# Thermal properties of samples prepared from polylactic acid by 3D printing

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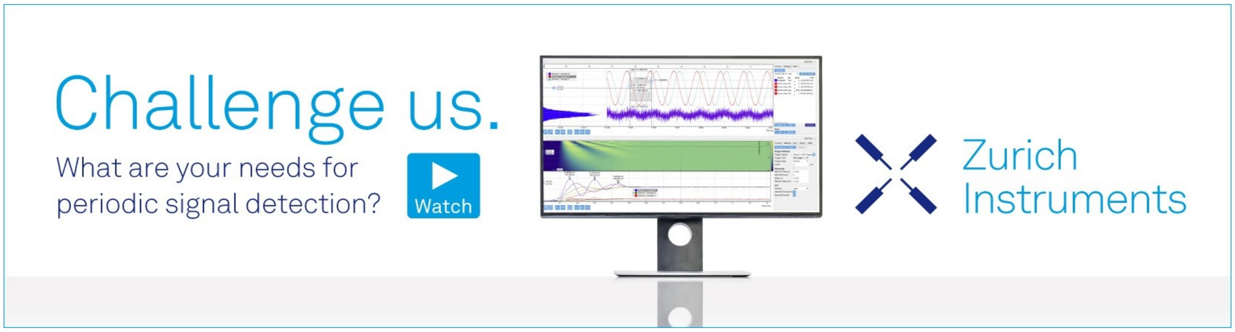

# Thermal Properties of Samples Prepared from Polylactic Acid by 3D Printing

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**Abstract.** In this contribution are described thermal properties (thermal conductivity, specific heat capacity and thermal diffusivity) of objects prepared from PLA (polylactic acid) filaments by 3D printing. The step wise transient method was used, which allows the determination of all parameters using a differential fractal heat transfer model. It was found that the determined thermal parameters depend on the thickness of the measured samples. This is due to the uneven heat transport through the sample. For the measurement of the thermal conductivity it is suitable to use thin samples, where the heat passes to the thermocouple evenly over the entire surface, unlike the measurement of the specific heat capacity where thick samples are used because heat is accumulated mostly in the sample volume. From the extrapolation/interpolation of the values of the determined thermal parameters, the correlation between the sample thickness and the thermal parameters can be found. Then **real** thermal parameters of the material can be determined. In our case for the transparent PLA, the value of the thermal conductivity and the specific heat capacity was determined as 0.12 W/m/K and 1200 J/kg/K, respectively, that agree with the tabulated values of the material.

## INTRODUCTION

The paper follows the article [1] and deals with the study of thermal properties of the material for 3D printing, namely polylactic acid (PLA). The quality of the final product depends primarily on the size of the printed objects, printing speed and printing resolution (see Table 1). Material selection and filament thickness also play an important role. We present here the thermal properties of PLA samples of different thicknesses.

The basic parameters of poly(methyl methacrylate) (PMMA) and PLA are presented in Table 2. The thermal conductivity was measured by Standard Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus (ASTM C518, C177), the specific heat capacity by Standard Test Method for Determining Specific Heat Capacity by DSC (ASTM E1269) [2]. Alternative values are taken from [3].

**TABLE 1**. Basic parameters of PMMA and PLA for 3D printing

| | chemical formula | glass temperature (°C) | melting temperature (°C) | print head temperature (°C) | substrate temperature (°C) |
|---|---|---|---|---|---|
| PMMA | $(C_5H_8\,O_2)_n$ | 85 – 165 | 160 | --- | --- |
| PLA [2] | $(C_3H_4O_2)_n$ | 60 – 65 | 180 – 220 | 190 – 210 | 50 |

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**TABLE 2**. Thermal properties of PMMA and PLA for 3D printing

| | ISO 1183 | ASTM E1269 | ASTM C177 | |
|---|---|---|---|---|
| | **Mass density ($kg/m^3$)** | **Specific heat (J/kg/K)** | **Thermal conductivity (W/m/K)** | **Thermal diffusivity ($mm^2/s$)** |
| PMMA | 1190 | 1450 | 0.19 | 0.110 |
| PLA [2] | 1240 | 1800 | 0.13 | 0.058 |
| PLA [3] | 1210 – 1240 | 1180 – 1210 | 0.12 – 0.15 | |

Samples of PLA were printed using the Prusa i3 MK3 3D printer (maximum printing size 250 × 210 × 200 mm). The printer used FFF technology (Fused Filament Fabrication). The minimal layer size was 0.05 mm and the resolution of the printed object depended on the type of the nozzle (hot end).

