MAGNETIC FORCE MICROSCOPY OF THE DOMAIN STRUCTURE OF HIGH-COERCIVITY (Nd-R)-Fe-B (R = Pr, Ce, Dy, Tb, Ho) PERMANENT MAGNETS

1938 ИМЕТ РАН

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The properties of Nd-Fe-B magnets are determined, along with the magnet composition, by the 2-14-1 phase grain size and alignment, presence of impurity phases, complex 2-14-1-phase grain structure (core-shell), width of Nd-rich phase layers, and the magnetic structure that should be studied along with the phase composition and microstructure of the magnets. The relationship between the grain structure and magnetic structure plays the crucial role upon magnetization and magnetization reversal.

The aim of the present paper is, using MFM, to study the domain structure at the surfaces perpendicular and parallel to the alignment axis of sintered Nd–Fe–B permanent magnets, which have the well-formed core-shell structure prepared in using REM hydrides as the constituent of powder mixtures. The domain structures are the characterization of the high performance and imperfection of the sintered magnets.

EXPERIMENTAL

The (R, R')-Fe-B magnets with R and R' = Nd, Pr, Dy, Tb, Ho were prepared by blending powder technique using (Nd,Pr,Ce)-Fe-B powders and rare-earth (Dy, Tb, Ho) hydrides as the constituents of the powder mixture (see Table, where [1]- DOI:10.1134/S2075113318030115 , [2] - DOI: 10.1134/S207511331303009X , [3] - DOI: 10.1063/1.5129896).

All samples (1-6) were prepared by traditional powder metallurgy technology that includes the hydrogen decrepitation of strip-casting alloys, milling of powder mixture with hydride additions in a ball mill; the milled powders were aligned in a magnetic field and pressed in air, sintered, and heat-treated. The heat treatment of sample 1 prepared with DyH_{2-3} included complete restoration of hysteretic properties after degradation of them with low-temperature annealings. The other magnets were studied in the thermally demagnetized state.

The hysteretic characteristics of the sintered Nd-Fe-B-based magnets under study are summarized in Table.

Figures 1-6 show the domain structure of magnet samples 1-6.

Hysteretic characteristics of sintered permanent magnets

Sample no. / hydride addition MeH_{2-3} (wt %)	B_r, T	$_jH_c$, kA/m	$_bH_c$, kA/m	H_k, kA/m	$(BH)_{max}$, kJ/m^3	Reference
1 / 2% DyH_{2-3}	1.29	1309	981	1262	322	[1]
2 / 4 % TbH_{2-3}	1.22	1900	930	1760	286	[2]
3 / 3% HoH_{2-3} (Pr-based)	1.11	1512	804	891	223	[3]
4 / 4% Tb-Co-Cu (Ce-containing)	1.114	1700	853.76	1530	236	This work
5 / 0.5% Tb-Co-Cu	1.299	1163.7	975.86	1094.8	328	This work
6 / (Ce-containing)	1.2327	875.46	831.11	798.66	292	This work

