FRACTOGRAPHIC EVALUATION OF PM MICRO-COMPOSITE MATERIALS

R. Bureš, M. Fáberová, I. Saxl

Abstract
Micro-composite materials based on iron powder with the addition of an electric insulator are generally used in electrotechnics and electronics. Inorganic and polymer coats are used for insulation. The paper focuses on evaluation of failure of micro-composite materials. Powder iron with an addition of thermosetting phenol-formaldehyde resin was compacted using powder metallurgy technology. A method for qualitative evaluation of fracture was proposed based on image analysis techniques and statistical processing of results. The fracture surface is cut by virtual cuts parallel with the fracture line. Parameters of the cuts are used to obtain information about changes in fracture surface characteristics and to compare fracture surfaces of materials produced by using different technological parameters.

Keywords: iron powder, thermosetting resin, statistics, image analysis

INTRODUCTION
Micro-composite materials with specific physical properties, intended for use in the electrotechnics industry and electronics, constitute a dynamically developing field of material research. Micro-composite materials, based on ferromagnetic powder electrically insulated with a thin polymer layer or inorganic insulator, are produced by powder metallurgy technologies [1-8]. Along with the development of new material systems and production technologies, one can observe progress in the evaluation of properties of micro and nano-composite materials [9-12]. An inseparable part of the evaluation of mechanical properties of materials is their fractographic analysis.

The present study analyses the possibilities of fracture surfaces evaluation of Fe – polymer micro-composite materials prepared by powder metallurgy, using image analysis and statistical methods [13].

EXPERIMENTAL MATERIAL AND METHODS
Experimental material consisted of commercial thermosetting phenol-formaldehyde resin with mineral filler (ATM Austria) to which powder iron ASC100.29 (Höganäs AB Sweden) was added. The phenolic resin powder was adjusted by mechanical milling using a knife mill. A powder mixture containing 50 vol.% resin and 50 vol.% powder iron was prepared by mechanical mixing in a Turbula homogenizer. The micro-composite material was prepared by compacting the powder mixture with a simultaneous application of pressure and temperature. The material was compacted in a Simplimet 3000 press using pressure 30 MPa, temperatures 120, 150 a 180°C and compaction times 3, 5, 10, 15 and 20 min. The obtained semi-finished products with circular cross-section were cut...
into rectangular-shaped specimens of the size 4x5x20 mm. They were subjected to the 3-point bending strength test according to ISO 3325 by testing machine ZD 10/90. Hardness was evaluated by Vickers. Fracture surfaces were observed using a scanning electron microscope JEOL 7000F.

The fracture images were processed and analysed by ImageJ software [14]. To ensure the best use of image information and achieve maximum contrast of displayed fracture characteristics, a bandpass filter using a Fourier transform was applied. The original picture in Fig.1 shows a characteristic narrow histogram which was changed after filtration to the entire range of gray scale as is shown in Fig.2. The filter corrects the shades of gray colour of "large objects" in the image and, at the same time, suppresses "small objects". The image profile is used as a characteristic of fracture image „ruggedness“. The profile is represented by pixel intensities on line selection. If planar selection is used instead of line selection (rectangular window e.g.) then pixel intensity in the X direction is calculated as mean of pixel intensity in Y direction. Pixel intensity is non-calibrated in regard to the nature of the image from SEM and it is a numerical parameter without physical mean. The image was filtrated by edge detector Sobel filter and thresholded to binary image for acquiring additional information about the character of fracture, Fig.3. Area fraction of edges to overall area of image was measured from inversion image.

Fig.1. SEM image of fracture surface of a micro-composite compacted at 150ºC for 5 min, histogram characterising distribution of shades of gray in the image.

Fig.2. Image of fracture from Fig.1 after application of a fast Fourier transform – bandpass filter, histogram characterising distribution of shades of gray in the image.
RESULTS OF THE EXPERIMENT AND DISCUSSION

The measured values of hardness and 3-point bending strength are summarised in Tab.1. The mechanical properties reflect changes in the technological history of the material. The basic process which takes place during compacting of the prepared micro-composite is hardening of the thermosetting resin by the simultaneous action of temperature and pressure. Hardening is accompanied by increasing density of the powder system, decreasing porosity and mechanical linking of resin-coated iron particles. According to Tab.1, affects temperature essentially micro-composite strength. Less pronounced but not negligible was the relation between the time of compacting and temperature. A temperature of 120ºC and compaction time up to 5 min was insufficient to ensure complete hardening of the thermosetting resin. A temperature of 180ºC, particularly in combination with long compaction time, ensured complete hardening of the resin. Thermosetting resin in such a form is most stable and exhibits the highest strength and hardness. High hardness gives rise to brittle behaviour, as indicated by the development of cracks during measurement of HV10 hardness.

