Influence of casting velocity on surface resistivity of epoxy-hard coal graded composites

J. Stabik, M. Chomiak*
Department for Processing of Metals and Polymers, Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Glivice, Poland
* Corresponding author: E-mail address: monika.szczepanik@polsl.pl

Received 04.10.2010; published in revised form 01.01.2011

ABSTRACT

Purpose: of this paper was to describe electrical properties of epoxy-hard coal functionally gradient polymeric materials. The experimental part describes preparation of the cylindrical samples of the polymeric gradient material by centrifugal casting method. Influence of casting velocity on electrical surface resistance and surface resistivity was measured and analysed.

Design/methodology/approach: The specimens were prepared using centrifugal casting method. Composites with epoxy resin as a matrix and with respectively 3; 4.32; 7.5; 10.68 and 12%vol of two hard coal types as a filler were cast. Surface resistivity was applied as a measure of electrical properties. The idea of the test was to define electrical resistivity in radial direction with different content and type of conductive component (hard coal). First, specimen’s diameter was measured and electrical resistivity was tested. Next, outer layer with thickness about 0.1 mm was removed by turning and electrical measurements performed. The procedure was repeated for all subsequent layers.

Findings: The experimental results demonstrated that casting velocity have pronounced influence on surface resistivity of these materials. Hard coal together with epoxy resin formed gradient composite material with different filler content in subsequent layers.

Research limitations/implications: The generated graded structure could be controlled only in limited extend by varying rotation speed and material characteristics of components, such as hard coal content and particles size and shape.

Originality/value: Hard coal particles were distributed in epoxy resin by centrifugal casting method in order to create functionally graded materials (FGMs). At present, no information is available on the influence of the casting velocity on the electrical properties of these composites. This paper is original also because in the research programme electrical properties of new type of polymeric gradient composites were tested and presented.

Keywords: Electrical properties; Functionally polymeric Gradient materials; Hard coal; Surface resistivity

Reference to this paper should be given in the following way:

1. Introduction

The paper presents electrical properties of new type of gradient materials produced by centrifugal casting technology. These materials are new because up till now no information was found on the graded composites made of epoxy resin as a matrix and hard coal particles as a filler.

Division of Metallic and Polymeric Materials Processing of Silesian University of Technology deals with polymeric graded materials for several years, still searching for new components and better methods of their production [1-7]. One of the problems searched in our Division is to develop polymeric material with gradient of electrical properties. Especially interesting are materials that are insulators on one side and conductors on the other. Insulating properties are ensured due to electrical characteristics of polymeric matrix and conductive properties are achieved due to application of conductive filler. Continuous change of electrical properties across the composite is a function of continuously changing volumetric content of filler. The general idea of structural gradient materials was for the first time developed for composites and polymeric materials in 1972. Various methods were suggested for achieving gradients in polymeric materials, mainly by changes in composition, in filament concentration, and in polymerization process kinetics and chemistry. Also possible applications for the resulting graded structures were proposed and in many cases tested. The gradient material of the definition is a composite material, which changes its properties as a function of at least one direction. On the other hand, in most of practical applications graded materials can be treated as layered composites. The basic aim of performed researches was to obtain materials with gradient structures in which volume fractions of two or more components vary continuously as a function of position along certain direction(s) of the structure to achieve a required functional changes in properties [8-13].

The idea of graded materials is not new and graded materials were not invented by humankind. Many researchers have found that gradient of phases or otherwise of their graded distribution is responsible for the often unique properties of materials existing in nature. Bamboo, barley, maize are plants which stem structure changes gradually in radial position. The specific gradient structure gives these plants’ stems amazing flexibility together with very high strength. The overall concept of polymer gradient materials with concentration distribution is to receive material in which shares of the polymer and filler components vary smoothly in certain direction, while concentration of one component grows, the other decreases. Polymer graded materials are manufactured using different methods. Graded materials manufacturing processes can be classified, depending on the phase transition during the synthesis of materials, processes in the gas phase, liquid or solid. Methods for producing in the gas phases allow obtaining very thin gradient coatings. In the case of processes occurring in the liquid phase achievement of the gradient distribution of components is difficult, because due to the diffusion phenomenon the system strives hard to homogenization. For the production of massive graded materials most widely used processes are in the solid phases or in solid-liquid phases. Gradient of specific functional characteristics of materials is obtained primarily through changes in parameters such as chemical composition, density, grain size and microstructure as a function of position. A method of controlling the scheduled gradient is one of the main problems in the design of graded composites. At the turn of XX and XXI century scientists elaborated many preparation methods that allow making effectively Functional Graded Materials (FGMs) which also can be applied to Functional Graded Polymeric Materials (FGPMs), for instance: solutions diffusion, corona discharge curing, compression moulding, selective laser sintering, in situ polymerization, gravitational or rotational casting, powder metallurgy techniques, reaction bonding and other technologies. One of the effective methods applied in production of FGPM is the centrifugal technique. The casting method and parameters depend upon the type of processed material which can be in form of liquid, paste or powder. The material is located close to heated mould’s wall. Mould with processed mixture is rotated around one or two perpendicular axis. As a consequence of applied centrifugal force one of the components sediments gradually toward the mould walls [14-18].

