

# Crystal and magnetic structure of hexagonal anisotropic polycrystalline ferrites $\text{SrFe}_{12}\text{O}_{19}$ obtained by radiation-thermal sintering

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## Abstract

Hexagonal anisotropic strontium ferrite has found wide application in the fabrication of permanent magnets, active media of UHF electronic devices, and photonics. The performance of hexagonal strontium ferrites depends largely on the synthesis technology. Anisotropic  $\text{SrFe}_{12}\text{O}_{19}$  samples have been for the first time synthesized using radiation-thermal sintering (RTS) in a fast electron beam from an electron accelerator. The RTS temperature was varied between 1200 and 1400 °C, and the process time, from 10 to 90 min. The phase composition and lattice parameters of the samples have been monitored using X-ray diffraction and Mössbauer spectroscopy. The X-ray spectra have been recorded with a DRON-8 diffractometer in  $\text{CoK}_{\alpha 1}$  radiation, and the Mössbauer spectra, using an MS1104E spectrometer with a constant acceleration at room temperature, the radiation source being  $\text{Co}^{57}$  in a chromium matrix. The density of the specimens has been studied in accordance with Archimedes' law on a UW620H electronic balance with a density measurement attachment. The X-ray diffraction and Mössbauer spectroscopy data show that the samples are single-phase and have the  $P6_3/mmc$  (No. 194) space group, corresponding to the hexagonal ferrite structure. The unit cell parameters  $a$  and  $c$  and the volume  $V$  of the samples have been studied as a function of RTS temperature for the sintering time  $t = 30$  and 60 min. Also, the unit cell parameters  $a$  and  $c$  and the volume  $V$  of the samples have been studied as a function of sintering time for the RTS temperature  $T = 1300$  °C. The lattice parameters  $a$  and  $c$  of the samples exhibit opposite dependences, suggesting anisotropic lattice distortion. The maximum magnetic texture degree achieved using RTS in anisotropic  $\text{SrFe}_{12}\text{O}_{19}$  proves to be about 92%. It has been concluded on the basis of the Mössbauer spectroscopy data that the optimum magnetization degree of the  $\text{SrFe}_{12}\text{O}_{19}$  samples is achieved for the following RTS modes (RTS temperature, °C / RTS time, min): 1300/60 and 1350/40. We show that the sintering temperature plays a considerably greater role in the RTS technology than the sintering time. Conclusion has been made that RTS can be used as an alternative technology of synthesizing polycrystalline anisotropic hexagonal  $\text{SrFe}_{12}\text{O}_{19}$  ferrite. As compared to the conventional ceramic technology, RTS proves to be highly energy-efficient and cheap.

## Keywords

radiation-thermal sintering, anisotropic polycrystalline hexagonal  $\text{SrFe}_{12}\text{O}_{19}$  ferrites, X-ray diffraction, Mössbauer spectroscopy, crystal structure, magnetic texture degree, unit cell, unit cell lattice parameter, ceramic technology, fast electrons, electron accelerator

## 1. Introduction

The crystal structure of hexagonal *M*-type  $\text{SrFe}_{12}\text{O}_{19}$  strontium ferrite (SrM) is isomorphic to that of magnetoplumbite  $\text{PbFe}_{12}\text{O}_{19}$  [1]. Due to the difference in the ionic radii of barium and strontium, replacement of the former ion for the latter one leads to an increase in the crystallographic magnetic anisotropy constant  $K_1$  by 10% [2]. Moreover, SrM hexaferrites have higher processability as compared to BaM ones [2].

SrM ferrite has found general application in permanent magnets [1, 2], active media of millimeter wavelength range UHF electronics [3], and THz photonics [4] due to its combination of magnetic properties and performance. Of special interest are anisotropic SrM hexaferrites [1–7]. Researchers and engineers are still also interested in the development of UHF electronic devices based on thick hexagonal ferrite films [5, 6]. Anisotropic SrM hexaferrites are currently commercially synthesized using the ceramic technology [1, 2, 5]. Schematic diagram of a typical anisotropic polycrystalline hexagonal SrM ferrite process based on the ceramic technology is presented in Fig. 1. Along with the multiple commonly recognized advantages, the latter technology has a number of drawbacks, the main one of which is its high energy consumption.

Alternative to the ceramic technology of anisotropic SrM hexaferrites is radiation-thermal sintering (RTS) which has been well tested for a number of ferrites [7–9], including barium hexaferrite (BaM) [7]. The RTS technology differs from the conventional ceramic one in that the thermal sintering of raw workpieces is replaced for sintering in a fast electron beam from an electron accelerator [7–9].

The aim of this work was to explore the possibility of synthesizing high-quality  $\text{SrFe}_{12}\text{O}_{19}$  hexaferrites using the RTS technology and to study the crystal and magnetic structure of the samples.

## 2. Experimental

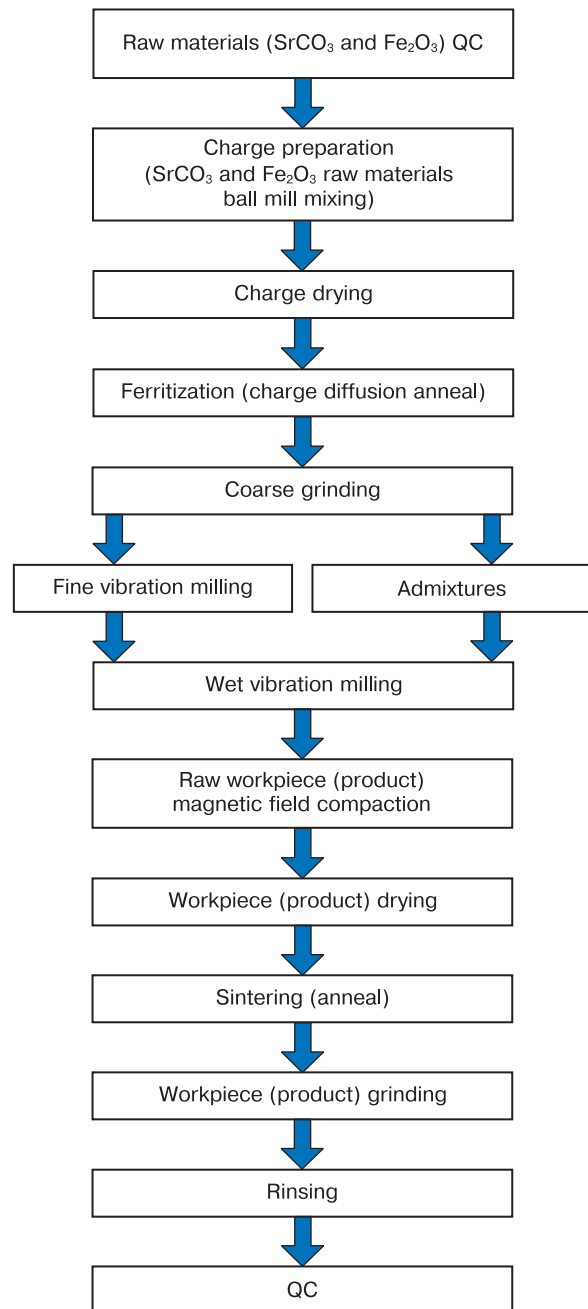
The iron-containing raw material for test sample preparation was MM-2 Grade  $\text{Fe}_2\text{O}_3$  iron oxide (TU6-09-4816-80, impurities (wt.%): 0.01  $\text{SiO}_2$ ; 0.01  $\text{Al}_2\text{O}_3$ ; 0.01  $\text{Cr}_2\text{O}_3$ ; 0.01  $\text{Na}_2\text{O}$ ). The strontium-containing raw material was crystalline strontium carbonate (Ch Grade, 99.0% purity) from the Isfara Chemical Plant, Tajikistan. The raw workpieces were produced following the chart shown in Fig. 1, except for the thermal sintering operation which was replaced for RTS. Replacement of thermal sintering for RTS in a fast electron beam offers advantage due to the considerably lower power consumption and higher sintering quality of RTS [7–9].

Along with temperature, an important factor of the RTS technology is radiation-enhanced diffusion. As a result, sintering occurs at lower temperatures and in a shorter time [7–9].

The samples were RTS processed with fast electrons in an ILU-6 linear accelerator designed for 2.5 MeV radiation technologies at the G.I. Budker Institute of Nuclear Physics of the Siberian Branch of the Russian Academy of Sciences [10]. Schematic of the accelerator is shown in Fig. 2.

