Thermal conductivity of the epoxy resin filled by low melting point alloy

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ABSTRACT

Purpose: The aim of the work was to determine the effectiveness of the modification of epoxy resin's thermal conductivity by Wood's alloy particles addition.

Design/methodology/approach: The manufacturing method of thermosetting polymer matrix composite materials reinforced with the Wood's alloy particles was searched. Firstly, the mixing conditions were differentiated to obtain good dispersion of the reinforcement in polymeric matrix. The thermal conductivity coefficient of composites has been investigated by using of the quasi-static test.

Findings: The suggested method of thermal conductivity measurement allows to avoid the procedure of solving complicated equations. The developed measuring device enables the measurements of thermal conductivity of polymer composites in a form of a beam of 4x20X120 mm dimension. The results of the thermal conductivity tests and their analysis are presented.

Research limitations/implications: The presented research was limited to composites in the form of small samples. The developed technology of this type of composites' preparation is limited to small volumes because of the sedimentation process. The second limitation results from the necessity of matching the shape of the test specimen to the size of the cooler and heater on the device for measuring thermal conductivity.

Practical implications: Materials with low melting point alloy content show many possible applications. First of all, these are the materials of higher thermal conductivity and electrical conductivity than the standard polymeric materials.

Originality/value: The preparation method of thermosetting polymer matrix composite materials reinforced with the Wood's alloy particles fabricating method was developed. Composites with good thermal properties were obtained. The obtained results confirm the utility of the applied investigation methods in the thermal analysis of polymer composites.

Keywords: Engineering polymers; Composites; Low-melting-point alloy; Fusible alloys; Epoxy resin

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MATERIALS

1. Introduction

Thermal conductivity is one of the main fundamental properties of materials such as density, strength properties, chemical resistance and etc.

In a solid material, there are two main mechanisms of heat conduction: through elastic waves, which are called phonons, and through free electrons. Polymer materials generally do not have freely moving electrons. So, heat transfer has be effected only via elastic waves [1,2]. The electron conductivity is dominant in metals and absent in insulators.

For one-dimensional and rectilinear heat flow, the steady-state heat transfer in polymeric materials can be described by the Fourier's law of heat conduction [3]:

$$Q = -\lambda \frac{dT}{dx} \tag{1}$$

where Q is the heat flux, x is the thickness of the material, dT/dx is the temperature gradient per unit length, and the proportionality constant λ is known as the thermal conductivity.

Thermal conductivity, usually written as λ or k, is the most commonly used property that helps to quantify the transport of heat through a material. Accordingly, thermal conductivity follows the relation [4]:

$$\lambda = C_p p u i \tag{2}$$

where: u is the speed of sound, i the molecular separation, C_p the specific heat, p is density of material.

In some publications [5,6] modification of the equation (2) for the materials, where phonons conduction mechanism is dominant (example: polymer materials), can be found:

$$\lambda = \frac{C_p v I}{3} \tag{3}$$

where: v is the phonon group velocity, I is the phonon mean free path.

Amorphous polymers show an increase in thermal conductivity with the increasing temperature up to the glass transition temperature (T_g). Above this temperature, the thermal conductivity decreases with increasing temperature. Due to the increase in density upon the solidification of semi-crystalline thermoplastics, the thermal conductivity is higher in the solid state than in the melt state. However, in the melt state, the thermal conductivity of semi-crystalline polymers is reduced to the values typical for amorphous polymers. Unfilled thermosets have thermal conductivity which is very similar to amorphous thermoplastics [4,16].

Thermal conductivities of polymer materials depend on many factors, such as chemical constituents, strength of bonding, structure type, molecular weight of side groups, molecular density distribution, type and strength of defects or structural faults, size of intermediate range order, processing conditions and temperature, etc [6]. For thermoset polymer materials, like epoxy resisns, the cross-link distance as an important parameter [7].

Although the value of thermal conductivity coefficient for pure materials was obtained theoretically or experimentally, there is not enough information and data available on the thermal conductivity of polymer composites.

Methods for determining the value of thermal conductivity are different. The most popular methods of thermal conductivity measurements are classified as stationary and nonstationary [8].

All stationery methods are based directly on Fourier's law, with a constant temperature difference on a sample. On contrary to the stationary methods, the non-stationary or so called dynamic methods are based on time varying temperatures [9].