Relatively new class of composite materials are polymeric composites filled with hard coal particles. The main problem with hard coal is its complicated structure and properties changing with every coal mine and even with every lot. Hard coal is a coal humus, heterogeneous in its structure, forming a mixture of several varieties of petrographic structures (with different hardness and gloss), forming a single strand. These include: fibrous coal, satin coal, semi glossy coal and glossy coal. Hard coal is one of the coals containing carbon element from 78 to 92%. Special type of hard coal is anthracite in which the carbon content is between 92% and 98%. Hard coal is mostly black in colour and with compact and brittle structure. Due to their complicated structure there are several types of coals, defined by properties such as sinter ability, the contents of volatile components, the expansion pressure and heat of combustion. It occurs mainly in the deposits of Paleozoic (Carboniferous, Permian) and Mesozoic. Hard coal is widely used around the world mainly for energetic purposes. Apart from energetic applications it is used also in medicine and chemical industry, where as a result of hard coal processing many compounds with different functions were developed [19-20].

Polymeric materials and polymeric composites among them are widely used in electrical engineering and electronics as insulators. Due to their high electrical resistance, especially surface resistance, they are susceptible to pick up static electricity on their surface. They may be subject to a breakdown, and exhibit varying creeping current resistance. Electrical properties of polymeric materials are defined by the resistance, dielectric strength, arc resistance, creeping current resistance and electric permittivity. The study of electrical properties of polymeric materials, intended for the manufacture of insulators, makes it possible to determine their future use. In the present study measurements of electrical surface resistivity were performed and because of this definitions of basic concepts are given [21].

Surface resistance, $R_s$, is defined in all of the aforementioned literature sources as the ratio of a DC voltage, $U$, to the current, $I_s$, flowing between two electrodes of specified configuration (1) that are in contact with the same side of a material under the test (Fig. 1):

$$R_s = \frac{U}{I_s}$$  (1)
2. Experimental

Influence of centrifugal force on the particles contained in liquid resin while mould rotating caused formation of gradient of filler concentration on the cross-section of the sample prepared from composites with different concentration of filler (hard coal). The aim of experimental research was to determine the electrical resistance on the subsequent layers of material and then to determine the effect of casting velocity and filler concentration on the profile of electrical resistance across the samples. The compositions were mixed with different filler contents (3-12%vol) and at different rotational velocities (535.3-617.7 rpm). Performed research had a preliminary character because only one polymeric resin and two types of hard coal as a filler were applied.

2.1. Materials

Epoxy resin, Epidian 6, cured at room temperature with hardener Z-1, both produced by „Organika - Sarzyna” Chemical Plant S.A. (Poland) were used in this study as thermosetting polymeric matrix [25]. Two types of hard coal were used as a filler and electric properties modifier. The anthracite coal filler mined in Kuzbas of the Kuznetsk Basin and the second type of hard coal acquired from “Zofiówka” coalmine. Basic characteristics of component materials are shown in Tables 1-4.

Surface resistivity, $\rho_s$, on the other hand, is determined by the ratio of DC voltage, $U$, drop per unit length, $L$, to the surface current, $I_s$, per unit width, $D$ (2):

$$\rho_s = \frac{(U/L)}{(I_s/D)}$$

Fig. 1. Basic setup for surface resistance measurement [23]

Surface resistivity is a property of a material. Theoretically it should remain constant regardless of the method and configuration of the electrodes used for the surface resistivity measurement. A result of the surface resistivity measurement depends on both the material and the geometry of the electrodes used in the measurement. The physical unit of surface resistivity is Ohm (Ω). The legitimate unit of the surface resistance is also Ohm [22-23].