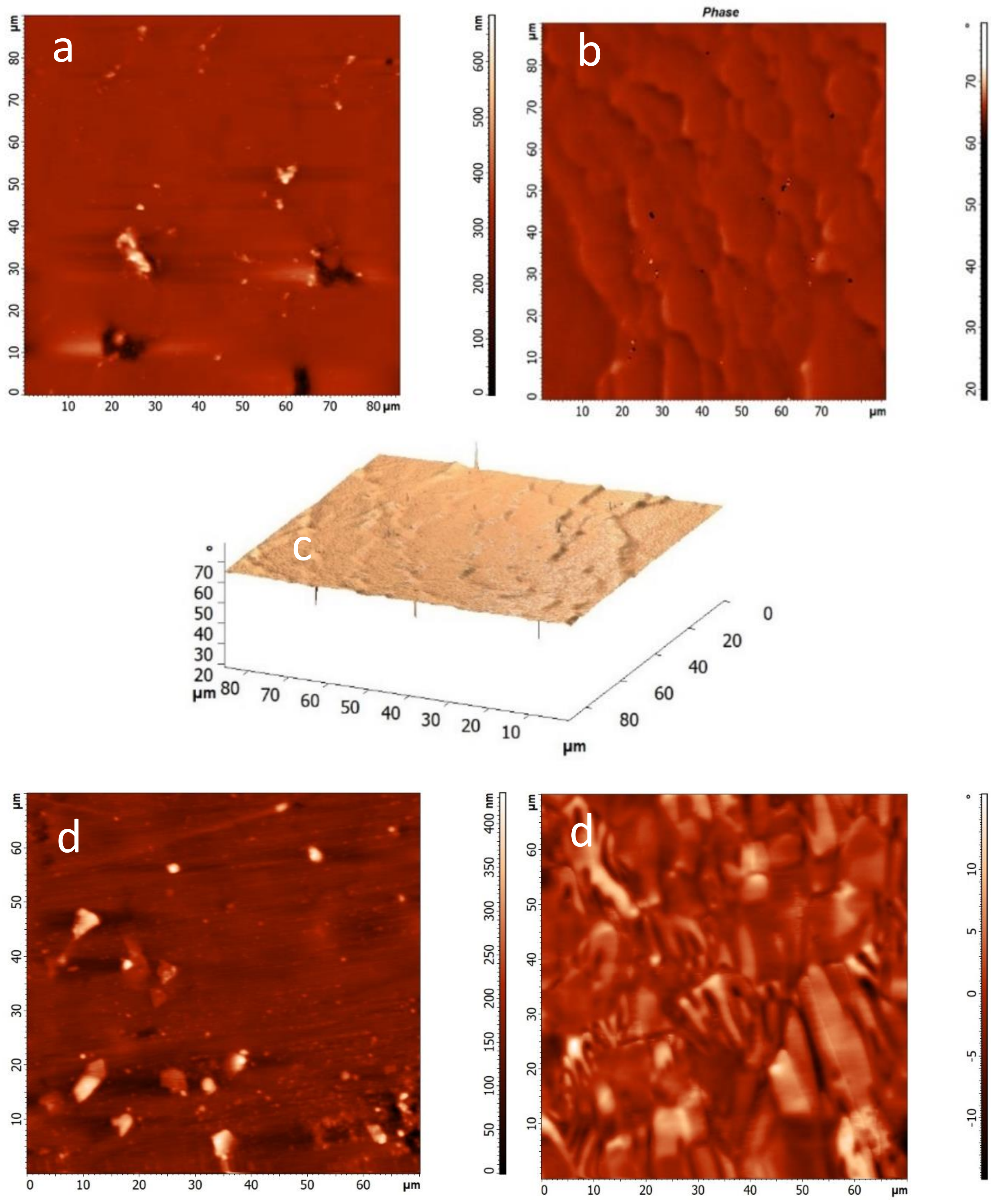
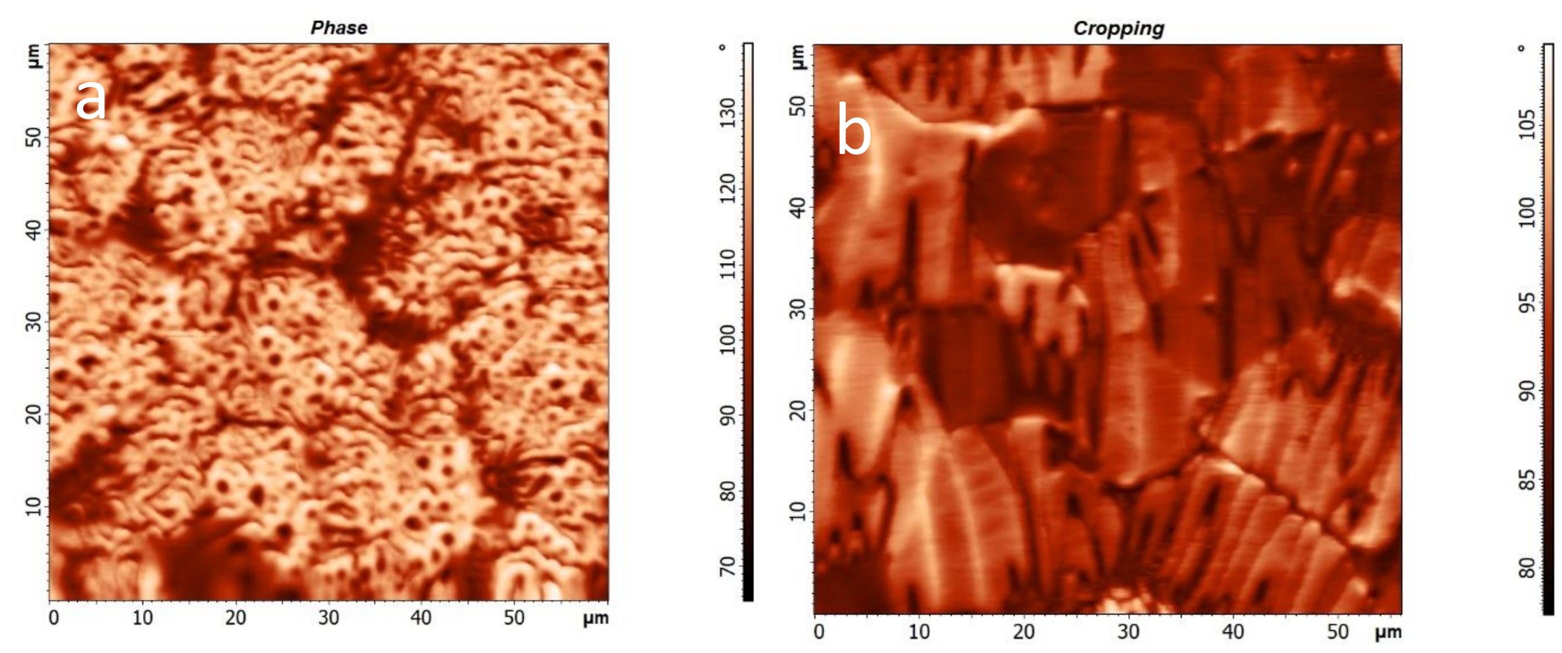


