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# FEATURES OF THE NANOSTRUCTURE FORMATION IN THE ND-PR-FE-B-SYSTEM ALLOYS: A STUDY BY AFM AND MFM

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**Abstract.** In this work, polycrystalline samples of hard-magnetic materials of $(Nd_{1-x}Pr_x)_2Fe_{14}B$ general formula have been prepared and subjected to severe plastic deformation (SPD). Thereafter the features of nanostructure formation in the compounds were investigated before and after SPD using atomic force microscopy and magnetic force one (AFM & MFM). The differences in texture formation were revealed when studying the surface microstructure of both the initial and deformed samples. The initial ones prepared by the Czochralski method, exhibited the columnar structure, while the SPD samples exhibited the concentric rings containing elongated nanosized crystallites. The magnetic domain structure was visualized using the MFM that clearly demonstrated the relationship between the microstructural features and the magnetic domains' configuration.

**Keywords:** atomic force microscopy, magnetic force microscopy, hard-magnetic material, nanostructure

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# ОСОБЕННОСТИ ФОРМИРОВАНИЯ НАНОСТРУКТУРЫ В СПЛАВАХ СИСТЕМЫ ND-PR-FE-B, ИССЛЕДОВАННЫЕ МЕТОДАМИ АТОМНО-СИЛОВОЙ И МАГНИТНО-СИЛОВОЙ МИКРОСКОПИИ

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**Аннотация.** В работе получены и исследованы на микро- и наноуровне поликристаллические образцы магнитотвердых материалов общей формулы $(Nd_{1-x}Pr_x)_2Fe_{14}B$, подвергнутые процедуре интенсивной пластической деформации (ИПД). Для контроля особенностей формирования наноструктуры соединений до и после ИПД использовались методы атомно-силовой и магнитно-силовой микроскопии (АСМ и МСМ). При изучении поверхностей как исходных, так и деформированных образцов были выявлены различия в текстурообразовании. Для исходных образцов, полученных с помощью метода Чохральского, оказалась характерной столбчатая структура, тогда как после процедуры ИПД структура состояла из концентрических колец, которые содержали вытянутые наноразмерные кристаллиты. Структура магнитных доменов была визуализирована с помощью метода МСМ, который четко продемонстрировал связь между особенностями микроструктуры и конфигурацией магнитных доменов.

**Ключевые слова:** атомно-силовая и магнитно-силовая микроскопия, магнитотвердый материал, наноструктура, доменная структура

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## Introduction

Developing new high-tech devices including computers, motors and generators, magnetic resonance imaging, etc. [1–3] generates great demand for high-energy hard magnetic materials capable of operating stably at various temperatures. Special attention is focused on $(Nd,Pr)_2Fe_{14}B$ materials [1–3]. It is known that permanent magnets based on $Nd_2Fe_{14}B$ compound show the best properties in the narrow temperature range of 150–400 K [4, 5]. The highest experimentally obtained energy product is 475 kJ/m3 (59.6 MGsOe) [9], which is close to the predicted theoretical limit $(BH)$max for the $Nd_2Fe_{14}B$ compound. Moreover, at $T$ = 135 K, the magnetic anisotropy type of $Nd_2Fe_{14}B$ compound changes from "easy axis" to "cone of easy magnetization axes", in other words, a spontaneous spin-reorientation transition (SRT) occurs with decreasing temperature [6, 7]. The observed SRT leads to remanence and energy product decreasing, which limits the applicability of the material based on the $Nd_2Fe_{14}B$ compound at low temperatures. Partial substitution of Nd by Pr allows decreasing of SRT temperature expanding working temperature range of the magnets based on $(Nd,Pr)_2Fe_{14}B$ compounds. It should be noted that the level of properties of magnets strongly depends on microstructure peculiarities. For example, it is known that magnetic properties can be increased by nanostructure formation [8,9]. Alloys with nanocrystalline structure could be obtained by various methods, both conventional (powder metallurgy [10,11] strip casting [12, 13], severe plastic deformation [14–18], etc., and their combination [9, 14, 19–21]). There are also new modern additive methods for the production of highly efficient magnetic materials, including magnetic materials [22–24]. Regardless of the chosen method, the mechanisms of magnetic properties formation and their dependence on structural features are mostly the same. Thus, the methods of structural analysis of nanostructured materials can be applied to samples obtained by any of the above methods, including modern advanced methods, such as selective laser melting (SLM) of Nd-Fe-B materials [25–27]. The SLM process is characterized by high cooling rates ($10^6$ K/s [28]) and rapid solidification, which can lead to the formation of nanostructures.

