



UDC 621.762

<https://doi.org/10.17073/1997-308X-2024-5-5-12>Research article
Научная статья

Structure and magnetic properties of strontium hexaferrite powder after milling in a beater mill in a magnetoliquefied layer followed by annealing

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Abstract. This study examines the effectiveness of processing dispersed strontium hexaferrite material in a beater mill within a magnetoliquefied layer formed by magnetic fields – an inhomogeneous alternating field with a frequency of 50 Hz and an induction gradient of 90 mT/m, and a constant field with an induction of 15.3 mT – under conditions where milling is accompanied by particle aggregation. The magnetic field lines are mutually perpendicular and parallel to the plane of the milling bodies. A comprehensive investigation of the changes in the dispersed composition and structural characteristics of the strontium hexaferrite powder with increased milling duration was conducted using scanning electron microscopy and X-ray diffraction analysis. The results show that processing the strontium hexaferrite dispersed system with an initial average particle size of 1558.5 μm in a magnetoliquefied layer for 120 min does not alter the phase composition of the powder. However, milling reduces the average particle size to 0.57 μm , decreases the size of the coherent scattering regions, increases the lattice microstrain of the $\text{SrFe}_{12}\text{O}_{19}$ phase, and raises the dislocation density. Magnetic properties of the powder samples before and after annealing were studied using a vibrating sample magnetometer at room temperature and normal atmospheric pressure. The conducted research allows for the assessment of the technological outcomes of processing the dispersed system in a magnetoliquefied layer, considering the collective effects that accompany milling.

Keywords: powder metallurgy, strontium hexaferrite, mechanical milling, X-ray diffraction analysis, scanning electron microscopy, vibrating sample magnetometer

For citation: Egorov I.N., Egorov N.Ya., Rusalev Yu.V., Egorova S.I. Structure and magnetic properties of strontium hexaferrite powder after milling in a beater mill in a magnetoliquefied layer followed by annealing. *Powder Metallurgy and Functional Coatings*. 2024;18(5):5–12. <https://doi.org/10.17073/1997-308X-2024-5-5-12>

Структура и магнитные свойства порошка гексаферрита стронция после измельчения в бильной мельнице в магнитоожигенном слое с последующим отжигом

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Аннотация. В работе рассмотрена эффективность обработки дисперсного материала гексаферрита стронция в бильной мельнице в магнитоожигенном слое, образованном в магнитных полях – неоднородном переменном с частотой 50 Гц, градиентом индукции 90 мТл/м и постоянном с индукцией 15,3 мТл – в условиях, когда измельчение сопровождается агрегацией частиц. Линии магнитной индукции полей взаимно перпендикулярны и параллельны плоскости вращения бил. Методами растровой электронной микроскопии и рентгеноструктурного анализа проведено комплексное исследование особенностей изменения дисперсного состава и структурных характеристик порошка гексаферрита стронция при увеличении продолжительности измельчения. Показано, что при обработке дисперсной системы гексаферрита стронция со средним размером частиц 1558,5 мкм в магнитоожигенном слое в течение 120 мин не происходит изменения фазового состава порошка, измельчение приводит к уменьшению среднего размера частиц порошка до 0,57 мкм, снижению размера областей когерентного рассеяния, увеличению микродеформации решетки фазы $\text{SrFe}_{12}\text{O}_{19}$ и плотности дислокаций. С помощью вибрационного магнитометра при комнатной температуре и нормальном атмосферном давлении изучены магнитные характеристики порошковых образцов до и после отжига. Проведенные исследования позволяют оценить технологический результат обработки дисперсной системы в магнитоожигенном слое с учетом совокупности основных явлений, сопровождающих измельчение.