Fig. 1. AFM images (height and phase contrast modes) of sample 1 (a, b, c) perpendicular (almost single-domain grains) and (d) parallel to the magnet texture axis (strip domains)



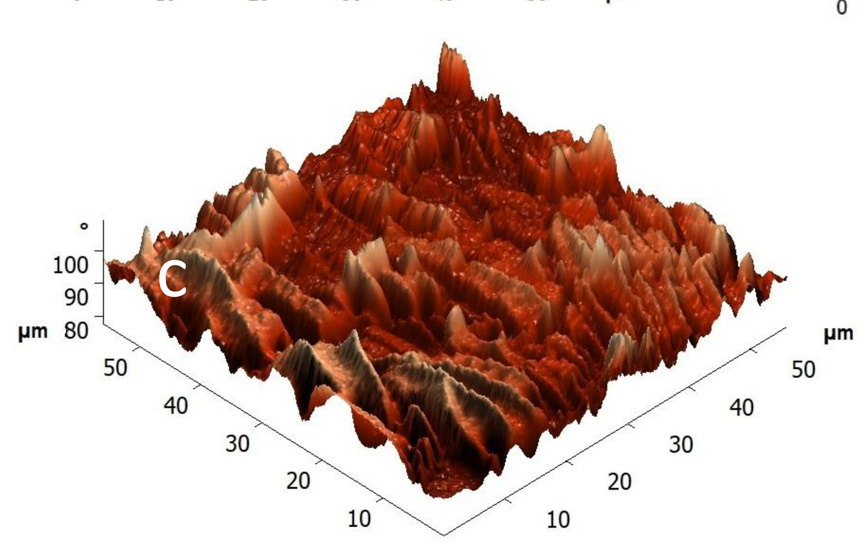


Fig. 2. AFM images (phase contrast mode) of sample 2 (a) perpendicular (maze-like and spike domains) and (b, c) parallel (strip domains) with 3D image (c) to the magnet texture axis

