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OBTAINING ANISOTROPIC HEXAFERRITES FOR THE BASE LAYERS OF MICROSTRIP SHF DEVICES BY THE RADIATION-THERMAL SINTERING

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Радіаційно-термічним спіканням (РТС) отримані зразки анізотропних полікристалічних гексаферитів $\text{BaFe}_{12}\text{O}_{19}$, $\text{BaFe}_{12-x}\text{Al}_x\text{O}_{19}$ (з добавками Ni , Ti , Mn), $\text{SrFe}_{12}\text{O}_{19}$ і $\text{SrFe}_{12-x}\text{Al}_x\text{O}_{19}$ (з добавками Ca , Si) для феритових розв'язуючих приладів. Суть технології РТС полягає в пресуванні в сильному магнітному полі сирих заготовок і їх подальшому спіканню в пучку швидких електронів

Ключові слова: гексагональний ферит, радіаційно-термічне спікання, пресування, магнітне поле, текстура

Радиационно-термическим спеканием (РТС) получены образцы анизотропных поликристаллических гексаферритов $\text{BaFe}_{12}\text{O}_{19}$, $\text{BaFe}_{12-x}\text{Al}_x\text{O}_{19}$ (с добавками Ni , Ti , Mn), $\text{SrFe}_{12}\text{O}_{19}$ и $\text{SrFe}_{12-x}\text{Al}_x\text{O}_{19}$ (с добавками Ca , Si) для ферритовых развязывающих приборов. Сущность технологии РТС состоит в прессовании в сильном магнитном поле сырых заготовок и их дальнейшем спекании в пучке быстрых электронов

Ключевые слова: гексагональный феррит, радиационно-термическое спекание, прессование, магнитное поле, текстура

1. Introduction

The method of radiation-thermal sintering that consists in the heating of original components by the beams of high-energy electrons without involving external sources of heat has been of increasing interest to researchers in recent years [1].

The advantages of the radiation-thermal method (simultaneous exposure to radiation and temperature) consist in the rapidity and low inertia of heating of materials, the absence of contact between the heating body and the heater, uniformity of material heating throughout entire volume [2]. The types of electron accelerators in the range of $E=0,01-13$ MeV that exist today make it possible to heat solid bodies to the temperature of their melting [3].

To improve the properties of ferrites, it is necessary to obtain single-phase temperature-stable compositions with low dielectric and magnetic losses [4]. The properties of ferrites depend not only on their chemical and phase compositions but also on the process of firing and cooling in the process of obtaining [5]. Porosity, violation of stoichiometry, existence

of second phases or incomplete progress of the reaction of ferritization lead to the reduction in the chemical and structural homogeneity of material. As a result, distortion of the magnetic anisotropy of ferrites occurs, which causes worsening of magnetic characteristics and their reproducibility.

The results obtained in the present work made it possible to develop the technology of obtaining polycrystalline hexagonal ferrites by the method of radiation-thermal sintering (RTS). Such hexagonal ferrites are used for the base layers of the subminiature microstrip ferrite untying instruments of the short-wave part of the centimeter and millimeter wavelength range.

2. Literature review and problem statement

Among the materials, obtained by ceramic technology, the articles made of polycrystalline ferrites are widely spread, which are the compounds of iron oxide with the oxides of other metals [6]. Possessing the unique combination

of magnetic, electrical and other properties, they relate to the class of electronic, which ensures their wide application in the areas of science and technology that determine technical progress [7, 8].

The properties of ferrites are determined, besides the chemical and phase composition, by the technology of their obtaining, and especially – by the regimes of ferrite formation and sintering [9].

In recent years, in the course of obtaining different ceramic materials (alkali-halide crystals, high-strength ceramics, oxides, steels, hard alloys, ferrosinels, ferrogarnets, hexaferrites), the method of radiation-thermal sintering with the aid of powerful flows of accelerated particles has been developing successfully [10, 11].

A large number of papers [6, 9–11] is devoted to the study of the processes of ferrite formation and to the formation of magnetic properties in Li- and Li- substituted (Li-Zn, Li-Ti, Li-Ti-Zn) ferrosinels, which are the base of a large group of thermostable SHF ferrites with a straight hysteresis loop (SHF), as well as promising material of the cathodes of lithium batteries.

