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Material investigations of components used in the production of composites for the electronics and electrotechnical industries

Abstract*. One of the great achievements of the last 50 years is the development of high-quality composite materials. By definition, classic composites are materials that are a combination of at least two components (phases), e.g. embedded particles, fibers, fabrics in a polymer, metal or ceramic matrix. Composites exhibit many unique properties if compared with solid metal and ceramic materials, as well as pure polymers and copolymers. This is typical to their stiffness and strength to weight ratios (strength and relative stiffness). The properties of composites depend to a large extent on the size, shape and form of the filling fibers and particles, in particular with their large share in the volume of the composite. This also applies to composite materials manufactured for the electronics and electrotechnical industries, where apart from appropriate mechanical properties, appropriate electrical properties are required, e.g. high ability to shielding of electromagnetic field in a wide frequency range.*

Streszczenie. *Znaczącym osiągnięciem ostatnich 50 lat jest rozwój badań nad wytwarzaniem wysokiej jakości materiałów kompozytowych. Z definicji klasyczne kompozyty to materiały będące kombinacją co najmniej dwóch składników (faz), np. cząstki, włókna, tkaniny w matrycy polimerowej, metalowej lub ceramicznej. Kompozyty wykazują wiele unikatowych właściwości w porównaniu z litymi materiałami metalowymi i ceramicznymi, a także czystymi polimerami i kopolimerami. Dotyczy to zwłaszcza stosunku ich sztywności i wytrzymałości do masy (wytrzymałość i sztywność właściwa). Właściwości kompozytów zależą w dużej mierze od wielkości, kształtu i postaci włókien i cząstek je wypełniających, w szczególności w przypadku ich dużego udziału w objętości kompozytu. Dotyczy to również materiałów kompozytowych wytwarzanych dla przemysłu elektronicznego i elektrotechnicznego, gdzie oprócz odpowiednich właściwości mechanicznych wymagane są odpowiednie właściwości elektryczne, m.in. wysoka zdolność ekranowania pola elektromagnetycznego w szerokim zakresie częstotliwości.* ***Badanie właściwości materiałów wykorzystywanych w produkcji kompozytów dla przemysłu elektronicznego i elektrotechnicznego****.*

Keywords: Composites, electromagnetic screen, SEM, XRD.
Słowa kluczowe: Kompozyty, ekran elektromagnetyczny, SEM, XRD.

Introduction

Modern powder composites, and in particular Soft Magnetic Composites (SMC), have recently become the subject of intense research [1-7]. SMCs are used in magneto conductors of electrical machines and devices, for the construction of chokes, and in the field of modern electric vehicles and electric motors with high power density. SMC materials allow the construction of complex three-dimensional objects with a magnetization topology unattainable for traditional transformer sheets [8].

Research on the impact of the processing process on the structure, density and magnetic properties of the obtained products can be found in the works of Gilbert et al. [9]. Other publications contains important information about the influence of heat treatment methods and particle size and packing of iron-based particles on energy losses and mechanical properties of the produced cores [10]. Kollár et al. [11] and Lauda et al. [12] focused on studying of the dynamic magnetic properties of Fe-based composites in a wide range of excitation frequencies. The freedom of shaping of electrotechnical composites properties enables their use as shielding of electromagnetic fields and unwanted electromagnetic radiation (EMI). These types of materials are highly desirable in medical, aerospace and military applications [13]. Also, there are more and more sources of high-frequency electromagnetic radiation in the daily environment.

From the point of view of technological process, it is very important to know the properties of materials used for production of composites, in particular, chemical and phase composition, shape and size of particles and fibers. In this paper, selected materials used in production of soft composite magnetic materials and screens for electromagnetic radiation [14, 15] were described and characterized. They were selected from a wide range of available materials, differing in their chemical and phase composition, shape and size. They come in the form of powders and their agglomerates, plates, flakes, chips and granules of materials such as aluminum, graphite, iron and steels, as well as ceramics including oxides. It should be emphasized that these materials were entirely waste materials from various production processes.

Investigations

Selected components for the production of composites in the form of powders, strips, chips and scale, including materials after an additional milling, were visually tested to determine their size, form and shape. Moreover, their chemical and phase composition, including domains and crystals size were determined with use of modern techniques of materials investigations.

