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Microwave Sintered Fe/MgO Soft Magnetic Composite

R. Bureš[a,*], M. Fáberová[a], P. Kollár[b], J. Füzer[b], S. Dobák[b], F. Onderko[b] and P. Kurek[a]

[a]Institute of Materials Research, Slovak Academy of Sciences, Watsonova 47, 040 01 Košice, Slovak Republic

[b]Institute of Physics, Faculty of Science, P.J. Šafárik University in Kosice, Park Angelinum 9, 041 54 Košice, Slovak Republic

Micro/nano soft magnetic composite based on the Fe microparticles and the MgO nanoparticles was prepared by cold pressing followed by microwave sintering. Magnetic and mechanical properties of the green compact as well as sintered samples were measured. Coercivity, permeability, resistivity, elastic modulus and transverse rupture strength values in dependence on MgO content were investigated. The influence of MgO content ratio on properties was different in the case of as pressed green samples in comparison to sintered bodies. Microstructure formation and its influence on mechanical and magnetic properties are discussed. The coercivity of the green compacts with 1–5 wt% of MgO exhibits approximately 460 A/m and after sintering decreases to approximately 290 A/m. The real part of complex permeability at the frequency of 100 kHz exhibits a maximum for 2 wt% of MgO in green compacts, while for 10 wt% in sintered samples. It was observed that increase of the content of MgO causes decrease of the permeability. Properties of the sintered composite are related to formation of magnesium ferrite as well as volume distribution of residual MgO in dependence on initial MgO ratio.

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1. Introduction

Soft magnetic composites (SMC) are produced by the powder metallurgy method from ferromagnetic particles coated with a thin electrically insulating layer. Widely used shaping method is cold pressing, which introduces an elastic and plastic deformation to the composite. Structural discontinuities, imperfections as well as residual stresses induce changes of magnetic and mechanical properties [1]. Inorganic coatings, based on MgO, Al_2O_3 and SO_2, with high thermal stability are investigated to allow stress relaxation heat treatment at temperature higher than 600 °C [2, 3]. Advanced compaction methods are investigated with focusing on optimization of intrinsic structure of SMC's and other functional materials [4, 5]. Microwave sintering is progressive heat treatment technique. Microwave heating is fundamentally different from conventional heating in the way how thermal energy is delivered to the material. It has been observed improvement of microstructure, physical and mechanical properties of microwave sintered composites in comparison to conventionally sintered materials [6]. Subject of this work is the microwave sintered micro–nano composite based on Fe/MgO. Microstructure formation, magnetic and mechanical properties were investigated.

2. Experimental material and methods

Technically pure Fe microparticles (ASC 100.29, Höganäs AB, mean size of powder particles d = 100 μm) were dry coated by MgO nanoparticles (MTI Corp., mean size d = 30 nm) using the Resonant Acoustic Mixing method in Resodyn LabRAM mixer. Fe/MgO powders with 1, 2, 3, 5, 10 and 13.85 wt% of MgO content were uniaxially cold pressed at pressure of 600 MPa. Sintering was provided in multimode microwave (MW) cavity with controlled power from 100 W to 3 kW at constant temperature of 800 °C, for 15 min, in dry air atmosphere. Multimode MW oven Hamilab V3000 (Synotherm Corp.) was equipped by IR pyrometer Optris.

Microstructure and EDS analyses were provided by scanning electron microscope (SEM) JEOL 7000F and inverse light microscope Olympus GX-71 (OM). For the investigation of magnetic properties, the samples were used in the form of a ring with one toroid. Complex permeability (real and imaginary part of complex permeability) were measured with an impedance analyzer (HP 4194A) at 100 kHz. Coercivity (H_c) of green compacts and sintered samples was measured using Koerzimat HCJ 1.097 (Foerster). The Young modulus (E) was measured by impulse excitation technique using Buzz-o-sonic system (BuzzMac). Flexural strength was characterized by measurement of transverse rupture strength (TRS) using three point bending test on universal testing machine Tiratest 2300.

3. Results and discussion

Magnetic and mechanical properties of the green compact as well as sintered samples were measured. Coercivity, permeability, resistivity, elastic modulus and transverse rupture strength values in dependence on MgO content were investigated. Relative density of the green compact and sintered sample, in Fig. 1, is nearly constant up

*corresponding author; e-mail: rbures@saske.sk

to 3 wt% of MgO. Relative densities decrease with MgO addition more than 3 wt%. Slightly lower values of the sintered density in comparison to green density is result of $MgFe_2O_4$ formation in process of the sintering in air atmosphere as it is presented in our previous work [7].