It should be noted that the development of the modern high-energy permanent magnets based on $R$-Fe-B ($R$ = Nd, Pr) is possible in case of proper distribution of rare-earth reach phase along with optimal microstructure and texture formation. The presence of random oriented or elongated grains within the microstructure, as a rule, decreases final magnetic characteristics. That is why deep analysis and understanding of microstructure formation is needed to reach new results in the Nd-Fe-B system qualitatively.

The present work aims to investigate $(Nd,Pr)_2Fe_{14}B$ nanostructure formation peculiarities during severe plastic deformation (SPD) using atomic and magnetic force microscopy. The work is carried out as a continuation of early studies [18, 29], which develop methods and approaches to the analysis of nanostructured magnetic materials. These approaches are planned to be applied in further studies for Nd-Pr-Fe-B samples obtained, for example, by selective laser melting.

## Experimental details

$(Nd_{1-x}Pr_x)_2Fe_{14}B$ ($x$ = 0.5 and 0.75) alloys were synthesized by a modified Czochralski method in a tri-arc furnace under an inert atmosphere [30]. As a result, polycrystalline samples with a directed crystal structure, as well as small single crystals, were obtained. It is polycrystals that we used in this work for further research. The SPD process was performed using Bridgman anvils under 6 GPa pressure and anvils' rotation (3 full turns). SMENA-A scanning probe microscope on the Solver platform (by NT-MDT, Russia) was used for atomic and magnetic force microscopy (AFM and MFM, respectively) investigations. Samples were investigated in the semi-contact method at room temperature using HA_NC ETALON standard silicon cantilevers with resonance frequencies from 110 to 235 kHz and 10 nm radius of curvature of the tip of the needle. MFM was performed using MFM 01 cantilevers with Co coating with 50–85 kHz frequencies. Elemental analysis was performed using scanning electron microscopy (SEM) TESCAN Vega 3 (Tescan Analytics, Fuveau, France).

## Results

Previous investigation [30] showed that $(Nd_{1-x}Pr_x)_2Fe_{14}B$ solidifies with tetragonal crystal structure ($P4_2/mnm$ space group) with $Nd_2Fe_{14}B$ type. The main phase in samples varied from 86 to 98 %. The high content of α-Fe (up to 13 %) was found in alloy with $x = 0.75$. Content of iron within grains was around 70.41 wt.% which corresponds to the $Nd_2Fe_{14}B$ phase, while iron content at grain boundaries was far lower (30–130 times less). Elements such as Cu and Ni were present mainly at the grain boundaries and were considered by us as impurities in the boundary region, while oxygen was present mainly in the inclusions. Thus, a small amount of oxygen found in inclusions rich in iron indicates the presence of oxides in the alloy structure.

Fig. 1 shows surface structures of $(Nd_{1-x}Pr_x)_2Fe_{14}B$ with $x = 0.5$ before and after the SPD procedure. These samples contain a minimum amount of the second phase thus are the most interesting for research. The columnar structure may be seen in Fig. 1,*a* associated with the samples' synthesis method. This method is usually used to grow large single crystals; however, in this work, as noted above, the single crystal structure was not predominant. However, the directional heat dissipation inherent in this method has led to the formation of some uniaxial texture, which may appear as a columnar structure. The columns had a width of about 0.8 μm and were primarily perpendicular to the investigated surface. The surface structure of the initial sample consisted of slightly elongated subgrains (with an aspect ratio of about 1.5) with the size of 100 - 200 nm order. It is the high temperature gradient (and hence the high rates of cooling and crystallization) that can explain the formation of such small structural elements on the surface of the initial sample during synthesis. It should also be noted that the observed inclusions, which consist mainly of iron atoms, should crystallize first due to the higher melting point. Thus, iron inclusions act as nucleation centers for main phase grains.

The surface microstructure of $Nd_1Pr_1Fe_{14}B$ after the SPD procedure differs significantly (see Fig. 1,*b*). Crystallite agglomerations with a „swirling vortex" shape are seen wherein the columnar structure still exists. It is better observed in Fig. 1,*c*, which shows another surface area of the same sample.

