NEUTRON-DIFFRACTION STUDY OF ANISOTROPY IN GAS-ATOMIZED Nd-Fe-B MAGNETS POWDERS

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A modern high-energy permanent magnet of the Nd-Fe-B system is a complex physicochemical system in which the magnetic properties depend on the orientation of the grains, the presence of foreign phases (including non-metallic inclusions and intermetallic compounds), surface modified layers of grains of the main phase $Nd_2Fe_{14}B_1$ (T_1), intergranular layers, grain scattering fields and many other features of the structural-phase state of the material at the nano- and microlevels. The maximum magnetic hardness and the magnetic energy correlating with it of a classical permanent magnet are determined by the chemical composition of the alloy and its structural-phase state, including those associated with the features of the technological process and the quality of the initial charge materials. For anisotropic sintered magnets, improved functional properties are achieved by optimizing the size and shape of the grains of the T_1 phase (L×H×T ≈ 6×3×3 μm), their high crystallographic wear (texture), the presence of a uniform intergranular boundary (with a thickness of ≈ 1 μm) and minimizing the contacts of other phases (primarily intermetallic compounds) with the main magnetic phase (T_1). The textured state increases the free energy of the magnet, which is compensated by the development of a domain structure within the grains. The process of magnetization reversal (demagnetization) of the domain structure can be slowed down by creating a certain microstructure of the alloy, which prevents the nucleation of domains of the opposite sign and hinders the movement of domain walls, thereby increasing the coercive force of the magnet.

Nonequilibrium crystallization under conditions of quenching from the liquid state or, in English terminology, Melt Quenching (MQ) can significantly affect the structural-phase state of the alloy (up to its amorphization) and opens up technological possibilities for controlling the magnetic properties of the material and products made from it. In the work, the MQ is implemented using the technology of spraying a jet of melt with a flow of inert gas, or Gas Atomization (GA). This produces a powder material (GAP) with a fractional composition that meets the requirements of both classical processing (mechanical crushing and grinding of the ingot to particles 6÷10 μm in size; compaction in a magnetic field; sintering and heat treatment in a protective atmosphere) and modern additive technologies of direct growth (60÷120 μm). In fact, the additive process involves the melting of an individual powder particle and the subsequent crystallization of a drop of comparable size under conditions of directed heat removal through a gaseous medium in a protective atmosphere.

Between the classical technology of producing sintered anisotropic magnets and the additive technology of direct growth of magnetic microsystems of complex shape, there is a large area of applied problems when the dimensions of the permanent magnet approach the dimensions of the GAP and the spherical shape of the magnet is a functional requirement.

At the same time, remelting of a separate GAP (heating above Tm ≥1250°C), as an independent technological operation for the production of a finished product (in the cycle of a single technological process) is often not allowed. Moreover, the implementation of such a micrometallurgical process requires significant correction of the alloy composition to achieve the required level of functional magnetic properties. In this work, a solution is proposed by implanting a separate GRP with surface melting (technological soldering) and volumetric recrystallization of T_1 phase grains [1].

In the academic aspect, the solution comes down to proving that in any individual GAP there is always an average anisotropy of the spatial orientation of T_1 grains due to the random nucleation of crystallization centers in the volume of an individual drop of supercooled melt and the spherical symmetry of the heat sink of the solidifying (crystallizing) melt.

In the materials science aspect, such random heterogeneity can be enhanced by recrystallization (by analogy with primary recrystallization) during special heat treatment: 1) thermal cycling in the Curie temperature region (T_c≈310°C), when the volumetric changes of an individual grain of the T_1 phase reach 6%; 2) heating and holding above the melting point of the ternary eutectic (T_e≈510°C), when, under the conditions of a developed pseudo-capillary system of the intergranular space, an anomalous mass transfer of matter occurs and selective directional recrystallization is realized.