The aim of this research was to analyse dependence of the surface resistivity on two parameters, namely the filler content in the composite and the angular velocity in centrifugal casting. Statistical experimental design methodology was used to determine filler content and angular velocity levels. The rotatable central composite design was chosen [24-25].

2.1. Materials

Epoxy resin, Epidian 6, cured at room temperature with hardener Z-1, both produced by „Organika - Sarzyna” Chemical Plant S.A. (Poland) were used in this study as thermosetting polymeric matrix [25]. Two types of hard coal were used as a filler and electric properties modifier. The anthracite coal filler mined in Kuzbas of the Kuznetsk Basin and the second type of hard coal acquired from “Zofiówka” coalmine. Basic characteristics of component materials are shown in Tables 1-4.

Surface resistivity is a property of a material. Theoretically it should remain constant regardless of the method and configuration of the electrodes used for the surface resistivity measurement. A result of the surface resistivity measurement depends on both the material and the geometry of the electrodes used in the measurement. The physical unit of surface resistivity is Ohm (Ω). The legitimate unit of the surface resistance is also Ohm [22-23].

The aim of this research was to analyse dependence of the surface resistivity on two parameters, namely the filler content in the composite and the angular velocity in centrifugal casting. Statistical experimental design methodology was used to determine filler content and angular velocity levels. The rotatable central composite design was chosen [24-25].

At this stage of research fillers were tested without fractions separation and in the state the such as they were received from the supplier.

### Table 1. Physical and chemical properties of epoxy resin Epidian 6 [26]

<table>
<thead>
<tr>
<th>Form</th>
<th>Pale yellow high-viscosity liquid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Odour</td>
<td>weakly sensible</td>
</tr>
<tr>
<td>Value pH</td>
<td>about 7</td>
</tr>
<tr>
<td>Combustibility</td>
<td>combustible</td>
</tr>
<tr>
<td>Ignition point</td>
<td>&gt;200°C</td>
</tr>
<tr>
<td>Autoignition point</td>
<td>&gt;300°C</td>
</tr>
<tr>
<td>Boiling point</td>
<td>&gt;200°C</td>
</tr>
<tr>
<td>Density in 23°C</td>
<td>1.17 g/cm³</td>
</tr>
<tr>
<td>Viscosity in 25°C</td>
<td>10000-15000 MPa-s</td>
</tr>
</tbody>
</table>

### Table 2. Physical and chemical properties of curing agent Z-1 [26]

<table>
<thead>
<tr>
<th>Form</th>
<th>Pale yellow liquid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Odour</td>
<td>typical of triethylenetetramine</td>
</tr>
<tr>
<td>Value pH</td>
<td>about 7</td>
</tr>
<tr>
<td>Boiling point</td>
<td>277°C</td>
</tr>
<tr>
<td>Ignition point</td>
<td>122°C</td>
</tr>
<tr>
<td>Autoignition point</td>
<td>335°C</td>
</tr>
<tr>
<td>Density in 23°C</td>
<td>about 0.981 g/cm³</td>
</tr>
</tbody>
</table>

### Table 3. Basic characteristics of anthracite coal

| Carbon contents | 87.9 %     |
| Ash contents    | 3.37%      |
| Specific surface| 0.146 m²/g |
| Range of grain size* | 20.12 μm |
|                 | 132.64 μm  |
|                 | 358.36 μm  |
| Density         | 1.37 g/cm³ |

### Table 4. Basic characteristics of hard coal from “Zofiówka” coalmine

| Carbon contents | 88.5%     |
| Ash contents    | 16.78%    |
| Specific surface| 0.939 m²/g|
| Range of grain size* | 2.92 μm  |
|                 | 17.93 μm  |
|                 | 64.25 μm  |
| Density         | 1.40 g/cm³ |

*) Average values of particle size were determined with Mastersizer device of the Malvern company.