Paper [12] presents experimental study of high-temperature diffusion and radiation-thermal effects in the alkali-halide crystals (KBr and LiF) during heating by high intensity electron beams with energy from 0.01 to 1–2 MeV. The effect is discovered of the acceleration of diffusion mass transfer of metal ions in the alkali-halide crystals during radiation-thermal treatment by electrons in the range of $E=1.4-2$ MeV.

Difference in the processes of diffusing oxygen in polycrystalline ferrites during the thermal (T) and radiation-thermal (RT) firing is connected to the change in the defective state of ferrite as a result of excitation of the electron and nuclear subsystems of the lattice caused by irradiation [12].

The technique of heating the samples by electron beam makes it possible to obtain oxide ceramic materials with the uniform phase composition and low elastic stresses, which ensures an increase in the operating characteristics. The studies, carried out on ferrosinels (Mg-, Ni-, Li-, Li-substituted), synthesized in the beam of accelerated electrons, convincingly attest about the increase in the rate of diffusion of original components, which leads to the higher efficiency of the formation of magnetic properties under conditions of RT-firing in comparison to the T-firing.

MnZn- and NiZn- ferrites were obtained previously by the method of radiation-thermal sintering with the aid of powerful streams of accelerated particles [5, 6]. However, despite the fact that there is a large volume of information in this area, we did not find in the open sources any data on the technology of obtaining polycrystalline hexagonal ferrites by the method of radiation-thermal sintering.

3. Aim and tasks of the study

The purpose of the work was to obtain anisotropic polycrystalline hexagonal ferrites for the base layers of the microstrip SHF-instruments of the millimeter wavelength range by the thermal radiation sintering.

To accomplish the set aim, it was necessary to solve the following tasks:

- to develop technology of obtaining, by the thermal radiation sintering, of anisotropic polycrystalline hexagonal ferrites on the basis of classic ceramic technology;

- based on the developed technology, to obtain the samples of anisotropic polycrystalline hexagonal ferrites by the thermal radiation sintering and to determine their magnetic parameters.

4. Materials and methods of research

4.1. Obtaining the objects of research

The technology of the fabrication of the billets of polycrystalline hexagonal ferrites (HF) of barium (HB) and strontium (HS) was based on the principles of classical ceramic technology. The formation of composition of hexaferrite took place by mixing original components in the process of wet grinding in the ball mill at the ratio of charge, spheres, deionized water =1:2:1, respectively, during 24 hours (Fig. 1).

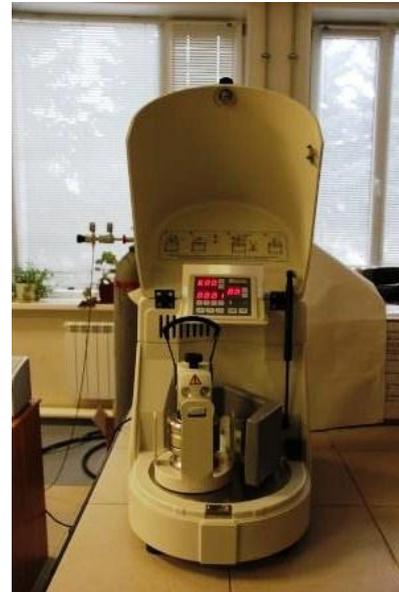


Fig. 1. Exterior view of the planetary mill “Pulverisette 6”

