

TRANSFERRING HEAT THERMAL PULSE THROUGH POLYMER MATERIALS

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ABSTRACT

The aim of this work was to determine the influence of temperature on thermal properties of polymer materials, as well as investigate the heat flux transfer in semicrystalline and amorphous polymers. The method is based on American standard ASTM D5334:2008 using a dual needle sensor; which was adapted to these materials. The thermal conductivity was determined due to its accuracy and reliability in the used technique. The polymers studied were polyamide-6, polymethyl

methacrylate and polyvinyl chloride. The obtained results indicated that thermal conductivity of investigated polymer increased when the temperature increased. Major factor which influence on heat transfer is the crystallinity of the sample. Semicrystalline polymers had higher value of thermal conductivity than the amorphous polymers, the reason was more efficient vibration of phonon in semicrystalline lattice. Other important factors which affected the thermal properties of polymers were dimension and orientation of sample.

KEYWORDS: Polymer, thermal properties, thermal conductivity, crystallinity, amorphous.

INTRODUCTION

Nowadays polymers are widely used materials due to their versatile properties in comparison with metal like steel or aluminium. They offer many advantages like light weight, flexibility, resistance to corrosion and fouling, low cost and easy of production (Joen et al., 2009).

Polymers have also well-known insulating properties. Low heat transfer in polymers is because of the large energy distance between the valence and conduction layer, low atomic density, anharmonicity in molecular vibrations and complex crystal structure (Shindé and Goela, 2006); i.e. anharmonicity results in additional oscillations with frequencies, which modifies the profile of the resonance curve, provoking phenomena such as the *Foldover effect* and super harmonic resonance. Their low thermal conductivity might limit their use, especially in heat transfer devices. Therefore, many efforts are made in order to increase the thermal properties of polymers.

Therefore, the aim of this work was to determine the influence of temperature on thermal properties of polymer materials, as well as to observe the influence of chemical structure, morphology, dimensions and orientation of the polymer chains on thermal conductivity.

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Theoretical considerations

Heat transfer takes place when thermal energy is exchanged between two physical objects. Thermal energy can be defined as a sum of kinetic energy of atomic motions and potential energy of distortion of interatomic bonds (Askeland et al., 2010). Heat is transferred from high to low temperature areas of the material and can occur in three different modes: conduction, convection and radiation. The main focus on this research is put on heat transfer in solid state polymers by means of conduction. Conduction is a result of interactions between electrons, vibrating atoms and molecules. Transferring heat by conduction requires direct contact and occurs in all phases of matter (Vlachopoulos and Strutt, 2002).

Thermal conductivity λ can be defined as the ability of material to conduct the heat. It appears primarily in Fourier's Law for heat conduction (Equation 1). The unit of thermal conductivity is $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$.

$$q = -\lambda \frac{\partial T}{\partial x} \quad (\text{Eq. 1})$$

Where: q – heat flux, the amount of thermal energy transferred through a unit of area per unit of time, λ – thermal conductivity ($\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$), $\frac{\partial T}{\partial x}$ – temperature gradient.

Thermal conductivity is defined as well as the quantity of heat ΔQ , transmitted during time Δt through a thickness x , in a direction normal to a surface of area A , per unit area of A , due to the temperature difference ΔT (Equation 2) (Bicerano, 2002).

$$\lambda = \frac{\partial Q \cdot x}{\partial t \cdot A \cdot \partial T} \quad (\text{Eq. 2})$$

Heat in materials are transferred by electrons and phonons (waves of lattice displacement). This transport is limited by elastic Rayleigh scattering of acoustic phonons by lattice defects. Since polymers are non-metallic substances with no free electrons the heat transfer occurs mainly due to lattice vibrations (Askeland et al., 2010). Debye in 1912 described the relationship between thermal conductivity and lattice oscillations by Equation 3 (Klein, 2011).

$$\lambda = K \cdot \rho \cdot C_p \cdot v \cdot l \quad (\text{Eq. 3})$$

Where: K – dimensionless constant around 0.33, ρ – density, C_p – heat capacity ($p = \text{const.}$), v – transfer speed for elastic oscillation, l – free length of elastic oscillation, the atomic distance for amorphous thermoplastic region.

Eventually, and according to Poulikakos (1994), there are important factors that might influence thermal conductivity; which include the temperature, the pressure, the chemical phase, the thermal anisotropy, density, magnetic field, morphology, type and strength of the bonds located in the direction of heat transfer orientation, additives, impurities and moisture.

MATERIALS AND METHODS

Samples required to carry out the experimental research were one semi-crystalline polymer and two amorphous polymers as follow;

Polyamide-6

Polyamide-6 (PA6) is semi-crystalline thermoplastic. The white color of sample comes from height level of crystallinity. It can be characterized by good mechanical strength and high

wear resistance. The literature value of the thermal conductivity at 25 °C falls into range from 0,22 to 0.33 W·m⁻¹·K⁻¹ (McKeen, 2012).

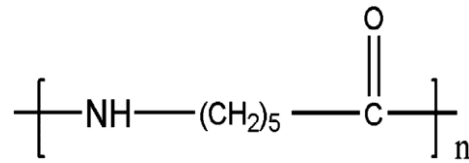


Fig. 1: Chemical structure of polyamide-6.

Polymethyl Methacrylate

Polymethyl methacrylate (PMMA) is transparent, amorphous thermoplastic. General properties of polymethyl methacrylate are high stiffness and hardness. This polymer has also good transluance and good insulation properties. The literature value of the thermal conductivity at 25 °C falls into range from 0.16 to 0.25 W·m⁻¹·K⁻¹ (Kaiser et al., 2015).

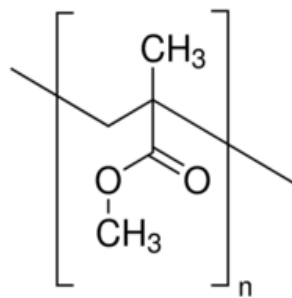


Fig. 2: Chemical structure of polymethyl methacrylate.