### 2.2. Samples preparation

The specimens were obtained using centrifugal casting methods which is one of the technologies relying on centrifugal force without applying pressure. In the first stage, the walls of cylindrical mould were covered with release agent. Next composition was prepared. Filler was added to epoxy resin and thoroughly mixed after that hardener was added and once more thoroughly mixed. After mould and composition preparation ready mixture was poured directly into the mould and mould was mounted on the device for casting. Next rotation around mould axis started. It was found that the time between mixing and pouring the composition into the mould should not be longer than 20 minutes. Outer surface of the specimen was shaped by the mould surface, while the inner surface of the specimen was shaped by the centrifugal force and gravity. Rotation was maintained until solidification of the material occurred. As a result of trials it was found that the best distribution of composition in the mould was achieved, if at the beginning of the casting rotational speed was small, which allows the initial distribution of material on the inner surface of the mould, and after about 1 minute the speed was increased to previously calculated. This procedure was necessary especially for higher filler contents (9, 12%), because the mixture was more viscous and difficult to evenly distribute inside the mould. When casting begun with high angular velocities, pronounced distortion of inner wall of the sample was observed. As a result of attempts to set minimum time of mould rotation it was set that two hours provided initial curing of the composition. Rotational speed of the mould allowing casting without visible influence of gravity force was in the range from 535.3 to 617.7 rpm. As a result of centrifugal casting cylindrical samples were produced. The procedure of specimens preparation is shown schematically in Fig. 2. Figure 3 shows an apparatus for samples casting. In Figure 4 an example of centrifugal cast sample with visible gradient of filler content is shown.

![Fig. 2. Scheme of specimen’s preparation](image)

![Fig. 3. Photograph of centrifugal casting device](image)

![Fig. 4. Specimen of gradient material (cured epoxy resin with hard coal filler) made by centrifugal casting method](image)

![Fig. 5. Geometric interpretation of applied experimental design](image)

Independent variables were set at five levels (coded by: -1.414; -1; 0; +1 and +1.414) (Table 5). Figure 5 show schematic geometric interpretation of the applied experimental design.

![Table 5. Coded and applied values of composite parameters for design of experiment](image)
The required number of experimental points is \( N = 2^4 + 4 + 3 = 11 \) (Table 6). There are four factorial experiments (2 factors on two levels, 2\(^2\)) with added 4 star points and centre point (average level) repeated 3 times to calculate the pure error. That experimental design enables to obtain the following second order polynomial model:

\[
y = a_0 + a_{12}(x_1)^2 + a_{22}(x_2)^2 + a_{12}(x_1)(x_2) + a_1(x_1) + a_2(x_2).
\]

Table 6.
Coded factors for the rotatable central composite design of experiments

<table>
<thead>
<tr>
<th>Test No</th>
<th>Coded factors</th>
<th>Volume fraction</th>
<th>Rot. velocity</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-1 -1</td>
<td>4.32</td>
<td>605.3</td>
</tr>
<tr>
<td>2</td>
<td>1 -1</td>
<td>10.68</td>
<td>547.7</td>
</tr>
<tr>
<td>3</td>
<td>-1 1</td>
<td>4.32</td>
<td>605.3</td>
</tr>
<tr>
<td>4</td>
<td>1 1</td>
<td>10.68</td>
<td>547.3</td>
</tr>
<tr>
<td>5</td>
<td>-(\lambda) 0</td>
<td>3</td>
<td>576.5</td>
</tr>
<tr>
<td>6</td>
<td>(\lambda) 0</td>
<td>12</td>
<td>576.5</td>
</tr>
<tr>
<td>7</td>
<td>0 -(\lambda)</td>
<td>7.7</td>
<td>535.3</td>
</tr>
<tr>
<td>8</td>
<td>0 (\lambda)</td>
<td>7.5</td>
<td>617.7</td>
</tr>
<tr>
<td>9</td>
<td>0 0</td>
<td>7.5</td>
<td>576.5</td>
</tr>
<tr>
<td>10</td>
<td>0 0</td>
<td>7.5</td>
<td>576.5</td>
</tr>
<tr>
<td>11</td>
<td>0 0</td>
<td>7.5</td>
<td>576.5</td>
</tr>
</tbody>
</table>

Table 7 shows the volumes of all ingredients together with hardener for all volume fractions resulted from experimental design.