Ключевые слова: порошковая металлургия, гексаферрит стронция, механическое измельчение, рентгеноструктурный анализ, растровая электронная микроскопия, вибрационный магнитометр

Для цитирования: Егоров И.Н., Егоров Н.Я., Русалев Ю.В., Егорова С.И. Структура и магнитные свойства порошка гексаферрита стронция после измельчения в бильной мельнице в магнитоожигенном слое с последующим отжигом. *Известия вузов. Порошковая металлургия и функциональные покрытия*. 2024;18(5):5–12. <https://doi.org/10.17073/1997-308X-2024-5-5-12>

Introduction

Hexagonal ferrites are widely used in powder metallurgy for the production of sintered magnets and magnetoplasts [1; 2]. Compared to barium hexaferrite magnets, strontium ferrite magnets provide superior magnetic properties [3–6]. The quality of the magnetic properties of strontium hexaferrite powder is influenced by the granulometric composition and the degree of uniformity in particle size within the dispersed system [7; 8].

With the increase in the production of permanent magnets, there is also a rise in the amount of waste generated during various technological operations, which needs to be recycled. The recycling process of magnet production waste or used sintered magnets involves stages of milling and powder production [9; 10]. The most common methods for producing fine and ultrafine powders of ferromagnetic materials are wet and dry milling [11–14]. There are also methods

to enhance the milling process efficiency by transferring the milled material into a fluidized state [15–17].

The primary goal in producing strontium hexaferrite powder is to obtain a dispersed system with a specified set of physical-technological and structural characteristics. The development of high-performance equipment for milling ferromagnetic materials is of great importance, as the duration of the milling process and the specific energy consumption are crucial economic factors. The efficiency of dispersing can be influenced by changing the conditions under which milling is carried out, particularly by applying alternating inhomogeneous and constant magnetic fields to the dispersed ferromagnetic material in a beater mill, with the magnetic field lines being mutually perpendicular and parallel to the plane of the milling bodies' rotation [18]. Under electromagnetic influence, a magnetoliquefied layer is formed from the dispersed material, where particles and aggregates move within an inhomogeneous alternating magnetic field, ensuring the principle of mul-

multiple returns of particles and aggregates to the area of the rotating milling bodies and significantly increasing their collision frequency with the milling bodies. Milling of dispersed media in a magnetoliquefied layer requires additional energy consumption for powering the electromagnets. As the induction of the constant field and the gradient of the alternating magnetic field increase, the energy consumption for power supply also rises. However, only when the induction of the constant magnetic field reaches 15.3 mT and the gradient of the alternating magnetic field reaches 90 mT/m is there intense movement of coarse ferromagnetic particles. The process of reducing particle size in the magnetoliquefied layer in the beater mill is observed only with the intense movement of the dispersed system's particles. Due to the significant reduction in dispersion time in the magnetoliquefied layer, energy consumption is reduced [19].

When processing dispersed substances in any milling apparatus, the resulting powder not only has a specific granulometric composition but also various structural defects that increase the particles' reactivity. In producing fine powders of strontium hexaferrite in powder metallurgy, high demands are placed on both the dispersed composition and the structural characteristics of the dispersed system [20]. Therefore, studying the milling process is necessary to determine effective operating conditions for the apparatus in terms of changing the powder's dispersed composition and structural characteristics.

The aim of this work was to study the effect of mechanical processing of a coarse-grained strontium hexaferrite system in a beater mill in a magnetoliquefied layer on the granulometric composition, structural characteristics of the powder, and its magnetic properties before and after annealing.

The tasks were carried out using research methods such as scanning electron microscopy, X-ray diffraction, and magnetic measurements of powder samples.

Experimental

To conduct the research, coarse strontium hexaferrite material with an average particle size of 1558.5 μm , a most probable size of 1420 μm , a dispersion of 497 μm , and a median of 1476.9 μm was milled in a beater mill, where the milling bodies rotated at a frequency of 15.0 ± 1.6 thousand rpm in a horizontal plane. During the milling of magnetic materials in a beater mill, two processes occur: particle fragmentation due to their interaction with the milling bodies and particle aggregation. As the particle size of the milled dispersed material decreases, their tendency to aggregate increases, and the milling intensity significantly