Polyvinyl chloride

Polyvinyl chloride plasticized (PVC-P) refers to polyvinyl chloride with plasticizer molecules embedded between the polymer chains; i.e. a low-molecular-weight polymer that increase the spacing between chains to make them more flexible and, thereby, tougher. This polymer is black amorphous thermoplastic with good chemical resistance and good insulation properties. The literature value of the thermal conductivity at 25 °C falls into range from 0.13 to 0,20 W·m⁻¹·K⁻¹ (Kaiser et al., 2015).

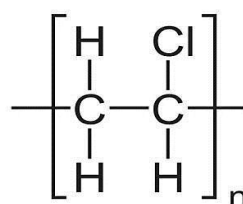


Fig. 3: Chemical structure of polyvinyl chloride.

Thermal transitions characterization

Thermal analysis of presented polymers was performed using a differential scanning calorimetry (DSC) and modulated differential scanning calorimetry (MDSC). Both techniques through Q-20 and RCS-40 cooling module from TA Instruments, Inc. Measurements were carried out in the temperature range from -40 °C to 250 °C with a heating rate of 5 °C·min⁻¹. DSC is the most frequently thermoanalytical technique for polymer investigation in which the difference in heat flow rate between a sample and an inert reference is measured as the sample is heated, or cooled and determined as a function of time or temperature. MDSC is an enhancement to common DSC. This technique employs a sinusoidal temperature modulation with underlying heating or cooling rate. The total heat flow is separated clearly into reversing and non-reversing signals. In reversing curve, we can observe effects related with heat capacity changes as glass transition and melting. Non-reversing curve shows kinetics events like recrystallization, crystallization, decomposition, relaxation, curing, and evaporation. MDSC technique can overcome the limitation of DSC technique and can give more information about thermal properties of polymers (Gracia-Fernández et al., 2012; 2014).

Thermal conductivity characterization

The method is based on American Standard ASTM D5334 using a heat pulse sensor. The thermal conductivity measurements were taken using dual-needle sensor SH-1, and measurements were collected through reader-logger KD2 Pro, both from Decagon Devices Inc. The dimensions of the sensor are; 1.3 mm diameter, 3 cm long and 6 mm spacing between the two needles. The methodology used in this research was based on the transient heat flux method of the hot wire, and adapted by Rubio et al. (2016). The framework of the method is based on the fact that the temperature of a thin hot wire rises exponentially when a constant power is applied while it is drawn in the centre of a sample of infinite length. This allows calculating the thermal conductivity of the material based on the heat flow, the characteristics of the heating wire and the increase of temperature in the wire in a certain time (Villar and Aroz, 2012).

The measurements were carried out at temperature range from 25 °C to 100 °C, for 5 thermal points: 25 °C, 50 °C, 65 °C, 80 °C and 100 °C. Five measurements were taken for each sample. The read time was set for 2 minutes. The interval between successive measurements was 30 minutes, minimizing thus the thermal drift. The reader logger KD2 Pro was used in high

power mode to gather all the data. Two different dual-needle sensors were used to take measurements. The sensors reliability and accuracy were verified and calibrated.

For each polymer 9 samples with different dimensions were analysed (Table 1). Data were collected with three different heat flow directions: parallel, perpendicular and trans. Orientation of the sensor was in regard to the direction of polymer flow. Figure 4 shows how the sensor was inserted into the sample, indicating with the arrows the flow of the polymer and its relation to the orientation of the heat pulse. TRANS orientation (Figure 4B) showed a sampling design similar to the perpendicular orientation, although it presented an angle of 90° not only with the flow of the polymer, but also with the axis of the sample.

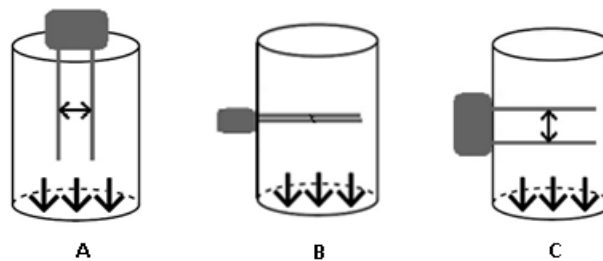


Fig. 4: Orientation of the sensor. (A) Perpendicular, (B) Trans, (C) Parallel.

Table 1: Sampling design.

Comparison of thermal properties in samples with different height			
Sample	Diameter [mm]	Height [mm]	Orientation
PA6, PMMA, PVC	50	10	Parallel
		15	
		25	
		40	
Comparison of thermal properties in samples with different diameter			
Sample	Diameter [mm]	Height [mm]	Orientation
PA6, PMMA, PVC	10	35	Perpendicular
	15		Parallel
	25		Trans
Comparison of thermal properties in samples with different direction of sensor			
Sample	Diameter [mm]	Height [mm]	Direction
PA6, PMMA, PVC	50	40	Perpendicular
			Parallel
			Trans

RESULTS AND DISCUSSION

Results of the polymer samples were analyzed in terms of their response to the temperature increase observing the first and second order transitions, and on the other hand in terms of the behaviour of heat transfer through the polymer matrix.

Thermal transitions characterization

Polyamide-6

Figure 5 shows the DSC thermogram of polyamide-6 (PA6). The glass transition of analysed sample was at 86.6 °C and melting temperature was observed at 220.7 °C using DSC analyser.