The key parameters of the ILU-6 accelerator are as follows:

- maximum electron energy: 2.5 MeV;
- working frequency: 117 MHz;
- HF generator pulse power: to 2.5 MW;
- maximum pulse beam current: 450 mA;
- maximum average beam current: 8 mA;
- beam current pulse length: 0.5 ms;



**Figure 1.** Typical process diagram of ceramic technology of anisotropic polycrystalline hexagonal  $\text{SrFe}_{12}\text{O}_{19}$  ferrites

**Table 1.** RTS synthesis conditions of anisotropic SrFe<sub>12</sub>O<sub>19</sub> samples

| # | Raw workpiece # | Sample # | RTS temperature (°C) | RTS time (min) | Density (g/cm <sup>3</sup> ) |
|---|-----------------|----------|----------------------|----------------|------------------------------|
| 1 | 1-p6_an         | GSA_#1   | 1200                 | 60             | 4.53                         |
| 2 | 2-p6_an         | GSA_#2   | 1250                 | 60             | 4.96                         |
| 3 | 4-p6_an         | GSA_#4   | 1300                 | 10             | 4.95                         |
| 4 | 5-p6_an         | GSA_#5   | 1300                 | 30             | 5.01                         |
| 5 | 3-p6_an         | GSA_#3   | 1300                 | 60             | 5.00                         |
| 6 | 6-p6_an         | GSA_#6   | 1300                 | 90             | 4.98                         |
| 7 | 7-p6_an         | GSA_#7   | 1350                 | 40             | 5.08                         |
| 8 | 29-p6_an        | GSA_#29  | 1400                 | 40             | 5.09                         |

- pulse repetition frequency: to 50 Hz;
- height of ILU-6 accelerator vacuum tank with HF generator: 2+ m;
- scan horn window length: 980 mm;
- irradiated area width: 90 cm;
- electron beam power output to atmosphere: to 16 kW.

Fast electron beam sintering of hexagonal ferrites was carried out in the RTS cell for ferrite ceramics and ferrite products specially designed at the Chair of Electronics Materials Technology of the National University of Science and Technology “MISIS” [11]. Table 1 summarizes RTS synthesis conditions of anisotropic SrM samples.

The test samples were in the form of thin wafers (thickness  $h = 200$  mm) cut in a plane perpendicular to the hexagonal axis  $c$ .

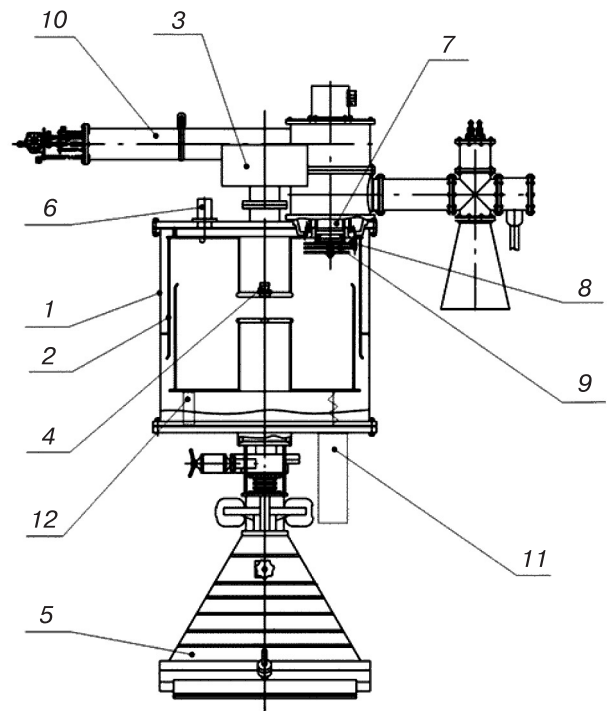
X-ray phase and structure studies were carried out on a DRON-8 X-ray diffractometer (Burevestnik Research and Production Co., St. Petersburg, Russia) in CoK<sub>α1</sub> radiation, wavelength  $\lambda = 0.178897$  nm. The beam was focused using the Bragg–Brentano method with two Soller slots. The measurements were conducted at room temperature.

The average coherent scattering region sizes ( $L_C$ ) for all the phases detected in the test specimens were calculated based on the X-ray diffraction patterns using the Debye–Scherrer formula:

$$L_C = \frac{k\lambda}{B \cos \theta}, \quad (1)$$

where  $L_C$  is the average coherent scattering region size, nm,  $k$  is the constant equaling 0.89,  $B$  is the HWHH of the first diffraction peak at the reflection angle  $\theta$  in the series of reflections for each of the detected phases, rad, and  $\lambda$  is the CoK<sub>α1</sub> radiation wavelength,  $\lambda = 0.178897$  nm.

The magnetic texture degree was determined by measuring the basis and reference reflection intensities of the test samples installed in a holder with a plane perpendicular to the sample's hexagonal axis which must be coincident with the texture direction. For hexaferrite, the most suitable reflections are the <008> and <107> ones as their intensities are the highest. Similar measurements were carried out for isotropic samples of the same



**Figure 2.** Schematic of ILU-6 linear accelerator: (1) vacuum tank, (2) resonator, (3) NMD type magneto-discharge pump (NMD series), (4) electron injector, (5) scan horn, (6) measuring circuit, (7) HF generator lamp anode, (8) HF power input circuit support, (9) HF power input circuit, (10) cathode bus, (11) 7 kV bias input, (12) resonator bottom section supports

composition. The texture degree was calculated using the following formula:

$$f = \frac{P - P_0}{1 - P_0} \cdot 100\%, \quad (2)$$

where  $P_0 = I_{008}^0 / (I_{008}^0 + I_{107}^0)$ ,  $P = I_{008} / (I_{008} + I_{107})$ , and  $I_{008}^0$ ,  $I_{107}^0$ ,  $I_{008}$  and  $I_{107}$  are the basis and reference reflection intensities for the textured and texture-free samples, respectively. The Mössbauer spectra of the samples were recorded on an MS-1104E spectrometer with a constant acceleration at room temperature, the  $\gamma$  radiation source being Co<sup>57</sup> in a chromium matrix. The isomeric (chemical) shifts were calculated relative to  $\alpha$ -Fe. The spectra were mathematically processed with the Univem Ms software.

The density of the samples was measured in accordance with Archimedes' law on a UW620H electronic balance with a density measurement attachment. The density calculation formula was as follows:

$$\rho = \rho_0 \left( \frac{W_a}{W_a - W_l} \right), \quad (3)$$

where  $\rho$  is the sample density ( $\text{g/cm}^3$ ),  $\rho_0$  is the known density of the liquid ( $\text{g/cm}^3$ ), and  $W_a$  and  $W_l$  are the sample weights in air and in the liquid, respectively (g).

The X-ray density of the hexaferrites was calculated based on the X-ray diffraction data (the average density was  $5.1 \text{ g/cm}^3$ ):

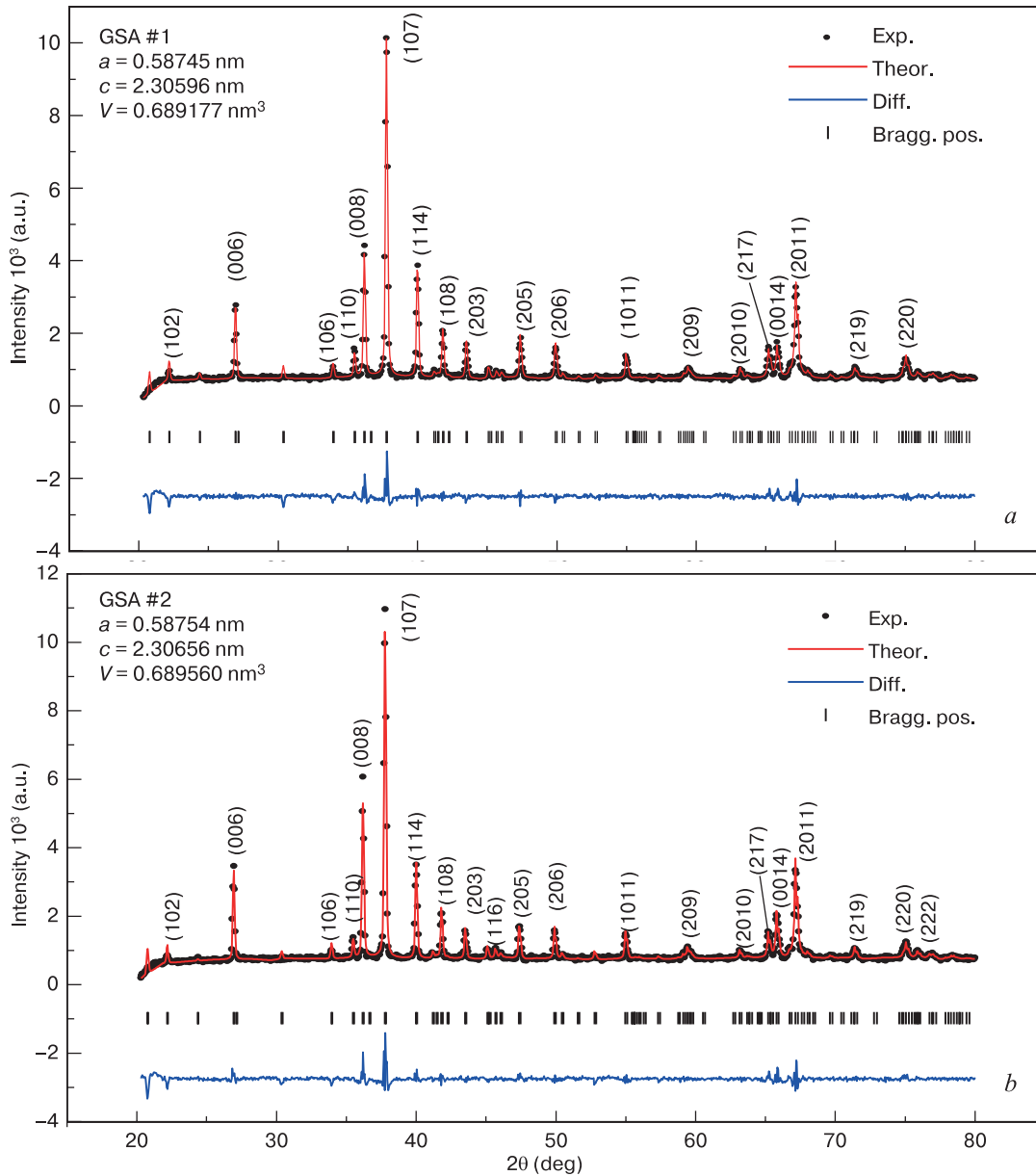
$$\rho_{\text{X-ray}} = \frac{2M_A}{VN_A} = \frac{2M_A \cdot 2}{ca^2 \cdot 3\sqrt{3} \cdot N_A}, \quad (4)$$