For obtaining 1 kg of charge, we used china drums with a capacity of 4 l. For obtaining polycrystalline hexaferrite of barium with the base composition $BaFe_{12-x}Al_xO_{19}$, we used the following components and alloying additives: $BaCO_3$; Fe_2O_3 ; Al_2O_3 ; TiO_2 ; NiO ; $MnCO_3$. For obtaining polycrystalline hexaferrite of strontium with the base composition $SrFe_{12-x}Al_xO_{19}$, we used the following components and alloying additives: $SrCO_3$; Fe_2O_3 ; Al_2O_3 ; SiO_2 ; $CaCO_3$. To increase the value of the inner field of crystallographic anisotropy H_A , we applied the substitution by ions Al^{3+} that substitute Fe^{3+} iron ions in the crystal lattice of hexaferrite. The introduction of titanium and nickel additives was conducted for the purpose of reduction in the temperature withdrawal of the field of anisotropy from 10 % to 4 % from the mean value in the temperature range $(-60 \div +85) ^\circ C$. The addition of manganese increases electrical resistance of ferrites and decreases dielectric losses due to the substitution (binding) of the ions of bivalent iron, which are the rapidly relaxing ions and serve as the sources of losses. The additions of silicon and calcium are used for hexaferrite of strontium. Introduction of silicon atoms into the composition of hexaferrite makes it possible to detain an increase in the crystals growth in the liquid phase, and the addition of calcium improves magnetic

parameters of the material due to the decrease in magnetic losses. Based on the need for the calculation of milling yield from the metallic spheres, and that the optimum combination of electromagnetic properties is observed in the samples of hexaferrite of barium (similarly for HF of strontium), whose composition differs from the stoichiometric ($\text{BaO} \cdot 6\text{Fe}_2\text{O}_3$) by the reduced content of iron oxide Fe_2O_3 , we examined ferrites with the chemical formula $\text{BaO} \cdot (5,5+5,75)\text{Fe}_2\text{O}_3$. After mixing, the charge was poured out into the steel cuvette and dried in a drying cabinet at $T=150^\circ\text{C}$ until complete drying. We sifted the dried charge through the sieve and poured out into a nickel cuvette, after which we placed it into the furnace, where the process of ferritization was conducted. The duration of curing comprised 5 hours at a temperature 1150°C for the strontium and 1250°C for the barium hexaferrites. After ferritization, the charge was exposed to wet grinding in the ball mill at the ratio of charge, spheres, deionized water = 1:2:1, respectively, for 96 h. This duration of grinding provided for obtaining the powder with the average particle size of the order $0,3 \div 0,5 \mu\text{m}$. The charge in the china drum was washed by deionized water and poured out into an idle tank. The obtained suspension of the powder of hexaferrite was kept for three days, after which the excess water was removed. Humidity of the suspension during pressing amounted to $30 \div 35\%$.

For the fabrication of anisotropic hexaferrite billets, the pressing was conducted in the magnetic field, applied along the direction of pressing (Fig. 2).

RTS of the samples was conducted using fast electrons on the pulse linear accelerator ILU-6 (Fig. 3, 4) made by IYaF named after G. I. Budker SO RAN (Russia) (energy of electrons $E_e=2,5 \text{ MeV}$).

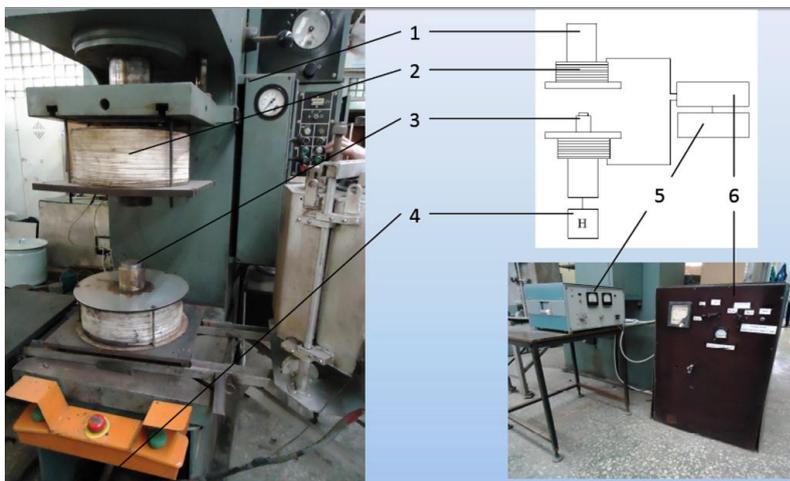


Fig. 2. General view and schematic of the press:
 1 – press, 2 – magnetic coil, 3 – mold, 4 – pump, 5 – power source,
 6 – control unit of magnetic field and the pump