decreases. When the dispersed system is processed in a beater mill in a magnetoliquefied layer, the intensity of movement of the ferromagnetic elements and the frequency of their collisions with the rotating milling bodies increase. In the magnetoliquefied layer of ferromagnetic dispersed systems, under certain electromagnetic conditions, the destruction of aggregates occurs [21; 22]. To form a magnetoliquefied layer from the dispersed system in the mill, which ensures the deflocculation of ferromagnetic elements and the return of the powder to the area of the rotating milling bodies, the material was subjected to magnetic fields: an alternating field with a frequency of 50 Hz and an induction gradient of 90 mT/m, as well as a constant field with an induction of 15.3 mT.

The study of the patterns of change in the dispersed composition and structural characteristics during the milling of the initial dispersed strontium hexaferrite system was carried out by analyzing samples taken after 10, 30 and 120 min of milling.

The granulometric composition of the powder samples was analyzed using a Supra 25 scanning electron microscope (Carl Zeiss, Germany). The structural characteristics of the milled dispersed material were investigated using X-ray diffraction analysis on a XRD-7000 diffractometer (Shimadzu, Japan) with CuK_α radiation (wavelength $\lambda = 1.5406 \text{ \AA}$) and Bragg-Brentano focusing (θ – 2θ). Given the complex appearance of the $\text{SrFe}_{12}\text{O}_{19}$ phase diffraction pattern, the PowderCell software version 2.3, based on the Rietveld full-profile refinement method, was used to process the diffraction spectra of the powder samples [23; 24]. The ICSD database was used for analyzing and refining the structural characteristics. X-ray measurements were conducted at room temperature in step-scan mode (step size 0.03°) over the angular range of $2\theta = 5^\circ$ – 90° . The exposure time at each step was 8 s. The profile R-factor was selected as the criterion for good agreement between the structural model and the actual experiment.

The magnetic properties of the strontium hexaferrite powder samples were investigated using a LakeShore vibrating sample magnetometer (USA) at room temperature and normal atmospheric pressure. The magnetic field measurements were conducted with a precision of 0.1 Oe. The hysteresis loops were recorded in the "Continuous" mode to ensure consistent and detailed measurement of the magnetic behavior.

Results and discussion

The average particle size after 10, 30 and 120 min of processing the dispersed system in the beater mill was 12.48 μm , 1.09 μm and 0.57 μm , respectively.

