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Studying microstructure of sintered magnets Co-25% Sm

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Abstract. Metallographic study of sintered rare-earth Co- 25% Sm magnets microstructure of KS-25 brand was carried out by scanning electron force microscopy (EDS-analysis). A dendritic structure with three phases was found: branches of dendrites are SmCo_5 ; interdendritic space is a mixture of two phases SmCo_5 and $\text{Sm}_2\text{Co}_{17}$. Crystals of 1–5 μm of $\text{Zr}_5\text{Co}_3\text{FeSm}$ compound and inclusions of Sm_2O_3 samarium of a globular form of 2–10 μm in size were founded also in the microstructure. Study of the domain structure on surfaces perpendicular to magnetization axis by magnetic force microscopy (MFM) showed presence of strong magnetocrystalline anisotropy. Comparison of magnetic images with electron microscopic images of surface made it possible to conclude that SmCo_5 dendrites correspond to large domains $\sim 30\text{--}50 \mu\text{m}$ in size and the interdendritic space consisting of a mixture of two phases SmCo_5 and $\text{Sm}_2\text{Co}_{17}$ correspond to a domain structure in labyrinth form with a size of $\sim 3\text{--}5 \mu\text{m}$.

1. Introduction

Sintered magnets based on the SmCo_5 intermetallic compound are used widely in engineering due to the large coercive force up to 40 kOe and magnetic energy up to 24 MGE [1-3]. Improvement characteristics of these magnets are impossible without a scientific study of principles formation of a highly coercive state, disclosure of a relationship between characteristics of microstructure, phase composition and magnetic properties. Microstructure of sintered SmCo_5 magnets includes fragile intermetallic compounds that can be destroyed both during manufacturing process and during operation. The study of the microstructure of magnets is also aimed at improving their mechanical properties.

The microstructure formation of sintered Co-25% Sm magnets occurs at several technological stages of manufacturing: 1) obtaining an alloy of the necessary composition, 2) preparing a fine powder, 3) pressing the powder in a magnetic field for formation of a texture, 4) fritting of powder billet and 5) carrying out thermal treatments to increasing of coercive force. Formation of a highly coercive state in sintered Co-25% Sm magnets is due to a change in surface of grains of main phase of SmCo_5 . There are dispersed $\text{Sm}_2\text{Co}_{17}$ precipitates on the surface of phase SmCo_5 grains during heat treatment process (slow cooling or low-temperature tempering) in the cobalt enriched magnets. Formation of $\text{Sm}_2\text{Co}_{17}$ phase precipitates coherently connected with main SmCo_5 phase leads to a smoothing of grains surface, a decreasing in number of defective places that are nucleation centres of reverse magnetization domains and it increase coercive force in thermoheating magnets. To obtain high hysteresis properties sintered Co-25% Sm magnets should be somewhat enriched with samarium compared to stoichiometric of SmCo_5 composition. An excess of Sm is needed in order to compensate for its loss in manufacture, sintering of powders as a result of evaporation, oxidation and binding of Sm with impurities. Samarium enrichment is also necessary for formation of a non-equilibrium microstructure that ensures growth of coercive force in the sintered magnets during their heat treatment [4-5]. Study of local crystal structure of high-coercive SmCo_5 alloys by EXAFS spectroscopy using synchrotron radiation discovered to formation of disordered defects such as stacking faults during enrichment of alloys with samarium [5].

Machining of magnets (EDM and laser cutting, grinding) gives a deterioration in magnetic properties and even leads to destruction. Local heating and saturation of surface layers with hydrogen gives a



decrease in magnetic properties of magnets and their destruction also. Sintered Co-25% Sm permanent magnets contain significant amounts of samarium rare earth element. Concentration of samarium at grain boundaries (in boundary phases, which are largely responsible for preservation of highly coercive state of magnets) reaches 70-98 wt.%. High affinity of rare-earth elements to oxygen and hydrogen is known, it leads to formation of oxides and hydrides. Under adverse climatic conditions of operation (high humidity and air temperature, in salt fog, hydrogen-containing atmosphere) magnetic and mechanical properties of magnets can significantly decrease, up to destruction. Additional mechanical loads in real conditions aggravate all this factors [6-7].

