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Effect of process parameters on the thermal properties of material extruded AM parts

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Abstract. This research focuses on the thermal characterization of 3D-printed parts obtained via Material Extrusion Additive Manufacturing using various process parameters. Differential Scanning Calorimetry and Thermal Conductivity measurements were used to evaluate the samples' thermal characteristics and heat transport behavior. The experimental results showed a significant influence of some parameters, such as wall layer count and layer thickness, on the thermal behavior of the printed part.

Introduction

Additive manufacturing (AM) is a widespread research technique for engineering and biomedical applications. Unlike traditional machining, AM utilizes either an energy source or an extruder to build parts by selectively melting and solidifying materials. An energy source such as a laser or an electron beam can fuse metal powders or wires, resulting in a solid part. Alternatively, an extruder can dispense feedstock materials like plastic, ceramic, or metal, which are then fused using heat or chemical reactions to form a solid part. In either approach, the material is built up layer-by-layer until the final shape is achieved [1]. This technique allows for close control of the fabrication process and greater design freedom for traditional machining, introducing challenges related to functional properties. While AM has long been used for rapid prototyping, the recent focus has been on building functional parts capable of withstanding thermal/mechanical loads. Filament-based Material Extrusion (MEX), according to ISO/ASTM 52900 classification, is a commonly used AM process relying on an extruder dispensing polymer above its glass transition temperature. However, given the AM complexity, it is essential to understand the fundamental transport processes and the dependence of functional properties on process parameters to optimize the process [2]. MEX-built parts have been reported to have anisotropic mechanical properties with the highest strength in the direction of the material [3][4]. The merging of adjacent polymer lines, known as roads, plays a critical role in determining the microstructure and functional properties of the built part [5]. This merging process depends on various process parameters and material properties, which have been studied previously [2][6]. It is vital to comprehend and enhance this merging process to attain high-quality parts.

While there is significant research on the mechanical properties of MEX parts [2]-[4],[6]-[8], a relatively small amount of work has been done on the thermal properties and their correlation with microstructure and process parameters. Thermal conductivity is a crucial property governing heat flow through the part. It is essential in engineering applications where heat generation and flow are critical. The thermal conductivity of the MEX part differs from that of the original material, expecting to vary between the raster and build directions. For this reason, a direct measurement of the thermal properties of the final part is essential. The in-plane and out-of-plane thermal conductivities of MEX-built parts were measured by Shemelya et al. using the transient plane source method [9]. Defects in the printing process can cause variations in thermal properties. Improper adhesion caused by poorly designed process parameters can determine inadequate heat flow, leading to low thermal conductivity. Chung et al. [10] have conducted measurements to investigate the impact of random voids on thermal properties. Ravoori et al. [11] investigated the effects of process parameters on the thermal properties of AM parts by using a one-dimensional heat flux method to measure the thermal conductivity in the build direction. Their analysis revealed a significant correlation between thermal conductivity, raster speed, and layer thickness, further supported by high-speed imaging of the printing process conducted at varying levels of these parameters. The anisotropy of thermal conductivity in AM components is challenging, as highlighted by Prajapati et al. [12]. Their study reveals the fundamental reason for this anisotropy is the strong interfacial thermal contact resistance in the build direction. They provide valuable data on the dependence of this parameter on process conditions. The measurements show significant anisotropy in thermal conduction, with thermal conductivity in the Z-direction being significantly lower than in the X-direction. This anisotropy is influenced by the air gap between roads during deposition. Thermal conductivity is an essential property of many materials, and various methods have been developed to measure it. Thermal conductivity describes the mode of heat transfer via conduction and can be affected by multiple factors such as temperature, pressure, and material composition. The measured property may be referred to as effective thermal conductivity if other modes of heat transfer, such as convection and radiation, are significant. Thermal conductivity can be measured through steady-state or transient methods using a specimen of simple geometry in contact with a heat source and one or more temperature sensors. Transient methods may be contact or non-contact and involve generating a dynamic temperature field within the specimen and measuring the temperature response over time. Depending on the setup, one or more thermo-physical properties can be obtained. The response is analyzed using a model and a set of solutions designed for the specific geometry and boundary conditions. The Transient Hot Wire (THW) method and the Laser-Flash Analysis (LFA) are commonly used to measure thermal conductivity. In recent research, an excellent agreement was found between THW and LFA values measured on the same components in the same conditions [13][14]. The THW method offers a low-cost alternative to LFA with reduced accuracy. The Temporary Hot Bridge (THB) is an evolution of the THW. It provides fast, precise measurements of thermal conductivity, thermal diffusivity, and volumetric specific heat from a single experiment on solids.