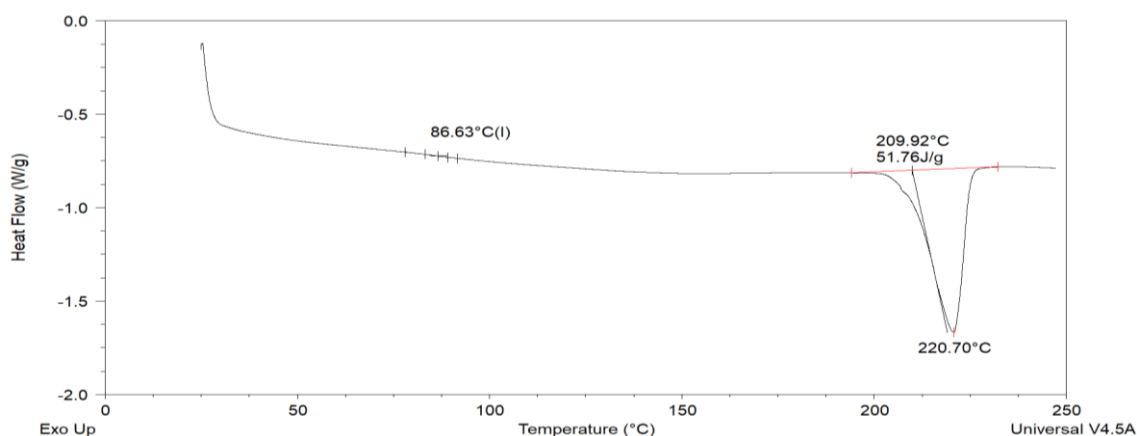


Fig. 5: DSC thermogram of polyamide-6 (PA6).

Polymethyl methacrylate

The results from DSC analysis for polymethyl methacrylate (PMMA) are plotted in Figure 6. The glass transition of analysed sample was at 117.9 °C (syndiotactic) using DSC analyser.

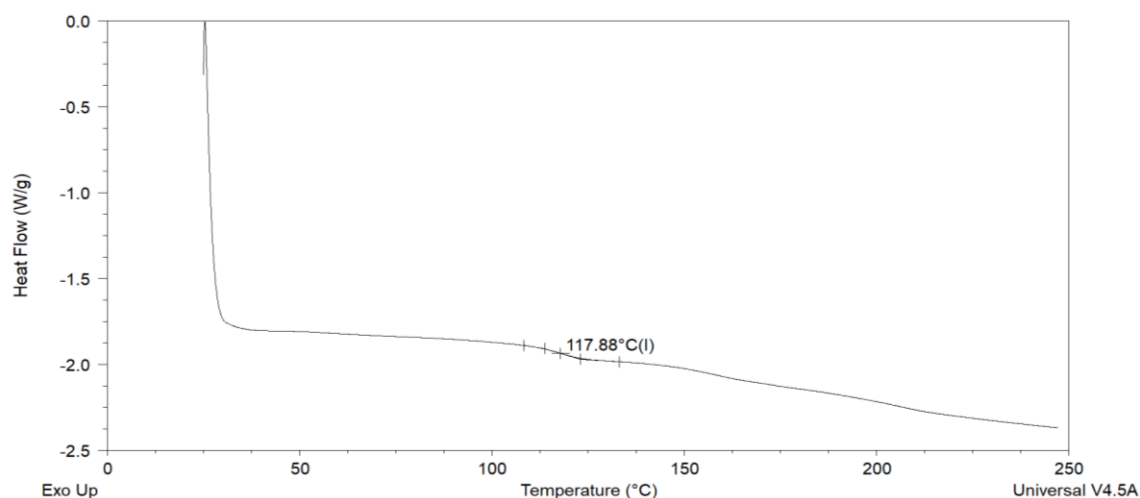


Fig. 6: DSC thermogram of polymethyl methacrylate (PMMA).

Polyvinyl chloride

The results from MDSC analysis for polyvinyl chloride (PVC-P) are plotted in Figure 7. The glass transition of analysed sample was at 66.9 °C and melting plasticizer temperature at 109.4 °C. We applied MDSC technique due to weak peaks observed on DSC thermogram; and no crystallization peaks were observed, because of amorphous features of the polymer.

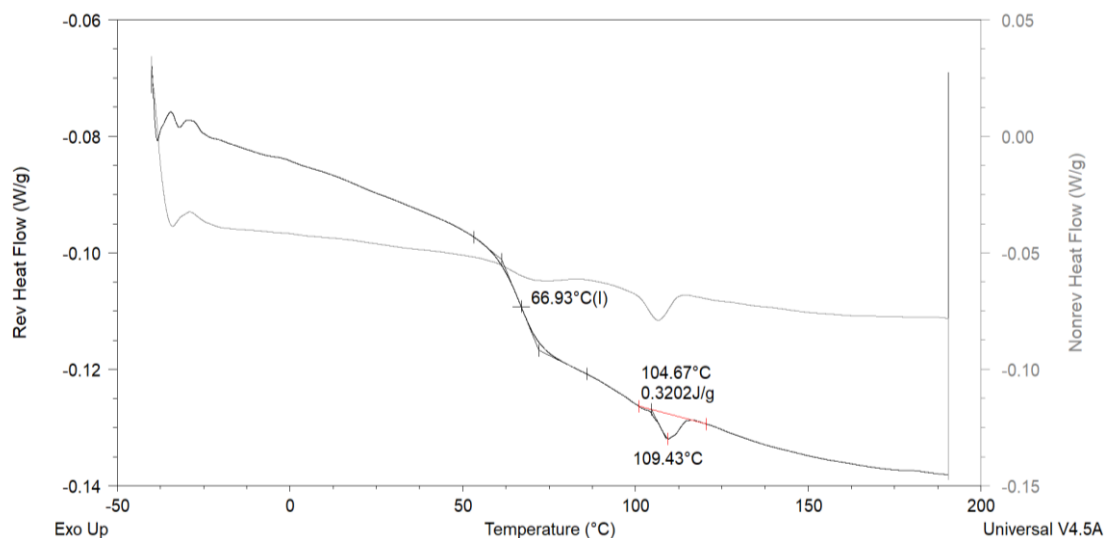


Fig. 7: MDSC thermogram of polyvinyl chloride (PVC). Black line reversing; and grey line non-reversing mode.

