Effect of annealing on recrystallization of GAP particles of Fe-Nd-B system alloy

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Abstract. The magnetic and mechanical properties magnets of the Fe-Nd-B system depend more on the size and shape of the grain structure. The influence of annealing on the microstructure of gas atomized powder (GAP) particles of Fe-Nd-B alloy was investigated. Conditional three types of microstructure are allocated and their changes in the process of recrystallization are analyzed. It is shown that the process of recrystallization is accompanied by an increase in the intergranular phase and the formation of grains in it. Increasing the annealing time enhances these processes.

1. Introduction
Magnetic materials of alloys of the iron-neodymium-boron system have unconditional very high practical significance. Operating conditions and high heat sometimes lead to irreversible processes and loss of material properties. The theory of boride ferromagnetism of stoichiometry TM₁₄REM₂B does not have a complete accurate quantitative model, requires verification, refinement and further development. The magnetic properties of the Fe-Nd-B alloy material and products made of such an alloy are sensitive to the type of grain structure and the size of the grain size. Annealing of the gas atomization powders (GAP) causes recrystallization and changes in crystallographic parameters of grain structure, size and shape, which is associated with changes in magnetic properties [1-5].

2. Experimental
Gas atomized powders (GAP) were obtained by gas atomization a melt jet with an inert gas stream at the URZHMV-3 liquid metal atomization unit (Ukraine). In this case, the pressure of the energy carrier gas reached 12 atm. and the nozzle “Laval nozzle” was used. The modes of the gas atomization process are described in detail in [4]. Fractures of composition Fe₇₇.₅Nd₁₄.₅B₈.₀ were investigated in the work.
Powders with a fraction from 0.0 to 0.630 mm were taken for the investigation. Separation into fractions was performed by sieves with numbers: 0.020, 0.056, 0.100, 0.160, 0.400 and 0.630 mm. The analysis of grain size, shape and structure was performed by quantitative metallography at the SIAMS-800 scanning station for panoramic applications in reflected light, including the OLYMPUS BX-51 metallographic microscope and SIAMS Drive System.

3. Results and discussion
The determination of the fractional composition by sieve analysis of the investigated compositions shows that the maximum mass fraction falls on the fraction of 0.160 - 0.400 mm (Fig.1).
To identify the microstructure, several types of etchants based on sulfuric acid and nitric acid with the addition of glycerol and phosphoric acid were used. Analysis of the microstructure of the studied alloys revealed some features. The main feature of particle formation during gas spraying is the difference in crystallization rates of particles of different sizes. Fracturing is characterized by the formation of particles into which other smaller particles were embedded during solidification. During the preparation of micro-grinding and etching, the boundary between the embedded particle and the base is destroyed, and the embedded particle falls out, leaving a cavity. Since the cooling rate of the embedded particle and the substrate differ, the microstructures also differ. Due to this feature, there is an uneven distribution of the neodymium containing phase, which causes differences in the microstructure of the particles. Three conventional structures are accepted for consideration: 1) coarse-grained structure with anisotropy of grain shape greater than three (Fig. 2a); 2) fine-grained structure with grain shape anisotropy less than three (Fig. 2b); grain block structure with radial gradient of grain growth direction (Fig. 2c).

The formation of the block structure is explained by the excellent solidification temperature of the different phases of the Fe-Nd-B system. When the fracturing particles cool down, the regions enriched with Nd with a fine-grained structure first harden. For figure 2c these areas look like small rounded nucleation centers of elongated grains with a radial direction of growth. The centers themselves are a collection of a large number of equiaxial grains with an average size of about 0.5 microns.

When annealing in air for 1 hour at 600 °C in all fractions there is a change in the shape of the grains. On the surface of the particles, a region saturated with oxygen is formed, which is colored with a characteristic light gray color (Fig. 3). Inside the particles, the intergranular space is also highly oxidized and expanded.

Analysis of the microstructure of particles after annealing for 1 hour at 600 °C in argon protective medium shows that different structures change differently. In the coarse-grained structure, grain associations and an increase in the intergranular space are manifested (Fig. 4a), in which there is the origin and crystallization of a new phase. In the fine-grained structure, the grains are crushed until the grain boundaries disappear, and the intergranular phase is formed in the form of fine particles (Fig. 4b). The dissolution of grain boundaries is observed in the block structure (Fig. 4c).
Figure 2. Microstructure of fracturing alloy particles системы Fe$_{77.5}$Nd$_{14.5}$B$_{8.0}$.

Figure 3. Microstructure of Fe$_{77.5}$Nd$_{14.5}$B$_{8.0}$ fracturing alloy particles after air annealing.

Increasing the annealing time to 2.5 hours increases the phenomenon of grain fusion and phase crystallization in the intergranular space in all fractions (Fig. 5).
Figure 4. The microstructure of particle fracturing of the alloy Fe\textsubscript{77.5}Nd\textsubscript{14.5}B\textsubscript{8.0} after annealing in argon.
Figure 5. Microstructure of Fe$_{77.5}$Nd$_{14.5}$B$_{8.0}$ fracturing particles after 2.5 hours annealing in argon.

4. Conclusions
1. The recrystallization process has a different mechanism for different initial microstructure.
2. During annealing, there is always a change in the morphology and size of the intergranular structural component in the Fe-Nd-B fracturing alloy particles.
3. In the intergranular structural component for all fractions of the studied powder, the separation of new structural formations is observed.

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