This work aimed to analyze the thermal conductivity of samples made with MEX in Polylactic acid (PLA) using the THB method. The key parameters were the infill percentage, which indirectly affects the air gaps between the lines and the number of top/bottom layers. During the study, the variation of the two factors above was analyzed to investigate their effect on the thermal properties while keeping the other process parameters constant. Additionally, an analysis was conducted to evaluate the surface roughness.

Materials and methods

A commercial black PLA 1.75 mm filament (AzureFilm d.o.o., Slovenia) was used as the material for producing the samples. The filament was dried for 4 hours at 40°C in an oven to prevent issues related to potential moisture presence and then stored in a vacuum-sealed bag before starting the printing process. This study did not consider the influence of additives on base PLA material [15]. The samples were produced on a commercial 3D printer, Flying Bear Ghost 4S (Zhejiang Flying Bear Intelligent Technology Co., Ltd.), with a build volume of 255×210×210 mm^3. The printer has a 0.4 mm brass nozzle and a Polyetherimide (PEI) print bed to ensure high adhesion while limiting warping phenomena.

The process parameters to investigate were selected after some preliminary testing. A line width equal to 0.4 mm followed the nozzle diameter of 0.4 mm, and the layer height was set to half the line width to maintain dimensional accuracy and acceptable quality. The crosshatch raster strategy with angles ±45° helped slightly contain anisotropy among the top and bottom layers. The wall layer was set to three in the slicer (Ultimaker Cura) to ensure the shell could clamp with a vise. In MEX, the wall layer refers to the outer layer of the printed object. Increasing the number of wall layers can make a part more robust and resistant to external forces. Lower values were considered as not strong enough. During the printing of the samples, the number of top and bottom layers and the infill percentage were adjustable, while the other process parameters remained constant. The top and bottom layers refer to the layers of material that make up the top and bottom surfaces of the printed object, respectively. Infill percentage refers to the material used to fill the object's interior. Table 1 reports the values of the process parameters.

Table 1. ***Process parameters of MEX***

Process Parameter	Value	Unit size
Nozzle temperature	210	[°C]
Bed temperature	60	[°C]
Printing speed	60	[mm/s]
Layer height	0.2	[mm]
Line width	0.4	[mm]
Wall layer count	3	-
Infill strategy	Grid	-
Layer raster strategy	-45°/+45°	-

To explore how the number of layers and infill percentage impacted the thermal conductivity of 70×40×10 mm^3 prismatic parts, a complete factorial Design of Experiments (DoE) was employed, as shown in Table 2.

Table 2. Complete factorial Design of Experiment

Factors	Name	Level 1	Level 2	Level 3	Level 4
Layer number	L_N	2	4	6	8
Infill percentage [%]	I_P	20	40	60	80

As a result of the material's properties, the sample surface was measured with the scanControl 2900-50 BL (Micro-Epsilon Messtechnik GmbH & Co. KG), a 2D/3D blue laser scanner and profilometer designed for measuring semi-transparent, red-hot glowing, and organic materials with a 405 nm wavelength laser. The scanner features a resolution of 4 µm on the Z-axis, considering the object's height growth direction, and 1,280 points/profile resolution on the X-axis. The specimens' top surface roughness Ra was also measured using the Surtronic 3P surface roughness profilometer (Taylor-Hobson, UK) with a cut-off value of 0.8mm to confirm the previous measurements.

The THB instrumentation (Linseis Messgeräte GmbH, Germany) detected the thermal conductivity without sample preparation. The THB method measured the time for a thermal signal to propagate through a material sample. The measurement was performed dynamically by applying a short-duration heat pulse to the thermal bridge, which exchanged heat with the sample. The propagation time of the thermal signal through the sample was then used to calculate the material's thermal properties. The instrumentation consisted of a flexible sensor (THB-B type) pressed manually between two flat surfaces of the specimen (Figure 1-left) to ensure good thermal contact without air inclusions. The specifications of the sensor are as follows: dimensions of 42×22 mm^2, a thermal conductivity range of 0.1÷2 W/m×K, as well as a temperature range of -150÷200 °C. The tests were conducted at room temperature according to UNI-EN ISO 22007 standard, with an execution time of 160 s and a pulse supply current of 50 mA. A non-uniform temperature profile is generated, driving a Wheatstone bridge off-balance to produce an offset-free output. A heater R_H created a time-dependent, distant-dependent temperature field along the sensor and the material under test. Additional resistors were connected to a Wheatstone bridge with a bridge voltage *UB* dependent on the temperature difference ΔT. Measuring the temperature difference ΔT enabled the calculation of thermal conductivity λ using the following formula:

$$\boldsymbol{\lambda} = \frac{\boldsymbol{\Phi}}{\mathbf{2} \times \boldsymbol{\pi} \times \Delta \boldsymbol{T}} \times \boldsymbol{k} \tag{1}$$

The heater R_H generated the applied thermal power Φ supplied with current, while k was the sensor calibration factor. The THB evaluation is refined to enable anisotropic materials' direction-dependent thermal diffusivity measurement. This new method requires only plate-shaped specimens and allows the determination of thermal diffusivity in all three directions [16][17]. It is important to note that the tests were conducted on the specimen's top surface, corresponding to the last printed layer, due to warping phenomena observed during the printing process. Running the tests on the top surface ensured more accurate results, using four different infill percentages (Figure 1-right).