Chemical composition

Morphology, size of particles and chemical composition was investigated with use of JEOL JSM-6610LV scanning electron microscope (SEM) with attached X-MAX 80 module (Oxfords Instruments) by means of energy dispersive spectroscopy (EDS). Investigation of samples morphology and their chemical composition was carried out with following parameters: accelerating voltage 20 kV, high vacuum and spot size (beam current) of medium value for best focus of image. Additional maps of chemical composition for selected samples were studied with resolution of 1024 (number of pixels in the X dimension over which the beam scans) and pixel dwell time up to 100 µs. The table 1 presents the results of the quantitative analysis of SEM-EDS.

The following elements were identified in the chemical composition of the tested samples: Fe, O, C, Al and Si in the case of the Fe strip and scale. High oxygen content is typical for scale and iron oxide powder. Due to the limitations of the SEM-EDS method, and in particular, the large error in the simultaneous measurement of light and heavy elements, their shares were given with accuracy to percentages.

Table 1. Assumed chemical composition of powders:

Element	Milled Fe tape	Powder scale	Iron oxides
	Chemical composition [wt. %]		
O	2	25	26
Si	8	3	-
C	<1	<1	<1
Al	<1	<1	<1
Fe	Balance	Balance	Balance

Phase analysis, particles and domains size

Powders' phases and crystals size identification was carried out by means of SEM imaging in secondary electron analysis mode and X-Ray diffraction in the Theta/Theta configuration on Empyrean XRD diffractometer (Panalytical) equipped with cobalt anode and with following parameters: K-Alpha radiation λ = 1.78901 Å, generator settings 45 mA, 40 kV. Diffractograms were recorded in range of 20° to 150° with step size 0.05° and scan step time 60 s working in continuous mode. Further data processing was performed using HighScore Plus with ICDD PDF 4+Database software. The size of coherently diffracting domains was determined with the help of Williamson Hall plot based on a principle that the approximate formulae for size broadening and strain broadening depend substantially on the Bragg angle θ. Images of the cross-section of the Fe tape and its surface as well as images of iron oxide powders, powder scale and milled Fe tape are shown in the figures 1-4, respectively. Diffractograms with the identification of the phases are visible in the figures 5-7, while summarized identification of phases and measured and calculated sizes of powders/flakes and domains is collected in table 2. Not all peaks have been identified.

The thickness of milled Fe tape particles are in the order of 200-300 μm, and their size ranges from dozens to hundreds of microns. There is also visible fine dust with a micrometric size.

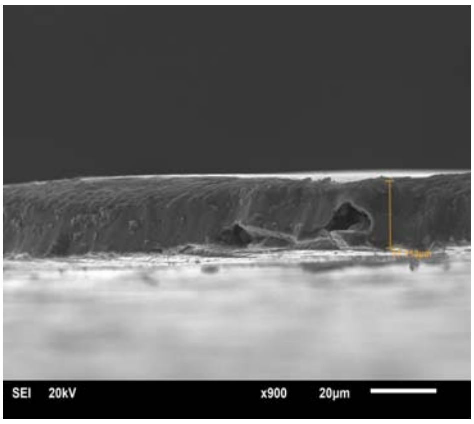

Fig.1. Fe tape before milling. Visible thickness on cross-section. SEM imaging.

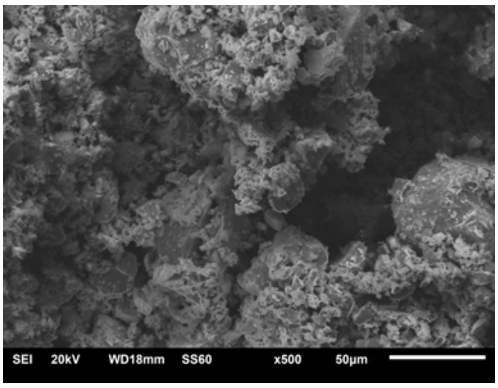

Fig.2. Powder and agglomerates of iron oxides. SEM investigation.