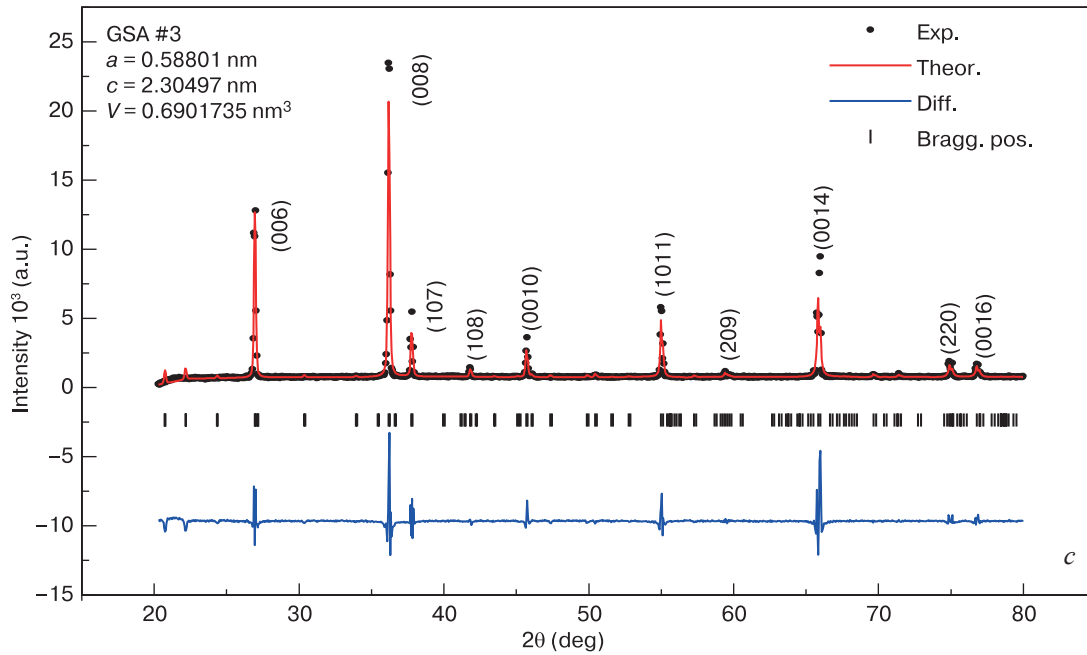
where  $M_A$  is the hexaferrite molecular weight,  $c$  and  $a$  are the hexaferrite unit cell parameters,  $N_A$  is the Avogadro number, and  $V$  is the unit cell volume.

### 3. Results and discussion

Analysis of the X-ray diffraction spectra of the synthesized samples showed that their structure was single-phase and had the  $P6_3/mmc$  (No. 194) space group, i.e., they were hexagonal ferrites. Figures 3 and 4 show X-ray diffraction spectra for some samples. Figure 3 shows the X-ray diffraction spectra of Samples ## 1–3 synthesized using RTS at 1200, 1250, and 1300 °C for 60 min. Figure 4 shows X-ray diffraction spectra of Samples ## 4–6 synthesized using RTS at 1300 °C for 10, 30, and 90 min.

Sintering time being the same (1 h), an increase in the RTS temperature from 1200 to 1300 °C leads to nonlinear and opposite changes in the unit cell parameters and volume (Fig. 5). However, the unit cell volume evolution (almost linear increase) suggests that the lattice parameter  $a$  increases faster than the lattice parameter  $c$  decreases.





**Figure 3.** X-ray diffraction spectra of anisotropic polycrystalline  $\text{SrFe}_{12}\text{O}_{19}$  hexaferrite samples (a) GSA #1, (b) GSA #2, and (c) GSA #3 synthesized using RTS for 60 min at 1200, 1250, and 1300 °C, respectively

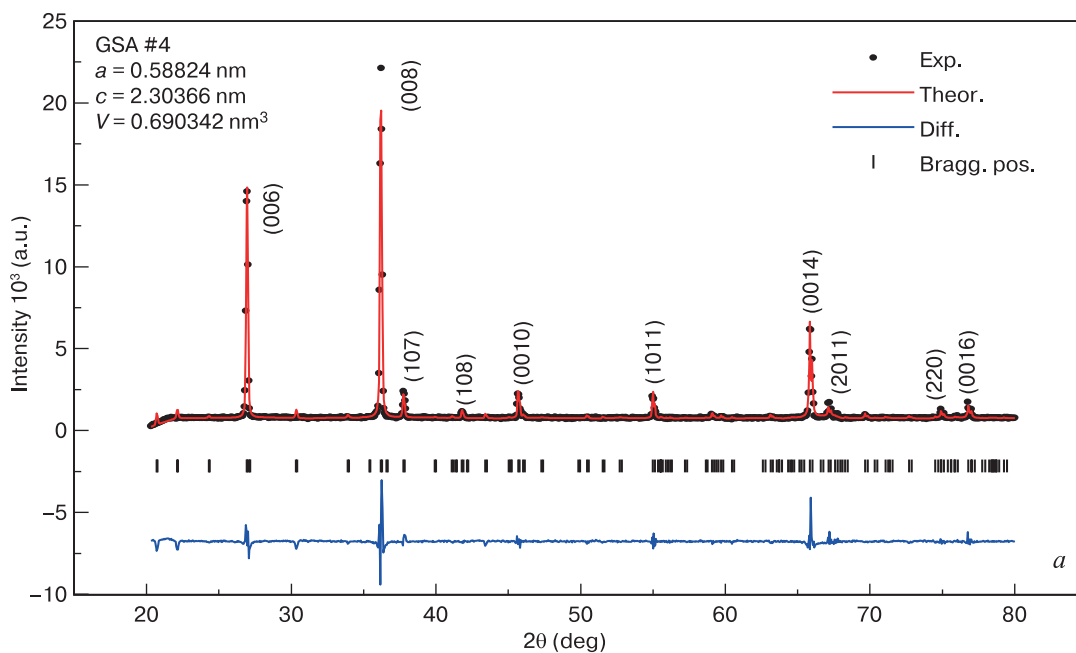
Sintering temperature being the same (1300 °C), an increase in the RTS time from 10 to 90 min also leads to nonlinear and opposite changes in the unit cell parameters and volume: the unit cell parameter  $a$  decreases whereas the unit cell parameter  $c$  increases (Fig. 6), indicating anisotropic unit cell distortion. The nonlinear unit cell volume evolution with process time at a constant temperature of 1300 °C (decrease in the 10–60 min interval and an abrupt increase for 90 min) originates from competitive changes in the unit cell parameters  $a$  and  $c$ .