Similar microstructure peculiarities in $(Nd,Pr)_2Fe_{14}B$-type alloys after strip casting were mentioned in [31]. To better visualize the vortex structure in direction perpendicular to the surface and detect its shape and typical sizes and typical sizes of the main structural elements, the samples were polished and etched using a 5% solution of nitric acid in ethyl alcohol. Images of such polished and etched surfaces are shown in Fig. 2. It can be seen that the swirling vortex consists of concentric rings set with a 150–300 nm thick (in one layer of crystallites) closely adjacent to each other. One can see crystallites of various sizes and shapes, oriented mainly along the vortex circumferences.

Possible reasons for such a swirling vortex structure are different deformation speeds and uneven heating of sample parts during the SPD procedure. Observed microstructure peculiarities may also be considered as a rounded induced texture because of deformation.

MFM method was applied to visualize magnetic domain structure on the samples' surface. Fig. 3 shows the MFM image of the $Nd_1Pr_1Fe_{14}B$ sample surface before and after the SPD procedure to show the difference arising from the deformation. A double domain structure was observed: striped domains with 5-10 μm width on the surface of the sample before SPD (Fig. 3,*a*) and with 5-7 μm width in case of the sample after SPD (Fig. 3,*b*); superimposed on these stripes a classical homogeneous branched domain structure on the alloy surface before SPD with a domain width of 1–3 μm (Fig. 3,*c*) and a more complex homogeneous structure on the alloy surface after SPD (Fig. 3,*d*) with a domain width of 1–2 μm are seen.

The observed complex domain structure of the sample after SPD (Fig 3,*b* and *d*) could arise because of a much more inhomogeneous microstructure and the possible presence of some amount of amorphous phase. Amorphous phase existence after SPD procedure was shown in [9,14]severe plastic deformation (SPD, and its volume content may exceed 50%, making it the dominant phase in the structure. The observed "dimple" domain structure means that the basal plane of most crystallites coincides with the surface of the sample before the SPD procedure confirms the texture. The initial sample shows mostly equiaxed domains (dark areas in Fig. 3,*a*), and the evenly dark fill of rounded domains means the single domain state of the corresponding crystallites.