In this study, we investigated a series of Nd-Fe-B-based MQ-GA-alloys alloyed with various elements. The first case of recrystallization was realized: thermal cycling near $T_c \approx 310$°C (ΔT = 200÷400°C, 10 cycles). To enhance the effect and provide a reducing environment, thermal cycling was carried out in flowing hydrogen.

The features of the emerging grain anisotropy and its relationship with magnetic properties are illustrated in Fig. 1.

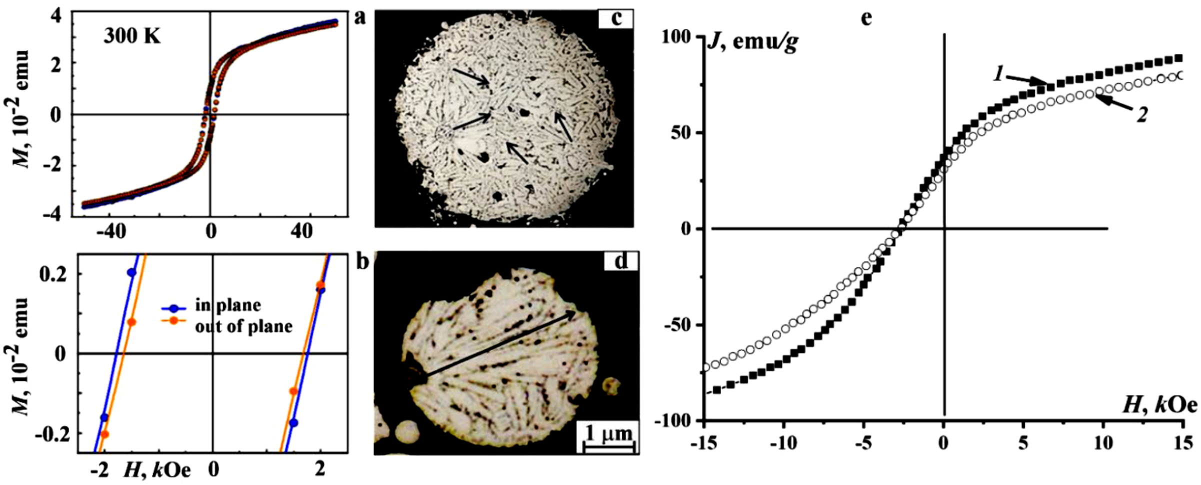

Fig. 1. Anisotropy of hysteresis properties of Fe-Nd-B GA-alloys: *a*, *b* - SQUID-magnetometry of $Fe_{78}Nd_{15}B_7$ alloy (No.4) in the longitudinal and transverse directions (*a* – powder on a silicon substrate obtained by laser cladding in a magnetic field perpendicular to the substrate surface; *b* – central part of the loop with color highlighting of property measurement data along and across the orientation of the powders); *c*, *d* – metallographic structure of the GAP alloy of the same composition; *e*, *f* – demagnetization curves of the sample (as a powder suspension, 20 g) on a vibrating magnetometer at room temperature: *e* – alloy No. 4, *f* - alloy No. 855 (identical to No. 4 in Nd and B), additionally alloyed (Mo+Ti+V+Al+In+Ga+Bi+Sc ≤ 2 at. %) [1]

Using X-ray and neutron diffraction methods, it was shown that the main phase in the studied GAP alloys is the $Fe_{14}Nd_2B$ boride (tetragonal phase T_1), space group $P4_2/mnm$ or $I\bar{4}3d$. Its quantity reaches 95±2 vol.%. Neutron diffraction analysis demonstrated the presence of magnetic, and therefore grain anisotropy in the GA-alloys. Thermal cycling in hydrogen environment near T_c increases this magnetic and grain anisotropy effect (by approximately 10%).

[1] V.V.SAVIN et al. (2025). Powder materials of REM-containing alloys obtained by gas atomization of the melt for laser surfacing technologies and additive processes. Materials Science Forum, 1151, 76-88.