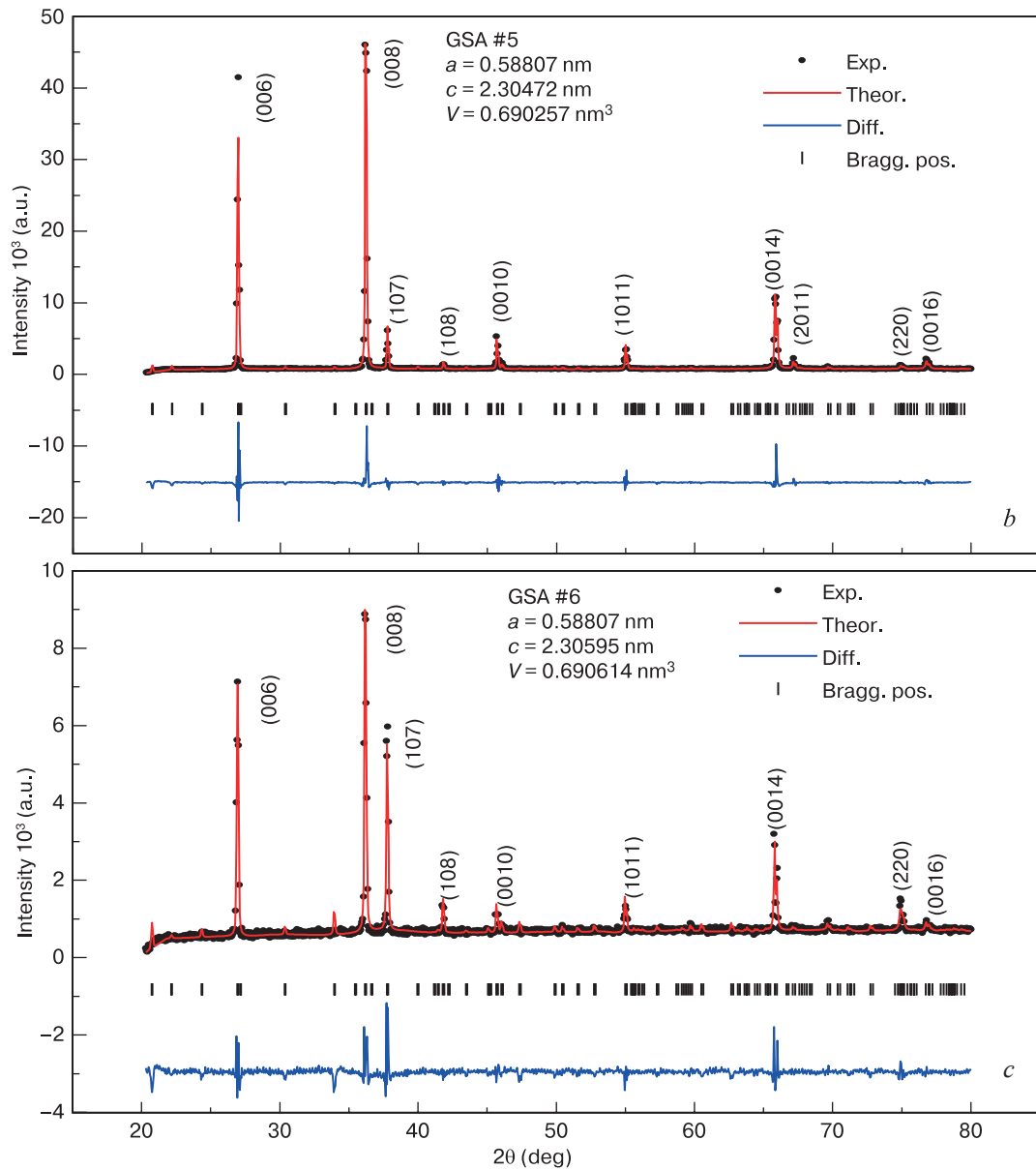
The data on the unit cell parameters and volume of the samples for the same RTS time (30–40 min) with an increase in temperature from 1300 to 1400 °C show a slight

increase in the unit cell parameters and volume with an increase in temperature (Fig. 7).

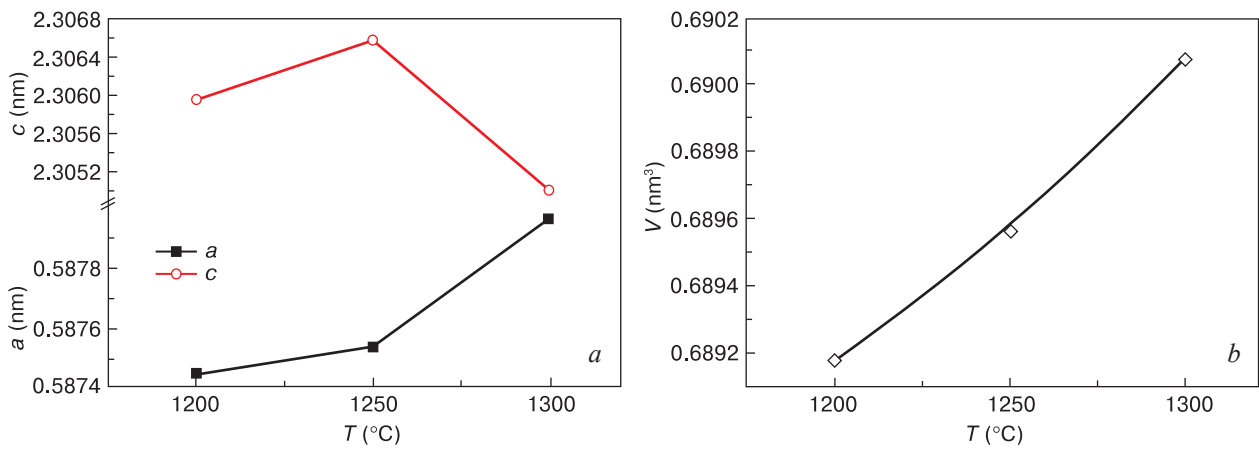
For the RTS temperature  $T = 1300$  °C, the X-ray diffraction spectra show a noticeable increase in the  $\langle 008 \rangle$  reflection intensity, suggesting the formation of a strong magnetic texture in the samples.

Figure 8 shows the magnetic texture degree  $f$  of the test samples as a function of ( $a$  and  $c$ ) RTS temperature and ( $b$ ) RTS time. As can be seen from Fig. 8  $f$  is the greatest (91.97%) for a  $\text{SrFe}_{12}\text{O}_{19}$  hexaferrite RTS synthesis temperature of 1300 °C and a time of 30 min. Further increase in the fast electron treatment time and the RTS temperature caused a decrease in  $f$ .

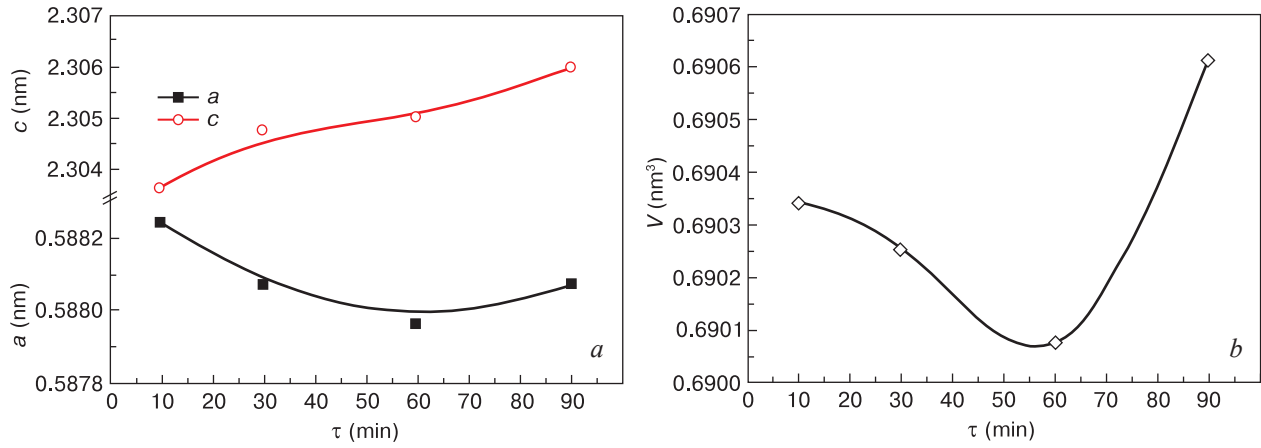




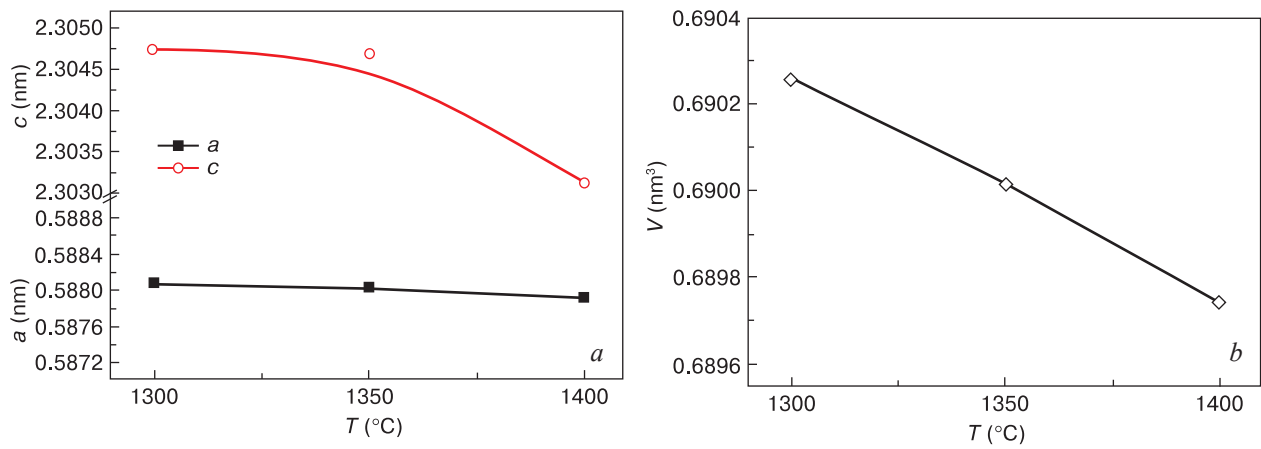
**Figure 4.** X-ray diffraction spectra of anisotropic polycrystalline  $\text{SrFe}_{12}\text{O}_{19}$  hexaferrite samples (a) GSA #4, (b) GSA #5, and (c) GSA #6 synthesized using RTS  $1300^\circ\text{C}$  for 10, 30, and 90 min, respectively



