Evaluation of measured and calculated thermal parameters of a photopolymer Kovács J. G., Körtélyesi G., Kovács N. K., Suplicz A.

This accepted author manuscript is copyrighted and published by Elsevier. It is posted here by agreement between Elsevier and MTA. The definitive version of the text was subsequently published in [International Communications in Heat and Mass Transfer, 38, 2011, DOI: 10.1016/j.icheatmasstransfer.2011.04.001]. Available under license CC-BY-NC-ND.

Evaluation of measured and calculated thermal parameters of a photopolymer

J. G. Kovacs^{a*}, G. Kortelyesi^b, N. K. Kovacs^a, A. Suplicz^a ^a Department of Polymer Engineering, Budapest University of Technology and Economic, Budapest H-1111, Hungary ^b Department of Machine and Product Design, Budapest University of Technology and Economic, Budapest H-1111, Hungary * Corresponding Author: Address: Department of Polymer Engineering, Budapest University of Technology and Economic, H-1111 Budapest, Muegyetem rkp. 3. T. bld. III. 35., Hungary; E-mail address: kovacs@pt.bme.hu Phone: +36-1-463-1440 Fax: +36-1-463-1440

Abstract:

Nowadays the designing and the production should continuously develop, so the old product development system has changed radically in the past decades and has been replaced by the so-called simultaneous planning, as the typical method. The bases of this method are the prototypes, on which evaluation is performed very simply and fast. To use these prototyped products as functional part, the thermal behavior of its material should be characterized. In this work we characterised all the thermal properties of a photopolymer used by PolyJet technology to the further finite element analysis. These parameters were the thermal conductivity, the specific heat and the density. We have concluded that the thermal conductivity and the specific heat are increasing in the function of the temperature. A simple method was introduced to compare the measured thermal gradients with a simulated and also with an analytical model. It was proved that the thermal properties measurements are imprecise, moreover the temperature dependence should be taken into consideration.

Nomenclature

А	cross-section	
c _{p(m)}	specific heat of the sample	
c _{p(s)}	specific heat of the standard	
m _{air}	weight of sample on air	
m _{EtOH}	weight of sample in ethanol	
m _m	weight of the sample	
ms	weight of the standard	
q	heat flux	
t	time	

T temperature)
---------------	---

- x distance
- y_m distance of the sample from the baseline
- y_s distance of the standard from the baseline
- Y proportional factor

Greek symbols

λ	thermal conductivity
ρ_{sample}	the density of sample

 ρ_{EtOH} the density of ethanol

Keywords: rapid prototyping, photopolymer, simulation

1. Introduction

In the industry the Rapid Prototyping is a widely used process to shorten the developmenttime and the "time to market". This technology is an innovative additive manufacturing process instead of the traditional subtractive processes. Its essence is that three dimensional physical parts can be fabricated directly from computer aided design data. All of the RP activities consist of mainly two main parts: data preparation and model production [1-3]. In the last decade a rapid growth can be seen in the field of Rapid Prototyping. The first commercial RP equipment was brought to the market in 1987. Since then, numerous of new apparatuses and processes were developed. According to the enabling technology, there are two main categories: one is based on high energy beam (for example: SL, SLS, LOM) and the other is based on the droplet jetting or slurry extrusion (for example: FDM, 3DP) [4-6]. There are three main reasons, to create prototypes. First is when there are no mechanical requirements the only purpose is to show the product shape and appearance [7]. Another reason is to investigate the suitability of models for applicability and assembly tests. These are the functional models. In this case mechanical strength is needed similar to the mass product. Thirdly, if just few models are required, a short series could be produced for short runs or overall functional tests [8, 9]. So the short run injection moulds can be created by Rapid Prototyping technologies [10]. It is clear, that these polymer based moulds have very different thermal properties, than the traditional steel or alumina moulds [11]. To use correctly the mould or simply fulfil a simulation, the known of these thermal parameters are

essential. They have a great influence to the cycle-time. To optimal it and raise the life-span of the mould, the correct determination of the parameters is needed.

There are numerous publications on the thermal measurement and simulation of photopolymers, but only on Stereolihography materials. Rahmati et al. [12] measured an SL tool's temperature during injection moulding. They appointed, that into a tool, which has low glass transition temperature, it is possible to inject polymer with high melting temperature. It is thanks to the low thermal conductivity and the short injection time. Colton et al. [13] measured the temperature change in SL mould 2.5 mm from the surface, during injection moulding. They investigated the necessary cooling time, when the temperature of the substance drops under a determined temperature, where the mechanical properties are suitable to moulding. It is clear that the thermal condition of the polymer moulds is necessary to be known to use them for injection moulding. Furthermore, the simulation of the thermal processes is easier than perform any complex and long measurements. Besides the technology developments there are several material improvements as well such as the curing process optimisation [14] or the additives and filler developments [15]. In our work the aim was to determine the thermal condition in the substance mathematically and with simulation. To perform it the thermal properties of the FullCure 720 photopolymer is needed, such as the specific heat and the heat conductivity in the function of temperature. Furthermore an apparatus was developed, to determine the real thermal-gradients. With the

help of it, the measured parameters can be confirmed or can be corrected with a failureanalysis.

