Tonkie Khimicheskie Tekhnologii = Fine Chemical Technologies.
2025;20(3):264–275 ISSN 2686-7575 (Online)

Chemistry and technology of inorganic materials

Химия и технология неорганических материалов

UDC 621.318.12
https://doi.org/10.32362/2410-6593-2025-20-3-264-275
EDN QSOBYJ

RESEARCH ARTICLE

Formation of the microstructure and properties of strontium hexaferrite magnets using powder injection molding

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Abstract

Objectives. The study set out to investigate the possibility of production strontium hexaferrite permanent magnets using powder injection molding (PIM) technology, which involves casting granules highly filled with ceramic powder. After obtaining the initial granulate based on organic binders and strontium hexaferrite powder, the material was cast in an injection molding machine to create the first intermediate (*green*) parts, followed by removal of the primary binder to obtain *brown* parts and final sintering.

Methods. Strontium hexaferrite powder was obtained by the ceramic method. The material underwent grinding in a planetary ball mill to obtain a powder having an average particle size of 13.4 µm, which is considered optimal for the applied PIM technology. Granulate materials, consisting of the obtained strontium hexaferrite powder combined with primary paraffin and secondary polyamide binders, were prepared by manual mixing of the components and used for creation of green parts in injection molding machine. Brown parts obtained following removal of binder from the obtained green parts were characterized by their higher brittleness and open pore structure. Permanent magnets with dimensions of 10 × 10 × 5 mm were obtained following sintering of brown parts in an oxidizing atmosphere.

Results. The more than 70% higher strength of the magnetic properties of the obtained strontium hexaferrite samples compared to isotropic barium hexaferrite-based magnets manufactured in accordance with GOST 24063-80 is due to the presence of pores after sintering.

Conclusions. The possibility of using the ceramic method for producing strontium hexaferrite powder for use in granulate manufacturing was demonstrated. This raw material can then be used to obtain strontium hexaferrite permanent magnets via PIM technology having 80% density.

Keywords
permanent magnet, strontium hexaferrite, PIM technology, granulate, microstructure, magnetic properties

Submitted: 06.10.2024
Revised: 17.01.2025
Accepted: 02.04.2025

For citation
Chernyshev B.D., Schetinin I.V. Formation of the microstructure and properties of strontium hexaferrite magnets using powder injection molding. *Tonk. Khim. Tekhnol. = Fine Chem. Technol.* 2025;20(3):264–275. https://doi.org/10.32362/2410-6593-2025-20-3-264-275

НАУЧНАЯ СТАТЬЯ

Формирование структуры и свойств магнитов на основе гексаферрита стронция, полученных с помощью технологии Powder Injection Molding

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Аннотация

Цели. Изучить возможность получения постоянных магнитов на основе гексаферрита стронция с помощью технологии Powder Injection Molding (PIM), заключающейся в литье гранулятов, высоконаполненных керамическим порошком. Данный процесс состоит из операций получения гранулята (исходного сырья на основе органического связующего и порошка гексаферрита стронция), литья гранулята в термопластавтомате для создания первых промежуточных («зеленых») деталей, последующего удаления связки из них, получения «коричневых» деталей и финального спекания.

Методы. Порошок гексаферрита стронция получен керамическим методом. Материал прошел стадию помола в планетарной шаровой мельнице до получения порошка со средним размером частиц 13.4 мкм, который считается оптимальным размером для PIM-технологии. На основе полученного порошка гексаферрита стронция, первичного связующего — парафина и вторичного — полиамида методом ручного смешивания компонентов подготовлен гранулят для создания «зеленых» деталей. Полученные детали подвергли операции удаления связующего — дебиндингу, в результате которого изготовили «коричневые» заготовки, отличающиеся более высокой хрупкостью и наличием структуры открытых пор. Постоянные магниты с размерами 10 × 10 × 5 мм получены методом спекания «коричневых» деталей в окислительной атмосфере.

Результаты. Уровень магнитных параметров образцов на основе гексаферрита стронция составил более 70% от значений, характерных для промышленных изотропных магнитов на основе гексаферрита бария в соответствии ГОСТ 24063-80, что обусловлено наличием пор в спеченных изделиях.

Выводы. Установлена возможность применения керамического метода для производства порошка гексаферрита стронция, который может быть использован при изготовлении гранулята. Использование данного сырья позволяет изготавливать магниты методом PIM-технологии с плотностью не менее 80%.

