://doi.org/10.1016/j.gaitpost.2020.05.009
6.NOFAR, M., UTZ, J., GEIS, N., ALTSTÄDT, V., RUCKDÄSCHEL, H. Foam 3D Printing of Thermoplastics: A Symbiosis of Additive Manufacturing and Foaming Technology. *ADV SCI*. **9**(11), 2022, 2105701. https://doi.org/10.1002/advs.202105701
7.DAMANPACK, A. R., SOUSA, A., BODAGHI, M. Porous PLAs with Controllable Density by FDM 3D Printing and Chemical Foaming Agent. *Micromachines*, **12**, 2021, 866 https://doi.org/10.3390/mi12080866
8.SUNDAR, A., BAIN, S., A Feasibility Study of Lightweight Polylactic Acid Fused Deposition Modeled Propeller Prototyping. *J. Stud. Res.*, **11**(3), 2022 https://doi.org/10.47611/jsrhs.v11i3.2797
9.CROWE, S., MAXWELL, S., BRAR, H. *et al.* Use of light-weight foaming polylactic acid as a lung-equivalent material in 3D printed phantoms. *Phys. Eng. Sci. Med.*, 2023, https://doi.org/10.1007/s13246-023-01318-4
10.KALAYCIOĞLU, S.G, Thesis - Mechanical Behaviour Of Polymeric Lattice Structures Produced By Additive Manufacturing, 2022, available at https://open.metu.edu.tr/handle/11511/101241 (accessed September the 14th 2023)

11.***Testing colorFabb varioShore TPU - Foaming 3D printing filament. CNC Kitchen. Available online:https://www.cnckitchen.com/blog/testing-colorfabb-varioshore-tpu-foaming-3d-printing filament (accessed on 26-10-2023)
12.***Datasheet ColorFabb VarioShore TPU – https://colorfabb.com/media/datasheets/tds/colorfabb/TDS_E_ColorFabb_varioShore_TPU.pdf
13.LEÓN-CALERO, M., REYBURN, V. S. C., MARCOS-FERNÁNDEZ, Á., RODRÍGUEZ-HERNANDEZ, J., 3D Printing of Thermoplastic Elastomers: Role of the Chemical Composition and Printing Parameters in the Production of Parts with Controlled Energy Absorption and Damping Capacity. *Polymers*, **13**, 2021, 3551. https://doi.org/10.3390/polym13203551
14.NACE, S.E., TIERNAN, J., HOLLAND, D., NI ANNAIDH, A., A comparative analysis of the compression characteristics of a thermoplastic polyurethane 3D printed in four infill patterns for comfort applications, *Rapid Prototyp. J.*, **27**(11), 2021, 24-36, https://doi.org/10.1108/RPJ-07-2020-0155
15.YARWINDRAN, M., SA'ABAN, N. A., IBRAHIM, M., PERIYASAMY, R., Thermoplastic elastomer infill pattern impact on mechanical properties 3D printed customized orthotic insole. *ARPN J. Eng. Appl. Sci*., **11**, 2016, 6519–6524
16.BATES, S. R. G., FARROW, I. R., TRASK, R. S., Compressive behaviour of three-dimensional printed thermoplastic polyurethane honeycombs with graded densities, *Mater. Des*, **162**, 2018, 130-142, https://doi.org/10.1016/j.matdes.2018.11.019
17.OMER, Y. G., RECEP, İ., BERAT, E. C., Effect of Printing Temperature on Mechanical and Viscoelastic Properties of Ultra-Flexible Thermoplastic Polyurethane in Material Extrusion Additive Manufacturing. *J. Mater. Eng. Perform*., **31** (5), 2022, 3679–3687. https://doi.org/10.1007/s11665-021-06510-9
18.PIVAR, M., GREGOR-SVETEC, D., MUCK, D., Effect of Printing Process Parameters on the Shape Transformation Capability of 3D Printed Structures. Polymers (Basel). 2021 (1):117, 2021, https://doi.org/10.3390/polym14010117

Manuscript received: 30.10.2023