Fig. 3. Pulse linear accelerator ILU-6

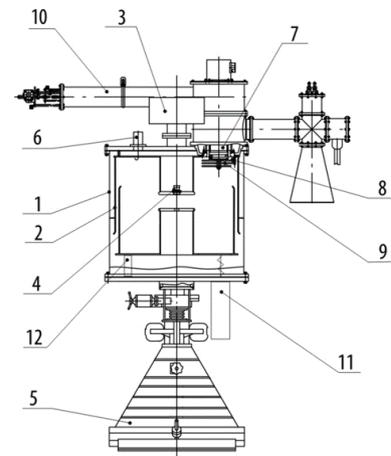


Fig. 4. Schematic of pulse linear accelerator ILU-6: 1 – vacuum tank; 2 – resonator; 3 – magnetic-discharge pump of the NMD type; 4 – electrons injector; 5 – outlet device; 6 – measuring loop; 7 – anode of the lamp of HF generator; 8 – support of the loop of HF power input; 9 – loop of HF power input; 10 – cathode stub; 11 – input of displacement voltage -7 kV ; 12 – supports of the lower half of the resonator

4. 2. Techniques of the experimental studies

The roentgen phase and X-ray diffraction analysis of the examined objects was carried out in the X-ray diffractometer DRON-8 (Russia) (Fig. 5).

When running the roentgen phase analysis, we used the $\text{CuK}\alpha$ -emission, as well as the tube with the iron anode (operating current – 25 mA, voltage – 25 kV). Wavelength

of emission is 0,193728 nm. When taking photographs of the samples, we used the filter from Mn. Focusing was performed according to the Bragg-Brentano method with two Soller slits. The measurements were carried at room temperature.



Fig. 5. Multifunctional X-ray diffractometer DRON-8

Identification of intensive peaks in the diffractogram was conducted with the help of the software PDWin 4.0 (NPO “Burevestnik” Russia). The roentgen phase analysis of the samples came down to determining a series of interplanar distances and their comparison with reference data of the powder diffraction database, which is based on the PDF2 card index.

Magnetic characteristics of the examined objects were recorded at room temperature at the vibration magnetometer of the sample VSM-250 made by Lake Shore Cryotronics, Inc. (USA) (Fig. 6).



Fig. 6. Vibration magnetometer VSM-250

The spheres $\varnothing 2,5-3,0$ mm obtained from the sintered specimens, served as the samples.

5. Results of research and discussion

Technological scheme of obtaining polycrystalline hexagonal ferrites by the RTS method is represented in Fig. 7.

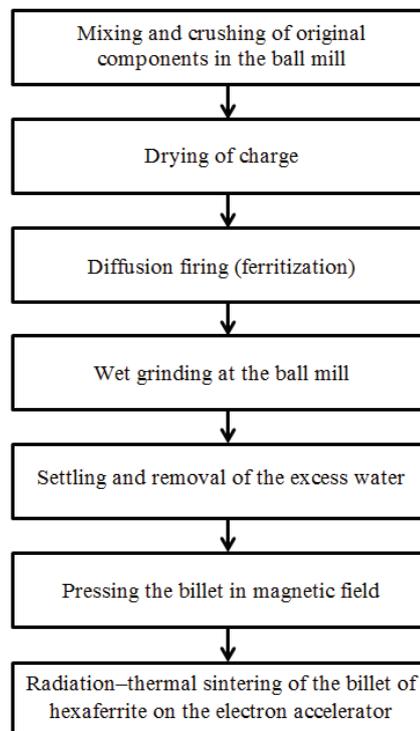


Fig. 7. Technology of manufacturing hexaferrites by the radiation-thermal sintering method

For the pressing of samples in magnetic field, a special press was designed, equipped with two coils (electromagnet), which create magnetic field. The upper coil accepts the press’s plunger with the tip fixed to it; its form contributes to the concentration of magnetic field. In the lower coil there is the base for the mold with the opening for water discharge, which ends with a coupling for fastening the hose, connected through the trap to the mechanical vacuum pump. The source that feeds the electromagnet provides for obtaining direct current to 10 A with voltage of up to 20 V.