RESULTS AND DISCUSSION

Samples 1-3 have the obvious core-shell structure [1,2,3]. It can be seen for sample 1 (Fig. 1a) as darker areas "cores" between white inclusions corresponding to triple junctions (white inclusions) of main-phase grains. The contrast of the areas is due to the different magnetic moment of the Dy-learned (core) and Dy-enriched (shell) areas.

The surface of the sample 1 exhibits the absence of maze-like structure (Fig. 1 b,c) at the surface perpendicular to the magnet texture axis and looks like the structure with single-domain grains. It is apparently to be true for majority of grains of the structure formed after the used complex heat treatment. 3D images of the domain structure (Fig. 1c) demonstrates the absence of domains (individual reverse spike domains are observed, which correspond to inclusions). The domain structure observed for the magnet at the surface parallel to the texture axis demonstrates the existence of strip domains often comparable in size with the grain size. Some grains observed in areas abundant with inclusions demonstrate the multidomain structure (Fig. 1e), the appearance of which is typical of the surface parallel to the magnet texture axis; however, the grain boundaries are not obviously observed. Such a domain structure is likely to be result from the used complex heat treatment.

The MFM image (Fig. 2) of the domain structure of Sample 2 demonstrates well-formed maze-like structure (on a scale of grains size), which is typical of high-anisotropy materials, with clearly observed spike domains (smaller domains within the main domains) at the surface perpendicular to the texture axis. Spike domains are dominant. Maze domains are bright and domain walls are dark. Spike domains are observed in the form of circles.

Stripe domains (Fig. 2) with straight domain walls parallel to the magnetization direction demonstrate the grain orientation (Fig. 2). The better magnetic alignment of grains, the higher the magnetization produced. At the same time, magnetic misalignment between grains leads to an increase in coercivity and a decrease in the remanent magnetization Mr with a reduction in the maximum energy product (BH)max.

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Sample 3 (Pr-Ho-Fe-B) has well observed maze-like structure at the surface perpendicular to the magnetic texture axis and strip domain structure with extended domain walls at the surface parallel to the axis; areas of maze-like structure alternate with spike domain areas (Fig. 3a, b). Both the images demonstrate the slightly imperfect alignment of grains. The magnetic domains at the grain boundaries seem to be terminated (Fig. 3b): some domain walls pass through several grains. The sample 3 is characterized by sufficiently low both remanence B_r and the hysteresis loop squareness H_k (Table).

The domain structure of sample 4 prepared with Ce and Tb-Co-Cu addition mainly is represented by spike and reverse spike domains within sufficiently small grains (~10 μm) (Fig. 4). Areas near triple junctions and grain boundaries correspond to lattice diffusion of Tb and Co. Copper ensures the improvement isolation of main magnetic phase grains and smoothing the grain boundaries. It is likely that just these areas mainly demonstrate the maze-like domain structure observed near grain boundaries. This magnet (sample 4) has the high coercive force (Table) among the samples under study.

Sample 5 is Ce-free magnet but also prepared with Tb-Co-Cu addition (0.5%). It is characterized by high remanence and magnetic energy product and enhanced coercive force. Its domain structure (Fig. 5a) is represented by spike domain structure and maze-like structure (that is not pronounced). The image (Fig. 5) taken allows us to compare the domain structure with the microstructure of the surface perpendicular to the texture axis. The multi-domain grain size that is 5-10 μm.