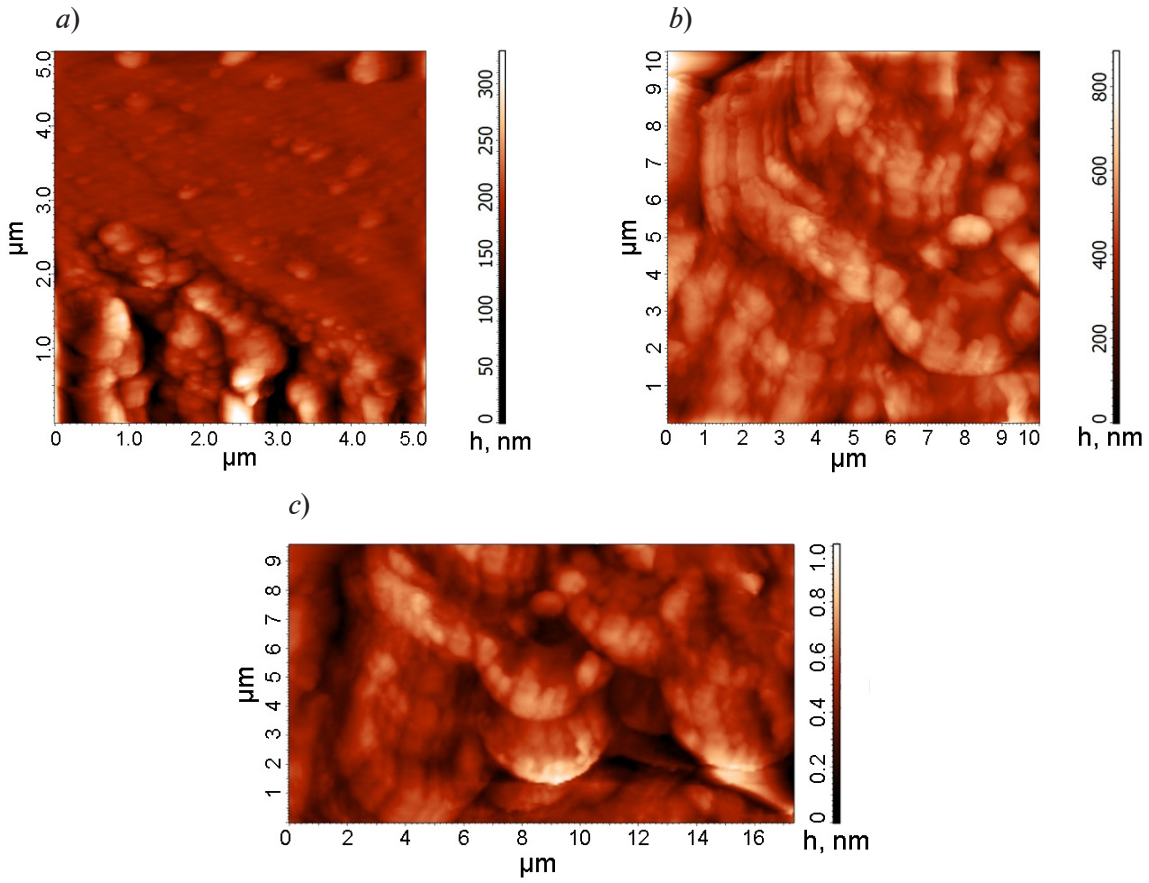


Fig. 1. AFM image for surface microstructure of $Nd_1Pr_1Fe_{14}B$ before (*a*) and after (*b*, *c*) severe plastic deformation; *c* shows another fragment of the sample surface (*b*) where swirling vortex structures are the most pronounced

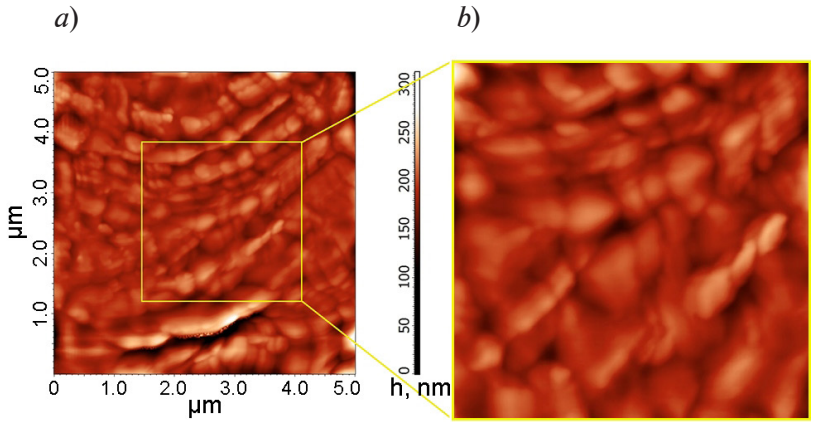


Fig. 2. AFM image for surface microstructure of $Nd_1Pr_1Fe_{14}B$ after SPD (see Fig. 1, c), surface polishing and acid etching (*a*); enlarged fragment (*b*)

Analysis of MFM images of samples after the SPD procedure does not give the same unambiguously interpretable domain structure as in the case of initial samples, which makes it difficult to describe the shape of crystallites, to identify foreign phases, inclusions, etc. However, it is quite obvious that the SPD procedure violates the texture of the initial samples and, most importantly, disorients the structural elements.