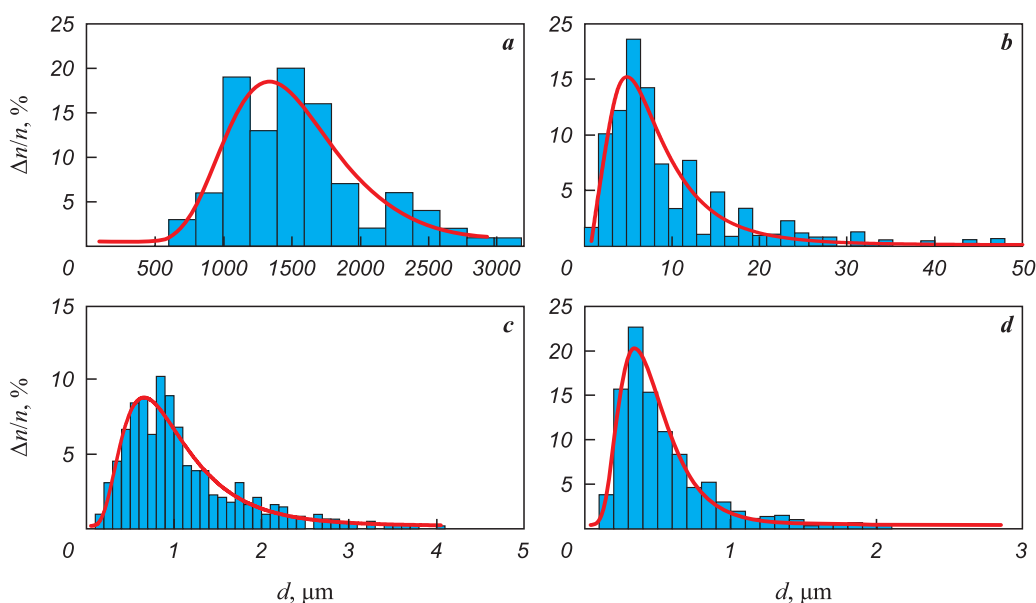


Fig. 1. Histograms and logarithmic-normal distribution functions of strontium hexaferrite powder particle sizes
a – initial powder, *b–d* – after milling for 10 min (*b*), 30 min (*c*), 120 min (*d*)

Рис. 1. Гистограммы и функции логарифмически нормального распределения порошка гексаферрита стронция по размерам
a – исходное состояние; *b–d* – после измельчения в течение 10 мин (*b*), 30 мин (*c*), 120 мин (*d*)

The experimental results indicate that after 10 min of milling, the degree of milling (the ratio of the average particle size of the initial dispersed system to the average particle size of the milled product) was 124.9. In the subsequent 20 min, the degree of milling decreased to 11.5 and after 90 min, it significantly reduced to 1.9. The rate of particle size reduction during the first 10 min of milling was 154.6 $\mu\text{m}/\text{min}$, during the following 20 min, it was 0.57 $\mu\text{m}/\text{min}$, and during the further 90 min, it was only 0.006 $\mu\text{m}/\text{min}$.

Thus, the most intense milling of the strontium hexaferrite dispersed material occurs in the first 10 min of milling. This result is consistent with studies showing that the strength of the particles and the work required for their fragmentation increase as the particle size decreases [25–27].

From the histograms and functions of the logarithmic-normal distribution of strontium hexaferrite powder particle sizes shown in Fig. 1, it is evident that for the initial dispersed material (Fig. 1, *a*), the most probable size is 1337.23 μm , and the width of the function maximum at half-height is 968.16 μm . After 10, 30 and 120 min of milling in the beater mill in a magnetoliquefied layer, the most probable particle sizes were 4.93 μm , 0.67 μm and 0.34 μm , respectively, and the width of the maximum at half-height of the particle size distribution function decreased to 7.68 μm , 0.91 μm and 0.40 μm , respectively (Fig. 1, *b–d*). The morphological features of the strontium hexaferrite powder particles after 120 min of milling in

the magnetoliquefied layer are shown in Fig. 2. Thus, after 30 min of milling, the homogeneity of the dispersed composition increases significantly.

The experimental results show that the process of milling the initial strontium hexaferrite dispersed system in a beater mill in a magnetoliquefied layer can be divided into three stages. During the first stage, lasting 10 min, the most intense milling occurs. In the second stage, which lasts 20 min, milling is less intense, but the homogeneity of the dispersed system distribution increases. In the third stage, lasting 90 min, the milling

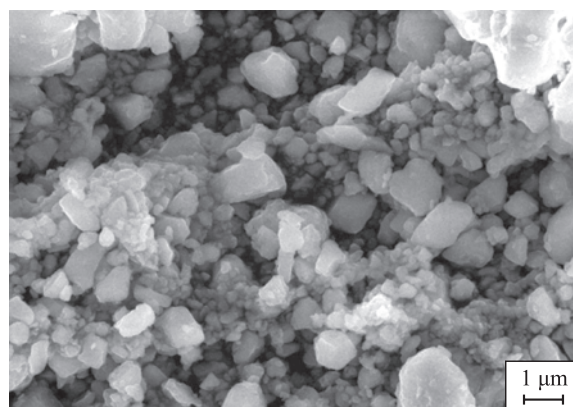


Fig. 2. Morphological features of strontium hexaferrite powder particles after 120 min of milling in a magnetoliquefied layer

Рис. 2. Морфологические особенности частиц порошка гексаферрита стронция после 120 мин измельчения в магнитооживленном слое