The measurement of these thermal properties includes a set of parameters that are common for different techniques and methodologies of determining the coefficient of thermal conductivity. In addition to changes due to the nature and type of the samples, all the methodologies require determination of the actual amount of heat transferred through the sample along and perpendicular to the heat flow path in a given thermal environment. Conductivity is a function of diffusivity, density and heat capacity. When through-thickness thermal conductivity for fixed-dimension solids is primarily measured in steady-state conditions, the accompanying transient diffusivity in the radial direction is taken into account by the use of the ratio of sample thickness to the total sample area as the heat flow path. The relationship is expressed as [8,10]:

$$\lambda = \frac{qh}{A(t_1 - t_2)} \tag{4}$$

where: q is time rate of the heat flow, h is thickness of the sample in the heat flow direction, A is an area of the sample, t_1 is the temperature of a hot surface, t_2 is the temperature of a cold surface.

Polymer materials are good thermal insulators. Commonly used polymer materials are materials of low thermal conductivity. The typical range of polymers is from 0.17 to 0.35 W/mK [11].

Improved thermal conductivity in polymers may be achieved either by changing the molecular orientation, by the addition of conductive fillers [12] or by synthesizing the polymer materials with enhanced thermal conductivity [13].

The only economically justifiable option to increase the thermal conductivity of the polymer materials is to fill them with particles of high conductivity coefficient. In consideration of the difficulty of synthetic techniques and many other factors, the other methods are unacceptable at present [13]. The fillers that conduct heat well and are also inexpensive are metal fillers. When compared to other solid materials, the metals have superior heat transfer capabilities.

The use of the fillers affects the heat transfer mechanism in the material. In particular, the heat transfer in metallic fillers will be characterized by the movement of free electrons. For these reasons, the heat conductivity in the composite material will be the sum of both mechanisms. Additionally, the value of the coefficient thermal conductivity of the composite will affect the thermal resistance at the boundary of the phases.

Polymer matrix composites filled with metal particles are of interest for many fields of science and engineering. This interest arises from the fact that the thermal, electrical and shielding

characteristics of such composites are close to the properties of metals, whereas the other, specially mechanical, properties and processing methods are typical for polymer materials [14]. These composites may be substituted for metals and alloys.

Primary benefits of the used heat conductivity polymer materials compared with fabricated metal include:

- relatively low density,
- higher thermal conductivity than pure polymers,
- customizable thermal performance with variety of fillers,
- large selection of polymer matrix,
- low thermal expansion - similar to metals,
- integrated flexibility (especially thermoplastic materials),
- excellent aesthetics,
- design flexibility,
- more complex shapes,
- lower production cost compared to metal materials,
- can be electrically insulative or conductive,
- lower shipping and transport cost,
- longer service life,
- more chemical resistance,
- heat dissipation can be superior to metals,
- greater reliability,
- eliminate/reduce secondary operations during production,
- less parts - consolidation few parts to one.

The most important benefit provided seems to be the possibility of making materials that are together thermally conductive and electrical insulators.

The advantage of the use of composites is the ability to influence its properties by selecting the ratio of its components. By entering more filler to the polymer matrix, we increase properties of the composite which are characteristic for the filler material. By reducing the amount of filler in the composite, characteristics of the matrix material become dominant. In this way, we can change the thermal conductivity of the composite. Of course, applying fillers not only affects the thermal conductivity but also electrical conductivity, stiffness and friction.

In the article [11], the following division due to the thermal conductivity of the polymer matrix composites was proposed:

- Composites with very low thermal conductivity. Coefficient of thermal conductivity of these materials is less than 0.1 W/mK. This low coefficient is achieved by filling the matrix polymer with microspheres. Commercially available glass, polymer, ceramic, and carbon hollow microspheres can be used as fillers in various polymer materials. The microspheres are used in a variety of applications because of the improved properties such as reduced density, increased stiffness, improved compressive and impact strength, reduced shrinkage and increased dimensional stability of products, good thermal and chemical resistance, low thermal conductivity and dielectric permeability;
- Polymer composites with relatively high thermal conductivity (0.1 W/mK $<\lambda<$ 10 W/mK). In the second category we may find most commonly used fillers. An example can be glass fillers which improve thermal conductivity by about 20-30%, but this filler is often used for non-thermal purposes.
- Ultra-high thermal conductivity polymer composites (10 W/mK $<\lambda$). The third category uses high aspect ratio fillers, such as graphite fibers, metallized glass fibers, silver

powders [15], aluminum fibers and flakes and etc. One approach to improve thermal conductivity has been to synthesize polymeric composites with high thermal conductivity nanoparticles [16,17], such as graphene and carbon nanotubes [18].