The samples were printed on the bed at 60°C. The temperature of the hot end was 215°C for the first layer and 210 °C for other ones. The print speed was 45 mm/s for the perimeters and 80 mm/s for infill. The thickness of all layers was 0.2 mm and the diameter of the nozzle was 0.4 mm.

Material of the samples was transparent PLA (Filament PM - Mladeč). The samples were square base 40 mm × 40 mm (and circles base of diameter 40 mm) and high 1, 2, 4, 8 and 16 mm. All samples were printed in single run of 3D printing.

**TABLE 3.** 3D printer Prusa i3MK3 specifications [4]

| | |
|---|---|
| Printer plates | heated plate (250 × 210) mm. |
| Print size | (250 × 210 × 200) mm. |
| Print speed | up to 200 mm/s. |
| Print substrate material | removable magnetic steel plate with PEI surface layer. |
| Filament diameter | 1.75 mm |
| Filament types | PLA, ABS, PET and other |
| Nozzle diameter | Standard 0.4 mm, quality 0.2 mm |
| Maximum temperature of hot end | 290 °C |

## THEORY

For the determination of PLA thermal parameters, mathematical model of Carslaw and Jaeger [5] is often used. In paper [6], this model was generalized for a the real source of heat with a defined power and inconsiderable losses. The new differential method was presented in [7]. The thermal conductance (in steady state thermal conductivity) $\lambda$ can be expressed by the equation

$$\lambda = \frac{P}{\Delta T}\frac{h}{S}, \tag{1}$$

where $P = UI$ is the power of electric heat source, $S$ is its surface area, $h$ is the distance of the temperature sensor from the heat source, and $\Delta T$ is the difference of the temperature between them.

The heat capacitance (for maximal slope of the dependence $T = f(t)$ - specific heat capacity) $c_p$ is defined by the equation

$$c_p = \frac{P\,dt}{m\,dT}, \tag{2}$$

where $dQ = P\,dt$ is the quantum of the heat required to heat the mass of material by a temperature of $dT$ and $m$ is the mass of the measured material.

The thermal diffusivity $a$, the thermal conductivity $\lambda$, and the specific heat capacity $c_p$ are bound by the equation $\lambda = c_p \rho a$, where $\rho$ is the mass density. The differential method for determining thermal parameters consists then of the calculation of the time dependencies of thermal conductance and heat capacitance by using Eqs. (1) and (2) i.e., from temperature responses related to the reference temperature (temperature of heat source). The inflection points of the dependence $c_p = f(\lambda)$ then allow determine the real heat transport and storage parameters (thermal conductivity and specific heat capacity) of the material.

## EXPERIMENTAL

### Studied materials

The samples were prepared from commercial PLA. Their mass, thickness, diameter, volume, and density are set in Table 4.

**TABLE 4**. Specifications of studied PLA samples

| | **Mass (g)** | **Thickness (mm)** | **Diameter (mm)** | **Volume ($cm^3$)** | **Density ($kg{\cdot}m^{-3}$)** |
|---|---|---|---|---|---|
| PMMA | 5.0 | 6.6 | 30.0 | 4.67 | 1072 |
| PLA | 1.91 | 1.0 | 40.0 | 1.60 | 1194 |
| | 3.80 | 2.0 | 39.9 | 3.18 | 1193 |
| | 7.65 | 4.0 | 40.0 | 6.40 | 1195 |
| | 15.42 | 8.0 | 40.1 | 12.86 | 1199 |
| | 30.65 | 16.0 | 40.0 | 25.60 | 1197 |