Structurally sensitive properties of magnetic material are initial and maximum magnetic permeabilities, coercive force, residual magnetization (residual induction) loss from hysteresis. These properties are extremely dependent on microstructure and sample size. The greatest influence on these properties is exerted by atoms of dissolved element, dislocations, grain boundaries, presence of second phase and its disparity. It is possible to influence these properties in a wide range by changing phase state and structure of alloys. Study of connection of the microstructure, the magnetic structure and the mechanical properties of magnets is theoretical and practical interest. The domain structure of magnetic materials mainly determines their magnetic properties. This work is devoted to the study of microstructure by means of scanning electron microscopy, magnetic structure by means of magnetic force microscopy of Co-25% Sm magnets of KS-25 brand in order to establish a connection between them.

2. Materials and Methods

Samples for study were selected from ready-made sintered magnets Co-25% Sm brand KS-25 according to GOST 21559-76. Chemical composition of the studied samples was Sm - 25 wt.%; Fe - 18wt.%; Cu - 9wt.%; Zr - 3wt.%; rest was Co. Before study the sample surfaces were grinded and polished by diamond abrasive down to 1 μm and colloidal silica during 20 min at the final stage.

A metallographic study was carried out on a Carl Zeiss AURIGA CrossBeam scanning electron microscope using the function energy dispersive spectroscopy (EDS). Scanning electron microscope AURIGA CrossBeam (Carl Zeiss, Germany) was equipped with the EDS system IncaEnergy (Oxford Instruments, UK) with 350X-MAX X-ray detector with spatial resolution about 1 μm and spectral resolution of 125 eV at $K\alpha$ line of Mn. The SEM was used for visualization of surface morphology and structural defects in secondary electron mode with the resolution down to 2 nm. The EDS was applied for revealing of elemental composition of defects and inclusions. The EDS data collection and processing were made by means of IncaEnergy software.

Study of the magnetic structure was performed using the Asylum MFP 3D SA scanning probe microscope. Resulting images are spatial distribution of the Z-component of magnetic field on sample surface. Measurements were carried out using a two-pass technique, where the "magnetic" images were separated from the relief images. It was necessary to minimize influence of relief on the image of the distribution of magnetic forces. On first pass, surface relief was determined by the discontinuous-contact method. In second pass, on each scan line, the cantilever was raised above surface for a specified distance dZ , and scanning was performed in accordance with previously obtained relief. Thus, on second pass, distance between scanned surface and fixed end of the cantilever was constant. As a result, the image of relief and the magnetic image are obtained simultaneously. To observe spatial periodic domain structure it is sufficient to know derivative of magnetic interaction force; magnitude of magnetic force

$\vec{F} = (\vec{m}\vec{\nabla})\vec{H}$, where m is effective magnetic moment of probe, H is the stray field of sample.

Registered force of magnetic interaction and magnitude of field during movement of probe over the domain will be almost constant. In process of passing the cantilever over the domain wall, a slightly smoothed phase jump and amplitude of resonant oscillations are observed, which corresponds to a change in force [8-10].

3. Results and discussions

Results of microstructure studying of sintered magnet Co-25weight.% Sm brand KS-25 by means of scanning electron microscopy, including EDS analysis are presented in Fig.1. Data of EDS-analysis confirmed presence in magnets of a mainly single-phase SmCo_5 structure.