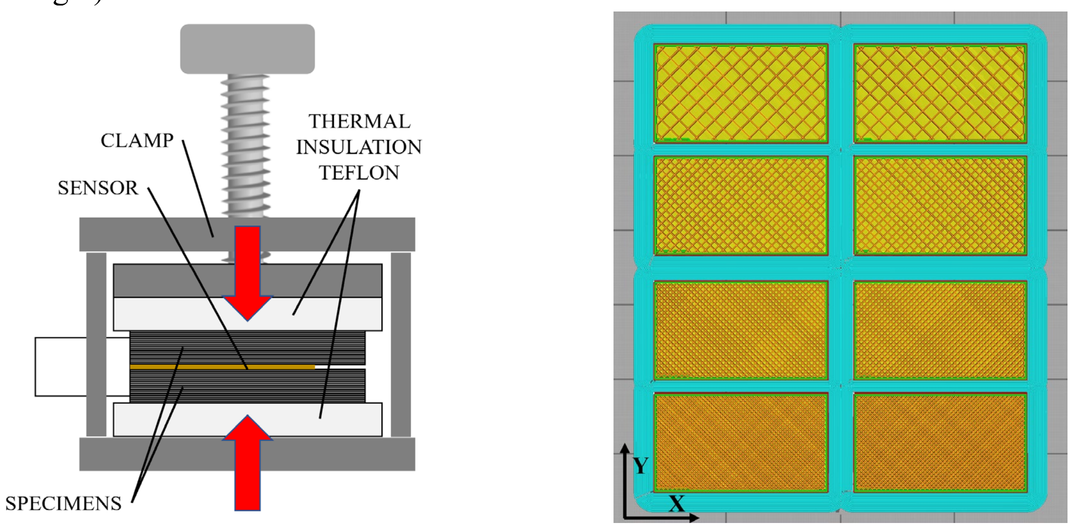

Figure 1. The functioning scheme of the THB system (left) and printing strategies (right).

Results and discussion

Before conducting tests to determine the thermal properties of the specimens, it was necessary to perform some preliminary studies on the roughness of the surfaces. This analysis was required to ensure that the specimen roughness values were within acceptable limits to avoid any potential influence on the measurement of thermal properties, as highlighted in the relevant literature. The samples were inspected as printed. Laser scanning was used to obtain a roughness profile, measured by sampling three sections (20×20 mm^2) of the specimen. The sections were taken in areas far from the edges to avoid measurement errors due to edge effects in the 3D-printed specimens. The average roughness values were R_z = 15.6 µm and R_a = 3.61 µm. To compare these values, measurements were taken with the Surtronic 3P profilometer, which yielded similar R_a values to those recorded with the scanControl 2900-50 BL. All measurements followed the UNI EN ISO 21920 standard to ensure accuracy and consistency. Preliminary tests were carried out on 100% infill specimens to evaluate the impact of roughness on the thermal conductivity values (Table 3). It was found that roughness was an influential factor, as specimens with higher roughness resulting from contact with the rough-textured PEI plate reported worse results in thermal conductivity. Instead, specimens printed on the smooth-textured PEI plate reported the highest values of the three measurements, like those obtained from printed top layers. Another purpose of this surface roughness analysis was to verify the samples' overall quality and test if the roughness values were evenly distributed. Moreover, it should be noted that the roughness values obtained through MEX processes are generally higher than those obtained through traditional manufacturing processes such as injection molding (1.06 µm) [18].

Table 3. Roughness and thermal conductivity values of 100% filled samples.