2. Experimental

2.1. Thermal properties measurement

In physics, thermal conductivity (λ) is the property of a material that indicates its ability to conduct heat. In this research the thermal conductivity was measured using the transient hot plate method. The point of the process is to reach a steady state condition, thus simplifying the measurement to a one-dimensional case to use the Fourier's law (Eq.1) [16]. Temperature is measured by built-in thermo-couples inside the heated and the cooled plate. The frame is heated by a separated device to minimize the heat loss and the tested models are placed symmetrically (Fig. 1).

$q(x,t) = -\lambda \cdot \nabla T(x,t) (1)$

where q [W] is the transmitted heat flux, λ [W/(m·K)] is the thermal conductivity and T [K] is the temperature.

The specific heat was measured with the Perkin Elmer DSC 2 equipment. To define this property, three measures were prepared. In each case the samples were simply heated up. To define the basic line, this measurement was performed with empty sample-holder. Secondly the sapphire sample was heated up (specific heat of sapphire in function of temperature is known) and thirdly the FullCure 720 photopolymer was analysed. The main set-ups are shown on Tab. 1.

During the evaluation, the absorbed power in function of temperatures, were plotted. As the next phase, the distance of the curve of the sapphire and the sample from the base line has been measured. The specific heat of the sample has been related to the specific heat of sapphire, and the coefficient was set as the distances from the base line and the rate of masses (Eq.2.). As a result, the specific heat of the sample has been determined at discrete points [15].

$$\frac{c_{p(m)}}{c_{p(s)}} = \frac{m_s \cdot y_m}{m_m \cdot y_s}$$
(2)

Density of the polymer sample was determined by the change of the lift force (Archimedes' principle) by the help of an analytical balance. The weight of sample was determined in air and in liquid. The liquid was watertight, analytical clean ethanol. The density of sample was determined at room temperature with the next equation (Eq.3.):

$$\rho_{\text{sample}} = \frac{m_{\text{air.}}}{m_{\text{air.}} - m_{\text{EtOH}}} \rho_{\text{EtOH}} \quad (3)$$

where ρ_{sample} [g/cm³] is the density of sample, m_{air} [g] is the weight of sample on air, m_{EtOH} [g] is the weight of sample in ethanol, and ρ_{EtOH} [g/cm³] is the density of ethanol.

2.2. Measuring configuration

To confirm the results of the transient thermal finite element analysis and the determined thermal parameters' correctness, a simple measure was fulfilled (Fig. 2).

Seven thermo-couples were placed into the specimen, at different distance from the heated surface. The localization of the boreholes is shown on Fig. 2. During the measurement the top point was not used. The block with the sensors was placed into the ram (Collin P200 E). Then the ram's plate was heated up, than cooled down. The temperature was recorded in every second.

2.3. Mathematical modelling and Finite Element simulation

In order to verify the value of the thermal conductivity, a transient thermal FE simulation was carried out. It was assumed that the value of the thermal conductivity does not depend on the

temperature. Only the half of the RP specimen was included into the simulation, because of the symmetry.

According to the places of the real sensors, in the FE simulation virtual sensors were generated, in order to make the comparison easier.

Performing transient thermal analysis beside the normal mesh generation procedure in the 3D space domain, also a time domain should be discretized. Mesh generation is a very crucial step in design analysis, so in order to avoid the numerical error; a convergence analysis was performed. The result of the convergence analysis shows, that in the space domain the second order 3D tetrahedral solid elements should have approximately 1mm global element size, and in the time domain accurate temperature values can be achieved applying 2 second time step. Supposing a general 2000 sec long measurement, 1000 FE calculation have to be performed including approximately 87 903 nodes in every simulation step. The calculation was performed in the SolidWorks/Simulation 2010 software environment.

The first simulation results, based on the measured thermal material properties, show good accordance with measurements close to the heated plate, but the sensors further away from the plate show deviation from the measurements. This is the reason why the initially supposed thermal conductivity was modified and varied between 0.178 and 0.33 W/(m·K). Numerical simulations were carried out, applying four different thermal conductivity parameters to determine the most suitable one.