Comparison of thermal properties in samples with different height

The thermal conductivity of the investigated samples with different height are plotted in Figure 8 for poliamide-6, Figure 9 for polymethyl methacrylate and Figure 10 for polyvinyl chloride, respectively. The thermal conductivity curves as a function of the temperature shown expected temperature dependence. The thermal conductivity increased with increasing temperature and increasing height of sample. Same tendency for all investigated polymer samples semi-crystalline poliamide-6 and amorphous polymethyl methacrylate and polyvinyl chloride, was observed.

We obtained the result that thermal conductivity is an increasing function of the temperature. Explanation of obtained results is the higher molecular energy and more molecular movement of polymer chains with increasing temperature. Consequently, more molecular movement leads to faster heat transport in material from more energetic to less energetic molecules. Especially high differences of thermal conductivity value were observed close to glass transition temperature for polyvinyl chloride ($T_g = 65.7$ °C) (Figure 10). Polyvinyl chloride exhibited much higher value of thermal conductivity for range of temperature higher than 65

°C. The cause of that behaviour is the fact that above glass transition temperature in flexible rubbery state large-scale movements of polymer chains are possible and polymer structure become more mobile in comparison with brittle glassy state with limited molecular motions.

The smallest samples – 10 mm exhibited low thermal conductivity value compared to higher samples – 15 mm, 25 mm and 40 mm. The cause of that behaviour is the results of the heat escaping from investigated polymer samples, especially observed for smaller samples. Quantity of polymer material in smaller samples was not enough, which consequently leads to significant heat escape.

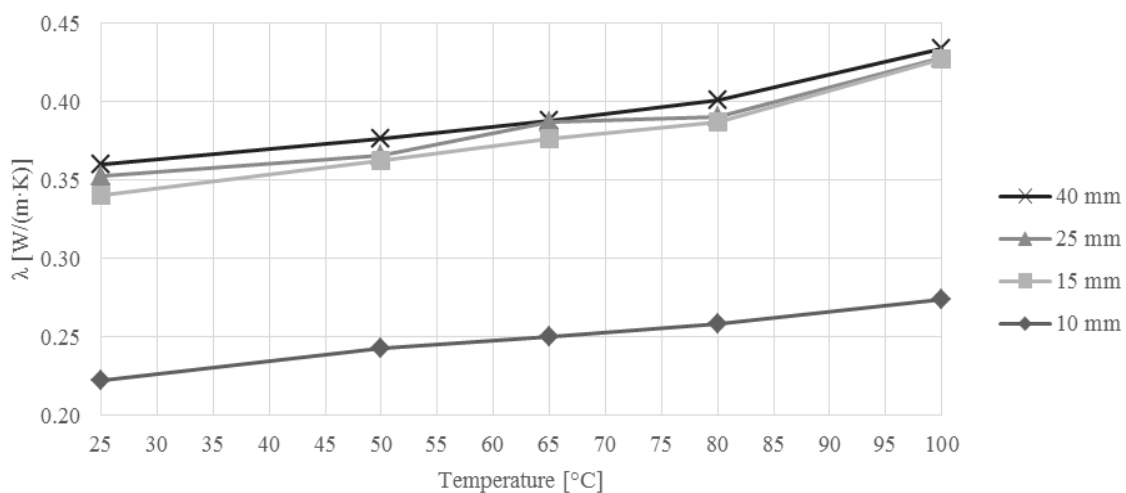


Fig. 8: Relationship between the thermal conductivity and temperature for sample polyamide-6 samples with different height.

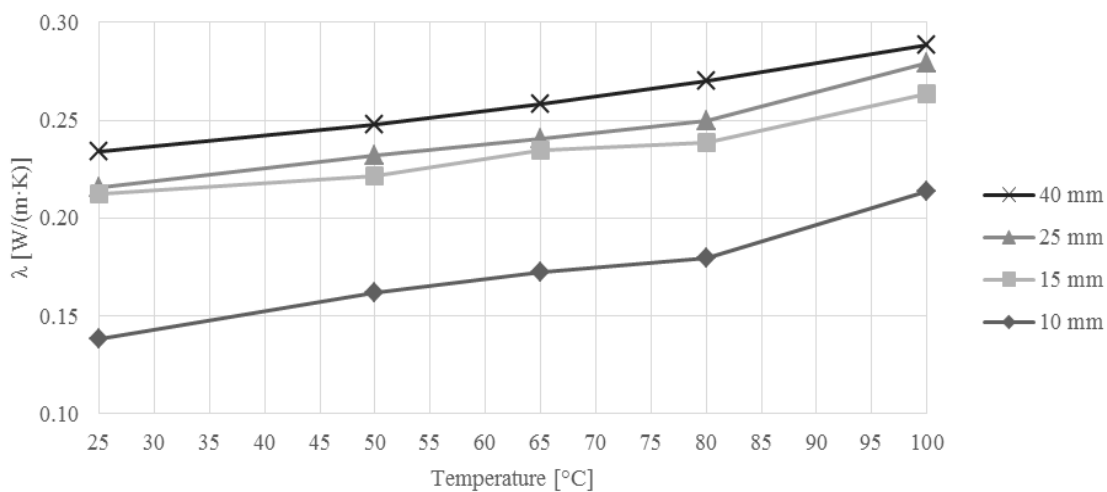


Fig. 9: Relationship between the thermal conductivity and temperature for sample polymethyl methacrylate samples with different height.