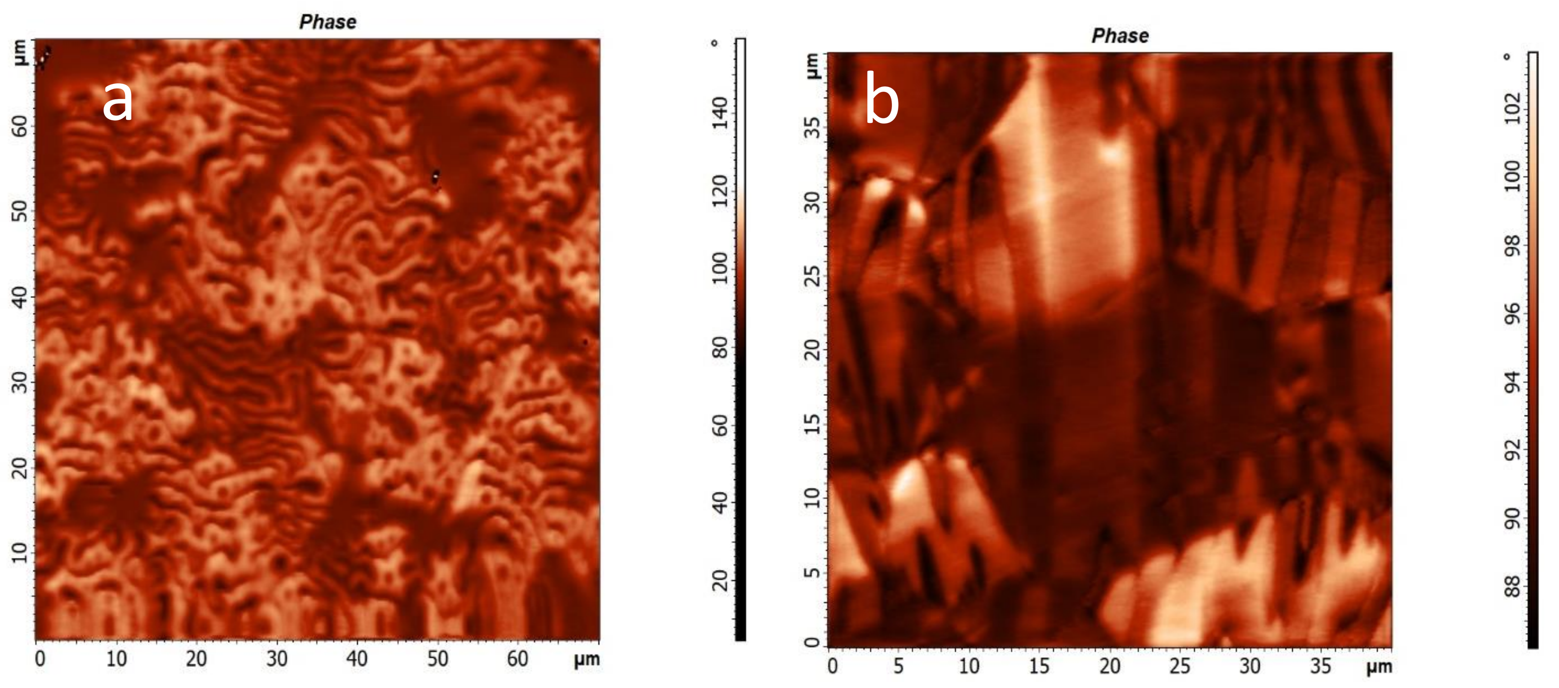

Fig. 3. AFM images (phase contrast mode) of sample 3 (a) perpendicular and (b) parallel to the magnet texture axis