For supplementation of the presented division, there is a necessity to implement another group of composites. It is a group of composites of ultra-low thermal conductivity. Further enhancement of thermal insulating quality can be achieved by forming polymer/gas composite, namely a polymeric foam.

The books [4,19] shows the comparison of the thermal conductivity of polymeric foams and polymer/metal composite. So, it is the comparison between ultra-low and ultra-high thermal conductivity materials (Fig. 1).

Metallic fillers should be qualified to the second and third groups. The most commonly used filler materials are shown in Table 1. For the comparison: the value of thermal conductivity for the perfect vacuum equals 0 W/mK while for monocrystalline diamond equals 2000 W/mK.

The low cost and high mechanical properties of metals, especially in a form of fibres compared to the traditional reinforcement like glass or carbon fibres, is the main motivation to explore the potential application.

Not only material of fillers affects the thermal conductivity of the composite. By the way, it does not make much sense to increase the thermal conductivity by changing the fillers materials beyond a certain level. Going from 40 to 400 W/mK increases the composite's thermal conductivity by only 5% [11]

The following parameters are important [11]: filler conductivity, polymer material conductivity, shape and aspect ratio, volume loading, packing and orientation, and bonding. Additionally, density and specific heat are affected. In the article [13], as equally important, the following parameters have been shown: the surface modification of filler and the selection and optimization of the conditions of molding and processing.
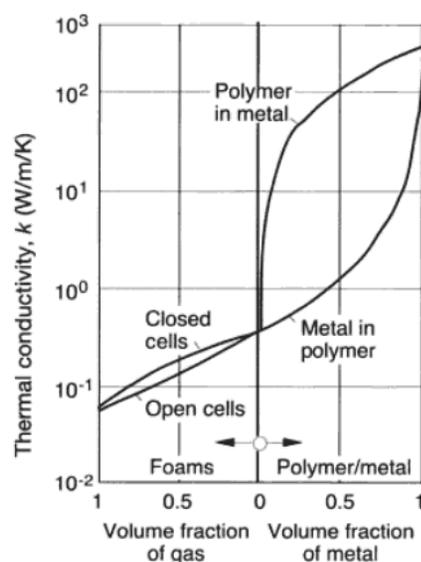
Fig. 1. Thermal conductivity of polymer materials filled by metal or glass [4]

Table 1.
Thermal conductivity of examples of filler material for polymer application [20,18]

Material	Thermal conductivity coefficient [W/mK]
C-Glass	1.10
E-Glass	1.30
Stainless Steel	1.24-30.0
Carbon	24.0
Graphite	6-150
Aluminium	210
Copper	385
Silver	419
Graphen	>3000
Carbon nanotubes	>3000

Most polymer materials are processed and fabricated at elevated temperatures, often above their melting temperatures.

Polymers filled with metal particles have worse processing properties than pure polymers. Rigid filler particles impede the flow of material. Metal powders are easily oxidized during the mixing process which affect the properties of the composites. During the formation of polymer, metal composites encounter problems resulting from the difference in density of both materials. One of the ways of avoiding this problem is the use of low melting point alloys as fillers.

The method of preparation of this type composite is typical for mixing the two liquids together. This is possible because the low melting point alloys have similar temperature range as the typical processing polymer materials. Low-melting alloy epoxy composites made with the application of the last method were presented in our previous papers [26,27].

The most traditional low melting point alloys are based on bismuth. They are usually alloys also with high proportions of lead, tin, sometimes with the participation of iridium, cadmium, thallium, copper, silver or antimony [28,29,30]. If we accept that a low-melting alloy is the alloy melting below 200°C, then a few solders alloys also meet this requirement. The melting point of some alloys with indium is even lower than 50°C (Table 2).

Soldiers alloys consist mainly of lead and tin, sometimes zinc, silver or copper. This is unique, especially as the parent metals in the pure state have their melting points high above alloy's. Although most of the low melting point alloys do not have high strength or hardness, they have many industrial uses.

Most literature describes compositions of low melting point alloys and thermoplastics matrix [32,32,33]. Authors of this article undertook an effort to create a heat transfer composite with a thermoset epoxy resin and low melting point Wood's alloy.

The epoxy resins are the second most widely used thermoset resins for composite applications. The most important of these is the application for coatings or adhesives. One reason for epoxy resins widespread success is their ability to accept fillers.

Epoxy resin is one of the common materials that is used for electrical insulation and packaging. In spite of the excellent performance of epoxy, its relatively low thermal conductivity cannot satisfy the increased demands of new electronic devices. Fillers are used to enhance the specific properties of materials [34].