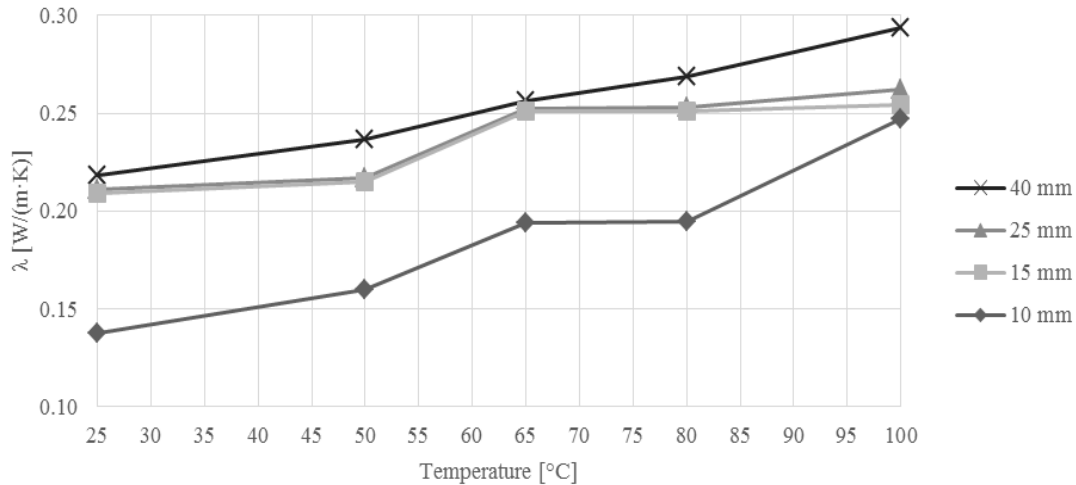


Fig. 10: Relationship between the thermal conductivity and temperature for sample polyvinyl chloride samples with different height.

Comparison of thermal properties in samples with different diameter

The thermal conductivity of the investigated samples with different diameter are presented in Figure 11 for poliamide-6, Figure 12 for polymethyl methacrylate and Figure 13 for polyvinyl chloride, respectively.

It can be observed, that thermal conductivity increased with increasing diameter of samples and with increasing temperature. Samples with the smallest diameter – 10 mm exhibit low thermal conductivity value compared to samples with a larger diameter – 15 mm and 25 mm. The reason of that behaviour is the same like that mentioned above for samples with different height. Quantity of ambient polymer material around the samples with smaller diameter was too small, which consequently leads to significant heat escape during the emitted heat pulse.

Especially for polyvinyl chloride was found. A 10 mm diameter presented the expected behaviour than other two polymers. However, since 50 °C temperature the heat flux underwent a dramatic increase, being its value close to those obtained in 15 mm of diameter. Once more the strong change in the slope curve occurred near to polymer glass transition. Thus, it can be understood as a strong mobility of the polymer chains.

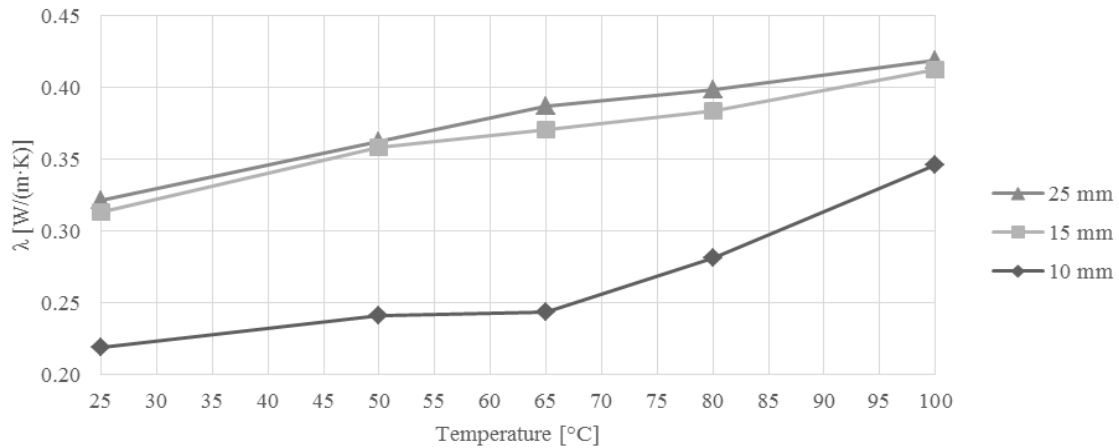


Fig. 11: Relationship between the thermal conductivity and temperature for sample polyamide-6 samples with different diameter.

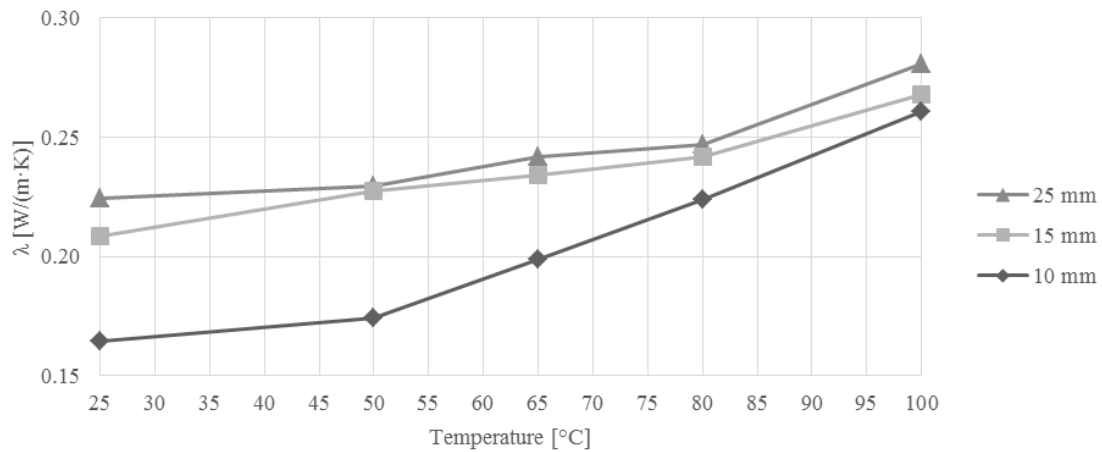


Fig. 12: Relationship between the thermal conductivity and temperature for sample polymethyl methacrylate samples with different diameter.