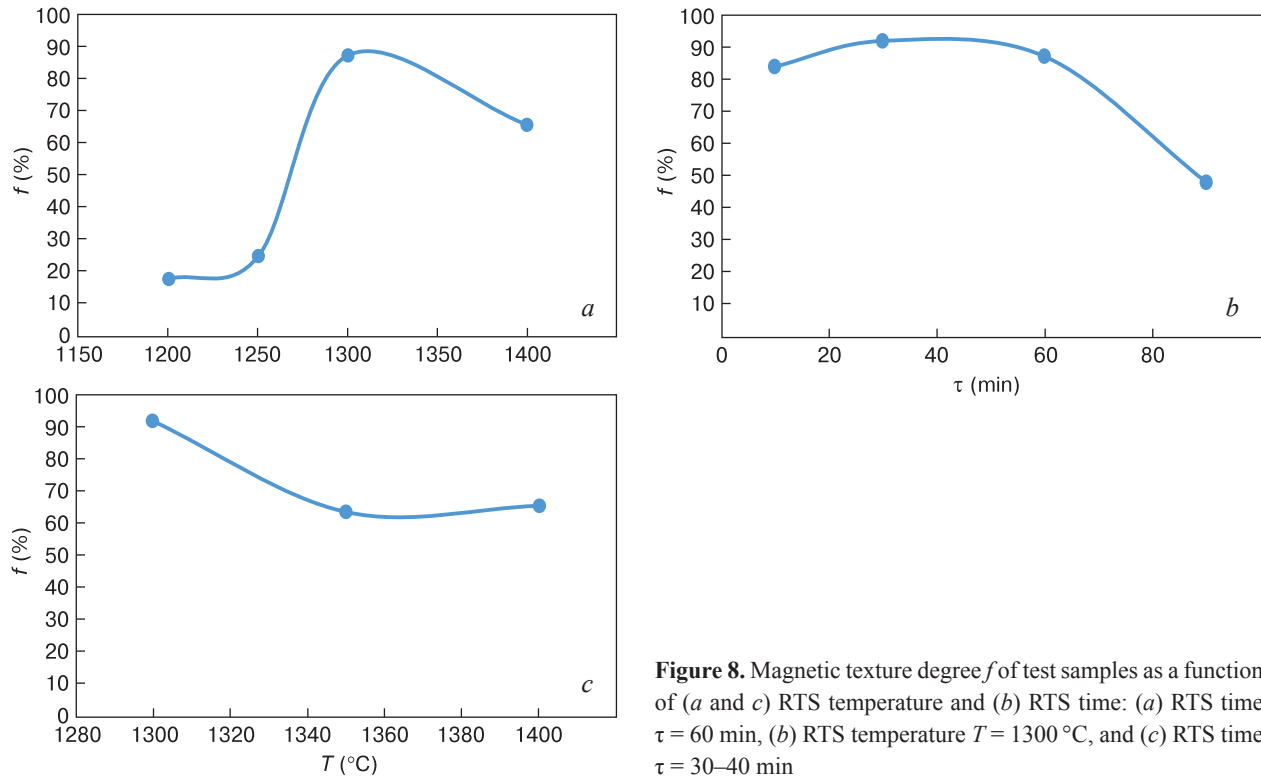
**Figure 5.** Evolution of (a) unit cell parameters and (b) unit cell volume of GSA samples synthesized using RTS at different temperatures (1200, 1250, and  $1300^\circ\text{C}$ ) for 1 h (## 1, 2, and 3, respectively)



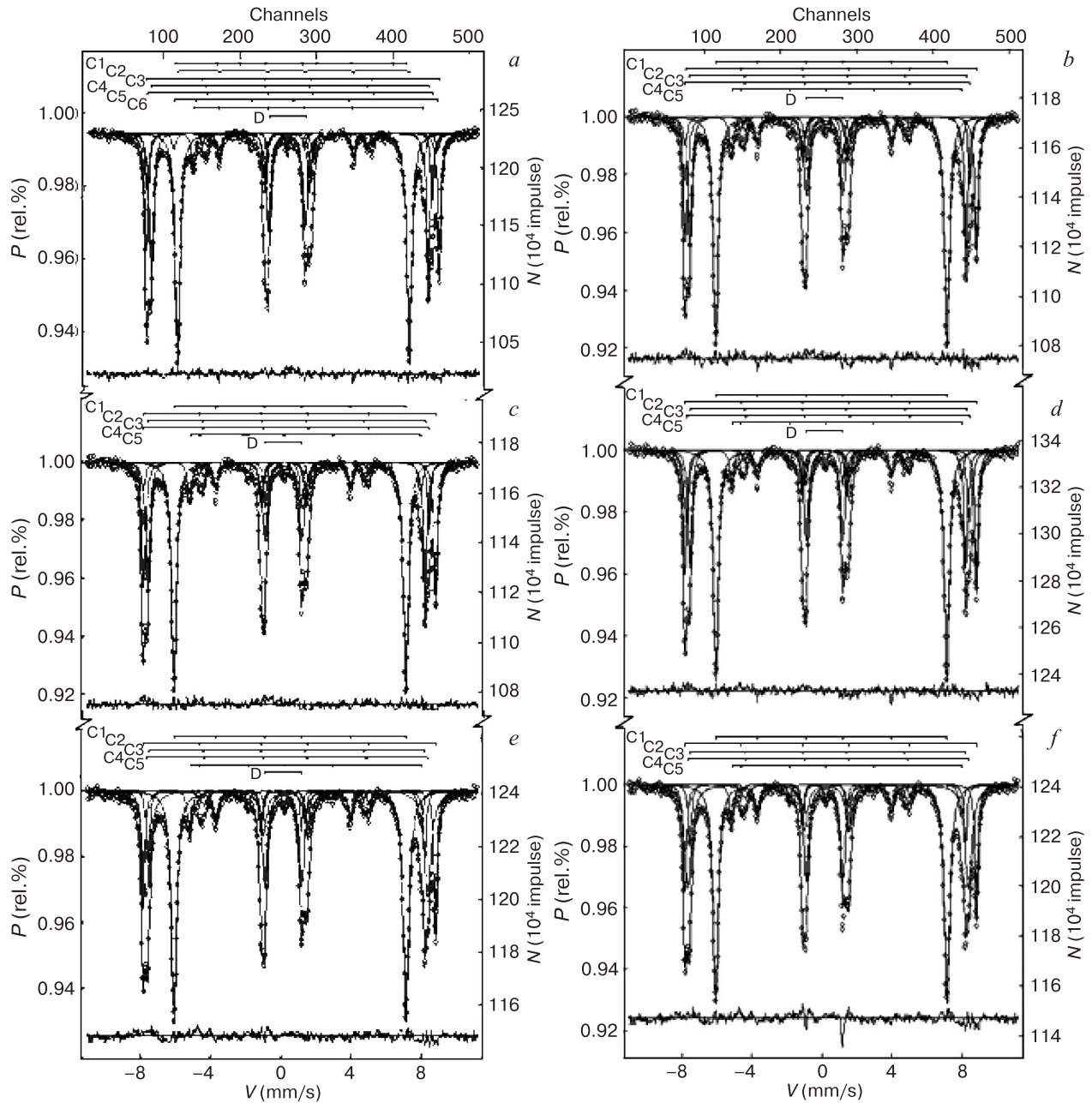
**Figure 6.** Evolution of (a) unit cell parameters and (b) unit cell volume of GSA samples synthesized with RTS for different times at 1300 °C (## 3, 4, 5, and 6, respectively)



**Figure 7.** Evolution of (a) unit cell parameters and (b) unit cell volume of GSA samples synthesized using RTS at different temperatures (1300, 1350, and 1400 °C) for 30 min (## 5, 7, and 29, respectively)



**Figure 8.** Magnetic texture degree  $f$  of test samples as a function of (a and c) RTS temperature and (b) RTS time: (a) RTS time  $\tau = 60$  min, (b) RTS temperature  $T = 1300$  °C, and (c) RTS time  $\tau = 30$ –40 min



**Figure 9.** Mössbauer spectra of anisotropic  $\text{SrFe}_{12}\text{O}_{19}$  hexaferrite wafers synthesized using RTS in the following modes ( $T, ^\circ\text{C} / \tau, \text{min}$ ): (a) 1200/60, (b) 1250/60, (c) 1300/60, (d) 1350/40, (e) 1400/30, and (f) 1400/30 (without doublet)

The Mössbauer spectra of the test samples were recorded using a standard technique [12]. Figure 9 shows the experimental Mössbauer spectra for different synthesis temperatures and times ( $T, ^\circ\text{C} / \tau, \text{min}$ ): 1200/60, 1250/60, 1300/60, 1350/40, and 1400/30.