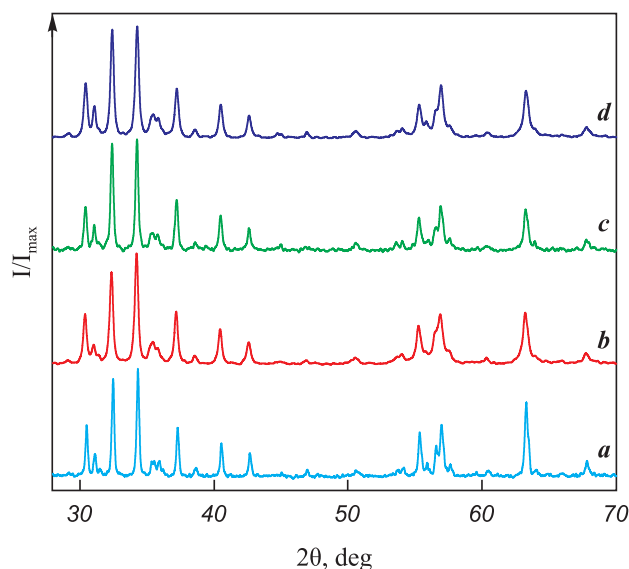


Fig. 3. X-ray diffraction patterns of the relative intensity of strontium hexaferrite dispersed material samples

a – initial powder, *b–d* – after milling for 10 min (*b*), 30 min (*c*), 120 min (*d*)

Рис. 3. Рентгенограммы относительной интенсивности образцов дисперсного материала гексаферрита стронция

a – исходное состояние; *b–d* – после измельчения в течение 10 мин (*b*), 30 мин (*c*), 120 мин (*d*)

intensity decreases significantly, but the homogeneity of the dispersed system increases substantially.

However, milling leads to the accumulation of crystal structure defects, which reduces the magnetic properties of the powder. It is evident that the most complete information about changes in the structural characteristics of the milled material can be obtained by studying the diffraction profiles of the X-ray patterns of powder samples of the initial dispersed material and after milling for $\tau = 10, 30$ and 120 min (Fig. 3). The duration of processing the dispersed material in the mill in a magnetoliquefied layer affects the sizes of coherent

scattering regions (CSR), the magnitude of additional relative microstrains $\Delta d/d_{hkl}$ (Δd being the average change in interplanar spacing d_{hkl} due to the presence of defects), and the dislocation density ρ .

According to X-ray diffraction analysis data (Fig. 3), milling the coarse strontium hexaferrite material in a beater mill for $\tau = 120$ min in a magnetoliquefied layer does not lead to a change in the phase composition.

Fig. 4 presents histograms showing changes in the sizes of the coherent scattering regions (D), lattice microstrains ($\Delta d/d$), and dislocation density (ρ) for samples of the initial strontium hexaferrite dispersed material and after processing in the mill for $\tau = 10, 30$ and 120 min. For the initial dispersed material, the CSR size was $D = 309.4$ Å, the dislocation density was $\rho = 3.13 \cdot 10^{11} \text{ cm}^{-2}$, and the relative deformation was $\Delta d/d = 3.49 \cdot 10^{-4}$. As the milling time increases, the size of the crystallites, determined from the analysis of the broadening of diffraction lines, decreases, while the lattice microstrain of the $\text{SrFe}_{12}\text{O}_{19}$ phase and the dislocation density increase. However, during the processing of the dispersed system in the magnetoliquefied layer, the intensity of changes in the structural characteristics of the powder varies. In the first stage of milling ($\tau = 10$ min), there is an intense reduction in particle size, but the contribution of defect formation processes is small. As seen in Fig. 4, during this stage of processing the initial strontium hexaferrite dispersed system, the change in lattice microstrain $\Delta d/d$ increased by $1.06 \cdot 10^{-4}$, the dislocation density ρ increased by $0.21 \cdot 10^{11} \text{ cm}^{-2}$, and the CSR size D decreased by 9.9 Å. In the second stage of processing the dispersed system for 20 min in the mill, the milling intensity decreases, and the defect formation process intensifies. During this stage, the change in lattice microstrain increased by $\Delta d/d = 7.50 \cdot 10^{-4}$, the dislocation density increased by $\rho = 1.15 \cdot 10^{11} \text{ cm}^{-2}$, and

