FRACTURE CHARACTERISTICS OF THE COMPOSITE MATERIAL BASED ON Fe – THERMOSETTING RESIN PREPARED BY POWDER METALLURGY

M. Fáberová, R. Bureš, E. Dudrová

Abstract
Powder metallurgy technologies are used in many fields of production and the metal/plastic combination opens a wide range of possibilities for preparation techniques. PM technologies enable one to prepare composite materials tailored to a particular purpose and thus ensure the superior flexibility of properties required for respective applications. This is true particularly in the case of soft magnetic microcomposite materials in which the ferromagnetic component of relevant size, composition and microstructure is electrically insulated by coating based on polymers. The ferromagnetic component consists typically of iron powder particles of the required purity, size and structure. The paper focuses on materials based on iron powder with the addition of thermosetting resin, exhibiting specific insulation properties. To reach the required properties of these materials, it is necessary to describe accurately the influence of temperature and hardening time on properties of bakelite and subsequently on properties of the composite material.

Keywords: iron powder, polymer coating, bakelite, fracture surface

INTRODUCTION
Powder metallurgy (PM) is a procedure of preparation of materials and shaped products based on the heat-activated process of binding individual particles of metal powders but also metals and non-metals, including ceramics, chemical compounds, graphite and various types of polymers. By targeted redistribution of powder particles of varying chemical character, size and shape before sintering, one can attain the required arrangement of structural components and obtain material with predetermined properties. This capability predestines PM for the production of various composite materials. This is motivated by the unique manner of production of materials with specific properties that cannot be prepared by any other technology. Composite materials can be tailored to a specific purpose and thus ensure superior flexibility of properties required for respective applications. Density and homogeneity of composite materials are very important factors governing the final properties. Many chemical concepts are available and various methods of compacting. Their use depends on the chemical character of the composite and its required properties [1,2].

One of the lucrative fields of research of composite materials involves applications for the electrotechnical industry. It focuses particularly on microcomposite, magnetically soft materials in which the ferromagnetic component of relevant size composition and structure is insulated by an electro-insulation coat based on polymers. The ferromagnetic component typically consists of particles of iron powder of sufficient purity, size and

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required structure. The electric-insulation component is usually a thermosetting or thermoplastic material, ceramics and such [3]. The produced composite powder is compacted by various methods, such as hot pressing, HIP, PIM, etc. [4,5,6].

The paper investigates properties of composite Fe – thermosetting resin, prepared by mixing and subsequent shaping under the action of temperature and pressure. The aim of the study was to observe the influence of hardening temperature on properties of bakelite, and subsequently on properties of the respective composite material.

**EXPERIMENTAL MATERIAL AND METHODS**

The experiments were carried out using commercial, water-sprayed iron powder ASC 100.29, Höganäs AB, with natural granulometric composition and a commercial thermosetting phenolic resin with mineral filler ( „bakelite“ thereafter). Bakelite was adjusted by high-energy milling to very fine powder with particle size under 40 μm. The mixture of iron powder and ground bakelite at a ratio 1:1 (vol.%) was homogenised by mixing in a homogeniser Turbula. Cylindrical specimens of diameter 25 mm and height 3 mm were prepared by a Simplimet 3000 press at elevated temperatures. The specimens were then compacted under pressure of 30 MPa at 120°C and compaction time 3 min, at 150°C and compaction time 5 min and at 180°C and compaction time 15 min. After compaction the specimens were cooled in a machine work chamber for 10 min. In this way prepared cylinders were used to prepare prism-shaped specimens of the size 4x5x20 mm³. Bending strength of specimens (ISO 3325) was tested employing test machine ZD 10/90 with a loading rate 0.5 mm/min. The test was conducted at room temperature. Then we analysed fracture surface (SEM, JEOL 7000F) with the aim to identify changes in bakelite and in its distribution in relation to conditions of compaction. HV10 hardness was measured on the surface of specimens. Microstructure and fracture profiles were analysed by a microscope Olympus GX 71 and fracture surfaces were examined by a scanning electron microscope JEOL 7000F.

**RESULTS OF THE EXPERIMENT AND DISCUSSION**

Values of bending strength and HV10 hardness in dependence on compaction conditions are presented in Table 1. According to processing conditions the hardness increased from the value of 50 HV10 for compaction at 120°C /3 min, to the value of 62 HV10 at 150°C /5 min and finally to 71 HV10 at 180°C /15 min. Increasing hardness was accompanied by a rise in bending strength, namely from 9 MPa for compaction at 120°C /3 min to 25 MPa at 150°C /5 min and finally to 33.4 MPa at 180°C /15 min. For the given material systems the measured values of hardness (apparent) depend on the hardness of the metal component, hardness of bakelite and porosity. Elastic deformation is a specific property of bakelite, therefore “cushioning” by bakelite may affect the measured values of hardness. Cushioning may result in smaller dimensions of indentation which can be interpreted as higher hardness. Decreased volume of pores at a higher temperature of compaction may be reflected in the value of apparent hardness which can be measured as higher hardness of the composite material.