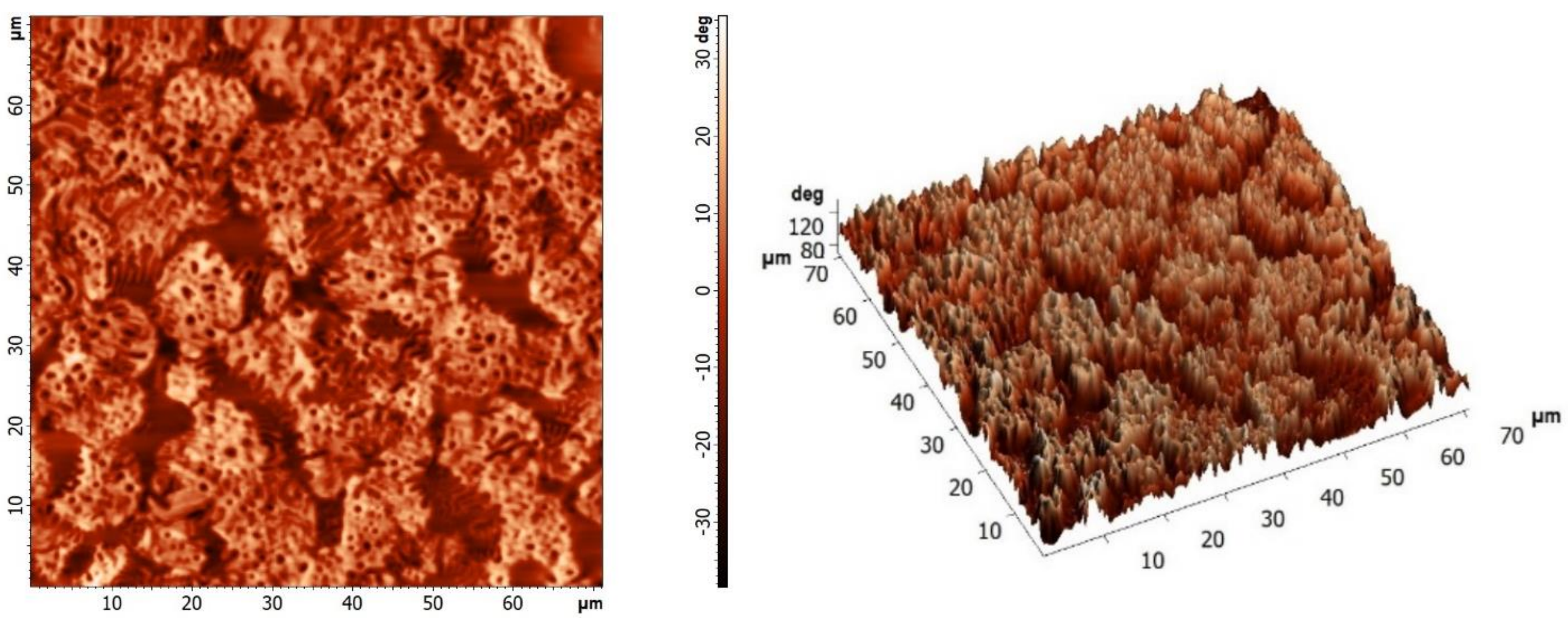

Fig. 4. AFM images (phase contrast mode + 3D view) of sample 4 perpendicular to the magnet texture axis