The uniformity of magnetic field is an important factor, since its distribution directly affects both the properties of the pressed material and their uniformity. The measurement of magnetic field strength in the working gap was carried out with the aid of teslameter, which employs the Hall effect. Graphic representation of dependency of the measured data is presented in Fig. 8.

Results of the measurements demonstrated that magnetic field strength during pressing was approximately 10 kOe. On the assumption that for the provision of quality magnetic texture (formation in one direction of magnetic

moments of most single-domain particles), magnetic field is required of the magnitude $3 \cdot H_C$ (H_C is the coercive force), and $H_C \approx 3$ kOe for hexaferrites of barium and strontium, then the field with magnetization of 10 kOe must be enough for creating the anisotropic material.

Fig. 8 also demonstrates that the field is distributed fairly uniformly, which must ensure the uniformity of properties of the pressed material. The lower value of the field in the boundary point may be possibly explained by screening from the housing of the press or by physical properties of the coils.

For obtaining the samples from hexaferrites, we used the mold with a matrix from the nonmagnetic material (brass) and the punches made of mild steel. The scheme of the mold is depicted in Fig. 9.

This set-up makes it possible to create magnetic field in the gap between the punches, where the textured charge is located. The lower punch has openings for water discharge through the felt filters, located on it. Two molds with diameters of 50 and 70 mm are used.

For orienting particles in the magnetic field it is necessary to create conditions, which make it possible for a particle to pivot fairly free around its axis, which is achieved by diluting the charge with the distilled water, which is removed after orientation in the process of pressing.

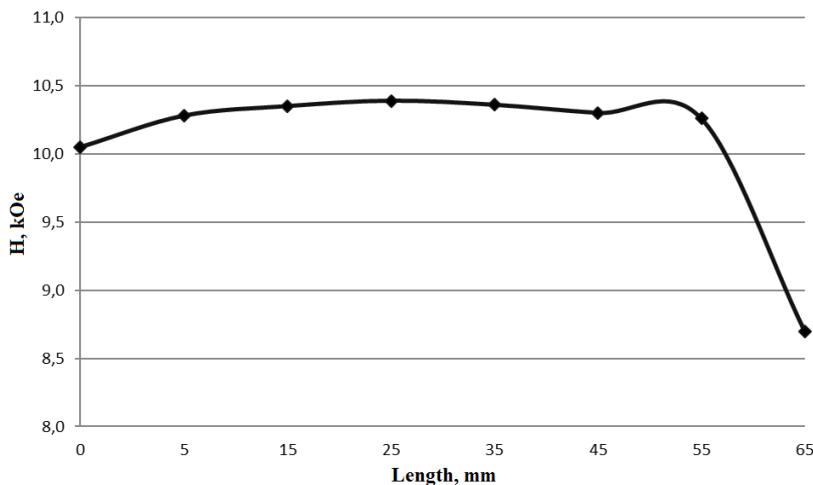


Fig. 8. Distribution of magnetic field H, kOe in the working gap of press for obtaining raw billets of hexaferrite

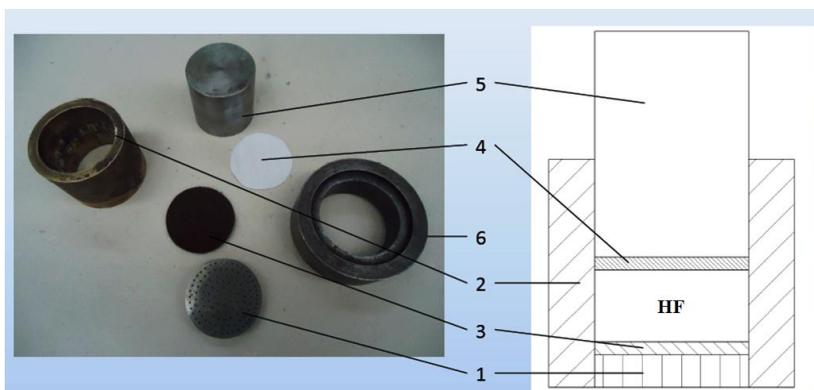


Fig. 9. Scheme of the mold for pressing raw anisotropic billets of hexagonal ferrites: 1 – lower punch, 2 – outer form, 3 – gasket of felt, 4 – fabric insert, 5 – top punch, 6 – squeezing ring

The process of pressing consists of the following stages:

- 1) loading the suspension of ferrite powder into the mold;
- 2) settling of suspension in magnetic field for the purpose of orientation of the powder particles by their mechanical turning by the axes of easy magnetization along the direction of the applied field;
- 3) preliminary discharge of moisture with the aid of the backing pump through the punch with filtering elements;
- 4) pressing of the suspension at active magnetic field and continuous discharging of the freed moisture;
- 5) pressing out the billet.