Ключевые слова

постоянный магнит, гексаферрит стронция, PIM-технология, гранулят, микроструктура, магнитные свойства

Поступила: 06.10.2024
Доработана: 17.01.2025
Принята в печать: 02.04.2025

Для цитирования

Чернышев Б.Д., Щетинин И.В. Формирование структуры и свойств магнитов на основе гексаферрита стронция, полученных с помощью технологии Powder Injection Molding. *Тонкие химические технологии*. 2025;20(3):264–275. https://doi.org/10.32362/2410-6593-2025-20-3-264-275

INTRODUCTION

Many areas of contemporary technical development rely on the use of hard magnetic materials. These materials are used to produce permanent magnets (PMs), which are used to create diverse products for special and general purposes, including electric motors, generators, sensors, acoustic systems and medical devices. Under industrial conditions, such materials are widely used in magnetic grippers, lifting mechanisms, mixers, and various kinds of sensor.

The strict quality requirements and continuous improvement of special-purpose products impose a number of limitations on magnetically hard materials for use in the creation of PMs. These include high operating—and consequent Curie point—temperatures, as well as higher values of tensile strength σ_B, relative elongation δ, and corrosion resistance. PMs based on rare-earth materials (REM) of Sm–Co system operate at temperatures of about 350–550°C. The class of magnetically hard materials based on alloys of

Fe–Cr–Co systems is characterized by the presence of an effective combination of magnetic parameters (residual induction B_r = 1.1 T and coercive force by induction H_C^B = 38 kA/m) and mechanical properties (tensile strength σ_B = 785 MPa and relative elongation δ = 3%) [1]. The achievement of a high level of magnetic properties of these types of PM is generally preceded by a stage of prolonged thermal and thermomagnetic processing.

Of all the PM materials produced onto the global market, Nd–Fe–B (neodymium) alloys have the highest magnetic energy product (the product of induction B and coercive force H $(BH)_{max}$), allowing the dimensions of manufactured equipment to be maximally reduced. This can be especially relevant in the production of various consumer goods. However, the significant disadvantages of materials of this system included its low operating temperatures (200–300°C, depending on the brand) as well as the high complexity of the production process, which is associated with the high activity of REMs [2, 3]. In view of these facts, a significant segment of the PM market (more than 25%) is occupied by products based on barium and strontium hexaferrites, which have a relatively low magnetic energy product compared to the Nd–Fe–B system, but are characterized by increased corrosion and chemical resistance. At the same time, the cost of raw materials for the production of ferrite PM is significantly lower as compared to REM-based materials, as well as those based on Co [4].

Strontium hexaferrite is a promising material for hyperthermic processes, which are only applicable in the case of nanosized particles [5]. In [6], particles having grain sizes of the order of 30–40 nm were obtained by using the sol–gel method. Nanoscale compositions can also be obtained by solution combustion of organo-nitrate precursors [7], synthesis from oxide glasses [8], self-propagating high-temperature synthesis [9], and hydrothermal methods [10]. However, the classical approach for obtaining strontium hexaferrite powders used in the creation of PMs is still the ceramic method, which consists of the operations of ferritization and grinding of the material to particles with the required fraction at the level of 1–10 μm [11].

PMs based on basic magnetically hard materials, among which the Nd–Fe–B, Sm–Co, Sr–Fe–O systems are distinguished, are generally produced using powder metallurgy methods [12, 13]. A number of materials based on Fe–Cr–Co and Al–Ni–Co alloys are obtained by investment casting [14]. The formation of a highly coercive state of these alloys is achieved in the process of spinodal decomposition of the main phase into strongly and weakly magnetic phases through the use of thermomagnetic processing technology. The use of these approaches is typical for the organization of large-scale production of magnets having predominantly simple shapes (ring, sector, etc.). However, miniaturization and complication of designs of existing devices and magnetic systems can complicate the product geometry [15]. When changing the configuration using traditional technological approaches, machining methods are applied. Their use not only requires the availability of facilities and a fairly large fleet of equipment, but also leads to a sharp decrease in the material utilization factor (MUF) to the level of about 40%. In order to reduce the costs of magnet manufacturing, the resulting large quantities of grinding waste containing expensive rare-earth metals must be re-extracted and reintroduced into the production cycle [16].