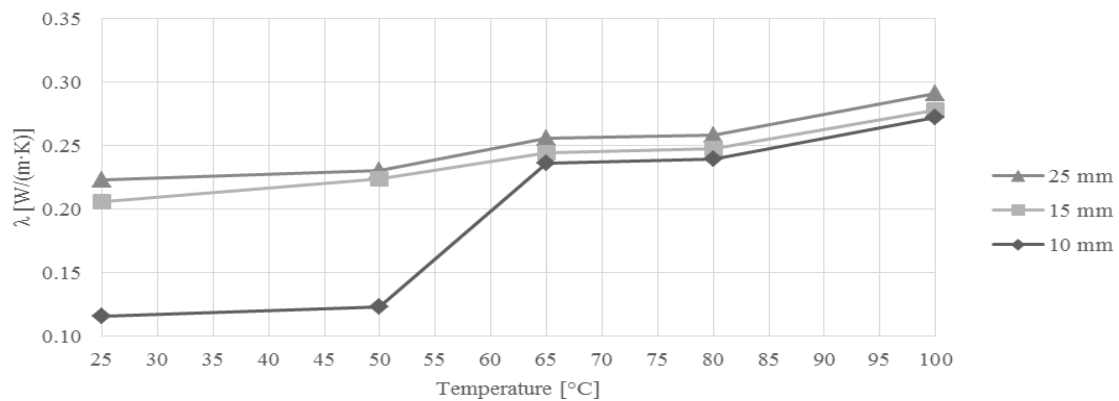


Fig. 13: Relationship between the thermal conductivity and temperature for sample polyvinyl chloride samples with different diameter.

Comparison of thermal properties in samples with different direction of sensor

The thermal conductivities of the investigated samples with different direction of the heat flow are plotted in Figure 14 to 16 for polyamide-6, polymethyl methacrylate and polyvinyl chloride; respectively. The anisotropy of thermal conductivity was observed for all samples. For parallel direction of the heat flow to polymer chains the values of obtained thermal conductivity were higher than for perpendicular and trans direction. Furthermore, when the direction of the heat flow was perpendicular the value of thermal conductivity was the lowest. The thermal properties of samples follow the trend of increasing thermal conductivity with increasing temperature.

The obtained anisotropy of thermal conductivity is attributed to two different heat transport in polymers. The higher thermal conductivity for parallel orientation of heat flow to polymer chains is owing to the fact that heat is transferred faster along polymer chain through strong covalent bonds C-C via phonon vibration, and because of longer phonon free paths along polymer chain. The lower thermal conductivity for perpendicular direction of heat flow to polymer chains is because of the fact that the heat is transported less effectively between chains by physical interactions as van der Waals force (Peterlin, 1969; Algaer et al., 2009).

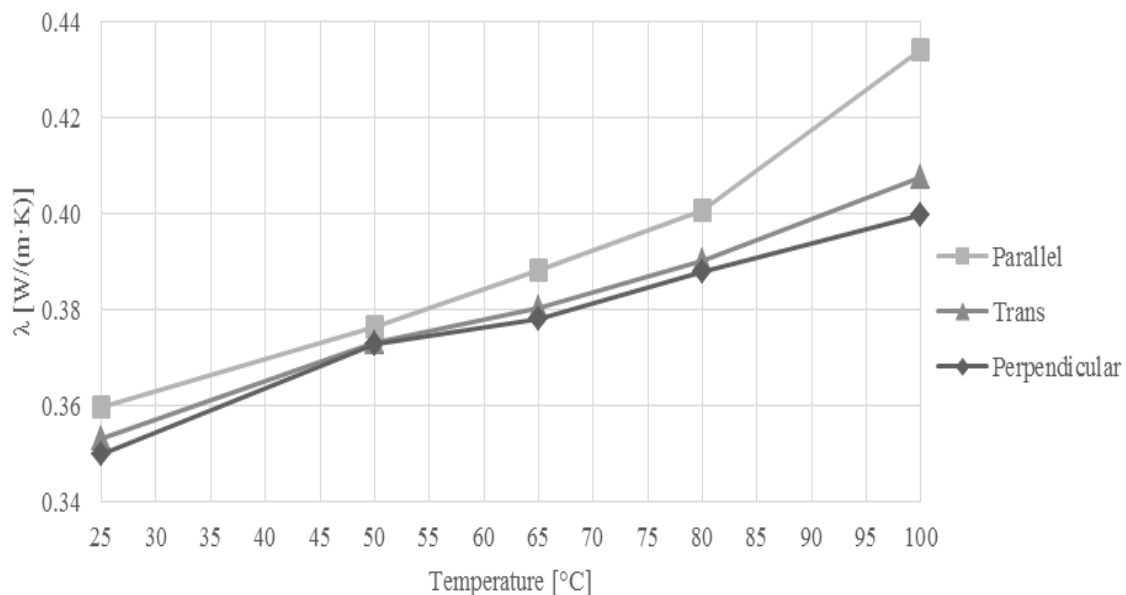


Fig. 14: Relationship between the thermal conductivity and temperature for sample polyamide-6 samples with different direction of sensor.