Table 7.
Filler content in prepared mixtures

<table>
<thead>
<tr>
<th>Filler content in the composite, % vol.</th>
<th>Components of the mixture, cm(^3)</th>
<th>Total volume of the sample, cm(^3)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Resin</td>
<td>Hardener</td>
</tr>
<tr>
<td>3</td>
<td>290.02</td>
<td>44.97</td>
</tr>
<tr>
<td>4.32</td>
<td>285.79</td>
<td>44.31</td>
</tr>
<tr>
<td>7.5</td>
<td>276.56</td>
<td>42.88</td>
</tr>
<tr>
<td>10.68</td>
<td>266.50</td>
<td>41.32</td>
</tr>
<tr>
<td>12</td>
<td>263.11</td>
<td>40.79</td>
</tr>
</tbody>
</table>

2.3. Measurements methodology

Surface resistivity was tested as a measure of electrical properties. Measurements were performed according to Polish standard PN-88/E-04405. Resistance Tester type Teraohm 5 kV of the Metrel company was used. The idea of the test was to define electrical resistivity in sequential layers with different content of conductive component (hard coal). First, specimens thickness was measured and surface resistivity tested. Next outer layer of about 0.1 mm thickness was removed by turning and surface resistance measured. Next, successive layer removements and resistivity measurements were performed. This procedure was repeated four times to achieve dependence of surface resistivity on the depth of layer. Apart from the first layer the thickness of removed layers was 0.05 mm. In the case of samples with hard coal from the "Zofiańska" coalmine removing the fourth layer of a material gave the resistance above measuring range of the resistance meter (5E+12 \(\Omega\)). Scheme of the system to surface resistance measurement is presented in Figure 6.

![Fig. 6. System to surface resistance measurement: 1 - resistance tester, 2 - measurement electrodes, 3 - specimen](image)

For the measurements of surface resistance three-electrodes system was used shown in Figure 7. The voltage was supplied to the electrode (2) and (3), while the electrode (4) functioned as protective electrode and was grounded. Measuring electrodes were cut from aluminium foil in strips form with width about 10 mm and a thickness of 10 \(\mu\)m. Protective electrode was made of cardboard tubes wrapped in aluminium foil. Electrodes were stuck to cleaned smooth surface of samples using a clean paraffin oil to provide precise electrodes adherence to tested surface.

![Fig. 7. Scheme of electrodes system to surface resistance measurement: 1 - voltage source and voltage measurement circuit, 2 - first measurement electrode, 3 - second measurement electrode, 4 - protective electrode, g - gap width, D - specimen diameter](image)

Parameters of the system for measurement of the surface resistance are presented in Table 8.

Table 8.
Parameters of surface resistance measurement

| Electric source voltage - \(U_s\) | 5000 [V] |
| Resistance measurement time - \(t\) | 60 [s] |
| Distance between the electrodes - \(g\) | 5 [mm] |
3. Results and their analysis

3.1. Surface resistivity

Based on the measured surface resistance values surface resistivity was calculated using appropriate formulas given in the Polish Standard PN-88/E-04405. Results of the surface resistance calculations are shown in Tables 9 and 10.

The hard coal fillers with specified concentration changed favourable surface resistivity on outer layers to the level allowing to avoid static electricity accumulation. On the outer layer of specimens anthracite coal filled (two layer) obtained electrical resistivity the order of $1 \times 10^9 \Omega$. According to the guideline which is related to avoiding the danger of ignition due to electrostatic charge to the material surface resistivity between ability to load up is weakened, while the values below $1 \times 10^9 - 1 \times 10^11 \Omega$ considered the material is no longer able to charge up.

The resistivity of composites with hard coal filler changed dramatically upon the addition of only 3% by volume of filler. The resistivity values decreased further with increasing volume concentration of filler. The observed decrease was more pronounced for the specimen with anthracite filler.

On the basis of obtained results dependences of surface resistivity on initial filler content and rotational velocity were determined in polynomial form. Mean values calculated from six measurements are taken into consideration. Obtained dependences are presented in 3D surface graphs. Surface plots were made using the STATISTICA software. Figures 8-11 show the dependences of surface resistivity on filler content and casting velocity for composites filled with anthracite coal. and Figures 12 to 15 show the same dependences for composites filled with hard coal from “Zofiówka” coalmine. The resistance results for the first not machined surface (Fig. 8) were slightly higher than for the next tested layer. Pure geometrical consideration show that filler particles concentration in layer adjacent to mould wall must be lower than that for deeper layers. It is the explanation for this phenomena. Subsequent layers of material were machined by turning. Research carried out on specimens containing anthracite coal (Figures 9-11) as a filler showed that both increasing the initial filler content as well as the increasing casting velocity caused decrease of the surface resistivity. It is in accordance with expected higher concentration of conductive filler together with increase of these variables. The introduction of highest percentage of filler (12%) resulted in a significant increase in viscosity of the mixture which has hindered the filler sedimentation during centrifugation. Therefore for 12%vol. an increase of resistance in relation to the 10.68% vol. content was observed.