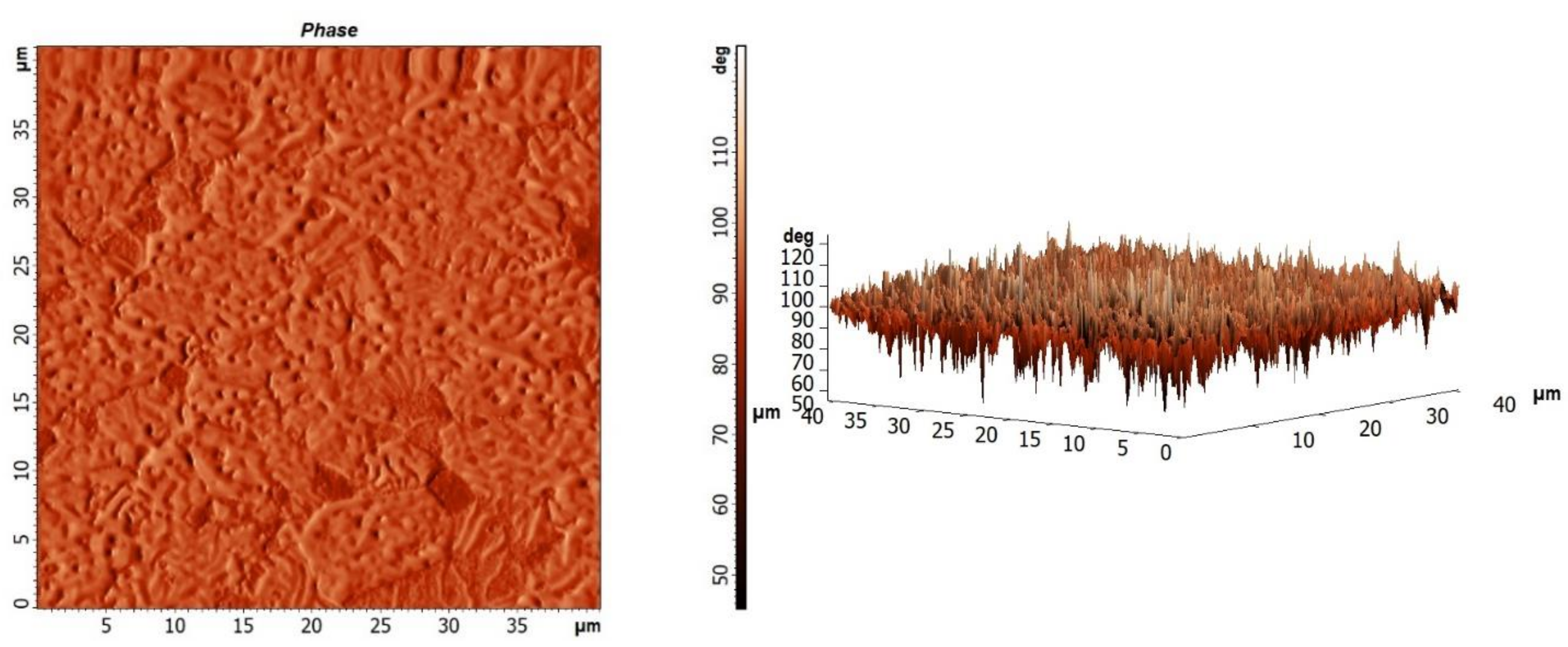

Fig. 5. AFM images (phase contrast mode + 3D view) of sample 5 perpendicular to the magnet texture axis