In order to maintain pressing in the magnetic field, the top punch of press descends to the contact with the top punch of the mold, decreasing the gap between the coils, thus making it possible to more effectively use magnetic field. Next is the exposure to the magnetic field. Then, at the applied necessary pressure, the billet is maintained still for a while.

The magnitude of magnetizing field in the process of pressing was 10 kOe.

The replacement of conventional thermal sintering with the radiation-thermal one (RTS) in the beam of fast electrons is caused by the substantially lower energy consumption of the latter and by the higher quality of sintering.

At RTS, in addition to the factor of temperature, there is such an essential factor as the radiation-stimulated diffusion. Due to this, the sintering takes place at lower temperatures and in shorter time.

Temperature in the sample in the course of conducting RTS was achieved by variation in the frequency of electron beam, its density and by exposure time.

Tables 1–4 display information about the RTS regimes of hexaferrites with different compositions.

The X-ray studies confirmed (according to the identified peaks in the diffractogram with the aid of the software PDWin 4.0) that as a result of using radiation-thermal sintering, we obtained polycrystals of anisotropic hexagonal ferrites of barium and strontium (both in the pure form and substituted). The characteristic X-ray diffractograms of hexaferrites $BaFe_{12}O_{19}$ and $BaFe_{12-x}Al_xO_{19}$ (with the Ni, Ti, Mn additives) are represented in Fig. 10, 11. Results of the magnetic parameters are given in Table 5.

As can be seen from Table 5, magnetic properties of hexagonal ferrites obtained by the RTS method are equal to or exceed the values of magnetic characteristics of hexaferrites, obtained by the traditional ceramic technology [13].

Thus, the RTS technology may effectively be used for obtaining not only ferrites-spinels [5, 6], but also hexagonal ferrites. The advantage of RTS is significant energy cost reduction, high values of performance characteristics of material [13] and low duration of the process. The shortcomings of this technology include essential financial investments at the initial stage due to high price of the electron accelerator.