The spectra of all the samples except the 1200/60 one were split into 5 sextets corresponding to five Fe ion positions in the  $M$ -type hexaferrite structure:  $12k$ ,  $4f_1$ ,  $4f_2$ ,  $2a$ , and  $2b$  [13] and an asymmetrical low-intensity doublet in which the  $\pi$ -component was predominant. The Mössbauer spectrum of the 1200/60 sample showed the best fit between the model and experimental curves when split into 6 sextets and a doublet. Mathematical processing data for the Mössbauer spectra of the sextets (isomeric shift ( $\delta$ , mm/s), quadruple splitting ( $\Delta$ , mm/s),  $\text{Fe}^{57}$  core

magnetic fields ( $H_{\text{eff}}$ , kOe), resonance bandwidths ( $G$ , mm/s), angle  $\theta$ , and sextet areas ( $S$ , rel.%) are summarized in Table 2.

It can be seen from Table 2 that for all the treatment modes, all the iron ions have a valence of  $3^+$  as suggested by the isomeric shifts, with all the parameters falling within the range typical of  $\text{Fe}^{3+}$  ions in the  $12k$ ,  $4f_2$ , and  $2a$  (octahedral),  $4f_1$  (tetrahedral), and  $2b$  (bi-pyramid) coordination positions. However, some parameter values differ from the theoretical ones. This is the case for the areas of  $\text{Fe}(4f_1)$  ion peaks that reach 23.3 rel.% (Sample 1400/30) vs the theoretical 16.7 rel.% figure. This can be accounted for by a higher probability of resonance for the tetrahedral  $\text{Fe}^{3+}$  ions as compared to the octahedral ones, as explained earlier [14]. On the other hand,

**Table 2.** Parameters of Mössbauer spectra for RTS-synthesized anisotropic SrFe<sub>12</sub>O<sub>19</sub> hexaferrite wafers

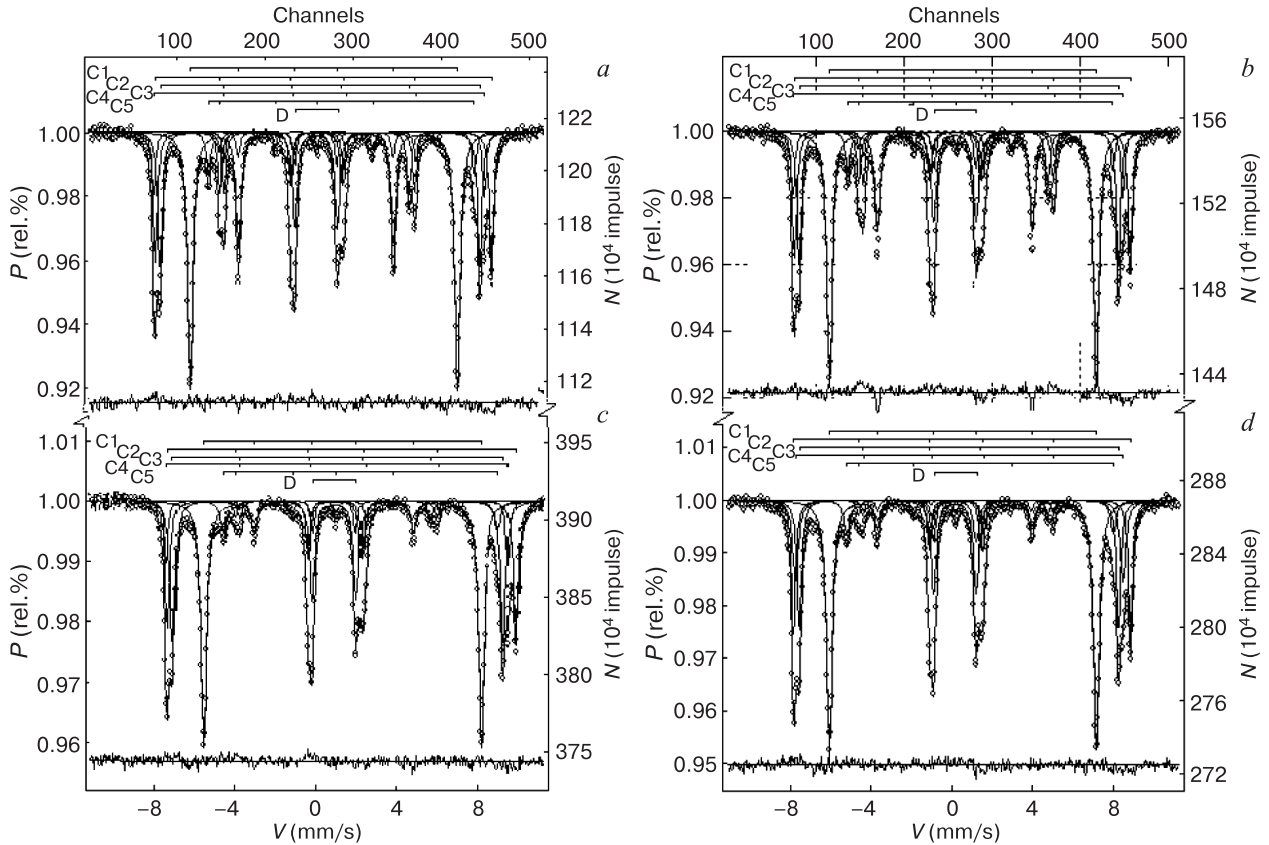
| SrFe <sub>12</sub> O <sub>19</sub><br>sample <i>T</i> / $\tau$<br>(°C/min) | Spectral<br>features         | Isomeric shift<br>$\delta$ (mm/s) | Quadruple splitting<br>$\Delta$ (mm/s) | Magnetic fields<br>$H_{\text{eff}}$ (kOe) | Feature areas<br><i>S</i> (%) | Bandwidth <i>G</i><br>(mm/s) | Angle $\theta$<br>(arc deg) |
|----------------------------------------------------------------------------|------------------------------|-----------------------------------|----------------------------------------|-------------------------------------------|-------------------------------|------------------------------|-----------------------------|
| 1200/60                                                                    | C1(12 <i>k</i> )             | 0.35                              | 0.39                                   | 410                                       | 43.6                          | 0.31                         | 45.1                        |
|                                                                            | C2(4 <i>f</i> <sub>2</sub> ) | 0.36                              | 0.28                                   | 517                                       | 17.5                          | 0.24                         |                             |
|                                                                            | C3(4 <i>f</i> <sub>1</sub> ) | 0.26                              | 0.18                                   | 490                                       | 18.8                          | 0.25                         |                             |
|                                                                            | C4(2 <i>a</i> )              | 0.35                              | 0.04                                   | 503                                       | 8.9                           | 0.25                         |                             |
|                                                                            | C5(2 <i>b</i> )              | 0.29                              | 2.23                                   | 407                                       | 5.6                           | 0.24                         |                             |
|                                                                            | C6(12 <i>k'</i> )            | 0.34                              | -0.39                                  | 237                                       | 3.0                           | 0.22                         |                             |
|                                                                            | D(Fe <sup>3+</sup> )         | 0.15                              | 2.07                                   |                                           | 2.6                           | 0.21                         |                             |
| 1250/60                                                                    | C1(12 <i>k</i> )             | 0.35                              | 0.39                                   | 410                                       | 44.9                          | 0.32                         | 39.7                        |
|                                                                            | C2(4 <i>f</i> <sub>2</sub> ) | 0.37                              | 0.32                                   | 516                                       | 18.6                          | 0.26                         |                             |
|                                                                            | C3(4 <i>f</i> <sub>1</sub> ) | 0.27                              | 0.17                                   | 490                                       | 19.8                          | 0.26                         |                             |
|                                                                            | C4(2 <i>a</i> )              | 0.33                              | -0.04                                  | 507                                       | 8.6                           | 0.23                         |                             |
|                                                                            | C5(2 <i>b</i> )              | 0.29                              | 2.23                                   | 407                                       | 5.6                           | 0.25                         |                             |
|                                                                            | D(Fe <sup>3+</sup> )         | 0.16                              | 2.07                                   |                                           | 2.5                           | 0.21                         |                             |
| 1300/60                                                                    | C1(12 <i>k</i> )             | 0.35                              | 0.39                                   | 410                                       | 46.0                          | 0.34                         | 23.4                        |
|                                                                            | C2(4 <i>f</i> <sub>2</sub> ) | 0.37                              | 0.32                                   | 516                                       | 17.3                          | 0.27                         |                             |
|                                                                            | C3(4 <i>f</i> <sub>1</sub> ) | 0.27                              | 0.17                                   | 491                                       | 19.6                          | 0.30                         |                             |
|                                                                            | C4(2 <i>a</i> )              | 0.34                              | -0.03                                  | 507                                       | 9.2                           | 0.21                         |                             |
|                                                                            | C5(2 <i>b</i> )              | 0.30                              | 2.23                                   | 408                                       | 5.4                           | 0.35                         |                             |
|                                                                            | D(Fe <sup>3+</sup> )         | 0.16                              | 2.03                                   |                                           | 2.5                           | 0.21                         |                             |
| 1350/40                                                                    | C1(12 <i>k</i> )             | 0.36                              | 0.39                                   | 410                                       | 43.6                          | 0.37                         | 23.2                        |
|                                                                            | C2(4 <i>f</i> <sub>2</sub> ) | 0.37                              | 0.30                                   | 518                                       | 16.8                          | 0.23                         |                             |
|                                                                            | C3(4 <i>f</i> <sub>1</sub> ) | 0.26                              | 0.18                                   | 490                                       | 19.6                          | 0.49                         |                             |
|                                                                            | C4(2 <i>a</i> )              | 0.32                              | 0.12                                   | 500                                       | 11.5                          | 0.21                         |                             |
|                                                                            | C5(2 <i>b</i> )              | 0.29                              | 2.23                                   | 409                                       | 5.2                           | 0.34                         |                             |
|                                                                            | D(Fe <sup>3+</sup> )         | 0.15                              | 2.08                                   |                                           | 3.3                           | 0.21                         |                             |
| 1400/30                                                                    | C1(12 <i>k</i> )             | 0.36                              | 0.39                                   | 410                                       | 43.8                          | 0.37                         | 22.5                        |
|                                                                            | C2(4 <i>f</i> <sub>2</sub> ) | 0.37                              | 0.30                                   | 517                                       | 14.8                          | 0.23                         |                             |
|                                                                            | C3(4 <i>f</i> <sub>1</sub> ) | 0.25                              | 0.18                                   | 489                                       | 23.3                          | 0.49                         |                             |
|                                                                            | C4(2 <i>a</i> )              | 0.33                              | 0.11                                   | 497                                       | 9.3                           | 0.21                         |                             |
|                                                                            | C5(2 <i>b</i> )              | 0.30                              | 2.21                                   | 408                                       | 5.2                           | 0.28                         |                             |
|                                                                            | D(Fe <sup>3+</sup> )         | 0.14                              | 2.10                                   |                                           | 3.6                           | 0.21                         |                             |

