Electric properties of Fe based soft magnetic composite.

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Abstract
Soft magnetic composites based on Fe powder with insulation coating were prepared. Uniaxial pressing and heat treatment was used. Changes of impedance characteristic in the frequency range 100 Hz – 10 MHz in dependence on content of insulation up to 30% and in dependence on compression pressure from 200 to 800 MPa was investigated. Distribution of insulation and porosity in composite was evaluated and quantified using image analysis. Correlation between microstructure characteristics, compaction parameters and electric properties were analyzed. An AC impedance spectroscopy technique was employed to investigate the effect of porosity and amount of resin insulator on specific electrical resistance of composite samples. It was shown that increasing of compaction pressure results in decreasing the specific resistance. An addition of resin in the range from 5 % to 25% causes an increase of the measured resistance from 1.2E-2 ohm.m to 6E+4 ohm.m for compression pressure of 400 MPa.

Keywords: SMC, resistance, impedance spectroscopy, microstructure, porosity

Results

Compressibility

Compressibility curves of composites

Image Analysis

Statistical evaluation of resin and pores area fraction according to the placement in sample.

<table>
<thead>
<tr>
<th>Sort of sample</th>
<th>Compressibility</th>
<th>R1</th>
<th>R2</th>
<th>%influenced</th>
<th>Pore + resin</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe/10ATM</td>
<td>0.88</td>
<td>1.07</td>
<td>10%</td>
<td>19.9</td>
<td>32.4</td>
</tr>
<tr>
<td>Fe/15ATM</td>
<td>0.95</td>
<td>1.21</td>
<td>15%</td>
<td>19.9</td>
<td>32.4</td>
</tr>
<tr>
<td>Fe/20ATM</td>
<td>0.97</td>
<td>1.52</td>
<td>20%</td>
<td>19.9</td>
<td>32.4</td>
</tr>
<tr>
<td>Fe/30ATM</td>
<td>0.94</td>
<td>1.86</td>
<td>30%</td>
<td>19.9</td>
<td>32.4</td>
</tr>
</tbody>
</table>

Experimental materials and methods
Powder mixtures were prepared from ASC 10022 iron powder and 5, 10, 15, 20, 25 vol.% phenol-formaldehyde thermosetting resin with mineral filler. Mixtures were uniaxially pressed in cylindrical die with inner diameter 10 mm at pressure of 200, 400, 800, 1600 MPa. After ejection from die the green compact was cured at a temperature of 165°C for 60 min. in an electric furnace in air atmosphere. Microstructure of compacts were analyzed by a LOM with polarized light. Compressibility was evaluated using a continuous measurement method. The pressure and the punch movement were monitored continually during the compaction process. Measured data were fitted by compressibility curve, \( P = \frac{K}{p + n} \) where \( P \) is porosity (\%), \( K \) and \( n \) are parameters of compressibility curve. In order to study the electrical properties, both flat surfaces of the sample were polished and electroded with high-purity silver paint and then dried at 130°C in oven for 30 min. to remove moisture. The impedance measurements were carried out at an input signal level of 500 mV in a wide frequency range of 100 Hz – 40 MHz using an impedance analyzer. A two-point configuration was used for the AC conductivity measurement. All experiments were performed at room temperature in air.

Conclusions
Fe-based soft magnetic composites of a core-shell structure with different content of phenol-formaldehyde thermosetting resin were prepared by uniaxial pressing of the mixture of iron powder and resin at various pressures. Particle refinement in the first stage of densification during pressing was suppressed due to agglomeration of Fe/ATM composite particles. Compressibility of the composite powder Fe/ATM increased with ATM content due to decrease of internal friction in powder and due to ability of elastic and plastic deformation of the ATM resin. Resin addition improves the compressibility and help to achieve higher density due to decrease of internal friction in powder and due to ability of elastic and plastic deformation of the ATM resin. Quantification of microstructure confirmed homogeneity of resin and pores distribution in the case of wet homogenisation followed with uniaxial cold pressing and curing as technology of preparation of micro-composites. The effect of resin addition along with compacting pressure on the electrical resistivity and Bode characteristics at room temperature was investigated. The experimental results showed that compressibility of the composites increases with increasing fraction of resin and follows a typical percolation behaviour. The percolation threshold was identified at 15% of resin added. The composites containing 25% of resin filler have the resistivity five orders of magnitude greater than that of the Fe/5ATM samples. For resin concentrations below the threshold, the specific electrical resistance decreased with increasing compacting pressure. AC impedance spectroscopy of the composites in the frequency range of 100 Hz – 100 MHz revealed that impedance spectra of the samples with an amount of resin below 20% show two relaxation processes, while as Fe/20ATM and Fe/25ATM display only one relaxation frequency in the resistivity plots. To describe a physical mechanism behind the electrical response of core-shell structure soft magnets, the impedance diagrams of composites will be further investigated at elevated temperatures. Also, a detailed study of structural aspects and dielectric properties of resin filler is required in order to understand the percolation effect in composites. Impedance spectroscopy is demonstrated as a powerful tool for non-destructive evaluation of quality of core-shell iron-based composites.

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