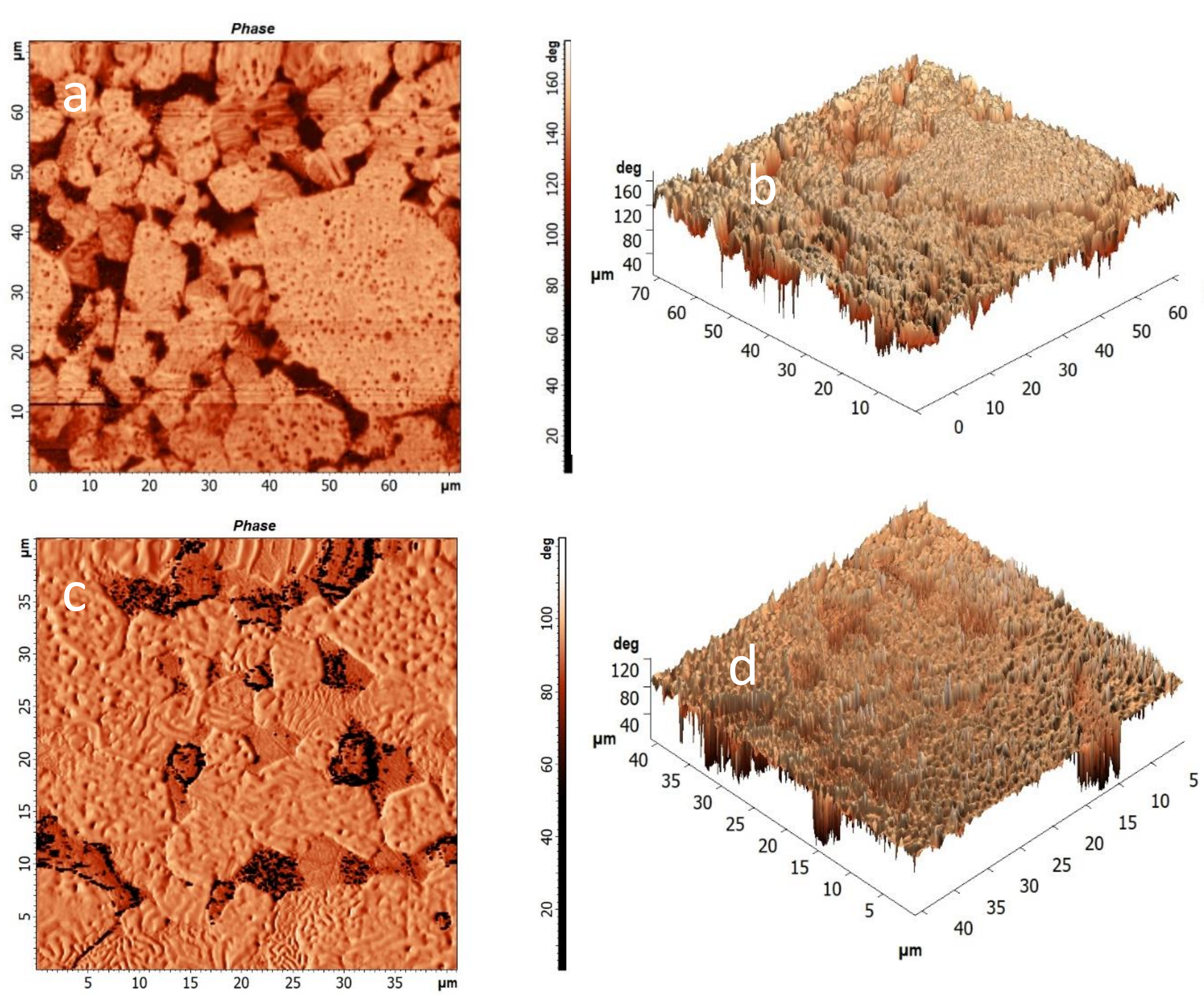

Fig. 6. (a,b,c,d) AFM images (phase contrast mode + 3D view) of sample 6 perpendicular to the magnet texture axis (different areas are shown)

Sample 5 has the high hysteretic characteristics, high density (7.502 g/cm3), and a hysteresis loop squareness ratio of 91.2%.
Sample 6 was prepared with Ce and without additions increasing its coercive force. Its domine structure irregular, grain misorientation takes place (grains with spike domains and stripe domains are observed). Grain sizes vary within a range of 5-25 μm. The structure is abundant with secondary phases (see Fig. 6b). Reverse spike domains are observed to be corresponding also to tripe junctions.

CONCLUSIONS

The MFM microscopy was used to study the domain structure of the Nd,Pr, Ce (Dy, Tb, Ho)-Fe-B magnets differing in composition. MFM images of the domain structure of the magnets were processed with Nova software to analyze domain structure micro-peculiarities. The maze-like structure with spike domains and strip domains typical of surface perpendicular and parallel to the alignment axis is observed, respectively. 3D views of the domain structure are used as images giving the more information about it. The formation of reverse spike domains related to both spike domains and inclusion of other phases was demonstrated. Features of the domain structure of Nd-Fe-B magnets, the properties of which were first degraded and after that restored by complex heat treatments, are studied for the first time. The formation of near single-domains state of grains is demonstrated.

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