One of the ways to transform the differential equitation of heat conduction into difference equitation is the explicit difference method. To solve the thermal problem, also an analytical model was used. At the case of one dimension and without inner heat source the (Eq.4.) describes the process in the substance.

$$\frac{T(x,t+\Delta t)-T(x,t)}{\Delta t} = a \cdot \frac{T(x+\Delta x,t)-T(x-\Delta x,t)-2T(x,t)}{\Delta x^{2}}$$
(4)

To measure the failure between the measured and the simulated or calculated results, a failure equitation (Eq.5.) was developed, which result is a proportional factor (Y). It is a relative value (Δt), the time-step and the distances of the measuring points from the heat source (x) are considered.

$$Y = \frac{1}{\bar{t}\bar{x}} \sum_{s=1}^{s_{max}} \left[\sum_{t=1}^{\bar{t}} \frac{\left| T^{sim}(s,t) - T^{measure}(s,t) \right|}{T^{ref}} \cdot \Delta t \right] x [-]$$
(5)

3. Results and discussion

In this paper the thermal properties of Objet PolyJet technology model material (FullCure 720 photopolymer manufactured by Objet Geometries) were analysed. Firstly, the heat conduction was investigated on $150 \times 150 \times 4$ mm specimens at different temperatures (26.5; 44.5; 61.1 and 80.5° C). As a result, the thermal conductivity is varying between 0.178 and 0.187 W/(m·K) (Fig. 3). The inaccuracy of the measurement is about 12%, so the measured values are in the range of dispersion. Hence, an average value should be used in the simulations [18]. Furthermore, according to the Archimedes' principle the density is 1.18 g/cm³. The specific heat was measured with Perkin Elmer DSC 2 equipment. As a result of the measurements and calculations, specific heat varied between 2000 and 3250 J/(kg·K) in the function of temperature (Fig. 4). It is clear, that by increasing the temperature, specific heat is increasing as well.

For the analysis constant parameters were used for the thermal conductivity and for the specific heat as well. Thus, the value of thermal conductivity was 0.18, 0.26, 0.28 and 0.33 W/($m\cdot K$), while the specific heat was constantly 2740 J/(kg·K) as an average value of the measured curve.

The real thermal processes, which were measured during the heating test (Fig. 5.), were compared to the simulated and the calculated curves. To determine the deviation between the measured and the calculated curves, Eq. 5. was used.

The values of the proportional factor can be seen on Tab. 2. at the case of the simulation and the calculation. Plotting the factors in the function of the thermal conductivity and fitting a trend line on the points (Fig. 6.), a minimal value of the curves can be noticed. Calculating with explicit difference method, the thermal conductivity is 0.286 W/(m·K) and with simulation 0.273 W/(m·K) at the minimum point of the error curves. Calculating the minimum deviation from the proportional factor function (Fig. 6.), the optimum point for the thermal conductivity can be determined as 0.28 W/(m·K).

Using the explicit method the minimum of the calculated proportional factor curves can not be lower than 0.14 (Fig. 7.). So if constant parameters are used, this failure always appears in the calculations. Hence temperature dependant thermal parameters should be used to decrease the deviation from the real processes.

Fig. 8 and Fig. 9. show the thermal processes in two points (4 and 9 mm from the surface), using the determined coefficients. It is clear, that the experimental results are very close to the measured curves. Comparing the simulation and the calculation, the calculated points show the best fit to the real process. On the other hand, for the further finite element analysis the

temperature dependent thermal parameters can be used to further minimize the deviation from the real process.

4. Conclusion

In this work all the thermal properties of a rapid prototyped part produced by PolyJet technology was measured. These parameters were the thermal conductivity, the specific heat and the density. We have concluded that the thermal conductivity and the specific heat are increasing in the function of the temperature. Based on hot plate measurements the thermal conductivity is varying between 0.178 and 0.187 W/(m·K) in the common temperature range while the specific heat based on DSC measurements is varying between 2000 and 3250 J/(kg·K). The calculated density was 1.18 g/cm³.

After determining the measured, the simulated and the calculated temperature-curves using discrete parameters, the proportional factors were determined. It was concluded that the calculated proportional factor curves can not be lower than 0.14 using the determined thermal conductivity of 0.28 W/(m·K). It was also pointed out that the heat dependent thermal properties should be used for the more accurate calculation results.

Acknowledgment

This paper was supported by the János Bolyai Research Scholarship of the Hungarian Academy of Sciences. This work is connected to the scientific program of the "Development of quality-oriented and harmonized R+D+I strategy and functional model at BME" project. This project is supported by the New Hungary Development Plan (Project ID: TÁMOP-4.2.1/B-09/1/KMR-2010-0002).