Tab.1. Values of hardness and bending strength in dependence on technological parameters of micro-composite preparation.

<table>
<thead>
<tr>
<th>Compaction temperature [ºC]</th>
<th>Compaction time [min]</th>
<th>Hardness HV10</th>
<th>TRS [MPa]</th>
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<tbody>
<tr>
<td>120</td>
<td>3</td>
<td>50.5</td>
<td>9.1</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>50.8</td>
<td>10.1</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>61.4</td>
<td>18.2</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>63.2</td>
<td>19.6</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>64.5</td>
<td>19.2</td>
</tr>
<tr>
<td>150</td>
<td>3</td>
<td>61.9</td>
<td>23.6</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>62</td>
<td>25.0</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>61.9</td>
<td>23.6</td>
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<tr>
<td></td>
<td>15</td>
<td>62.8</td>
<td>30.4</td>
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<tr>
<td></td>
<td>20</td>
<td>63.6</td>
<td>29.7</td>
</tr>
<tr>
<td>180</td>
<td>3</td>
<td>63.8</td>
<td>34.1</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>64.4</td>
<td>35.1</td>
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<tr>
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<td>10</td>
<td>65.8</td>
<td>33.1</td>
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<tr>
<td></td>
<td>15</td>
<td>70.6</td>
<td>33.4</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>73.6</td>
<td>37.1</td>
</tr>
</tbody>
</table>
The mechanical properties described are inevitably reflected in the character of the micro-composite fracture as observed by examination of fractures by SEM. Fractographic characteristics of fracture are discussed in the paper [15]. According to our objective to evaluate statistically the influence of compaction factors on fracture characteristics, we measured intensity profiles (shades of gray) of fracture surface images.

Fig. 4. Profiles of image in dependence on window size [pixel] of selection.

The first step was to optimise selection of the statistical set of data. The basic parameter of the data set, representing the fracture surface, was magnification of SEM images which was chosen on the basis of comparison of the total fracture area, real area of one image and size of characteristic formations in the fracture image. The magnification selected was used for analysis of all fractures.
Size of selection window used for analysis of the pixel intensity profile was chosen based on comparing statistical parameters of the selections in dependence on size of the sampling window. Influence of selection size on intensity profile is shown in Fig.4. Value of intensity is calculated as the mean of a large number of intensities choosing a large sampling window. A large sampling window provides global, but not a specific enough view on the changes of the fracture surface. The result of measurement is strongly influenced by the random location of the sampling window in image in the case of a small selection (line selection with 1 pixel thickness of line e.g.). This would be necessary to compensate by a large number of measurements in various parts of image. Selections of window size 1000 pixels of width and 5, 10 and 20 pixels of length were used for analysis, based on comparison of selections with window 1000, 600, 200, 100, 50, 20, 10, 5 and 1 pixel of width as shown in Fig.5.

About 100 images of fracture surfaces were measured. The statistical data file indicates that the used method detected some differences in nature of fracture. This method was not sensitive enough to detect smaller differences. For example in the framework of one curing temperature it was impossible to quantify differences between curing time 3, 5 and 10 minutes.

Moreover, random selection was replaced by selection based on known parameters of technology of sample preparing. The goal was to find out the sensitivity of methods in relation to differences between fracture surfaces of materials prepared using essentially modified compaction technologies. Temperatures 120, 150 and 180ºC and curing times 3 and 20 minutes were selected for observation.
Five window selection 5x1000 pixel sizes were used as virtual cuts of fracture surface in parallel direction with the fracture line. Window selection was located systematically in the same part of the image. Profile of intensity was measured for each sample window. A box diagram was chosen for presentation of statistical parameters of profile intensity. The box represents interquartile range (difference between 25% and 75% quantile), the line in the box stands for median value, a point in the box is an arithmetical mean, × points lies out of interval 1% to 99% quantile, I marked of scale of non-outlying values, _ marked of minimum and maximum. The result of analysis of the defined
statistical selection is shown in Fig.6. Each box in the diagram represents measurement of profile intensity of one sample window 5x1000 pixel size. 5 measurements were realised on every image. Sample window c1–c5 was located in image in an direction from the fracture line to the end of fracture. Differences between statistical selections in the frame of one fracture (one technology of preparing of micro-composite) were not statistically significant.