Powder injection molding (PIM) technologies are being widely introduced to increase MUF by reducing the need for machining. The PIM method is based on the pressing of products from a granulate consisting of an organic binder highly filled with metal or ceramic powder. Further, the pressed intermediate parts undergo binder removal (debinding) and sintering stages [17]. Using this approach, the production of complex configurations weighing up to 1 kg is ensured, allowing MUF to be increased to values of about 97–99% [18]. Consequently, it is rational to produce permanent magnets based on rare-earth metals using PIM technology.

Since this method is expedient for the production of large batches of products, it is possible to use additive manufacturing technologies to develop processes and obtain unique magnets on a laboratory scale. The results of using the selective laser melting method to create magnetic systems based on several materials have already been obtained [15]. The main disadvantage of selective laser melting is the cost of PM printing equipment, as well as high requirements as to the purity and size distribution of initial powders. The main competitor of this technology is stereolithography, which is successfully used to create ceramics by printing photopolymers highly filled with ceramic powders [19]. The process of adding the powder composition into the photopolymer, which is similar to the processes that take place during the preparation of granulates for PIM technology, can be performed using different methods for mixing the starting materials. Due to the high fluidity of photopolymers, this process does not require heating [20].

The aim of the present study is to investigate the microstructure and magnetic properties of powders based on strontium hexaferrite and permanent magnets obtained using PIM technology.

EXPERIMENTAL METHOD

Strontium hexaferrite powder was produced by solid-phase synthesis. At the first stage, initial components comprising hematite and strontium carbonate with a purity no less than 99.5 wt % were mixed in the Turbula mixer of the *Techno-Center* company (Russia), after which the synthesis of the required phase of strontium hexaferrite was carried out at 1200°C for 5 h. The obtained powder was milled in a planetary ball mill (*Techno-Center*, Russia) for at least 5 h. Control of the average size of powder compositions was carried out using an Analysette 22 MicroTec plus laser particle size analyzer (*Fritsch*, Germany). Analysis of magnetic properties of powder compositions was carried out using a VSM-250 vibromagnetometer (*Changchun*, China), which can analyze materials in fields up to 2 T.

The obtained strontium hexaferrite powder compositions were processed into a granulate to which a binder based on paraffin, polyamide, and additional technological additives was added manually and using an industrial granulator. Compacting of granulates was carried out on a thermoplastic automatic machine at the softening temperature of the binder. Investigation of the presence of internal defects of green parts was carried out using the tomography method.[1]

Removal of the primary paraffin-based binder from the intermediate parts was carried out by solution debinding methods. Acetone, hexane, and perchloroethylene (*Ekos-1*, Russia) were used as the main solvents. The change in the mass of parts and the amount of removed binder during debinding was determined using M-ER 123 ACFJR-600.01 Sensomatic TFT scales (*Mertech Equipment*, South Korea).

Sintering of the following intermediate parts, which are referred to as brown body samples, was carried out in an oxidizing environment in a PMV-1600p muffle furnace (*Bossert*, Russia) at a temperature not lower than 1210°C for 2 h. After binder removal and sintering, the samples had the shape of a parallelepiped with dimensions of 10 × 10 × 5 mm. The density of magnets after sintering was determined using a helium pycnometer (*Micromeritics*, USA).

The microstructure of the strontium hexaferrite powders, granulate, intermediate parts and sintered magnets was analyzed using TM-3000 scanning electron microscope (SEM) by *Hitachi* (Japan) and FEI quanta 200 F Feg 250 SEM (*FEI*, USA) with EDAX energy dispersive analyzer (*Octane Elect*, USA). Phase analysis was performed using a DRON-4 X-ray diffractometer (*Burevestnik*, Russia). Processing and analysis of the obtained data was carried out using a PDXL specialized software package (Rigaku[2]) and PDF-2 database[3]. Quantitative phase analysis was performed using the Rietveld method. Magnetic hysteresis loops of permanent magnet samples were measured using a MN-50 hysteresisgraph (*Walker Scientific Inc.*, USA). Chemical analysis of the samples was performed using an iCAP 6300 inductively coupled plasma atomic emission spectrometer (*Thermo Fisher Scientific*, USA). A LECO SC844 carbon and sulfur analyzer (USA) was used to study the contamination of permanent magnets with organic binder.

RESULTS AND DISCUSSION

According to the SEM analysis, the particle sizes of the powder, which passed the synthesis stage at 1200°C for 5 h, do not exceed 3–4 µm; the particles themselves often have the shape of hexagonal prisms (Fig. 1). However, in the process of high-temperature synthesis, sintering of the powder occurred with the formation of large agglomerates.