The Thermophysical Transient Tester 1.02 was used to measure the responses during the step wise heat. It was developed at the Institute of Physics, Slovak Academy of Science. The setup of the experiment is described in [8], (see Fig. 1, left). Heat was supplied to the system through a built-in resistor in the planar metal source. The power of the pulse generated by the Power Supply (Agilent 6622A) was changed. The measurement duration was 4 hours (2 hours of heating and 2 hours of cooling) for one heat power. Temperature sensors (thermocouples) were placed inside the thermophysical tester. The temperature of the heat source ($T_0$) was measured by a nanovoltmeter Agilent 34420A with an included thermocouple of type K. The temperature difference between the hot and cold surface of the sample was measured by two thermocouples of the type K. The measurement was carried out under vacuum to minimize heat losses. The temperature $\Delta T$ was calculated using a calibration curve. The temperature dependencies of $\Delta T$ on time of heating part of the cycle of studied PLA for different thicknesses and heat powers are shown in Fig. 1 (right).

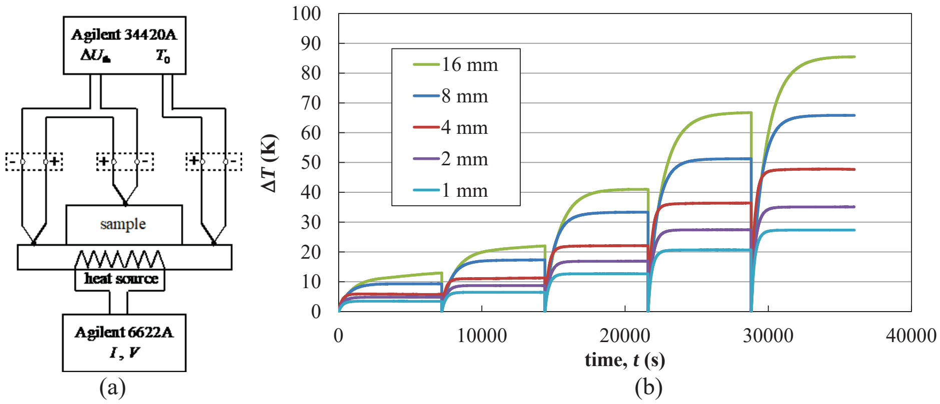



**FIGURE 1.** Measuring apparatus (a) and measured responses of temperature (b) for different powers of heat (0.539 W, 0.992 W, 1.930 W, 3.187 W, 4.187 W) and ~~the~~ thicknesses (1 mm, 2 mm, 4 mm, 8 mm, 16 mm); pulse duration of heating was 2 hours

Figure 2 shows the dependencies determined from the measured experimental data given in Fig.1, right. In Fig. 2a are plotted temperature responses for 16 mm thick samples exposed to different heat power. These dependencies are inversely proportional to the thermal conductance (at steady state thermal conductivity) according to Eq. (1). In Fig. 2b are plotted the corresponding dependencies of the temperature response derivatives on time, those are inversely proportional to the thermal capacitance (at the maximum derivation of the thermal capacitance) according to Eq. (2). The dependence of the temperature response derivation on the change of temperature is plotted in Fig. 2c. From these dependences the steady state values (asymptotes with the *x*-axis and *y*-axis) of thermal capacity, resp. thermal conductivity can be determined.