Nowadays, advanced filled epoxy resins challenge alternative thermal interface materials like silicones, greases, tapes, or pads [35]. Exemplary values for the thermal conductivity of epoxy composites are shown in Table 3.

Higher thermal conductivity of the commercial electrically conductive composites presented in Table 3 is the result of the conducting of heat and electricity. In the case of thermally conductive composites, they must also act as electrical insulators, which limits of the use some filler materials (Table 1) [36]. Thermal properties of materials are very important in many industrial applications. Implementation of composite materials by mixing methods in the liquid phase, which are devoid of existing restrictions, increasing applicability of thermally conductive polymer composites and metal.

Table 2.
Exemples of the low melting point alloys [21,22,23,24,25]

Inventor's name or trade name	Chemical composition	Melting point	Thermal conductivity
Indalloy 117, CS Alloys Low 117, CERRO LOW 117 Alloy	$Bi_{44.7}Pb_{22.6}In_{19.1}Sn_{8.3}Cd_{5.3}$	47°C	15 W/mK
Indalloy 136	$Bi_{49}In_{21}Pb_{18}Sn_{12}$	58°C	23 W/mK
Wood's alloy, Indalloy 158, Roto158F, CS Alloys Bend 158, AsarcoLo 158	$Bi_{50}Pb_{25}Sn_{12.5}Cd_{12,5}$	70°C	18 W/mK
Newton's alloy, Indalloy 203 Roto203F, CERRO SHIELD Alloy, CS Alloys Shield 203	$Bi_{50}Pb_{31.2}Sn_{18.8}$	96°C	13 W/mK

Table 3.
Thermal conductivity of modified epoxy resins [20]

Epoxy composites	Thermal conductivity coefficient [W/mK]	
	Min	Max
Epoxy composite type:		
Pure resin	0.140	1.208
Glass fiber filler	0.259	1.260
Mineral filled	0.700	1.500
Metal filled	0.724	9.090
Carbon fiber filled	0.450	400.0
Application of epoxy:		
Adhesive	0.140	9.400
Thermally Conductive	0.100	9.090
Electrically Conductive	0.550	29.00

2. Experimental

The experiment was based on the thermal conductivity of the epoxy resin filled by Wood's alloy particles performed by the of mixing the liquid composite components.

All the tests were performed in the laboratories of the Department for Processing of Metals and Polymers that is a part of the Institute of Engineering Materials and Biomaterials at the Silesian University of Technology.

The experiments were made with the Wood's alloy supplied by "Innovator" Company (Poland). The melting point of the alloy is 70°C. The particles of the Wood's alloy were dispersed in the epoxy resin system. The epoxy resin system was cured at ambient temperature with a hardener. We used epoxy resin Epidian 6 and hardener Z1 (mixed weight proportion 100:13), both by "Organika Sarzyna" Company (Poland). Epidian 6 is an epoxy resin obtained from bisphenol A and epichlorohydrin having an average molecular weight $\leq$ 700. Z1 is triethylenetetramine hardener. The main properties of the epoxy resin system are presented in the article [37].

Samples with 0, 20, 40, 60, and 80% (by weight) of the Wood's alloy were made from these materials.

Composites were prepared according to the following procedure:

- Measured portions of the Epidian 6, Z1 and Wood's alloy, for composites with various concentration of the low-melting alloy;
- Epidian 6 and Wood's alloy was mixed together at the temperature of 80°C at the time of 3 minutes, the rotation speed was 10 000 rpm. Peripheral speed is: 13,09 m/s. Mixtures were made to dissolver DISPERMAT LC30 by VMA-GETZMANN GMBH. Dissolver mixer fitted with a Z-blade with 25 mm diameter of disk. Mixing was done in a cylinder of 50 mm diameter. Manufacturer's recommendations as to form dispersions (Fig. 2) have been met in this way;
- After the cooling to the room temperature, the resin and alloy compositions mixed with the hardener Z1.

After the preparation, the compositions were cast in a mold of a size of 120x20x4 mm. In the next step, the samples were heated up in the dryer to 25°C. Times of the samples curing were 24 hours.

The research of thermal conductivity was performed on the device designed and built at the Department for Processing of Metals and Polymers and Division of Nanocrystalline and Functional Materials and Sustainable Proecological Technologies that is a part of the Institute of Engineering Materials and Biomaterials at the Silesian University of Technology [38,39].

