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# Tuning the structural and magnetic properties of electrospun strontium-iron-oxide nanofibers with different stoichiometry

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A R T I C L E I N F O	A B S T R A C T
Keywords: Ferrite nanofibers Electrospinning Microstructure Magnetic properties	Nanostructured ferrites such as SrFeO <sub>3-x</sub> , SrFe <sub>2</sub> O <sub>4</sub> and SrFe <sub>12</sub> O <sub>19</sub> have great potential applications as permanent magnets due to their dynamic structures and excellent magnetic properties. However, the crystal structures of SrFeO <sub>3-x</sub> and SrFe <sub>2</sub> O <sub>4</sub> nanomaterials remain controversial and their magnetic properties have large room for improvements. Attempting to tackle these problems, we fabricated strontium ferrite nanofibers with Sr:Fe ratios of 1:1, 1:2, and 1:12 using electrospinning followed by annealing at 850 °C in air. Scanning electron microscopy and transmission electron microscopy (TEM) examinations showed that the nanofibers with average diameters of 130–140 nm are made of nanograins. Through X-ray diffraction and TEM analysis, the crystal structures for nanofibers with Sr:Fe ratios of 1:1, 1:2, and 1:12 were identified as tetragonal SrFeO <sub>2.86</sub> , orthorhombic SrFe <sub>2</sub> O <sub>4</sub> and hexagonal SrFe <sub>12</sub> O <sub>19</sub> , respectively. Extensive high-resolution TEM examinations showed the formation of stacking faults and edge dislocations in SrFeO <sub>2.86</sub> nanofibers, and edge dislocations in SrFe <sub>12</sub> O <sub>19</sub> nanofibers. Hysteresis loop measurements showed the three nanofibers had a similar coercive force (H <sub>c</sub> ), but different saturation magnetization (M <sub>s</sub> ) and residual magnetization (M <sub>r</sub> ). SrFe <sub>12</sub> O <sub>19</sub> nanofibers possessed the largest M <sub>s</sub> and M <sub>r</sub> , while SrFeO <sub>2.86</sub> nanofibers showed the lowest. The experimental M <sub>r</sub> values agreed well with magnetic moments obtained from density functional theory calculations. This work clarifies the crystal structure of Sr-Fe-O nanofibers, and provides a good candidate for permanent magnet applications.

#### 1. Introduction

Permanent magnets are prevalent in modern life, which have been applied to motors, read-heads, and sensors in automobiles, computers and household electronics [1]. In the past decades, rare earth materials such as Nd-Fe-B alloys have dominated the commercial market due to their superior magnetic properties. However, long-standing drawbacks such as low coercivity, low temperature stability and poor corrosion resistance hinder their practical applications in certain areas [2]. Thus, people have been hunting for substitutes that are more tolerant to harsh environments. As a good alternative, ferrite magnets [3-5] like Pb-Fe-O, Ba-Fe-O and Sr-Fe-O have been found to possess high coercive force, good temperature stability and excellent corrosion resistance, which have triggered great interest in the research community.

Recently, researchers have carried out extensive research on fabrication, structure characterization and magnetic properties of nanostructured Sr-Fe-O ferrites. Most research focuses on helimagnetic SrFeO<sub>3</sub> and SrFeO<sub>3-x</sub> [6,7], ferrimagnetic SrFe<sub>2</sub>O<sub>4</sub> [8-12] and SrFe<sub>12</sub>O<sub>19</sub> [13-16]. It has been found that these ferrite nanostructures show

considerable enhancements in magnetic properties compared to their bulk counterparts. This opens up new prospects for the applications of ferrite nanostructures as permanent magnets.

For fabrication of these ferrite nanomaterials, researchers have used various techniques such as co-precipitation method [6,7], sol-gel method [8-10,14], citrate autocombustion technique [11], electrospinning [12,13], hydrothermal method [15], and ultrasound radiation [16]. Among these techniques, electrospinning has proven to be an easy and reliable method to produce ferrite nanofibers because their anisotropy, dimension and microstructure can be tuned to obtain excellent magnetic properties. Up to now, electrospinning has been adopted to fabricate SrFe<sub>2</sub>O<sub>4</sub> [12] and SrFe<sub>12</sub>O<sub>19</sub> [13] nanofibers, but no reports have been found for the fabrication of pristine SrFeO<sub>3-x</sub> nanofibers.