<table>
<thead>
<tr>
<th>Temperature [°C]</th>
<th>Compaction time [min]</th>
<th>HV 10</th>
<th>TRS [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>120</td>
<td>3</td>
<td>50.5</td>
<td>9.1</td>
</tr>
<tr>
<td>150</td>
<td>5</td>
<td>62</td>
<td>25</td>
</tr>
<tr>
<td>180C</td>
<td>15</td>
<td>70.6</td>
<td>33.4</td>
</tr>
</tbody>
</table>
Macroscopic appearance of the fracture

Figure 1 shows the macroscopic appearance of fracture surface of specimens after the 3-point bending test. On a macroscopic scale, the fracture surface of all analysed states was flat with little articulation. Morphology suggests development of agglomerates of a size up to approx. 500 μm. The agglomerates are separated by more or less connected pores which resemble a network.

![Macroscopy of the fracture surface of specimens, SEM.](image1.png)

Propagation of cracks

Trajectory of failure on the surface of specimens showed initiation and propagation of cracks at the metal/bakelite interface, more accurately along the interfaces of agglomerates produced during compaction, Fig.2.
Fig. 2. Trajectory of fracture on the surface, OM.

Microfractography of fracture surface

Fig. 3. Fracture surface, SEM.

The fracture surface exhibits considerable articulation, and according to its composition it has a complex character, Fig. 3. It consists of free surfaces of Fe particles.
which are in some cases covered by a bakelite layer, Fig.4. Fe particles are surrounded by zones of bakelite composed of fine and coarser, more or less connected particles. This indicates that the temperature and compaction time used were not sufficient to ensure complete hardening of the entire volume of bakelite. The appearance of the specimen surface suggests that the degree of hardening was higher on the surface than in the bulk. Failure of specimens during bending strength test occurred at interfaces of Fe particles or Fe particles and bakelite. The strength of bonds/connections between Fe particles or Fe particles and bakelite was low.

![Fig.4. Particle enveloped by bakelite, SEM.](image)

System 150ºC/5 min

![Fig.5. Fracture surface, SEM.](image)

Figure 5 shows fracture surface with a similar articulation as that obtained at 120ºC /3 min, it has a complex character, and just as the one obtained at 120ºC /3 min it contains free surfaces of Fe particles visibly covered by bakelite, Fig.6. The layer of bakelite smoothens the surface of particles but does not overlap the original surface micro-morphology. Fe particles are surrounded by bakelite zones, the morphology of which differs slightly from the state produced at 120ºC /3 min, particularly it does not contain very fine particles and is more continuous. In some places it acts as a connecting material of Fe particles. Hardness of the specimen indicates that the process of bakelite hardening was more complex than that at 120ºC /3 min. Increased hardness of bakelite and better enveloping of Fe particles by bakelite, eventually the joining of Fe particles by means of
bakelite, results in a higher than twofold increase in bending strength compared to the value obtained at 120ºC /3 min.

Fig.6. Detail of fracture surface, SEM.

System 180ºC/15 min

Fig.7. Fracture surface, SEM.

Fig.8. Detail of fracture surface after cracking and cleavage failure.
The fracture surface of this system also has a complex character, Fig.7. On this fracture surface one can identify original surfaces of Fe particles covered with a bakelite layer. Fe particles are surrounded by more or less continuous bakelite zones. It is obvious that during the hardening process bakelite envelops Fe particles and, in some cases, the bakelite surface even copies the surface of Fe particles. The bending strength test resulted in cracking failure of bakelite which acquired the character of brittle cleavage with numerous secondary cracks, Fig.8. The relatively marked increase in hardness and bending strength confirms that bakelite was hardened to a “glassy” state.

CONCLUSION

The study presents information about the influence of hardening temperature on properties of bakelite and subsequently properties of the composite material based on Fe powder and thermosetting resin. The results obtained allowed us to state the following:

- At the temperature of 120°C /3 min the strength of bonds/connections between Fe particles or Fe particles and bakelite was low,
- At the temperature of 150°C /5 min the specimen hardness indicated that the process of bakelite hardening was more complex than that at 120°C /3 min. Increased hardness of bakelite and better envelopment of Fe particles with bakelite, eventually the joining of Fe particles by means of bakelite resulted in a higher than twofold increase in bending strength in comparison with the value reached at 120°C /3 min.
- At a temperature of 180°C /15 min, bakelite enveloped the Fe particles during its hardening and in some cases the bakelite surface even copied the surface of Fe particles. During the bending strength test we observed cracking failure of bakelite that acquired the character of brittle cleavage with numerous secondary cracks and a relatively marked increase in hardness and bending strength indicated that bakelite was hardened to a “glassy” state.

The results obtained enable one to predict temperature and time of hardening for various fields of utilization of the examined experimental materials.

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REFERENCES