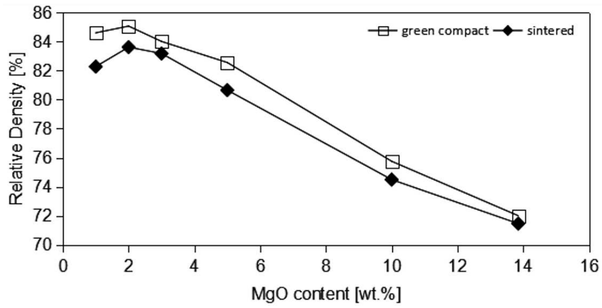

Fig. 1. Relative density versus MgO content.

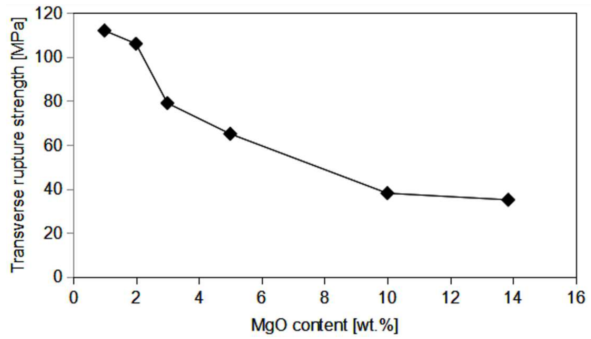

Fig. 2. Flexural strength versus MgO content.

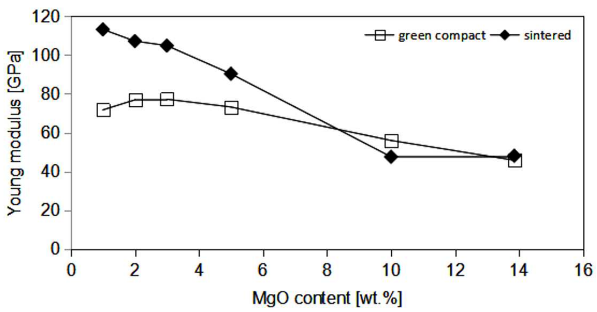

Fig. 4. Coercivity versus MgO content.

Flexural strength of the sintered composite, in Fig. 2, decreases with increase of MgO content. Dependence of the Young modulus of the green compact on MgO content is shown in Fig. 3 and is in agreement with relative green density. The Young modulus of the sintered composite decreases with increase of MgO content up to 10 wt% MgO. At this point, elastic properties of sintered composite are lower in comparison to green compact. MgO content higher than 10 wt% discontinues decrease of the elastic and mechanical properties.

Coercivity of the green compact, in Fig. 4 is nearly constant. Lower value of coercivity of the sintered composite

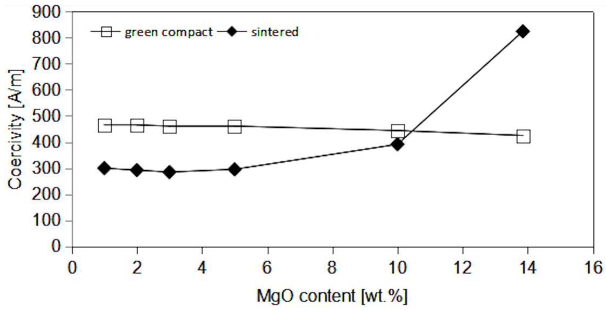

Fig. 4. The Young modulus versus MgO content.

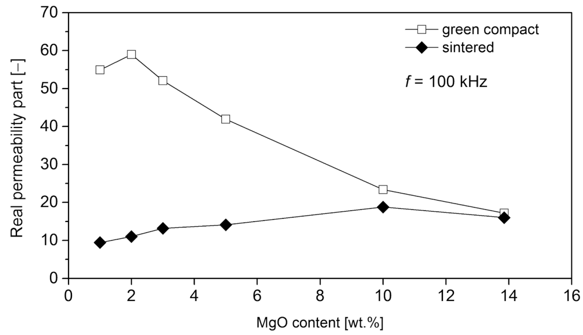

Fig. 5. Real part of complex permeability of Fe/MgO composites at 100 kHz.

is a result of recovery and stress relief processes induced by sintering. Coercivity of the sintered composite slightly increases with increase of MgO content higher than 5 wt%. MgO content higher than 10 wt% causes rapid increase of the coercivity value.