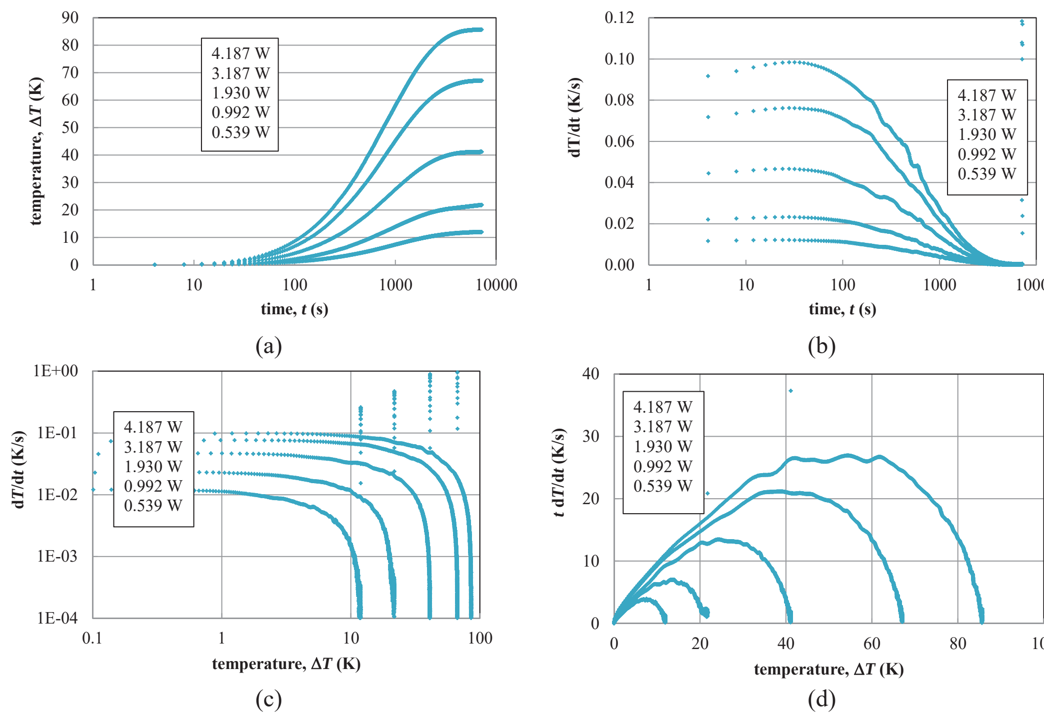



**FIGURE 2.** Dependence of temperature difference on the time (a), derivation of temperature response on time (b), dependence of d$T$/d$t$ derivation on temperature change (c), and dependence $t$ d$T$/d$t$ = $f$ ($\Delta T$) (d) for sample of 16 mm thick and different powers

In Fig. 2d similar dependences are plotted, on the vertical axis the derivative is multiplied by time. This graph resembles Cole-Cole diagrams used in impedance spectroscopy $R$ = f($X$), where $R$ is the resistance and $X$ is reactance of the sample.

From the dependence~~s~~ of the temperature change and its derivation on time was calculated using Eqs. (1) and (2) corresponding dependence~~s~~ of thermal conductivity and specific heat capacity on time (Figs. 1a and 1b), their mutual dependence~~s~~ (Fig. 1c), and dependence of thermal reactance 1 / $\lambda''$ on thermal resistance (1 / $\lambda'$) according to relations

$$\frac{1}{\lambda'} = \frac{\Delta T}{P}\frac{S}{h}, \quad \frac{1}{\lambda''} = \frac{t\,\mathrm{d}T/\mathrm{d}t}{P}\frac{S}{h} \tag{3}$$

where $\lambda'=\lambda$ is the thermal conductance (*effective* thermal conductivity), and $\lambda''=h^2\rho\, c_p/t$ is the quantity proportional to thermal capacitance (*effective* specific heat capacity). All figures show dependences for all input heat powers. It is apparent from all figures that the responses do not depend on the amount of heat supplied (the curves for the individual heat powers overlap), e.g. graphs form Fig. 2 aligns (see multiple points in all graphs on Fig. 3).