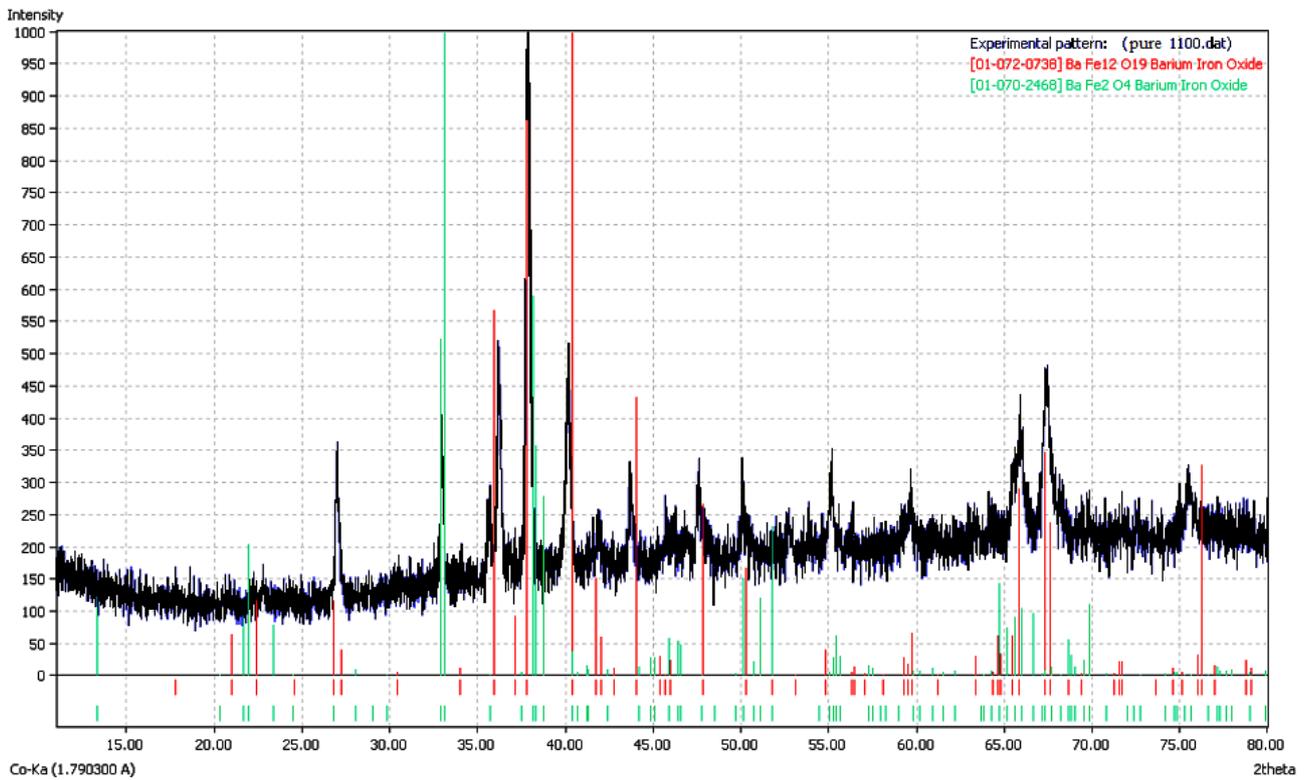


Fig. 10. Characteristic X-ray diffractogram of the sample $BaFe_{12}O_{19}$, obtained by the RTS method at the temperature of sintering 1100 °C