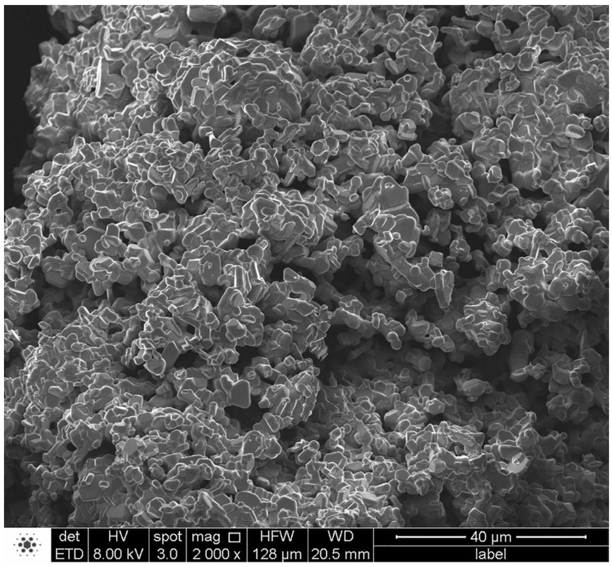

Fig. 1. Strontium hexaferrite microstructure after sintering during 5 h at temperature of 1200°C

[1] The names and manufacturers of the industrial pelletizer, thermoplastic automatic machine, tomograph, as well as the name and properties of the binder are trade secrets and cannot be published in the article.

[2] https://rigaku.com/. Accessed March 11, 2025.

[3] https://www.icdd.com/pdf-2/. Accessed March 11, 2025.

Since the use of the obtained material for obtaining products by PIM and stereolithography methods is impossible due to the obvious coarseness of the particles, their size should be reduced. For this purpose, fine grinding of the obtained materials was carried out. A snapshot of the microstructure of strontium hexaferrite powder after fine grinding is presented in Fig. 2.

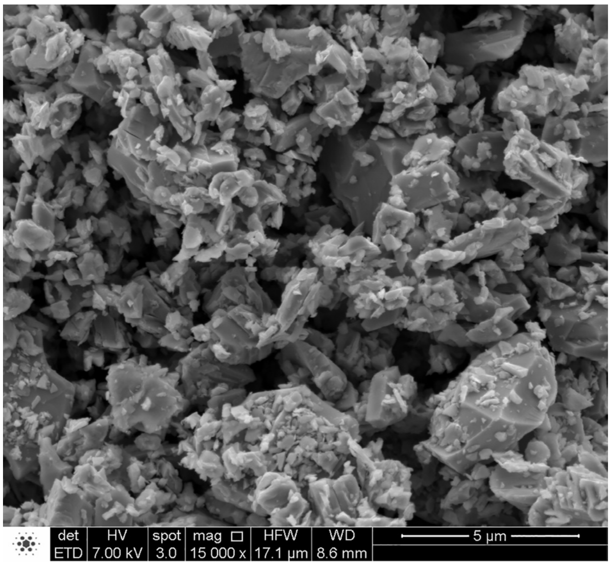

Fig. 2. Microstructure of strontium hexaferrite after grinding in a planetary ball mill

The shape of the powder based on strontium hexaferrite is predominantly splintered as a consequence of the material grinding process. It was found that crystallites with sizes less than 1 μm were formed during the grinding process, whose presence in the powder structure contributes to the achievement of high magnetic properties [21], which is due to the single-domain sizes of hexaferrite particles, which are in the range of 300–600 nm.

The magnetic properties of the strontium hexaferrite powder composition following fine grinding were as follows: specific saturation magnetization σ_s = 70.3 A·m^2/kg; specific residual saturation magnetization σ_r = 37.2 A·m^2/kg; coercive force by magnetization H_C^M = 303.9 kA/m (Fig. 3).