Regarding the crystal structure of Sr-Fe-O ferrites, although some progress on structural characterization has been achieved, there are still controversies for SrFeO3-x and SrFe2O4 nanostructures. For bulk SrFeO3x, with an increase of oxygen vacancies, four distinct phases were found with cubic (x  $\approx$  0), tetragonal (x  $\approx$  0.125), orthorhombic (x $\approx$  0.25), and brownmillerite (x  $\approx$  0.5) structures. [17] However, only cubic structures

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 $(a \approx 3.87 \text{ Å})$  were reported for SrFeO<sub>3-x</sub> nanoparticles with different oxygen stoichiometries (x = 0.135 [6], x = 0.05 [7]). For nanostructured SrFe<sub>2</sub>O<sub>4</sub>, there are huge discrepancies on its crystal structure. Studies on nanoparticles predominantly reported a cubic structure but with different lattice parameters (a = 8.25 Å [8], a = 6.62 Å [9]), while others claimed an orthorhombic [10] or monoclinic [11] structure. For nanofibers, quite few reports were found for SrFe<sub>2</sub>O<sub>4</sub>, in which an orthorhombic structure was proclaimed [12]. In contrast to above two ferrites, a hexagonal structure is widely acknowledged for SrFe<sub>12</sub>O<sub>19</sub> nanostructures [13-16]. Therefore, it is imperative to explore and clarify the crystal structures for  $\rm SrFeO_{3-x}$  and  $\rm SrFe_2O_4$  nanomaterials. In addition, the crystal structures for these three nanostructured ferrites were mainly determined using X-ray diffraction (XRD) analysis but lacking corroboration from transmission electron microscopy (TEM) examination. Thus, extensive TEM investigation is urgently required to clarify their structures and explore the microstructural defects in the nanostructured ferrites.

As for magnetic properties of ferrite nanostructures, most research focused on nanoparticles. For nanofibers, contrary to abundant investigations on ferrimagnetic  $SrFe_{12}O_{19}$ , research on helimagnetic  $SrFeO_{3-x}$  and ferrimagnetic  $SrFe_{2}O_{4}$  is very rare. For  $SrFe_{12}O_{19}$ , both nanofibers and nanoparticles show excellent magnetic properties [13–16]. For  $SrFeO_{3-x}$ , both cubic  $SrFeO_{3}$  and tetragonal  $SrFeO_{2.875}$  are quoted as helimagnetic [17–19], but hard magnetic properties, characteristic of helimagnetism, are not present in hysteresis loops for nanoparticles [6,7]. In addition, the magnetic parameters for  $SrFeO_{3-x}$  were all obtained from nanoparticles. For  $SrFe_{2}O_{4}$ , previous studies mainly focused on magnetic properties of nanoparticles [8,9,11], and no magnetic parameters were obtained for nanofibers. Thus, it is necessary to study and compare the magnetic properties of the three ferrite nanofibers. In addition, it is desirable to correlate magnetic properties with microstructure of the three ferrite nanofibers.

To understand the origin of magnetism in the ferrites, researchers have carried out extensive density functional theory (DFT) calculations [20-22]. For SrFe<sub>12</sub>O<sub>19</sub>, most calculations adopted a hexagonal structure with a ferrimagnetic state. For example, Hmŏk et al. [20] obtained a total magnetic moment of 36.7  $\mu_B$ . For SrFe<sub>2</sub>O<sub>4</sub>, most calculations focused on monoclinic structure with different magnetic states. Calculations of ferromagnetic, antiferromagnetic, and ferrimagnetic states yielded a total magnetic moment of 8.56  $\mu_B$ , 0  $\mu_B$ , and 0.03  $\mu_B$ , respectively [21]. For SrFeO<sub>3</sub> and SrFeO<sub>2.875</sub>, their helical magnetic structure cannot be reproduced due to limitations of theoretical modelling. Therefore, most studies used a ferromagnetic model to replace its helical magnetic ordering. Shein et al. [22] calculated a total magnetic moment of  $3.65 \mu_B$  for cubic SrFeO<sub>3</sub> using this approximation. Up to now, no DFT calculation has been found for the magnetic moments of orthorhombic SrFe<sub>2</sub>O<sub>4</sub> and tetragonal SrFeO<sub>3-x</sub>. Furthermore, there is no direct comparison of magnetic moments obtained from hysteresis loops and theoretical calculations in previous work.