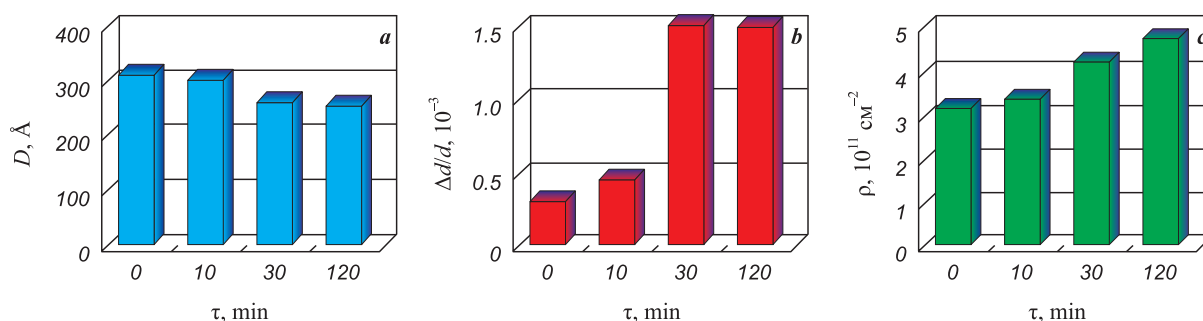


Fig. 4. Changes in the size of coherent scattering regions D (*a*), lattice microstrains $\Delta d/d$ (*b*) and dislocation density ρ (*c*) for samples of the initial dispersed strontium hexaferrite material and after milling with different durations (τ)

Рис. 4. Изменение размера областей когерентного рассеяния D (*a*), микроискажений кристаллической решетки $\Delta d/d$ (*b*), плотности дислокаций ρ (*c*) для образцов исходного дисперсного материала гексаферрита стронция и после обработки в мельнице с различной длительностью (τ)

the CSR size decreased by 41.0 Å. In the third stage of milling, lasting 90 min, the $\Delta d/d$ value increased by $1.27 \cdot 10^{-4}$, the ρ value increased by $0.21 \cdot 10^{11} \text{ cm}^{-2}$, and the CSR size D decreased by 5.96 Å.

The experimental results indicate that the most intense particle size reduction process during the treatment of the dispersed system in a magnetoliquefied layer occurs within the first 10 min, with the degree of milling reaching 271.2 at this stage. The most significant increase in lattice microstrain and dislocation density is observed between 10 and 30 min, during which the average size of the dispersed system changed from 12.48 μm to 1.09 μm . Between 30 and 120 min of milling, the intensity of the milling and defect formation processes decreases, but the homogeneity of the dispersed system increases significantly.

After 120 min of milling the initial strontium hexaferrite dispersed system, the average particle size was 0.57 μm , with a median of 0.46 μm , and the values of D , $\Delta d/d$ and ρ were 252.54 Å, $13.32 \cdot 10^{-4}$ and $4.70 \cdot 10^{11} \text{ cm}^{-2}$, respectively.

The magnetic properties of bulk samples made from strontium hexaferrite powder, processed in a beater mill in a magnetoliquefied layer for 120 min, before and after annealing can be evaluated from the magnetic hysteresis loops shown in Fig. 5. To restore the crystalline structure of the sample, annealing was performed for 3 h at a temperature of 950 °C.

The results of the magnetic studies on the sample made from powder processed in a beater mill in a magnetoliquefied layer for 120 min showed that the saturation magnetization M_s was 60.032 $\text{G} \cdot \text{cm}^3/\text{g}$, the remanent magnetization M_r was 29.991 $\text{G} \cdot \text{cm}^3/\text{g}$, and the coercive force H_c was 1656.6 Oe. As a result of annealing, the magnetic properties of the strontium hexaferrite powder improved, with the saturation magnetization,

remnant magnetization, and coercive force of the sample increasing by 8.7, 43.7 and 64.3 %, respectively.