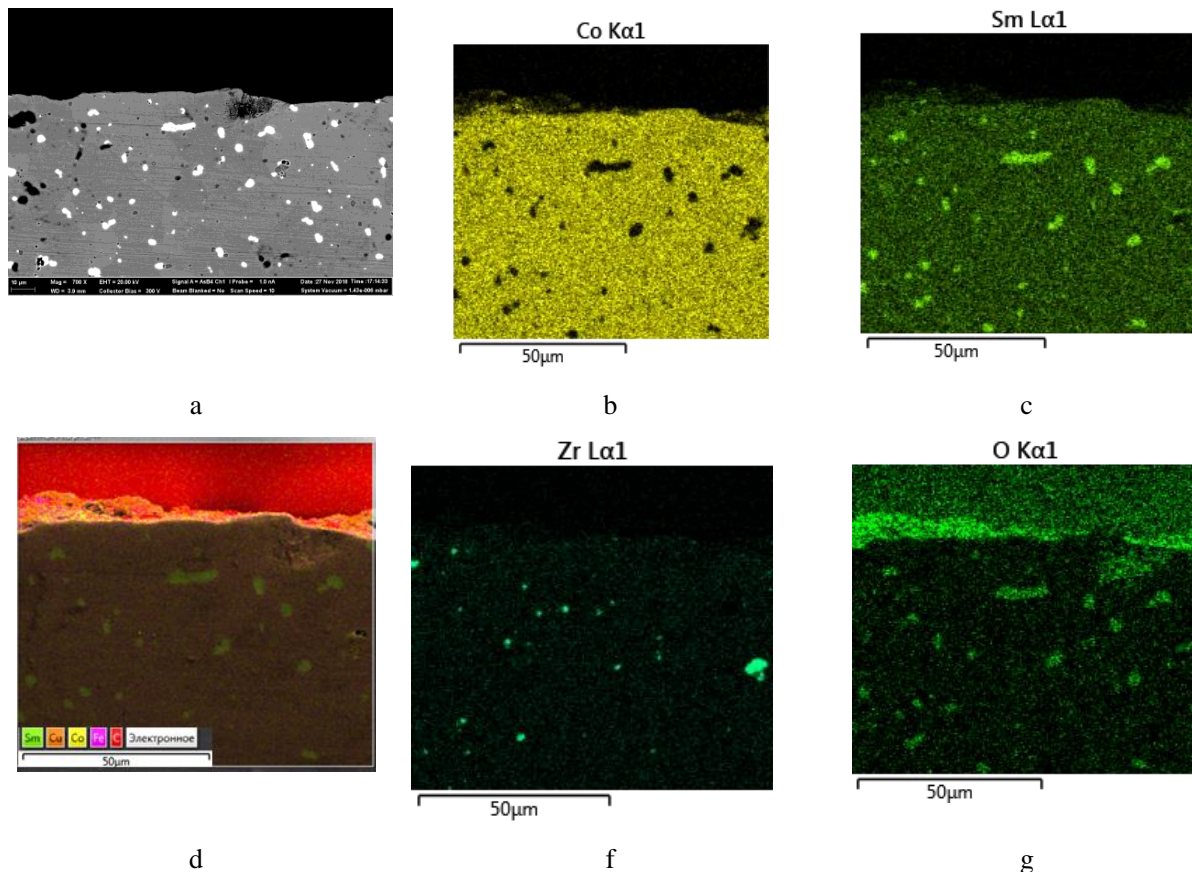


Figure 1. Microstructure of surface of sintered Co-25% Sm magnets of KS-25 brand. EDS-analysis in form of elements maps distribution (b, c, d, e, f, g) and image (a) in the back scattered electrons

Microstructure is generally represented by three components: SmCo_5 dendrites (samarium content is 11 at.%), the interdendritic space is a mixture of two phases SmCo_5 and $\text{Sm}_2\text{Co}_{17}$ (samarium content 18 at.%), the crystals $\sim 1\text{-}5\ \mu\text{m}$ in size $\text{Zr}_5\text{Co}_3\text{FeSm}$ (zirconium content is 47-52 at.%; cobalt is 25-28 at.%; iron is 11-13 at.%). Large inclusions of $2\text{-}5\ \mu\text{m}$ samarium oxide Sm_2O_3 (oxygen content is 58–57 at.%, samarium content is 38 at.%) it is white globules in Fig. 1 were also found. The $\text{Sm}_2\text{Co}_{17}$ phase has a lower hardness than the main phase of SmCo_5 ($\text{Sm}_2\text{Co}_{17}$ microhardness is 600 MPa, SmCo_5 microhardness is 840 MPa) and is considered as a binding additive that improves mechanical properties. Sm_2O_3 inclusions are defects of metallurgical origin. Non-metallic inclusions of Sm_2O_3 sharply deteriorate mechanical properties of magnets; reduce strength and ductility, and corrosion sites appear in places of localization of these inclusions.