The device was designed on the basis of some findings of PN-EN 12667:2002 Polish National Standard. Fig. 2 show scheme of device.
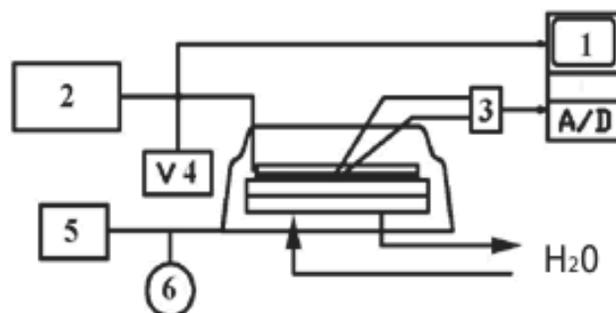
Fig. 2. Testing system for measuring the thermal conductivity [40]. The device consists of the following elements: 1 is a computer with A/D card, 2 is a DC power supply, 3 is a thermocouples, 4 is a universal meter of electrical properties, 5 is a vacuum pomp, 6 is a vacuum gauge

The test consists of the heating of the top surface and the cooling bottom surface of the sample. The temperature on the upper and lower side of the sample material was examined.

The previously prepared test sample of thermal conductivity was provided on the radiator block, then the heater was placed (The set heater power was 2 W) To ensure better adhesion surface, the heater was charged with two weights. Between the surface of the heater and the cooler of the sample thermal grease H by "AG TermoPasty" was applied.

The results of the test were the graphs of temperature changes on both surfaces of the material sample at the measure time. The test lasted until the temperature's stabilization.

The following formula was used to measure thermal conductivity :

$$\lambda = \frac{Ph}{lb\Delta T} \tag{5}$$

where: P - power heater, h - thickness of the sample, l - length of the sample, b - width of the specimen, ΔT - the difference between the temperature of the lower and upper side of the sample.

ΔT was averaged from the two readings of thermocouples. An example of the resulting graph for the pure resin Epidian 6 presented Fig. 3.

Table 4.
Thermal conductivity of tested composites

Epoxy composites	Pure Epidian 6/Z1	20% Wood's alloy + 80% Epidian 6/Z1	40% Wood's alloy + 60% Epidian 6/Z1	60% Wood's alloy + 40% Epidian 6/Z1	80% Wood's alloy + 20% Epidian 6/Z1
Top surface temperature [°C]	24.30	24.19	24.49	24.57	24.49
	39.22	37.42	36.81	35.95	32.52
Bottom surface temperature [°C]	38.36	37.03	37.63	35.13	32.05
Power of the heater [W]	1.96	1.98	2.00	1.92	1.85
Thermal conductivity coefficient [W/mK]	0.23	0.26	0.27	0.30	0.41
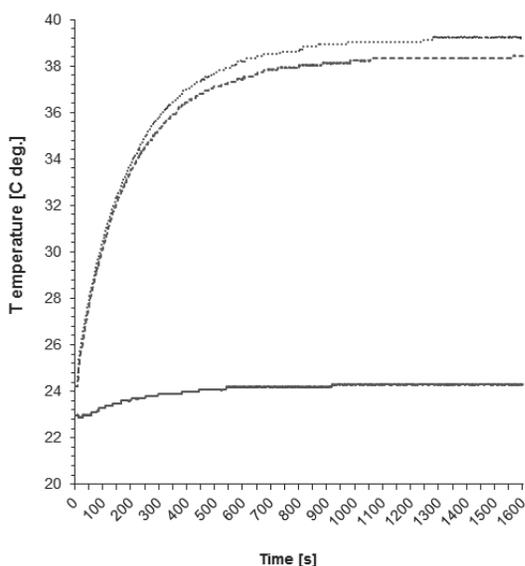
Fig. 3. Testing curve for the pure Epidian 6, where ---- and ····· are the temperatures measured by thermocouples from the top surface of epoxy resin sample (heated surface), — is the temperature measured from the bottom surface (cooling surface)

3. Results and discussions

After the measurements were completed, all the samples summarize the results of individual data as shown in Figure 4. Each sample was tested under the same conditions, where the temperature is equal to the initial ambient temperature of about 24°C.

There were small initial temperature variations between the samples. However, this did not affect the final results because the calculation of the average temperature considered the mean temperature from the last 100 measurements.