The coercivity of the green compacts within 1–5 wt% of MgO exhibits approximately 460 A/m and after sintering decreases to approximately 290 A/m. Figure 5 provides behavior of real relative permeability component versus MgO content at given magnetizing frequency of 100 kHz. In green compacts, this permeability possesses a maximum at 2 wt% MgO and then decreases. However in sintered cores, MgO content causes an increase in permeability up to 10 wt% of MgO.

Observation of microstructure by light microscopy in Fig. 6 shows that MgO ratio up to 5 wt% leads to distribution of the MgO nanoparticles to the voids within irregular shaped iron microparticles. MgO ratio about 1 wt% is not enough to continuous insulation of iron particles. MgO content from 2 to 5 wt% provide suitable insulation network in the composite. There is not significant increase of the porosity with increase of MgO ratio. Decrease of the green density with increase of MgO content is result of MgO distribution changes in process of cold pressing. On the other hand, decrease of sintered density is caused by formation of $MgFe_2O_4$ interlayer among iron and MgO particles. MgO ratio up to 3 wt% leads to formation magnesium ferrite without residual content of MgO in microstructure of sintered composite as it is shown in Fig. 7 (top). Higher MgO ratio leads to microstructure with residual MgO, which cases the degradation of mechanical and magnetic properties. These observations are related to result of study of the effect of iron island morphology and interface oxidation on the magnetic properties of Fe–MgO thin film composites published by Spurgeon et al. [8].

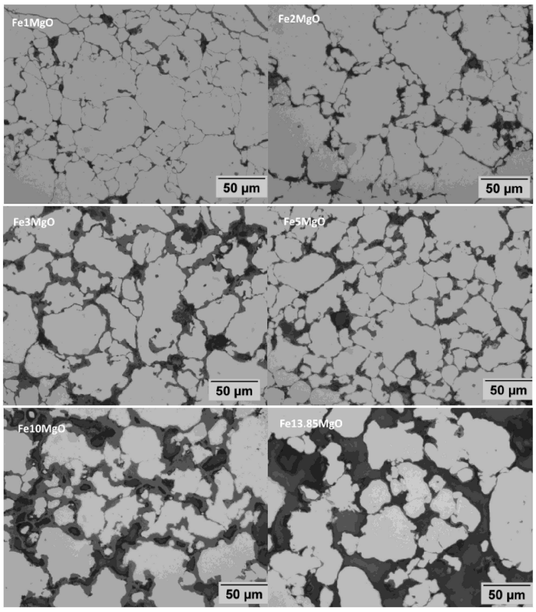

Fig. 6. Microstructure of sintered Fe/MgO composite, OM.

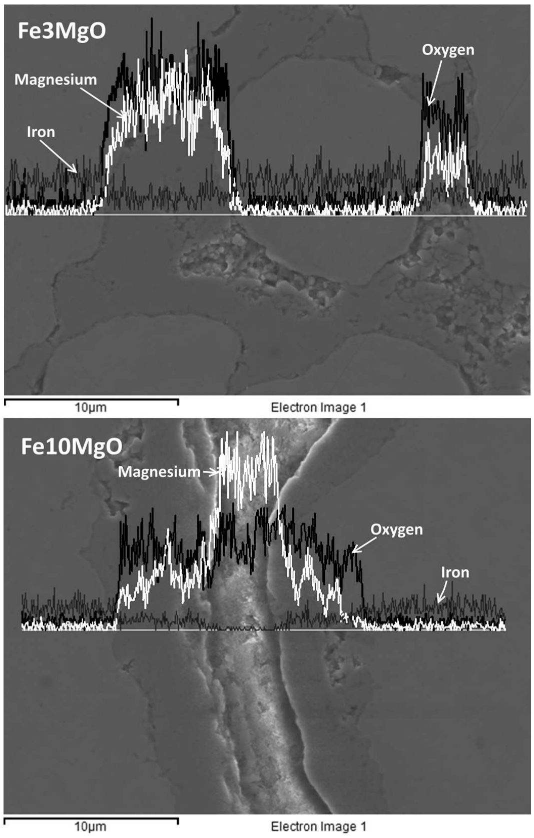

Fig. 7. SEM microstructure and EDS line analysis of sintered Fe/MgO composite.

4. Conclusions

Micro/nano soft magnetic composite based on the Fe microparticles and the MgO nanoparticles was prepared by cold pressing and by microwave sintering. The influence of MgO content ratio on properties was different in the case of as pressed green samples in comparison to sintered bodies.

Magnetic and mechanical properties of the sintered composite are related to formation of magnesium ferrite as well as volume distribution of residual MgO in dependence on initial MgO ratio.

Acknowledgments

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