A laser particle analyzer was used to determine that the average particle size of the powder composition based on the material of the Sr–Fe–O system was 13.4 μm. The particle sizes of this powder composition are in the range from 500 nm to 25 μm. Powder with this particle size distribution is considered as an optimal raw material for the formation of feedstocks according to the PIM method. This is due to the fact that powders with sizes up to 20–25 μm can be used to produce granulates with the required yield parameters on their basis [20]. In this case, nanosized particles will fill the pores between

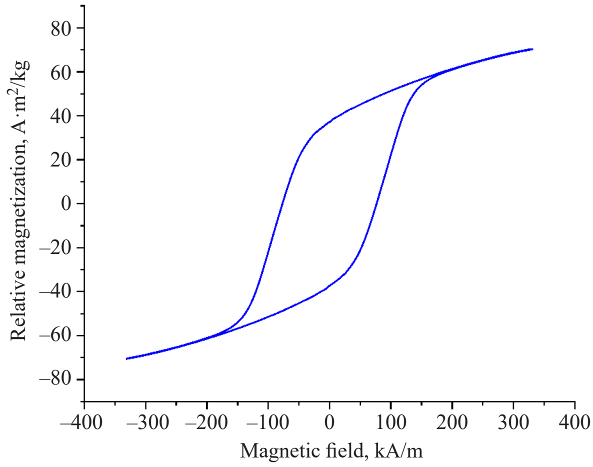

Fig. 3. Hysteresis loop of a strontium hexaferrite powder after fine grinding for 10 h

large particles. Powders of the obtained granulometric composition can also be used as a feedstock for the creation of photopolymers used in 3D printing of products by stereolithography. The particle size of this powder composition does not exceed the thickness of the printing layer, which is predominantly in the range of 20 to 50 μm [22, 23].

Figure 4 depicts an SEM image of the granulate showing particles of strontium hexaferrite powder located in a polymer matrix based on paraffin and polyamide. The contrast of light ceramic particles against the dark background of the binder shows a strong difference in the atomic numbers of chemical elements of the substances. From this it may be concluded that there are quite large areas in the feedstock structure, to which the powder was hindered in the mixing process. This is due to the peculiarities of granulate preparation, which was obtained by manual mixing of initial components.

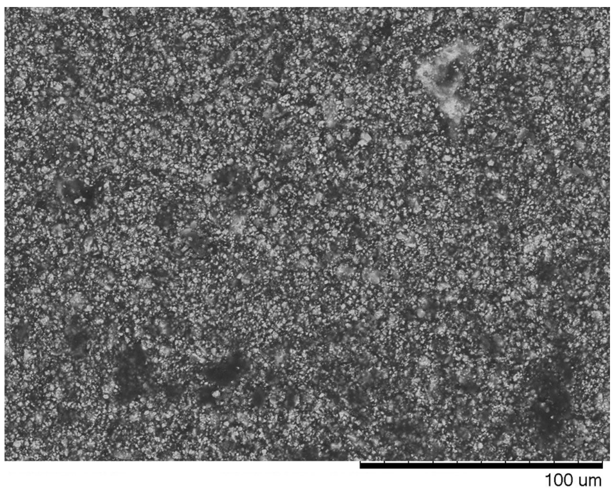

Fig. 4. Microstructure of granulate consist of strontium hexaferrite powder

The tomography results of the green body samples obtained from strontium hexaferrite are shown in Fig. 5.

(a) (b) (c)

Fig. 5. Tomography of strontium hexaferrite permanent magnet green body samples with lack of fusion (a, b) and without defects (c)

In the course of testing the process of obtaining green parts, the modes of granulate pouring into the mold of the injection molding machine were changed. Increasing the pressing pressure[4] allowed defects from the intermediate parts to be removed with lack of fusion, which were formed as a result of non-slip flows of molten binder of reduced liquid flowability (Figs. 5a and 5b). Green parts obtained under the modified regime were characterized by minimal porosity, indicating the possibility of their further use in order to obtain high-quality magnets at the final stage of the PIM process. Here, the magnetic (product of induction *B* and coercive force *H* $(BH)_{max}$, coercive force by induction H_C^B and magnetization H_C^M, residual induction B_r) and mechanical properties (tensile strength σ_B, relative elongation δ) will depend solely on the quality of the initial raw material. SEM images of the microstructure of the green parts, which were classified by tomography as having no internal defects, are presented in Fig. 6.