Figure 4 shows the temperature measured on the surface of the samples. Gradient bars represent the temperature difference between the upper and lower stable temperature. As it can be seen, the temperature gradient decreases with the increasing Wood's alloy content in the composite.
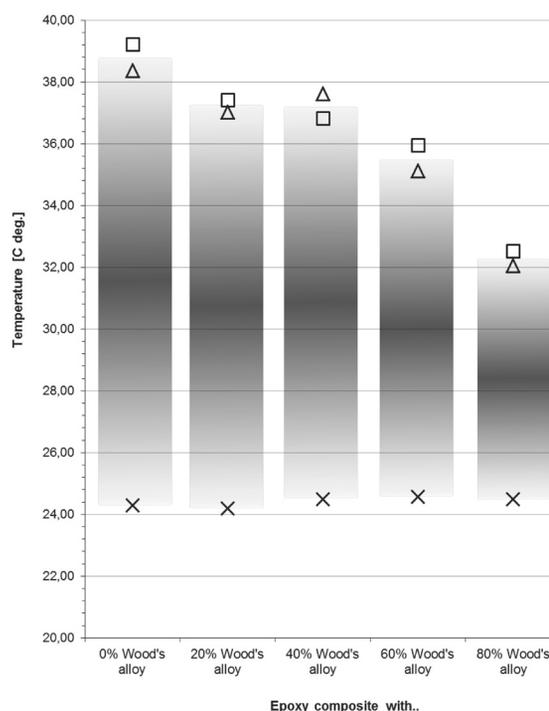
Fig. 4. Stable value of temperature from bottom and top surface of tested composites. Δ, □ - temperatures measured by thermocouples from the top surface, x - temperature measured from the bottom surface

As the Fig. 4 shows, one of the temperature of the heated (top) surface of the sample usually achieves a higher value. This may imply that a weaker adhesion of the surface to the heating element or thermocouple attached at this point has a lower accuracy. Temperature differences on the top surface are not huge, so that it can be concluded that flatness of the sample has been required there. The applied thermal grease H, increases also the adhesion of the surface to the surface of the boiler and radiator.

The values of thermal conductivity was calculated by the formula (5) and presented in the Table 4.

Figure 5 shows the analysis of the thermal conductivity changes depending on the content of Wood's alloy in the

composite. Thermal conductivity for composites and pure resin allows to create an equation of correlation:

$$y = 0.0127x^2 - 0.0376x + 0.2656 \qquad (6)$$

where:
y is a coefficient of thermal conductivity [W/mK],
x is a Wood's alloy content in the composite [%].

This equation has a high correlation coefficient (R^2), which is 0.949.
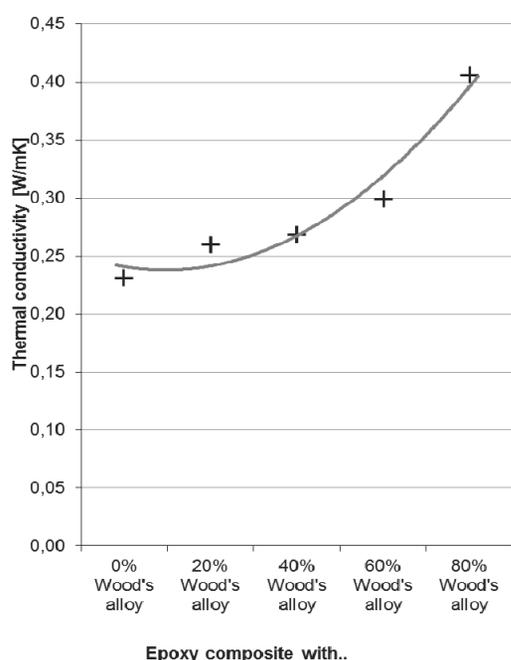
Fig. 5. Influence of Wood's alloy on the thermal conductivity of the composite

4. Conclusions

We have presented an experimental study of the thermal conductivity of polymer-metal composites. We have investigated Wood's alloy particles in the epoxy resin matrix with different particle concentration. The research has shown that the coefficient of thermal conductivity of composites decreases with the increasing concentration of Wood's alloy.

This work gives valuable insight into the behaviour of epoxy composite materials reinforced with Wood's alloy in mixing and curing processes, too.

The presented research was preliminary. In the future, researches with a higher temperature are planned. The determination of the conductivity of the composite at an elevated temperature, where one of the composite phase is liquid, enables the design and execution of a material with a variable thermal properties. As the there is a low melting point, alloy fillers can be

expected to increase the thermal conductivity of such composite materials. Such composites could be classified as so called smart materials.

In the future we are planning experiments with other low-melting alloys, too.

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