Results of the magnetic structure study on the surfaces perpendicular to axis of magnetization by magnetic force microscopy (MFM) are presented in Fig.2. Figure 2 displays spatial pattern of magnetic forces on surface sample. Images were obtained by measuring amplitude shift of the cantilever oscillation, which occurs under action of the magnetic force. The images contain information about location of the magnetic domains on sample surface. Grains of the main phase of SmCo_5 are dendrite branches with a characteristic size of $\sim 20\ \mu\text{m}$ in a magnetized state, they consist of one domain and correspond to lighter regions in Fig. 2. Observed domain structures are similar to those present in bulk

uniaxial crystals with strong magnet anisotropy [11]. On surface perpendicular to axis of magnetization a domain structure is also observed in a labyrinth form. Small-scale domains have a transverse size of 3-5 microns and are magnetized perpendicular to surface. Their appearance is explained by a decrease in the magnetostatic energy due to higher energy of the domain wall. Large domain structures of grains, as a rule, are independent of neighbors but in some cases a significant magnetostatic connection between neighboring grains may be observed [12]. Connection between the domain width and the internal coercivity is also known. If the domain width decreases from 2 to 1 μm , energy of the domain wall also decreases, and the internal coercivity increases [13]. Comparison of magnetic structure images (Fig. 2) with electron microscopic images of surface (Fig. 1) made it possible to conclude that SmCo_5 (the dendrites branches are $\sim 30\text{-}50 \mu\text{m}$ in size) consist of one domain, the mixture of two phases SmCo_5 and $\text{Sm}_2\text{Co}_{17}$ (the interdendritic space) have a domain structure in the labyrinth form with the size of magnetic phase components $\sim 3 \mu\text{m}$.

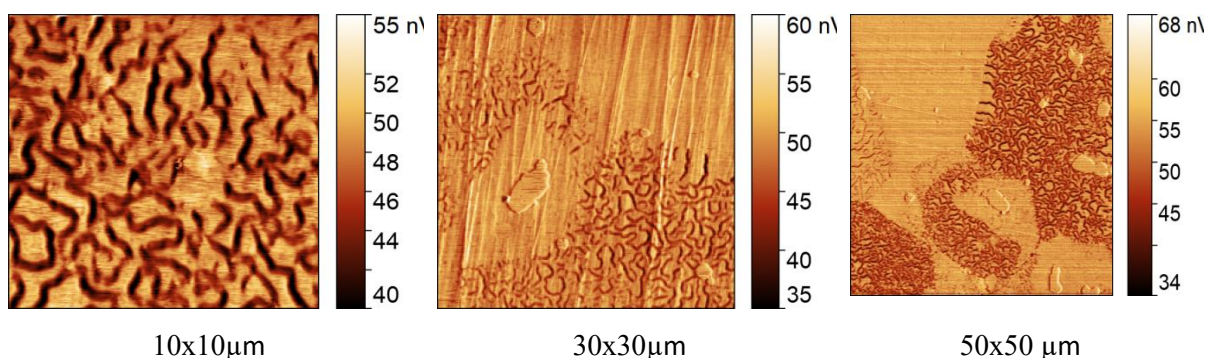


Figure 2. MFM-image of surface of sintered magnets Co-25% Sm of KS-25 brand (amplitude of resonant oscillations)

4. Conclusions

Microstructure of sintered magnet Co-25% Sm of KS-25 brand is represented by SmCo_5 dendrites (samarium content 11 at.%), the interdendritic spaces are a mixture of two phases SmCo_5 and $\text{Sm}_2\text{Co}_{17}$ (samarium content is 18 at.%). Microstructure also contains individual crystals of 1–5 μm of $\text{Zr}_5\text{Co}_3\text{FeSm}$ compound (zirconium content is 47–52 at.%; cobalt is 25–28 at.%; iron is 11–13 at.%) and large globules 2–10 μm of samarium Sm_2O_3 oxide. Inclusions of Sm_2O_3 reduce strength and ductility and in localization places of inclusions corrosion pockets appear. Magnetic structure of sintered permanent magnets Co-25% Sm of KS-25 brand on surfaces perpendicular to axis of magnetization is represented by large SmCo_5 domains with a characteristic size of 30-50 microns and a domain structure in the labyrinth form from small-scale domains of 3-5 microns in size. Comparison of MFM-images with SEM-images of Co-25%Sm magnet surface made it possible to conclude that domains correspond to SmCo_5 dendrites, and domain structure in form of a labyrinth corresponds to an interdendritic space of a mixture of two phases SmCo_5 and $\text{Sm}_2\text{Co}_{17}$.

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