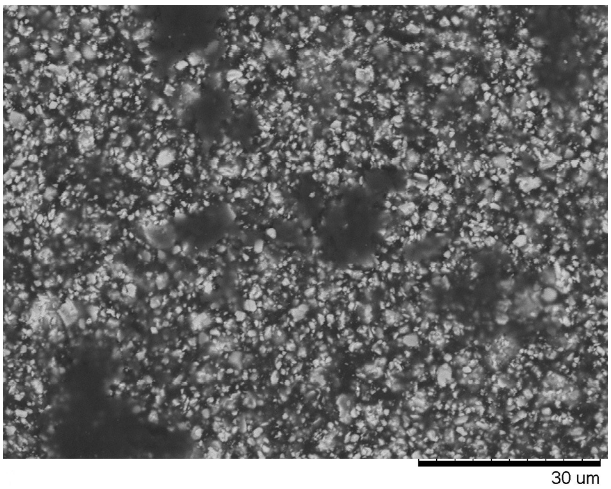

Fig. 6. Microstructure of strontium hexaferrite permanent magnet green body sample

These images show that the microstructure of the green part inherits the structure of the granulate: while particles of strontium hexaferrite powder are evenly distributed in the body of the intermediate part, there are significant areas to which the powder has not penetrated. The size of such areas reaches 30–40 μm in diameter. The presence of this defect is undesirable since the removal of the primary binder at the next stage of debinding can lead to the formation of large pores, which can not only remain in the final product, but also lead to warping and cracking of parts during shrinkage due to the weakening of the framework based on the secondary binder, mainly consisting of polyamide.

In the process of selecting the most effective medium and parameters for removing polyamide from the intermediate body samples, a plot of the green part mass variation was obtained over time, as shown in Fig. 7.

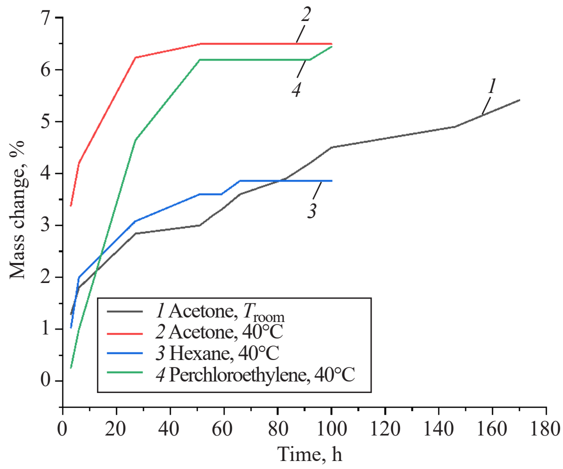

Fig. 7. Mass change of strontium hexaferrite permanent magnet green bodies samples during the debinding process depending on duration of debinding stage

Based on the obtained dependence, it can be concluded that the optimal media for removal of the primary binder are perchloroethylene and acetone. Nevertheless, it is desirable to accelerate the process of interaction between the green parts and the solvent by increasing the ambient temperature up to 40°C. Samples that underwent the debinding stage in acetone at room temperature (20°C) contained primary binder at the level of 1–2 wt % even after 170 h of exposure. In comparison with the results of works devoted to the study of effective debinding modes, the use of hexane did not lead to a positive result: soaking for 100 h allowed removal of only 4 wt % of the binder [24, 25]. Furthermore, the solvent interacted actively with the part, which led to the formation of a white scale on its surface. This is

[4] The nature of the change of granulate pouring modes and the value of increasing pressing pressure are commercial secrets and cannot be published in the article.

due to the fact that the dissolution of paraffin in hexane is a heterogeneous procedure, which is observed at the phase interface between liquid and solid substances with possible precipitation [26].

A snapshot of the microstructure of the brown body sample that was obtained from the green part during debinding is shown in Fig. 8.

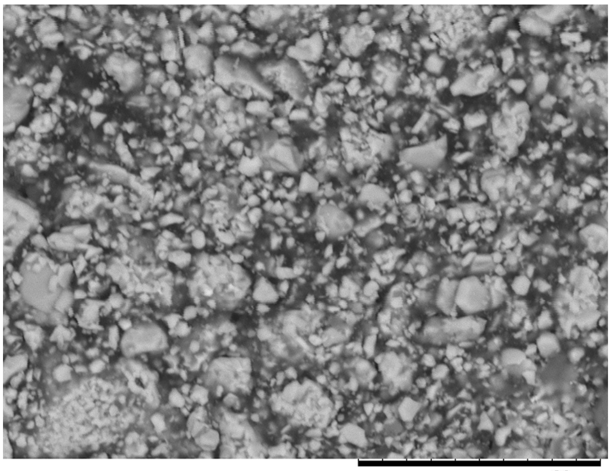

Fig. 8. Microstructure of strontium hexaferrite permanent magnet brown body sample

The resulting image shows that the second intermediate body samples also consist of strontium hexaferrite powder and organic binder. In contrast to the structure of the granulates and green parts, the brown body samples do not contain a primary binder. At this stage, the secondary binder acts as a framework, which makes the intermediate parts more brittle. The structure of the brown part is characterized by the presence of interconnected open pores necessary to ensure uniform shrinkage during the sintering stage.