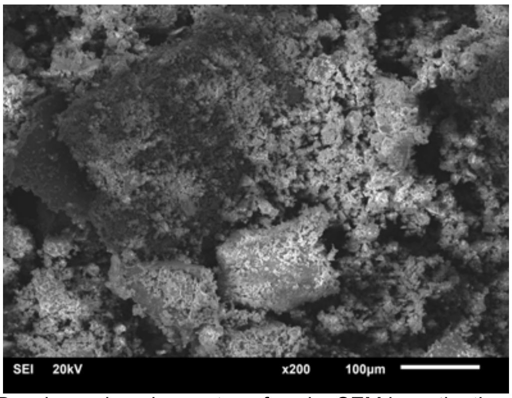

Fig.3. Powder and agglomerates of scale. SEM investigation.

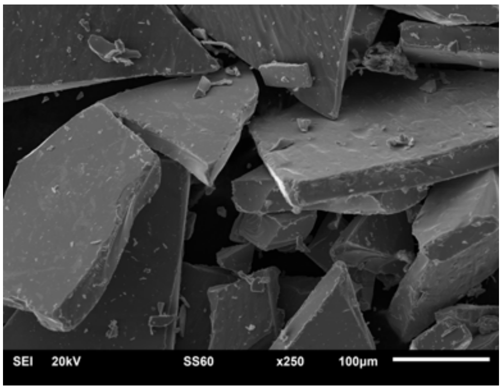

Fig.4. Flakes of milled Fe tape. SEM investigation.

The shape and particle size of the other two samples differ significantly from the flake scale. In the tested sample of the dust scale, it was noticed that it consists of irregularly shaped particles with a varied surface and sizes from a 2-5 μm to approx. 100 μm, coagulated to particles with a diameter of up to 500 μm. Similar particle sizes were found during the study of the iron oxide sample, but with a much higher share of particles with a size of a 2-5 μm.

Table 2. Measured and calculated sizes of powders/flakes and domains:

<table>
<tr><th>Parameter</th><th>Milled Fe tape</th><th>Powder scale</th><th>Iron oxides</th></tr>
<tr><td>Flake/powder size [μm]</td><td>30-500</td><td>10-500</td><td>10-150</td></tr>
<tr><td rowspan="6">crystalline domain size [Å]</td><td rowspan="3">Fe(Si)</td><td>Fe_3O_4</td><td>Fe_3O_4</td></tr>
<tr><td rowspan="2">310-390</td><td>380-500</td></tr>
<tr><td>Fe_2O_3</td></tr>
<tr><td rowspan="3">60-90</td><td>Fe_2O_3</td><td>300-500</td></tr>
<tr><td rowspan="2">200-440</td><td>FeO</td></tr>
<tr><td>250-310</td></tr>
</table>

XRD analysis showed that all tested materials showed a crystalline structure. Phases of Fe_3O_4 (ICDD reference card number: 04-002-5632), Fe_2O_3 (04-003-1445) and FeO (01-080-3819) were detected in the phase composition of the investigated iron oxide powders. The composition of the powder scale consists mainly of Fe_3O_4 (04-006-1668) and Fe_2O_3 (01-076-4579) oxides, while the milled Fe tape consists mainly of Fe (04-013-9776) with a small addition of Fe_3Si (04-015-3939) in the phase composition.

Based on the results of the analysis of the size of the domains by means of Williamson Hall plot, one can see that domains are much smaller (from a few to several dozen nanometers) than the size of the powders and plates of the tested materials.