Comparison of diagrams (technologies) does not allow one to draw unambiguous conclusions regarding differences in fracture characteristics in relation to the technological parameters of micro-composite preparation.

The mean of 5 measurements of selections c1–c5 of each image of fracture was evaluated, considering the statistical non-significant differences in measurements in the framework of one fracture. The results in Fig.7. indicate a tendency to differentiation of statistical parameters of fracture images in dependence on the technology of material preparation. Plastic fracture of the micro-composite compacted at 150ºC for 3 min is manifested by a narrow interval of pixel intensities in the profile and thus by the relatively narrowest box representing the quantile interval and, at the same time, a smaller range of minimal and maximal values. Contrary to that, brittle cleavage failure identified in the micro-composite fracture after compaction at 180ºC for 20 minutes was manifested as a wide quantile interval with a wide range of min-max values. Dark cracks and light, relatively smooth surfaces of embrittled thermosetting resin in the image are projected in a smaller range of shades of gray in comparison with the image of elastic failure. Regarding the image of fracture characteristic of elastic failure, one can observe bigger range of the shades of gray and a higher frequency of their alternation as a manifestation of articulation of surface formations after elastic deformation before the failure. Failure of an insufficiently compacted micro-composite, hardened at 120ºC, exhibits typical decohesion of thermosetting resin and iron particles and disintegration of the insufficiently hardened resin. Characteristics of the image of such failure resemble more the elastic failure and differ by shift of intensities towards lower values which are characteristic of brittle failure.

![Fig.7. Statistical characteristics of profiles of intensity in dependence on used preparing technology of micro-composite, temperature [ºC]/time [minutes], calculated as mean of measurements of window selections c1-c5 from Fig.6.](image-url)
Results obtained by the described method of measurement of intensity profiles point to the well known fact that development of fractures is a highly stochastic process. Any fracture characteristic is affected by this randomness. Despite that, by affecting the selection, for example by arranging data into groups with anticipated (e.g. technologically) similar parameters of the statistic data set, one can obtain information about fracture characteristics. The obtained information can serve as a quantifier of verbal description of fracture characteristics, for example when comparing modifications of technological parameters for the preparation of micro-composite materials. Contrary to that, the simple parameter of the type of area fraction of edges in Tab.2. is so affected by the stochastic character of the image that it is not usable for this purpose.

Tab.2. Ratio of area proportion of edges [%] and total area of image of fracture surface in relation to technological parameters of micro-composite preparation.

<table>
<thead>
<tr>
<th></th>
<th>120°C</th>
<th>150°C</th>
<th>180°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>3 min</td>
<td>19.63</td>
<td>26.11</td>
<td>23.22</td>
</tr>
<tr>
<td>20 min</td>
<td>23.46</td>
<td>22.86</td>
<td>22.10</td>
</tr>
</tbody>
</table>

The method of fracture evaluation of the described micro-composite material appears prospective, provided that its sensitivity to fracture characteristics can be increased. The sensitivity may be increased through searching for ways of suppression of the stochastic character of initiation of fractures in the areas where this approach is feasible. As an example we can mention changes in preparation of specimens for a 3-point bending test so that mechanical machining of the surface can be eliminated. A press tool for production of test specimens of the required shape and dimensions was manufactured. We managed to suppress the random location of fractures due to micro-notches after machining the specimen as witnessed by increased values of TRS without changes in HV10, for example at 120ºC/3min TRS = 16.2 MPa (49 HV10) compared to TRS = 9.11 MPa (50.5 HV10) by mechanical machining of the produced specimen.

CONCLUSION

The aim of the present study was to analyse the possibility of evaluation of the fracture surface of micro-composite materials Fe – polymer, prepared by powder metallurgy using statistical methods.

The method developed, based on images of fracture surfaces obtained by electron microscopy and subsequently processed by image analyser and subjected to statistical analysis of data appears prospective for obtaining a quantifier capable of describing fracture characteristics. Sensitivity of the quantifier is affected essentially by the stochastic character of the process of fracture initiation. Sensitivity of the method may be increased through suppression of the random character of influenceable conditions of initiation of fracture and its image. Success of the method can be increased by its application to a suitably selected statistical set of data.

Acknowledgement

The study was carried out within the work on projects VEGA 2/0149/09 and APVV-0490-07 MICOMAT.

REFERENCES