Table 9.
Surface resistivity versus volume percentage of anthracite coal and casting rotational velocity

<table>
<thead>
<tr>
<th>Filler content, % vol.</th>
<th>Rotational velocity, rpm</th>
<th>Surface resistivity, $\Omega$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1 layer</td>
<td>II layer</td>
</tr>
<tr>
<td>3</td>
<td>576.5</td>
<td>4.85E+10</td>
</tr>
<tr>
<td>4.32</td>
<td>547.7</td>
<td>2.08E+10</td>
</tr>
<tr>
<td>7.5</td>
<td>605.3</td>
<td>2.30E+09</td>
</tr>
<tr>
<td>7</td>
<td>535.3</td>
<td>9.18E+09</td>
</tr>
<tr>
<td>7.5</td>
<td>576.5</td>
<td>1.47E+09</td>
</tr>
<tr>
<td>7</td>
<td>576.5</td>
<td>1.67E+09</td>
</tr>
<tr>
<td>7.5</td>
<td>576.5</td>
<td>1.58E+09</td>
</tr>
<tr>
<td>7</td>
<td>617.7</td>
<td>1.15E+09</td>
</tr>
<tr>
<td>10.68</td>
<td>547.7</td>
<td>1.47E+09</td>
</tr>
<tr>
<td>10.68</td>
<td>605.3</td>
<td>1.34E+09</td>
</tr>
<tr>
<td>12</td>
<td>576.5</td>
<td>4.99E+09</td>
</tr>
</tbody>
</table>

Table 10.
Surface resistivity versus volume percentage of hard coal with “Zofiówka” coalmine and casting velocity

<table>
<thead>
<tr>
<th>Filler content, % vol.</th>
<th>Rotational velocity, rpm</th>
<th>Surface resistivity, $\Omega$ - average</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>I</td>
<td>II</td>
</tr>
<tr>
<td>3</td>
<td>576.5</td>
<td>6.82E+09</td>
</tr>
<tr>
<td>4.32</td>
<td>547.7</td>
<td>5.29E+09</td>
</tr>
<tr>
<td>4.32</td>
<td>605.3</td>
<td>3.24E+10</td>
</tr>
<tr>
<td>7.5</td>
<td>535.3</td>
<td>6.77E+09</td>
</tr>
<tr>
<td>7.5</td>
<td>576.5</td>
<td>3.79E+10</td>
</tr>
<tr>
<td>7</td>
<td>576.5</td>
<td>3.31E+10</td>
</tr>
<tr>
<td>7.5</td>
<td>617.7</td>
<td>2.91E+10</td>
</tr>
<tr>
<td>7.5</td>
<td>617.7</td>
<td>2.83E+10</td>
</tr>
<tr>
<td>10.68</td>
<td>547.7</td>
<td>3.65E+10</td>
</tr>
<tr>
<td>10.68</td>
<td>605.3</td>
<td>4.44E+10</td>
</tr>
<tr>
<td>12</td>
<td>576.5</td>
<td>3.54E+11</td>
</tr>
</tbody>
</table>
It is in accordance with Stokes law describing particles movement in viscous media due to external forces action. The higher is viscosity and the smaller are particles the lower is sedimentation velocity.