Magnets with the required geometry of 10 × 10 × 5 mm were obtained by sintering of brown parts based on strontium hexaferrite. Analysis of the content of impurities in the samples after sintering showed that the material was not contaminated with carbon, which is often included in the parts made of various materials from the binder: its percentage was 0.014 wt %. This fact indicates that the binder based on paraffin and polyamide can be used to create clean materials by PIM technology.

The density of magnets based on strontium hexaferrite after sintering was 4.2 g/cm^3, which is 80% of the theoretical value [27]. It is likely that a density close to the theoretical one can be achieved by using industrial equipment for mixing powders and organic binder, which will permit uniform distribution of particles inside the granulate and green parts.

Magnetic properties of bulk samples based on strontium hexaferrite powder for the alloy obtained by PIM technology were as follows: residual induction B_r = 0.12 T; coercive force by induction H_C^B = 85.7 kA/m; coercive force by magnetization H_C^M = 298.4 kA/m. The magnetization coercive force exceeded the value established by the requirements of normative and technical documentation for isotropic permanent magnets based on barium hexaferrite[5]. However, the values of residual induction and coercive force by induction remained at the level of 70% of the values specified in the normative and technical literature. An increase in the level of coercive force can be achieved primarily by reducing the sintering time of magnets: by this means, the growth of individual crystallites, which in this case act as single-domain particles, will be prevented.

Despite the absence of major defects in the form of geometry changes or cracks in the parts based on strontium hexaferrite, the degradation of magnetic properties compared to the original powder is due to defects in the microstructure of the parts that have passed the sintering stage (Fig. 9).

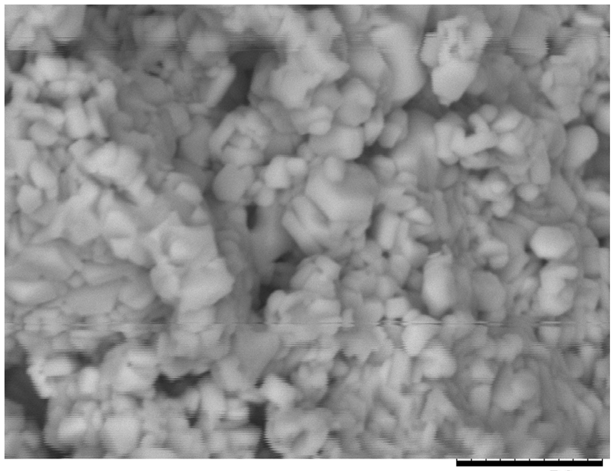

Fig. 9. Microstructure of sintered strontium hexaferrite permanent magnet obtained by PIM technology

The SEM images show that the final product is represented as a large number of large agglomerates, which are formed in the process of sintering of fine-grained single-domain powder. Between the agglomerates, pores with sizes of about 2–4 µm are noticeable, which could not be removed during high-temperature processing. The presence of pores, which negatively affect not only the

[5] GOST 24063-80. State Standard of the USSR. Magnetically hard ferrites. Brands and main parameters. Moscow: USSR State Committee for Standards; 1986. 14 p.