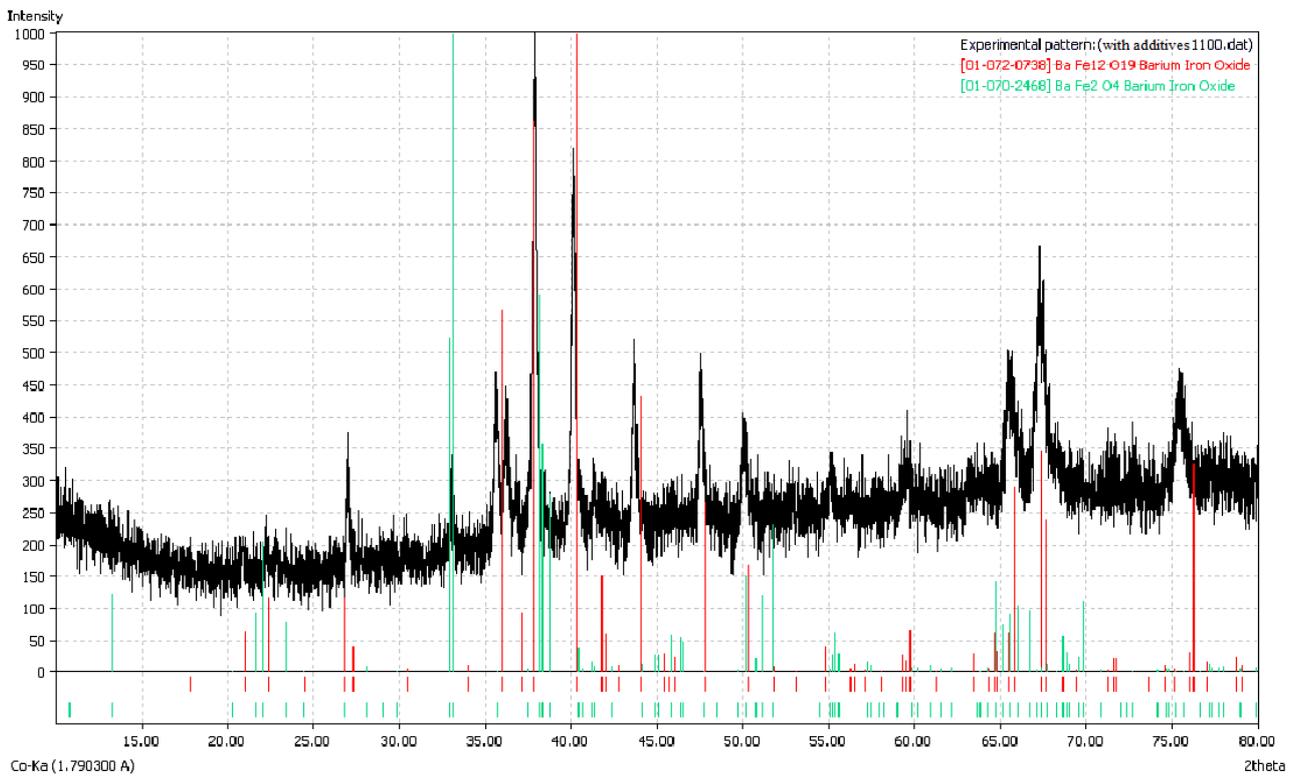


Fig. 11. Characteristic X-ray diffractogram of the sample $BaFe_{12-x}Al_xO_{19}$ (with the Ni, Ti, Mn additives), obtained by the RTS method at the temperature of sintering 1100 °C

Table 1

RTS technological regimes of the samples BaFe₁₂O₁₉

Sample No.	T _{sintering} , °C	t _{sintering} , min	Heating speed V, °C/min
1-1	1 100	60	50
1-2	1 200	60	50
1-3	1 300	60	50
1-4	1 400	60	50

Table 2

RTS technological regimes of the samples BaFe_{12-x}Al_xO₁₉ (adding Ni, Ti, Mn)

Sample No.	T _{sintering} , °C	t _{sintering} , min	Heating speed V, °C/min
2-1	1 100	60	50
2-2	1 200	60	50
2-3	1 300	60	50
2-4	1 400	60	50

Table 3

RTS technological regimes of the samples SrFe₁₂O₁₉

Sample No.	T _{sintering} , °C	t _{sintering} , min	Heating speed V, °C/min
3-2	1 200	60	50
3-3	1 300	60	50
3-4	1 400	60	50

Table 4

RTS technological regimes of the samples SrFe_{12-x}Al_xO₁₉ (adding Ca, Si)

Sample No.	T _{sintering} , °C	t _{sintering} , min	Heating speed V, °C/min
4-2	1 200	60	50
4-3	1 300	60	50
4-4	1 400	60	50

Table 5

Magnetic properties of the samples of barium and strontium hexaferrites

Chemical composition	Field of saturation H _{max} , kOe	Field of anisotropy H _k , kOe	Field of specific magnetization of saturation σ _s , emu/g	Field of residual specific magnetization σ _r , emu/g	Loop squareness ratio M _r /M _s	Coercive force at magnetization j _{Hc} , kOe
SrFe _{11,9} Al _{0,1} O ₁₉ (with additives Ca ²⁺ , Si ⁴⁺)	20,0	450,0	59,0	25,0	0,38	2,8
SrFe _{11,8} Al _{0,2} O ₁₉ (with additives Ca ²⁺ , Si ⁴⁺)	20,0	130,0	38,0	6,0	0,07	0,45
BaFe ₁₂ O ₁₉	20,0	430,0	68,0	44,0	0,64	1,98
SrFe ₁₂ O ₁₉	20,0	425,0	64,0	40,0	0,56	2,1

6. Conclusions

1. We developed the basics of technology for obtaining hexagonal barium and strontium ferrites of the M type by the method of radiation-thermal sintering.

2. Using the developed RTS method, we obtained the samples of anisotropic polycrystalline hexaferrites BaFe₁₂O₁₉, BaFe_{12-x}Al_xO₁₉ (with the Ni, Ti, Mn additives), SrFe₁₂O₁₉ and SrFe_{12-x}Al_xO₁₉ (with the Ca, Si additives) for the base layers of the microstrip ferrite untying instruments of the short-wave part of the millimeter wavelength range. The obtained samples fully comply with the demanded requirements.

3. Owing to high energy efficiency and low duration of the process of sintering, RTS may find its worthy place among existing methods of obtaining polycrystalline hexaferrites.

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