	Top layer	Smooth PEI	Rough PEI	Unit
Roughness R_a	3.62	2.19	7.96	[µm]
Thermal conductivity λ	0.186	0.189	0.141	[W/m×K]

Table 4. Statistical analysis results

	Estimate	Lower 95%	Upper 95%
Intercept	0.12985	0.12501	0.13468
layer_number(2,8)	0.02726	0.02501	0.0295
layer_number×layer_number	0.00318	-0.0015	0.00782
infill_percentage(20,80)	0.02642	0.02417	0.02867
infill_percentage×infill_percentage	-0.0044	-0.0091	0.00023
layer_number×infill_percentage	-0.0073	-0.0099	-0.0048
R^2	0.9928		
Mean squared error	0.0034		

Another critical aspect considered was the choice of the part infill. Test samples were not printed 100% filled because the main reason for MEX is the ability to obtain complex or lightweight structures. Therefore, for the reduction of material waste and better optimization, it was essential to study the thermal conductivity on lightweight samples with different infill percentages and top/bottom layer count to observe the correlation between the tested values.

The statistical analysis results are graphically in Table 4 and Figure 2-(right). The regression model's coefficient R^2 is 99.28%. The figure shows an analysis of the expected values compared to the observed ones. It can be noticed that these factors were linear, indicating that the model used to generate the predicted values was a good fit for the observed data. This linearity could be seen in the proximity of the data points to the regression line. Additionally, the slope of the regression line suggests that the observed values tend to be slightly higher than the expected values, but the difference was not significant. Figure 2-(left) proves that the model accurately predicted the observed values.

It is observed that there was a linear correlation between the infill percentage and thermal conductivity. Furthermore, the trend line for the data shows a convex increasing structure, indicating that as the infill percentage increased, the rate of change in thermal conductivity also increased at an increasing rate. This result suggested optimizing the infill percentage could significantly improve the material's thermal conductivity. This observation was consistent with the findings reported in the literature and could be explained by air having a very low thermal conductivity. As the infill percentage increased, the volumetric fraction of air increased, which reduced the overall thermal conductivity of the specimen (at the expense of the PLA fraction).

Similarly, as the number of surface layers increased, the thermal conductivity increased because heat encountered the insulating layer of air later. It was worth noting that the trend line of the data exhibits a concave increasing structure, suggesting that the rate of increase in thermal conductivity became progressively smaller as the number of layers increased. Therefore, it could be concluded that the more the component was filled, the better it could transmit heat through its thickness.

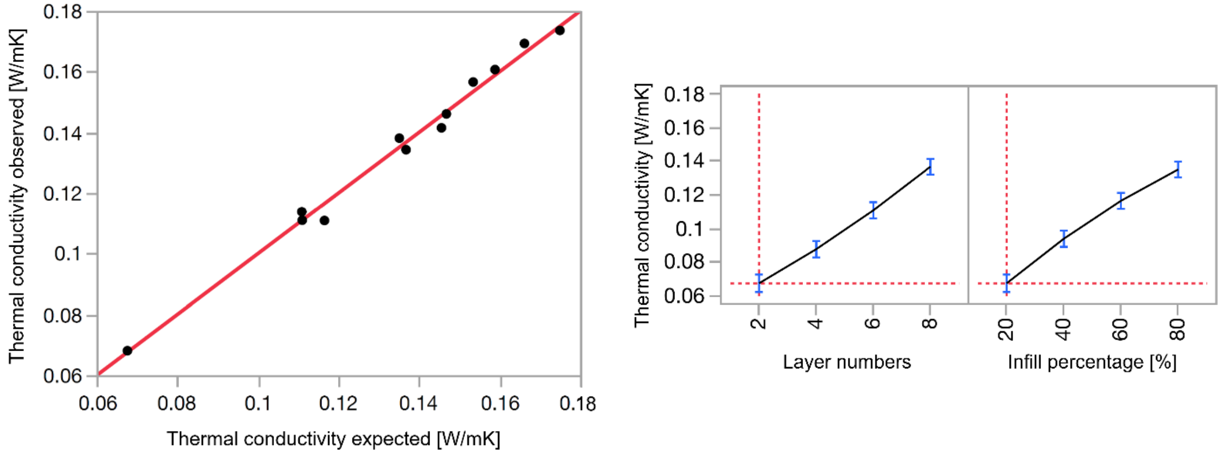

Figure 2. Thermal conductivity expected vs. observed analysis (left); Statistical analysis results (right)

Conclusion

This study investigated the thermal conductivity properties of samples made with filament-based MEX in PLA. The effect of infill percentage and the number of layers on thermal properties was examined while keeping the other process parameters constant. The results indicated a linear correlation between infill percentage and thermal conductivity, as well as between the number of layers and thermal conductivity. Moreover, further investigations are necessary to fully comprehend the impact of anisotropy on thermal properties and its correlation with the number of surface layers. The transient hot-bridge sensor provided fast and accurate measurements of thermal conductivity, thermal diffusivity, and volumetric specific heat from a single experiment. These findings can help optimize the additive manufacturing process to achieve the desired thermal properties in-built parts. This aspect is crucial for engineering applications with significant heat generation and flow.

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