In this paper, Sr-Fe-O nanofibers with different stoichiometries were prepared by electrospinning followed by high-temperature annealing. Their morphology, crystal structure, and microstructural defects were characterized using scanning electron microscopy (SEM), XRD, and TEM. The valence states of iron in three ferrite nanofibers were determined using X-ray photoelectron spectroscopy (XPS). The magnetic properties of the three ferrite nanofibers were investigated using hysteresis loop measurements, and compared with the magnetic moments obtained by DFT.

#### 2. Experimental

The ferrite nanofibers with different stoichiometries were fabricated by electrospinning technique followed by a high-temperature annealing. The gel solution for electrospinning was prepared by adjusting the molar ratios (1:1, 1:2, and 1:12) of strontium acetate [(CH<sub>3</sub>COO)<sub>2</sub>Sr] and iron acetylacetonate [Fe(acac)<sub>3</sub>], mixing them with 3 mL ethanol and 3 mL glacial acetic acid under stirring, and adding 0.630 g polyvinylpyrrolidone (PVP, molecular weight: 1,300,000) into the solution. The gel solution was stirred vigorously for 6 h before electrospinning. In the electrospinning process of each ferrite, the gel solution was filled into a plastic syringe with a stainless-steel needle with an inner diameter of 0.6 mm. The needle was connected to a variable voltage range of up to 25 kV. The collector of the nanofibers consisted of a steel plate wrapped with oil paper rotating at 550 rpm. A high voltage of 18 kV was applied between the needle and the collector at a distance of 15 cm. The injection rate was 0.35 mL/h; the temperature was 25 °C; the humidity was  $\sim$ 60%. After the electrospinning, the nanofiber membrane was first dried in air at 50 °C for 2 h to evaporate organic solvents and then annealed at 850 °C in air for 2 h with a heating rate of 5.0 °C/min. The annealing temperature of 850  $^\circ \mathrm{C}$  was chosen to ensure formation of single-phase pristine Sr-Fe-O nanofibers [12,13,23] and sufficient reduction of carbon content in these nanofibers. The obtained nanofiber membranes with Sr:Fe ratios of 1:1, 1:2 and 1:12 were labeled S-F-1, S-F-2, and S-F-12, respectively. Specimens for TEM observations were prepared by dispersing the nanofiber membranes in ethanol through ultrasonic treatment, and dripping several drops onto a holey-carbonfilm-supported Cu grid.

The morphology of the membranes was examined using a fieldemission SEM (FE-SEM, Sigma 500) operating at 10 kV. The crystal structures were analyzed by an X-ray diffractometer (XRD, Bruker D8 Advance) with Cu-K<sub>α</sub> radiation ( $\lambda = 1.5406$  Å). XPS (Thermo Scientific K-Alpha+) spectra were acquired with an Al K<sub>α</sub> X-ray source (h $\nu =$ 1486.6 eV). Bright-field (BF) images, selected-area electron diffraction (SAED) patterns, and high-resolution TEM (HRTEM) images were obtained using a field-emission-gun TEM (JEOL JEM 2100F). The magnetization *versus* magnetic field hysteresis loops were measured by a vibrating sample magnetometer (VSM) equipped on a physical properties measurement system (PPMS, Quantum Design) at room temperature.

DFT calculations were performed to obtain magnetic moments for three ferrites using Vienna *ab initio* Simulation Package (VASP). The Perdew-Burke-Ernzerhof (PBE) functional and projector augmented wave potentials were adopted to describe the exchange-correlation and electron-ion interactions. The cutoff energy for the plane-wave basis was set as 400 eV and the resolution for *k*-point meshes was set to be  $2\pi \times 0.03$  Å<sup>-1</sup>, ensuring that an equilibrium structure can be reached. The crystal structures were considered to be fully optimized until the corresponding energy change and the force acting on each atom were <1.0  $\times 10^{-5}$  eV/atom and 0.01 eV/Å, respectively. Based on the optimized structures, the most stable spin configurations were obtained through self-consistent calculations.