one can note a decrease in the angle  $\theta$  from 55 deg with an increase in the synthesis temperature at a constant time, resulting in an increase in the magnetic texture degree of the sample. For the lowest synthesis temperature 1200 °C, a sixth sextet emerges in the spectrum due to the formation of nonequivalent Fe<sup>3+</sup> ion positions (12*k'*) at the expense of 12*k* ones. Isomorphous substitutions being absent, the latter nonequivalent positions can be generated by structural defects.

For determining the effect of synthesis time on the magnetic parameters of the samples, Mössbauer spectra were taken for SrFe<sub>12</sub>O<sub>19</sub> hexaferrites synthesized at

1300 °C for 10, 30, 60, and 90 min (Fig. 10). Table 3 presents the respective Mössbauer spectrum parameters.

Figure 10 and Table 3 show that, by analogy with synthesis temperature variation, similar Mössbauer spectra of anisotropic SrFe<sub>12</sub>O<sub>19</sub> hexaferrites were obtained. One can also note the slightly greater peak areas of the C3 sextets for the Fe<sup>3+</sup> ions in the octahedral positions, especially for the 1300 °C/10 min sample. Another common feature of the sample spectra is the spectral doublet occurring for both synthesis modes. The doublet originates from the elevated  $\chi^2$  parameter, reaching 3 or greater. The latter parameter could therefore be reduced to 1.8



**Figure 10.** Mössbauer spectra of anisotropic  $\text{SrFe}_{12}\text{O}_{19}$  hexaferrite wafers synthesized using RTS at  $1300^\circ\text{C}$  for different times  $\tau$ , min: (a) 10, (b) 30, (c) 60, and (d) 90

or lower. Figure 9 shows, by way of comparison, the Mössbauer spectrum of the  $1400^\circ\text{C}/30\text{min}$  sample split in five sextets only. The doublet is distinguished by a weak isomeric shift ( $0.14\text{--}0.16\text{ mm/s}$ ), high quadruple splitting ( $2\text{--}2.1\text{ mm/s}$ ), and asymmetrical peaks with  $\pi/\sigma = 1.8$ . Comparing the anomalously high quadruple splitting of the Fe ions in the doublet with the respective parameters of the Fe ions in the  $2b$  positions (Tables 2 and 3), one can see their close values. This is most likely not an occasion. Furthermore, the total  $2b$  doublet and sextet peak area is close to the theoretical one within the experimental error,  $\sim 8.3\text{ rel.}\%$ . Thus, the presence of the doublet in the spectra can be accounted for by the formation of bipyramids with oxygen vacancies on the surface of the hexaferrite wafers. This reduces the coordination number and the observed isomeric shift. For the lowest  $2b$ -positioned  $\text{Fe}^{57}$  ion core field of  $408\text{ kOe}$ , only three magnetic bonds of the five will be active, since if there is one oxygen vacancy, one more oxygen atom will be at a large distance from the iron ion. The bond angles being unfavorable, those ions will undergo no Seeman magnetic splitting. Significant local distortions of such a tetrahedron will create a large electric field gradient and, accordingly, a high quadrupole splitting, which we observe for doublets in the studied spectra. It is impossible to interpret the doublet as a sextet with a low  $\text{Fe}^{57}$  ion core magnetic field of about  $63\text{ kOe}$ , since even if the second and fifth peak intensities were low due to the

low angle  $\theta$  and the third and fourth peak intensities were within the experimental error, the intensities of the fifth and sixth peaks would be equal; however, their actual intensity ratio is  $1:1.8$ .

The measured angle  $\theta$  is closer to the longitudinal magnetic field orientation and the  $\gamma$ -ray direction for which the second and fifth peak intensities decrease. Since the magnetic moment of hexaferrite is controlled by the difference between the magnetic moments of the ions in the  $12k$ ,  $2a$ , and  $2b$  positions and the magnetic moments of the ions in the  $4f_1$  and  $4f_2$  ones, the total area of the respective sextets and their difference  $\Delta S$  will be proportional to the resultant magnetic moment of the sample.  $\Delta S$  calculation from analysis of the Mössbauer spectra is presented in Table 4.

Analyzing the data summarized in Table 4, one should expect that the minimum magnetization of the sample will correspond to the minimum DS and hence the least favorable synthesis mode. The maximum difference between the iron ion sextet areas shown above for the  $1300^\circ\text{C}/60\text{ min}$  and  $1350^\circ\text{C}/40\text{ min}$  samples suggests that the magnetization will be the highest in those samples, and hence the respective process modes are the closest to the optimum synthesis mode.

Figure 11 shows the density  $\rho$  of the anisotropic  $\text{SrFe}_{12}\text{O}_{19}$  hexaferrites synthesized using RTS as a function of (a and c) sintering temperature and (b) sintering time for  $T = 1300^\circ\text{C}$ .

**Table 3.** Mössbauer spectrum parameters of anisotropic SrFe<sub>12</sub>O<sub>19</sub> hexaferrite wafers synthesized using RTS at 1300 °C for different times