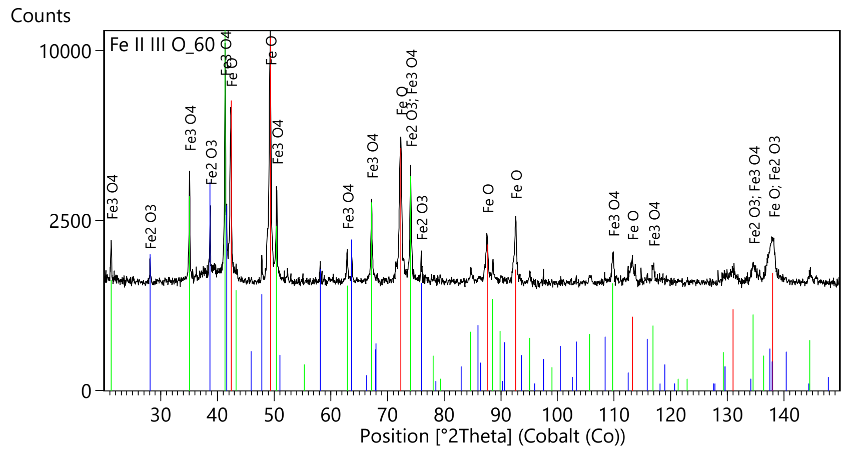

Fig.5. Diffractogram of investigated iron oxide powder.

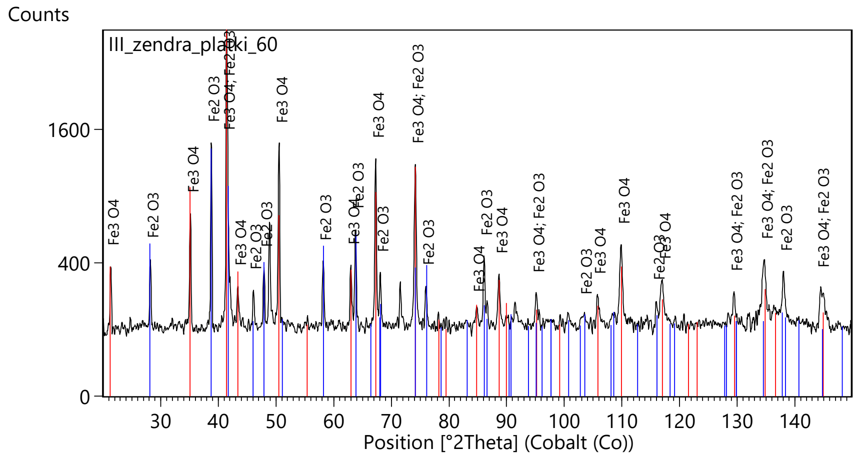

Fig.6. Diffractogram of investigated powder scale.

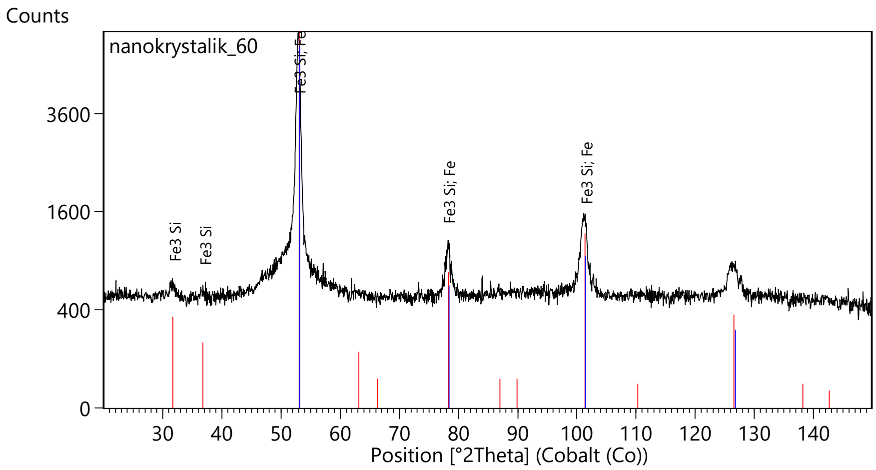

Fig.7. Diffractogram of investigated milled Fe tape.

Conclusions

Taking into account the increasing requirements for materials used in industry, including the electrotechnical and electronic industries, a very important factor is to determine the properties of these materials. This applies in particular to the production of composites, e.g. for the shielding of electric and magnetic fields. In order to reduce research costs and shorten the time needed to prepare the product, the first stage of work is based on modelling using the finite element method. Modern materials investigations methods used in materials engineering are able to provide relevant data necessary in the modelling process. In particular, this applies to the chemical and phase composition of the materials. Moreover, considering the dependence of the domain size and the macroscopic magnetic properties of materials, analysis of crystalline domains sizes is crucial in the case of designing of composites for magnetic and electric field screens.

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