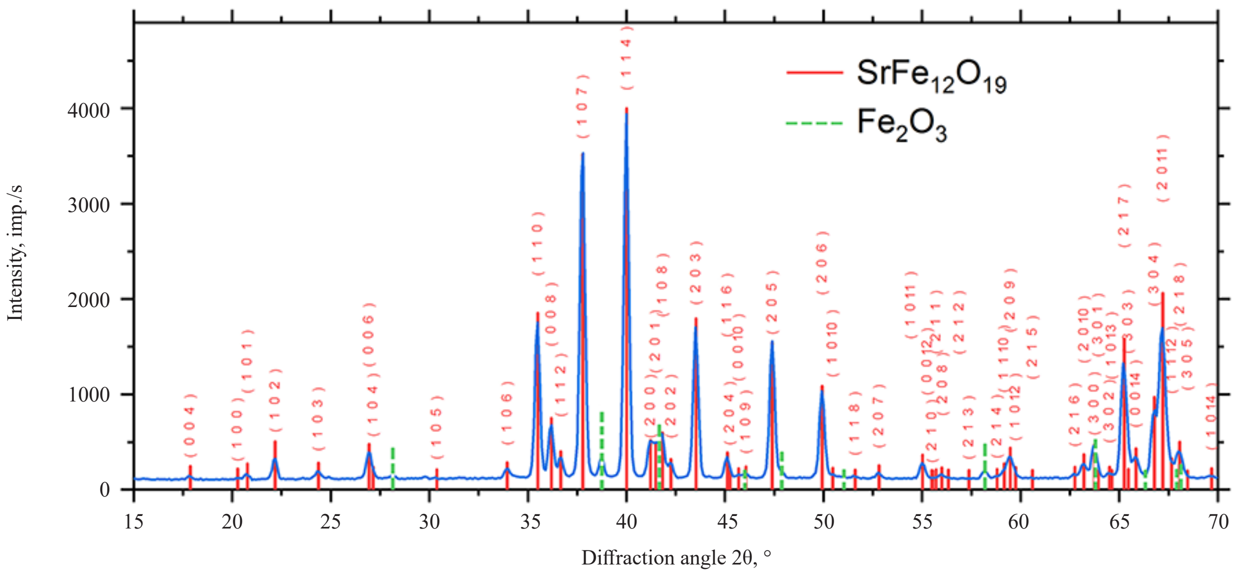

Fig. 10. X-ray diffraction of strontium hexaferrite permanent magnet obtained by PIM technology

mechanical and functional but also magnetic properties, can be prevented by preparing the raw material using sufficient powder quantity (from 4 kg) and industrial granulators. This problem can also be solved by hot isostatic pressing, which allows fixing microcracks and pores to increase the density and mechanical properties of the parts [28].

It is likely that magnetic parameters of the sintered product can be further enhanced by reducing the time of holding the material in the sintering process. This will prevent the growth of single-domain crystallites and preserve the structure of the final part to be more similar to that of the initial raw material.

In the process of studying the phase composition of magnets after sintering, an additional factor of magnetic properties reduction was revealed. On the X-ray diffractogram of the sample of permanent magnet based on strontium hexaferrite, as shown in Fig. 10, there are additional lines of hematite Fe_2O_3, the content of which in the sample was equal to 2 wt %. The amount of the main ferromagnetic phase $SrFe_{12}O_{19}$ amounted to 98 wt %.

CONCLUSIONS

In the process of studying the microstructure and magnetic properties of strontium hexaferrite powder, it was confirmed that the proposed technology used to obtain this material can be used for manufacturing raw materials for PIM technology and stereolithography.

The control of intermediate and final parts obtained by injection molding of granulates highly filled with ceramic powder led to the conclusion that the PIM method is a promising technology for the production of permanent magnets based on strontium hexaferrite. However, for the industrial implementation of this method, the granulate production processes will need to be optimized. This will be realized in further research through the use of specialized equipment for granulate production. Industrial equipment for production of raw materials, which should work in a continuous mode, requires powder loading in the amount of 2–3 kg to provide a more uniform distribution of strontium hexaferrite particles in the binder based on polyamide and paraffin. Furthermore, in comparison with the method of manual mixing, the use of a granulator will eliminate human influence on the homogeneity of the distribution of powder particles to obtain a high quality granulate. The sintering mode of brown parts may also be optimized in order to prevent the formation of pores and growth of single-domain particles of strontium hexaferrite. This can be achieved by lowering the sintering temperature and reducing the heating rate in the section up to 500°C, during which the removal of secondary binder and technological additives takes place.

By developing this process using granulate filled with strontium hexaferrite powders and solving the above problems, it will probably be possible to use this material as a binder for the development of feedstock based on Sm–Co alloy powders and for the production of grade KS25 rare-earth permanent magnets.

Acknowledgments

The work was financially supported by the Russian Science Foundation, project No. 23-73-00114.

Authors' contributions

B.D. Chernyshev—synthesis of strontium hexaferrite powder, production of permanent magnets by PIM technology, processing the results, and writing the text of the article.

I.V. Schetinin—analysis of the microstructure, phase composition, and magnetic properties of samples.

The authors declare no conflicts of interest.

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Translated from Russian into English by H. Moshkov
Edited for English language and spelling by Thomas A. Beavitt