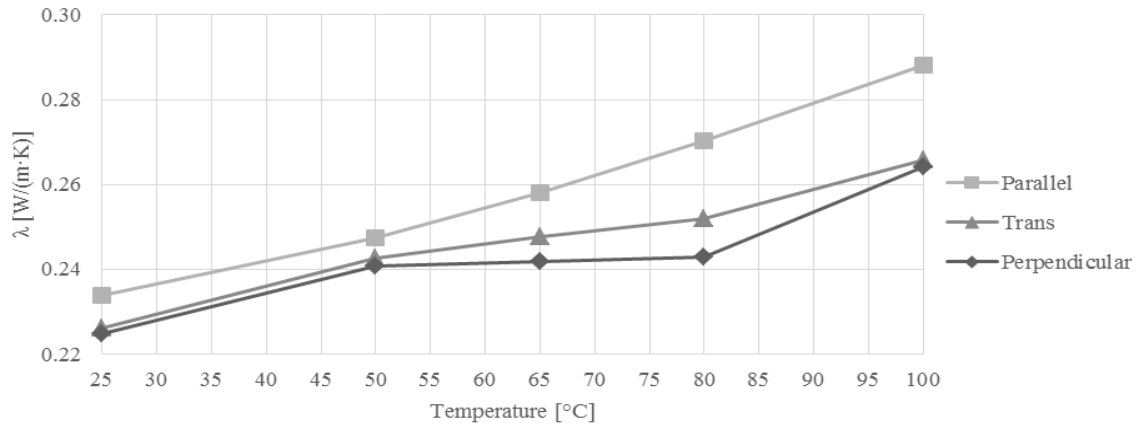


Fig. 15: Relationship between the thermal conductivity and temperature for sample polymethyl methacrylate samples with different direction of sensor.

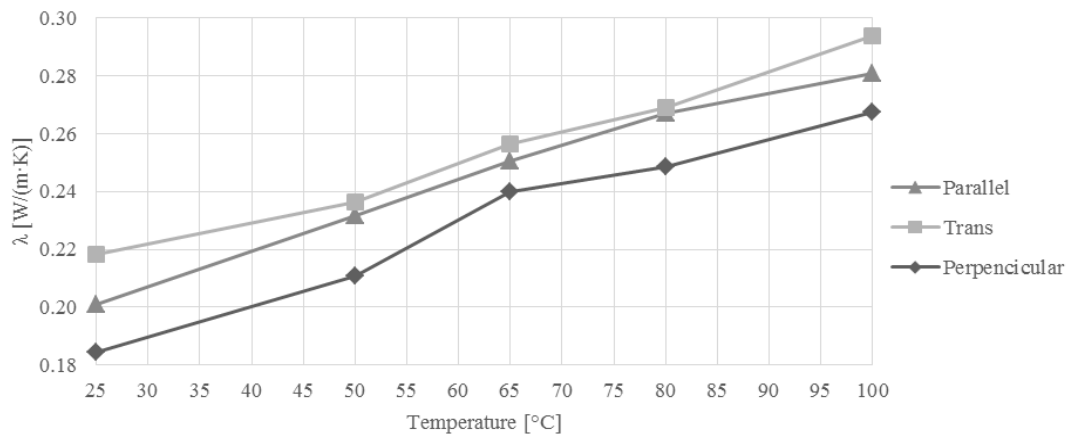


Fig. 16: Relationship between the thermal conductivity and temperature for sample polyvinyl chloride samples with different direction of sensor.

Comparison of thermal properties of different polymer materials

Figure 17, shows the thermal conductivity of different polymer materials. It can be seen that polyamide-6 exhibit the highest thermal conductivity value for the temperature from 25 °C to 100 °C. Amorphous polymers like polyvinyl chloride and polymethyl methacrylate have much lower thermal conductivity. At this point an important factor to take into account, which effect is clear on observed results are degree of crystallinity and ordering. High disordering of structure enhances the scattering of phonon and decrease the thermal conductivity (Radhakrishnan et al., 2004; Choy et al., 1980).

Polyvinyl chloride has the lowest thermal conductivity from investigated samples for temperature range from 25 °C to 50 °C, for higher temperature above glass transition

temperature thermal conductivity of polyvinyl chloride was higher than for polymethyl methacrylate. The polyvinyl chloride macromolecules have higher energy level in rubbery state than polymethyl methacrylate macromolecules in glassy state. This result clearly demonstrates that another factor, which influence on thermal properties is also the physical state of polymer materials.

Polyamide-6 is semicrystalline linear polymer with strong covalent bonds. These macromolecules contain carbonyl $-\text{CO}$ and amide groups $-\text{NH}$, which form hydrogen bonds between chains. Physical forces help to transfer heat more efficiently from one to other neighbouring chain (Brydson, 1999). The effect of mentioned properties is the highest thermal conductivity of polyamide-6 from investigated polymer materials. Polymethyl methacrylate is amorphous polymer with side groups, which increase the disorder of structure and decrease the thermal conductivity. Polyvinyl chloride is also amorphous polymer, investigated samples contained plasticizer between neighbouring chains, which higher the distance between chains, increase the disordering and decrease thermal conductivity (Peterlin, 1965), as well.