Furthermore, it is apparent from the figures that the thermal parameters depend on the thickness of the measured sample. The thermal conductivity determined from steady-state measurements (long times) with decreasing sample thickness decreases up to the tabulated value (see Fig. 3a). On the contrary, the specific heat capacity determined from the initial measurements (short times) decreases with increasing thickness up to the tabulated value (see Fig. 3b). Similar conclusions can be drawn also from the dependence on Fig. 3c, where thermal conductivity and specific heat capacity can be determined from the asymptotes of curves parallel to the $x$-axis and $y$-axis, respectively. Figure 3d is suitable for the optimization of the model of PLA sample. Points of the same color express the measurement results for different wattages. It is obvious that the set values do not depend on the heat input. Furthermore, it was found that the results did not depend~~s~~ on the heating and cooling procedure (in vacuum).

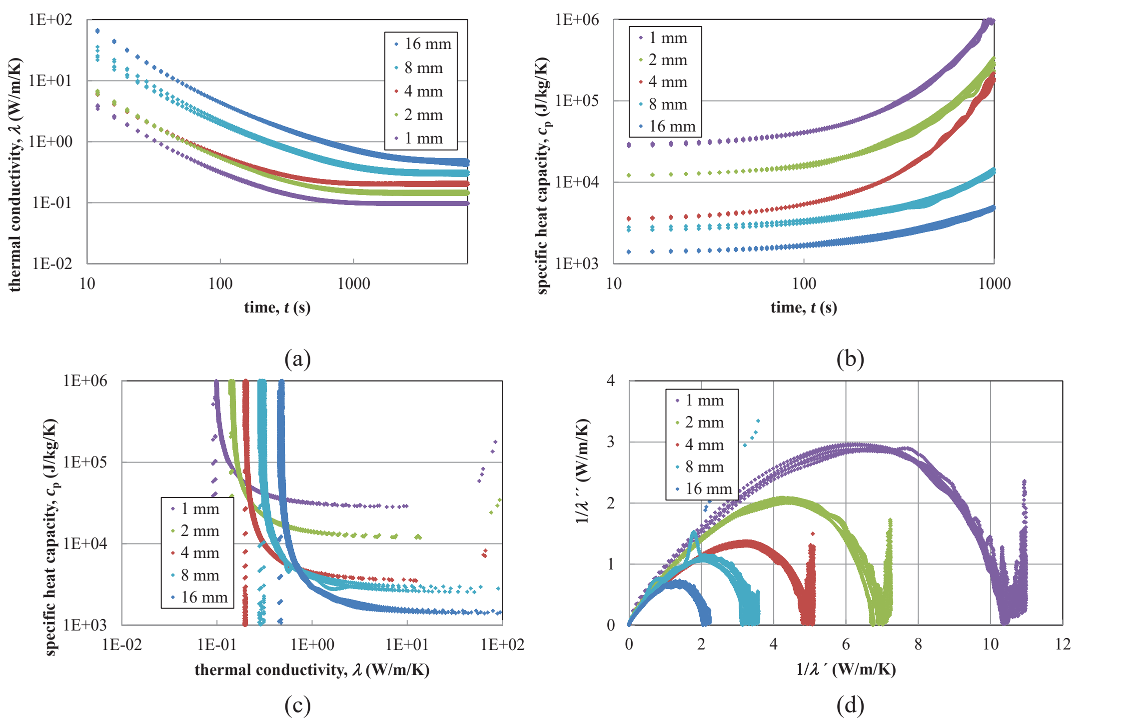



**FIGURE 3.** Dependences of effective thermal conductivity (thermal conductance) on ~~the~~ time (a), effective specific heat capacity (thermal capacitance) on time (b), mutual dependence of effective thermal conductivity on effective specific heat capacity (c), and dependence of reciprocal thermal reactance ($1/\lambda''$) on reciprocal thermal conductance ($1/\lambda'$) (d) for samples of different thicknesses

It is clear from Fig. 3 that the determined thermal conductivities calculated using the geometric parameters of the samples vary over a wide range that does not correspond to the real values (see Table 2). From the measurement of temperature responses, using the Fluke TI 55 thermal camera, we have found out the trend of the temperature development during the step heating to 60 °C of all samples. The results are shown in Fig. 4. There is the photograph of the samples (top), thermal photo (middle), and the 3D profile of temperature (bottom). Especially from the last figure it is clear that the samples do not heat evenly over the entire area: in the middle of the sample temperature is higher. This reality leads to the conclusion that the value of thermal conductivity measured on thin samples is closer to the real value, because the heating is relatively uniform over the entire area (sample on the left side of Fig. 4). On the contrary, the specific heat capacity of thick samples is closer to the real value, because there is no heat leakage through the thermostat. In order to obtain the real values of the thermal parameters of the material it is necessary to correct the values obtained from the experiment to real sample area (for the determination of thermal conductivity) or to real sample volume (for the determination of the specific heat capacity).