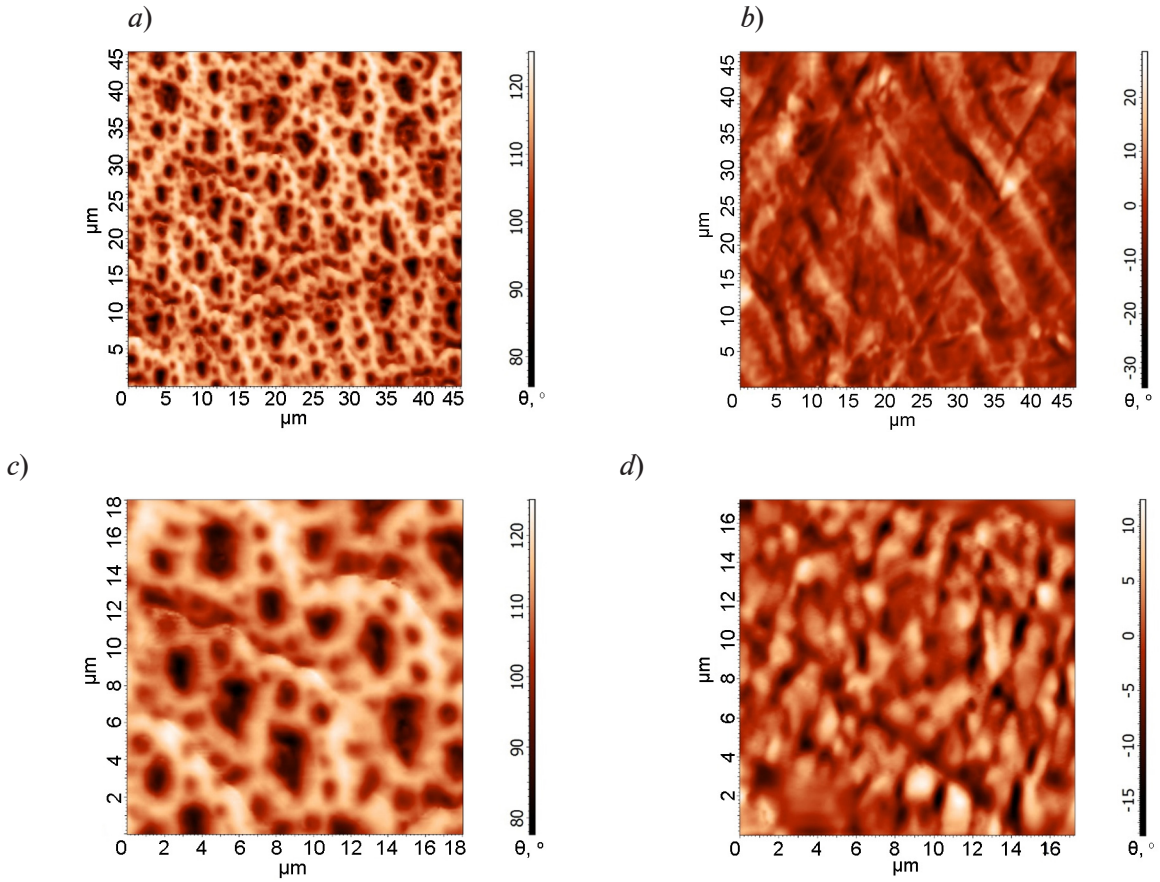


Fig. 3. MFM images for double domain structure of $Nd_1Pr_1Fe_{14}B$ sample surface before (*a*, *c*) and after SPD (*b, d*); the figure shows striped domains (*a*, *b*), classical branched domain structure (*c*) and more complex homogeneous structure (*d*)

## Conclusions

Samples of the $(Nd,Pr)_2Fe_{14}B$ type were synthesized to study the formation of their surface morphology in the initial state and after severe plastic deformation. The main phase of the initial samples was a tetragonal phase of the $Nd_2Fe_{14}B$ type with a certain amount of α-Fe, which is typical for the materials under study. The lowest content of α-Fe (up to 2%) was found in the $Nd_1Pr_1Fe_{14}B$ alloy, while in the $Nd_{0.5}Pr_{1.75}Fe_{14}B$ alloy the amount of the second phase reached 13%. The columnar structure (consisting, in turn, of rounded grains about 100–200 nm in size) was characteristic of the original $Nd_1Pr_1Fe_{14}B$ sample. On the contrary, for the samples after the SPD procedure, agglomerations in the form of a "swirling vortex" (sets of concentric rings 150–300 nm thick, consisting of elongated nanosized crystallites) were found.

Conducting the MFM study and domain structure visualization, on the one hand, confirmed the AFM analysis of the microstructure; on the other hand, they demonstrated the main features of the emerging structure of magnetic domains. While the original sample has a classical "dimple" domain structure with single-domain crystallites, the sample after the SPD procedure has a complex "smeared" domain structure, which can be explained both by the features of the microstructure and the appearance of an amorphous phase after severe plastic deformation.

The study demonstrates the convenience and informativeness of using the AFM and MFM methods in studying the surface of nanocrystalline hard magnetic materials, in particular, the $(Nd,Pr)_2Fe_{14}B$ materials considered in this work, and also makes it possible to reveal the main features of the formation of their structure before and after severe plastic deformation. AFM and MFM studies of the surface morphology of alloys make it possible to obtain information about the sizes of all the main structural elements and their shape at the nanolevel, about the domain structure, and make it possible to reveal differences in texture formation depending on the methods of obtaining alloys and their further processing, which is extremely important for the production of highly efficient magnetically hard materials and products from them for various functional purposes.

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