On the basis of obtained results dependences of surface resistivity on initial filler content and rotational velocity were determined in polynomial form. Mean values calculated from six measurements are taken into consideration. Obtained dependences are presented in 3D surface graphs. Surface plots were made using the STATISTICA software. Figures 8-11 show the dependences of surface resistivity on filler content and casting velocity for composites filled with anthracite coal. and Figures 12 to 15 show the same dependences for composites filled with hard coal from “Zofiówka” coalmine. The resistance results for the first not machined surface (Fig. 8) were slightly higher than for the next tested layer. Pure geometrical consideration show that filler particles concentration in layer adjacent to mould wall must be lower than that for deeper layers. It is the explanation for this phenomena. Subsequent layers of material were machined by turning. Research carried out on specimens containing anthracite coal (Figures 9-11) as a filler showed that both increasing the initial filler content as well as the increasing casting velocity caused decrease of the surface resistivity. It is in accordance with expected higher concentration of conductive filler together with increase of these variables. The introduction of highest percentage of filler (12%) resulted in a significant increase in viscosity of the mixture which has hindered the filler sedimentation during centrifugation. Therefore for 12%vol. an increase of resistance in relation to the 10.68% vol. content was observed. It is in accordance with Stokes law describing particles movement in viscous media due to external forces action. The higher is viscosity and the smaller are particles the lower is sedimentation velocity.

Under each graph is given the equation of second-degree polynomial functions specified for each test layer. In this equation the variable $x$ represents filler content and the variable $y$ - casting rotational velocity.

Surface resistivity $[\Omega] = 1.8003E11+1.6706E8*x-4.7301E10*y-5.8314E5*x*x+5.0145E7*x*y+1.0117E9*y*y$

Fig. 8. A graph showing dependence of surface resistivity on filler content and casting rotational velocity for the first test layer of the specimens filled with anthracite coal

Surface resistivity $[\Omega] = 2.2681E14-7.742E11*x+4.7635E11*y$

Fig. 9. A graph showing dependence of surface resistivity on filler content and casting rotational velocity for the second test layer of the specimens filled with anthracite coal

Surface resistivity $[\Omega] = 1.0223E12-2.198E9*x-7.3693E10*y+1.2743E6*x*x+5.8171E7*x*y-2.199E9*y*y$

Fig. 10. A graph presenting dependence of surface resistivity on filler content and casting rotational velocity for the third test layer of the specimens filled with anthracite coal

Surface resistivity $[\Omega] = 9.7422E13-2.054E11*x-5.5673E12*y+6.7836E8*x*x-2.9605E9*x*y+6.043E10*y*y$

Fig. 11. A graph presenting dependence of surface resistivity on filler content and casting rotational velocity for the fourth test layer of the specimens filled with anthracite coal
Considerably higher surface resistivity results were obtained for specimens containing the second type of filler (Figures 12-15). Hard coal from the “Zofiówka” coalmine is characterized by a greater size reduction of grains, what results in slower sedimentation of the filler during centrifugation. Mixture containing 12% vol. of filler had very high viscosity what made movement of filler grain towards the wall of the mould difficult (Fig. 12). The measurement results of the second layer show that as well as for anthracite coal, the greatest surface resistivity exhibit the specimens including the lowest content of filler (3%) and cast at the lowest velocity (Fig. 13). After removal of the third layer the resistance grew rapidly, reaching the order of 5E+12 Ω and for the lowest filler content (3%) reached a maximum value of the upper range of the meter equal to 1.3 E+13 Ω (Fig. 14).

By changing the particle size distribution of coal, by changing the proportions of the various fractions, it is possible to control concentrations of coal in the different layers of material. The grain size composition of used powders is not precisely known. Therefore, it is planned to carry out research for a mixture of narrow, well-defined grain fractions.

![Fig. 12. A graph showing dependencies surface resistivity on filler content and casting velocity for the first test layer of the specimens filled with hard coal from “Zofiówka” coalmine](image)

![Fig. 13. A graph showing dependencies surface resistivity on filler content and casting velocity for the second test layer of the specimens filled with hard coal from “Zofiówka” coalmine](image)

**4. Conclusions**

This study was carried out in order to evaluate the electrical properties of the gradient composites. On the basis of obtained results the following conclusions were drawn:

1. The hard coal fillers with specified concentration changed favourable surface resistivity on outer layers to the level allowing to avoid static electricity accumulation.
2. Lower values of surface resistivity were obtained for specimens filled with anthracite coal.
3. Bigger coal particles moved quicker towards mould wall what resulted in better packing of the filler on outer layers of the specimen.
4. Research have shown that the optimum content of filler at which it is possible to reach the lowest resistivity values is about 11%.
5. A continuation of research on polymer gradient composites filled with anthracite coal is planned. Research is also planned for mixtures of specific grain fractions.
Acknowledgements

The authors would like to thank Mrs. K. Dahlke for help in preparing specimens and her help during experiments.

References