References

[1] F. E. Wiria, N. Sudarmadji, K. F. Leong, C. K. Chua, E. W. Chng, C. C. Chan, Selective laser sintering adaptation tools for cost effective fabrication of biomedical prototypes, Rapid Prototyping Journal 16 (2) (2010) 90–99

[2] A. Rosochowski, A. Matuszak, Rapid tooling: the state of the art, Journal of Materials Processing Technology 106 (2000) 191-198

[3] X. Yan, P. Gu, A review of rapid prototyping technologies and systems, Computer Aided Design 26 (4) (1996) 307-316

[4] Y. Yongnian, L. Shengjie, Z. Renji, L. Feng, W. Rendong, L. Qingping, X. Zhuo, W. Xiaohong, Rapid Prototyping and Manufacturing Technology: Principle, Representative Technics, Applications, and Development Trends, Tsinghua Science and Technology 14 (1) (2009) 1-12

[5] F. Xu, Y.S. Wong, H.T. Loh, Toward Generic Models for Comparative Evaluation and Process Selection in Rapid Prototyping and Manufacturing, Journal of Manufacturing Systems 19 (5) (2000) 283-296

[6] N. K. Kovács, J. G. Kovács, Developments in the Field of Rapid Prototype Production, Materials Science Forum 589 (2008) 421-425

[7] N. K. Kovács, J. G. Kovács, The change of the 3D printing product mechanical properties in the function of different post-treatment, Materials Science Forum 659 (2010) 183-189
[8] Á. Oroszlány, J. G. Kovács, Gate type influence on thermal characteristics of injection

molded biodegradable interference screws for ACL reconstruction, International

Communications in Heat and Mass Transfer 37 (2010) 766-769

[9] Á. Oroszlány, P. Nagy, J. G. Kovács, Injection molding of degradable interference screws into polymeric mold, Materials Science Forum 659 (2010) 73-77

[10] J. G. Kovács: Construction of pre-deformed shapes for rapid tooling in injection molding, Macromolecular Symposia 239 (2006) 259-265

[11] J. G. Kovács, T. Bercsey: Mold properties influence on injection molded part quality, Periodica Polytechnica Mechanical Engineering 49 (2) (2005) 115-122

[12] S. Rahmati, P. Dickens, Rapid tooling analysis of Stereolithography injection mould tooling, International Journal of Machine Tools and Manufacture 47 (2007) 740-747

[13] J. S. Colton, Y. Lebaut, Thermal Effects on Stereolithography Injection Mold Inserts, Polymer Engineering and Science 40 (6) (2000) 1360-1368

[14] J. Zhang, Y. C. Xu, P. Huang, Effect of cure cycle on curing process and hardness for epoxy resin, Express Polymer Letters 3 (9) (2009) 534-541

[15] A. Allaoui, N. El Bounia, How carbon nanotubes affect the cure kinetics and glass transition temperature of their epoxy composites?, Express Polymer Letters 3 (9) (2009) 588-594

[16] W.J. Minkowycz, A. Haji-Sheikh, K. Vafai, On departure from local thermal equilibrium in porous media due to a rapidly changing heat source: the Sparrow number, International Journal of Heat and Mass Transfer 42 (1999) 3373-3385

[17] M. J. O'Neill, Measurement of Specific Heat Functions by Differential Scanning Calorimetry, Analytical Chemistry 38 (10) (1966) 1331-1336

[18] M. Rides: The Effect of Thermal Conductivity and Heat Transfer Coefficients onPolymer Processing, National Physical Laboratory [midas.npl.co.uk], Measurment note 023,1998



Fig. 1. Heat conduction measurement.



Fig. 2. Measuring configuration and localization of the borehole to place the thermo-couples



Fig. 3. Heat conduction in function of temperature.



Fig. 4. Specific heat of FullCure 720 in function of temperature.



Fig. 5. Result the measurement configuration.



Fig. 6. Proportional factor in the function of the thermal conductivity



Fig. 7. Proportional factor in the function of the thermal conductivity at different specific heats



Fig. 8. Comparing the results of the simulation and the explicit method to the measured curve (thermal conductivity= $0.28 \text{ W/(m \cdot K)}$; distance = 4 mm from the front surface)



Fig. 9. Comparing the results of the simulation and the explicit method to the measured curve (thermal conductivity= $0.28 \text{ W/(m \cdot K)}$; distance = 9 mm from the front surface)

value
320-550 K
10 K/min
one-time
29.05 mg
4.558 mg

Tab. 1. Parameters of the measurements.

	thermal conductivity	specific heat	
	$[W/(m \cdot K)]$	$[J/(kg \cdot K)]$	Y
pç	0.18	2740	0.776
late	0.26	2740	0.446
calculated	0.28	2740	0.447
ca	0.33	2740	0.56
p	0.18	2740	0.823
late	0.26	2740	0.214
simulated	0.28	2740	0.144
Sir	0.33	2740	0.275

 Tab. 2. Proportional factors for different thermal parameters