Conclusion

The experimental studies conducted allow us to observe the changes in the granulometric composition, crystallite size of the phases, lattice microstrains, and dislocation density during the production of $\text{SrFe}_{12}\text{O}_{19}$ powder through milling in a magnetoliquefied layer. It was established that processing the dispersed system of coarse strontium hexaferrite material for 120 min in a beater mill within an alternating inhomogeneous magnetic field with a frequency of 50 Hz and an induction gradient of 90 mT/m, along with a constant field with an induction of 15.3 mT, does not alter the phase composition of the powder. However, it leads to a significant reduction in particle size, a decrease in the size of coherent scattering regions, and an increase in the lattice microstrain of the $\text{SrFe}_{12}\text{O}_{19}$ phase and the dislocation density.

It has been shown that during milling, the intensification of changes in the granulometric composition and structural characteristics of the dispersed system of the material being processed significantly decreases once the average particle size reaches 1 μm . Further milling results in a substantial reduction in the intensity of changes in the granulometric composition of the material processed in the mill, while the homogeneity of the dispersed system increases significantly.

According to studies conducted using a vibrating sample magnetometer, the magnetic characteristics of the sample made from the powder obtained after milling – namely, remanent magnetization, saturation magnetization, and coercive force – were determined. The magnetic properties of the sample increased as a result of annealing.

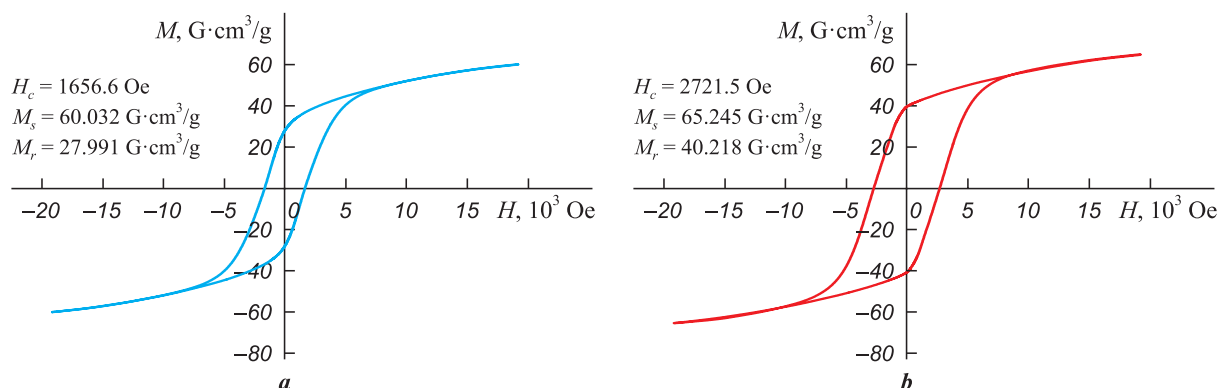


Fig. 5. Hysteresis loops of bulk samples of strontium hexaferrite powder after milling in a magnetoliquefied layer for 120 min (a) before annealing and (b) after annealing

Рис. 5. Магнитные петли гистерезиса объемных образцов порошка гексаферрита стронция после измельчения в магнитоожигенном слое в течение 120 мин до отжига (a) и после отжига (b)


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Н. Я. Егоров – проведение экспериментов по измельчению порошков, участие в обсуждении результатов.

Ю. В. Русалев – исследование магнитных свойств порошков.

С. И. Егорова – участие в обсуждении результатов, написание текста статьи.

Received 26.11.2023

Revised 24.01.2024

Accepted 29.01.2024

Статья поступила 26.11.2023 г.

Доработана 24.01.2024 г.

Принята к публикации 29.01.2024 г.