| RTS Mode (°C/min) | Spectral features    | Isomeric shift $\delta$ , (mm/s) | Quadruple splitting $\Delta$ (mm/s) | Magnetic fields $H_{\text{eff}}$ (kOe) | Feature areas $S$ (%) | Bandwidth $G$ (mm/s) | Angle $\theta$ (arc deg) |
|-------------------|----------------------|----------------------------------|-------------------------------------|----------------------------------------|-----------------------|----------------------|--------------------------|
| 1300/10           | C1(12k)              | 0.35                             | 0.39                                | 410                                    | 44.6                  | 0.34                 | 21.0                     |
|                   | C2(4f <sub>2</sub> ) | 0.35                             | 0.30                                | 517                                    | 18.5                  | 0.25                 |                          |
|                   | C3(4f <sub>1</sub> ) | 0.27                             | 0.18                                | 490                                    | 22.2                  | 0.29                 |                          |
|                   | C4(2a)               | 0.37                             | -0.01                               | 504                                    | 8.7                   | 0.24                 |                          |
|                   | C5(2b)               | 0.21                             | 2.21                                | 407                                    | 5.2                   | 0.31                 |                          |
|                   | D(Fe <sup>3+</sup> ) | 0.16                             | 2.                                  |                                        | 0.8                   | 0.21                 |                          |
| 1300/30           | C1(12k)              | 0.35                             | 0.39                                | 410                                    | 44.8                  | 0.33                 | 22.8                     |
|                   | C2(4f <sub>2</sub> ) | 0.36                             | 0.30                                | 518                                    | 18.0                  | 0.25                 |                          |
|                   | C3(4f <sub>1</sub> ) | 0.27                             | 0.17                                | 490                                    | 18.7                  | 0.27                 |                          |
|                   | C4(2a)               | 0.37                             | 0.02                                | 503                                    | 11.0                  | 0.29                 |                          |
|                   | C5(2b)               | 0.32                             | 2.19                                | 408                                    | 5.7                   | 0.33                 |                          |
|                   | D(Fe <sup>3+</sup> ) | 0.18                             | 2.04                                |                                        | 1.8                   | 0.21                 |                          |
| 1300/60           | C1(12k)              | 0.35                             | 0.39                                | 410                                    | 46.0                  | 0.34                 | 23.4                     |
|                   | C2(4f <sub>2</sub> ) | 0.37                             | 0.32                                | 516                                    | 17.3                  | 0.27                 |                          |
|                   | C3(4f <sub>1</sub> ) | 0.27                             | 0.17                                | 491                                    | 19.6                  | 0.30                 |                          |
|                   | C4(2a)               | 0.34                             | -0.03                               | 507                                    | 9.2                   | 0.21                 |                          |
|                   | C5(2b)               | 0.30                             | 2.23                                | 408                                    | 5.4                   | 0.35                 |                          |
|                   | D(Fe <sup>3+</sup> ) | 0.16                             | 2.03                                |                                        | 2.5                   | 0.21                 |                          |
| 1300/90           | C1(12k)              | 0.35                             | 0.40                                | 411                                    | 45.5                  | 0.30                 | 22.3                     |
|                   | C2(4f <sub>2</sub> ) | 0.37                             | 0.30                                | 518                                    | 17.2                  | 0.24                 |                          |
|                   | C3(4f <sub>1</sub> ) | 0.29                             | 0.18                                | 491                                    | 20.0                  | 0.28                 |                          |
|                   | C4(2a)               | 0.36                             | -0.02                               | 506                                    | 8.1                   | 0.29                 |                          |
|                   | C5(2b)               | 0.29                             | 2.22                                | 407                                    | 6.3                   | 0.31                 |                          |
|                   | D(Fe <sup>3+</sup> ) | 0.15                             | 2.10                                |                                        | 2.9                   | 0.21                 |                          |

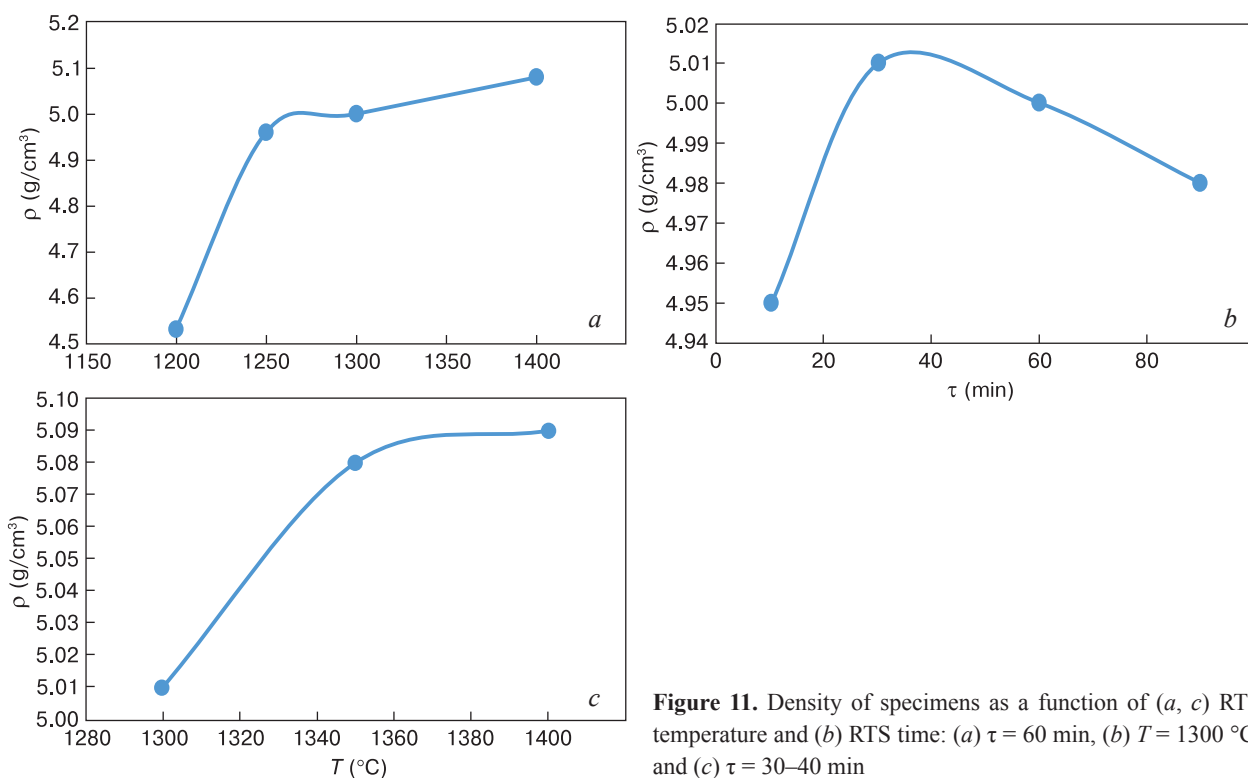
**Table 4.** Difference between sextet areas  $\Delta S$  for Fe<sup>3+</sup> ions with opposite magnetic moments 12k, 2a, 2b and 4f<sub>1</sub>, 4f<sub>2</sub>

| RTS Mode (°C/min) | 1200/60 | 1250/60 | 1300/60 | 1350/40 | 1400/30 | 1300/10 | 1300/30 | 1300/90 |
|-------------------|---------|---------|---------|---------|---------|---------|---------|---------|
| $\Delta S$        | 22.0    | 20.7    | 23.7    | 23.9    | 20.2    | 20.0    | 24.8    | 23.7    |

A common feature of the SrFe<sub>12</sub>O<sub>19</sub> specimens is a decrease in the density with an increase in the RTS time (for the RTS temperature 1300 °C) and an increase with the RTS temperature within the experimental RTS temperatures and times.

It should be noted that some of the above results for SrFe<sub>12</sub>O<sub>19</sub> strontium hexaferrite are similar to earlier ones for other ferrite materials [7–9, 15–23], especially barium hexaferrite [15]. Interestingly, RTS sintering of ferrites can be completed in several dozens of minutes. The key factor delivering this advantage is the RTS temperature which is specific for each material. Today, the physical processes underlying the acceleration of ceramic sintering

in fast electron beams are still incompletely clear [16–19, 22, 23]. The most widespread explanation, commonly accepted by experts, is that the predominant mechanism of radiation-enhanced mass transport in ferrite ceramics is surface recombination. Irradiation of ceramic materials in the form of grains and compacted powders generates electron excitations which tend to localize at grain or phase boundaries, where they undergo recombination and release energy and heat. The above processes produce temperature gradients that trigger diffusion flows and thereby tangibly accelerate the ferrite solid-state synthesis reactions.



**Figure 11.** Density of specimens as a function of (a, c) RTS temperature and (b) RTS time: (a)  $\tau = 60$  min, (b)  $T = 1300$  °C, and (c)  $\tau = 30$ –40 min

## 4. Conclusions

Anisotropic polycrystalline hexagonal  $\text{SrFe}_{12}\text{O}_{19}$  ferrites were for the first time synthesized using RTS. The crystal and magnetic structure and some physical properties of the samples were studied. The results suggest the following conclusions.

The  $\text{SrFe}_{12}\text{O}_{19}$  samples synthesized using RTS in this work were single-phase.

RTS can be used as an alternative technology for synthesizing anisotropic hexagonal  $\text{SrFe}_{12}\text{O}_{19}$  ferrite. As compared to the conventional ceramic thermal sintering technology, RTS proved to be highly energy efficient and cheap.

The sintering temperature plays a greater role in RTS than the sintering time.

RTS allows synthesizing anisotropic hexaferrites with a high (> 90%) magnetic texture degree.

Anisotropic unit cell distortion in RTS-synthesized  $\text{SrFe}_{12}\text{O}_{19}$  hexaferrites seems to originate from oxygen vacancy generation by fast electrons in the lattice. Quite probably, improving the parameters of  $\text{SrFe}_{12}\text{O}_{19}$  samples will require additional final short-time annealing in an oxygen gas atmosphere.

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## Conflict of interests

The Author declares there is no conflict of interests.

## Additional information

No AI tools were used in the making of this article.

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