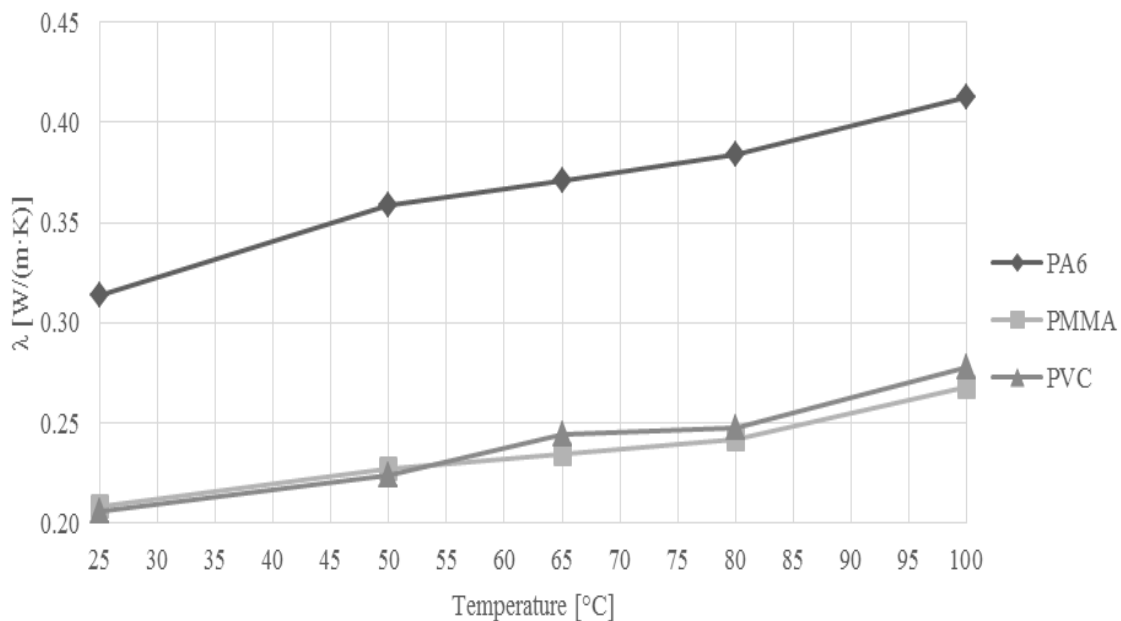


Fig. 17: Relationship between the thermal conductivity for different polymer samples.

Moreover, ambient temperature is important factor with great impact on thermal properties. Heat can be transferred in polymer material trough bonds via phonon vibration when the electrons on highest occupied valence layer have enough energy to cross up the distance to

higher conduction layer. Thus, heat conduction depends on energy level of electrons (Joel et al., 2009). Higher temperature, which indicate higher vibration energy of phonon and more molecular movement of polymer chains affect on higher thermal conductivity of polymers (Algaer et al., 2009; Choy et al., 1981). Especially high thermal conductivity value can be observed close to glass transition temperature. The reason of this phenomena is the fact that polymer structure become more mobile in flexible rubbery state in comparison with brittle glassy state with limited molecular motions. Thus, thermal conductivity significantly grows in glass transition region (dos Santos et al., 2013).

Degree of crystallinity and ordering significantly influence on heat transfer in polymer materials. Amorphous polymers have much lower thermal conductivity than semicrystalline. High disordering enhances the scattering of phonon and decrease the thermal conductivity. Thus, increasing amount of crystallinity increase the thermal properties of polymers. Furthermore, size and amount of side groups attached to polymer backbone decrease regularity and effectiveness of heat conduction. As reported before also presence of additional chemical components as plasticizer decrease the order of structure and decrease thermal properties (Radhakrishnan et al., 2004; Choy et al., 1980).

Chain orientation has great impact on thermal properties (Rubio et al., 2016). Polymers exhibit anisotropy of thermal conductivity, this fact is attributed to two different heat transport in polymers. The higher thermal conductivity for parallel orientation of heat flow to polymer chains is due to the fact that heat is transferred faster along polymer chain through strong covalent bonds C-C via phonon vibration, and because of longer phonon free paths along polymer chain (Rubio and Rodriguez, 2017). The lower thermal conductivity for perpendicular direction of heat flow to polymer chains is because of the fact that the heat is transport less effectively between chains by physical interactions as van der Waals force. It has also been observed that presence of hydrogen bonds between chains in polyamide-6 significantly increase the thermal conductivity (Peterlin, 1969; Algaer et al., 2009).

Dimension of sample determine the effectiveness of heat transport in polymers. The quantity of ambient polymer around the sensor should not be too small to prevent significant heat escape from the sample. The smallest samples have the major drop of heat flux from the core. Thus, decrease of the samples diameter and height affect on decrease of thermal conductivity value (Baird and Collias, 2014).

CONCLUSION

The chemical formula, crystallinity, morphology, temperature, chain orientation, diameter and height of different samples (PA6, PMMA, PVC) were investigated to study the heat transfer in polymer materials.

In summary, the obtained results suggest that important factor, which affects on thermal properties is degree of crystallinity of polymer. Higher degree of crystallinity and ordering influence on higher thermal conductivity.

The obtained results have indicated that thermal properties of polymers strongly depends on temperature. Heat is transferred through bonds via phonon vibration. Higher temperature affect on higher vibration energy of phonon. Thus, thermal conductivity increase when the ambient temperature increase.

Great impact on thermal properties has also polymer chain orientation. Thus, thermal conductivity is higher for parallel direction of heat flux to polymer chains than for perpendicular direction. The obtained anisotropy of thermal conductivity is attributed to the fact that heat is transferred faster along polymer chain trough strong covalent bonds via phonon vibration, that between polymer chains by physical interactions. It has also been observed that presence of hydrogen bonds between chains increase the thermal conductivity.

Moreover, another important factor, which has also significant influence on thermal properties is dimension of sample. Decrease of the samples diameter and height affect on decrease of thermal conductivity value. The smallest samples have the lowest value of thermal conductivity. Thus, the dimension of ambient polymer should not be too small to prevent major drop of heat flux from the sample.

The present research work clearly demonstrates that thermal properties depends on many factors like chemical formula, degree of crystallinity, morphology, temperature, chain orientation, and dimension of polymer sample. It was shown that there is a possibility to control thermal properties and enhance the heat transfer through the polymer materials. The obtained data suggest that it would be essential to control properties and parameters of sample to obtain better thermal properties using the same polymer.

The study of thermal properties has increased knowledge of the heat transport mechanisms in polymers, and moreover has contributed to improve the methodology using the ASTM

D5334 in polymers. However, to fully understand mechanism of heat transport in polymers materials many research still have to be done in this field of science.

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