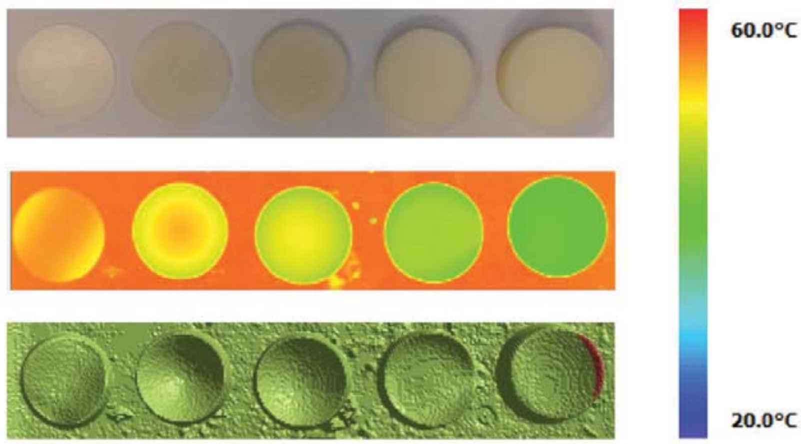
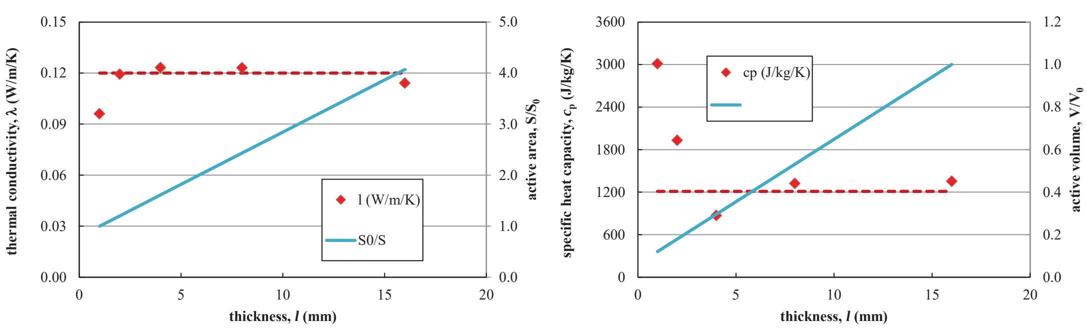



**FIGURE 4.** Measured samples photo and thermo and 3D profile of temperature for different sample thicknesses

The corrected results together with the linear correction curves determined from the experimental data are shown in Fig. 5. It is clear from these figures that after the correction the values of thermal parameters acquire real values corresponding to tabulated values in the literature; see Tab. 2.




(a) (b)

**FIGURE 5.** Dependences of thermal conductivity (a) and specific heat capacity (b) on the thickness of measured samples PLA

## CONCLUSION

In this paper are presented thermal properties of printed 3D layers of PLA (polylactic acid) as obtained by transient (step wise) method. For the samples of different thicknesses, the thermal parameters were determined using a differential method. The results obtained from the experimental data had to be corrected to the sample effective area (for the thermal conductivity due to uneven heat transfer through the sample) and to the sample volume (to determine the heat capacity due to heat removing from the sample). Inhomogeneous heat transport throw the samples is demonstrated by thermo-photographs. The thermal parameters obtained after the correction correspond to the parameters other authors [2, 3].

## ACKNOWLEDGMENTS

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