# 3. Results and discussion

SEM examinations were carried out to investigate the morphology of three ferrite nanofibers. Fig. 1 shows low-magnification and enlarged SEM images of the nanofibers with different stoichiometries. From Fig. 1, we can see that the nanofibers of S-F-1, S-F-2 and S-F-12 are all randomly oriented due to the bending and whipping movements of the electrospinning jet. From Fig. 1(a), (c) and (e), the average diameters of S-F-1, S-F-2, and S-F-12 are measured to be 131 nm, 139 nm, and 137 nm, respectively. The average diameters for three different nanofibers are very close, which results from the same conditions adopted in the electrospinning and annealing process. Compared to the rigid nanofibers in Fig. 1(a) and (c), most nanofibers in Fig. 1(e) are bent, indicating that the nanofibers in S-F-12 are more flexible. In addition, branching occurs in some fibers of S-F-1 and S-F-2, but not in S-F-12. From the enlarged SEM images in Fig. 1(b), (d) and (f), we can see that all three nanofibers are composed of nanograins. It is believed that the formation of grain boundaries results from the rapid growth of crystal grains during the crystallization process at high temperatures, which can partially relieve the strain energy [24].



Fig. 1. SEM images of S-F-1 (a, b), S-F-2 (c, d), and S-F-12 (e, f).

Fig. 2 shows the XRD patterns of the nanofiber membranes with different stoichiometries. All peaks obtained from S-F-1, S-F-2, and S-F-12 are sharp and intense, indicating that they all have good crystallinity. In Fig. 2(a), all the peaks are consistent with those of tetragonal SrFeO<sub>2.86</sub> (JCPDS card no.: 39–0954). Therefore, we can identify S-F-1 as a tetragonal phase with lattice parameters a = 10.934 Å, c = 7.705 Å. In Fig. 2(b), a good match can be found between the peak positions of

the S-F-2 and orthorhombic SrFe<sub>2</sub>O<sub>4</sub> phase (JCPDS card no.: 48–0156). Thus, S-F-2 is identified as the orthorhombic SrFe<sub>2</sub>O<sub>4</sub> with lattice parameters *a* = 8.031 Å, *b* = 18.208 Å, *c* = 5.454 Å. Careful examination of Fig. 2(b) shows that several peaks have different intensities with those in the standard XRD pattern. The largest difference can be found for the diffraction peak corresponding to (002) plane, which shows a much higher intensity than that in the standard pattern. This increase in



Fig. 2. XRD patterns obtained from S-F-1 (a), S-F-2 (b), and S-F-12 (c).

intensity results from the preferred orientation along [002] direction for the nanograins in the nanofibers, which is confirmed by the SAED pattern hereafter. From Fig. 1(c), the peak positions and intensities of S-F-12 are highly consistent with those of hexagonal phase SrFe<sub>12</sub>O<sub>19</sub> (JCPDS card no. 33–1340). Thus, S-F-12 can be identified as hexagonal SrFe<sub>12</sub>O<sub>19</sub> with lattice parameters a = 5.887 Å, c = 23.037 Å.

XPS spectra were acquired to analyze the surface composition and valence states of the elements in three ferrite nanofibers. Fig. 3 shows typical XPS spectra of Fe 2p, O 1s, and Sr 3d in the three nanofibers. For all three nanofibers, the Fe 2p peak consists of a doublet corresponding to Fe  $2p_{3/2}$  and  $2p_{1/2}$ , whose peak positions and splittings are summarized in Table 1. From Table 1, it can be seen that the Fe 2p peak positions and splittings vary for different fibers, which is ascribed to the fact that Fe possesses different valence states and chemical environments in the three ferrites. For SrFe<sub>2</sub>O<sub>4</sub> and SrFe<sub>12</sub>O<sub>19</sub>, the valence state of Fe is +3, and part of the Fe<sup>3+</sup> ions on the surfaces can be reduced into Fe<sup>2+</sup> under XPS measurement conditions. Therefore, the doublet peaks are attributed to  $Fe^{2+}$  and  $Fe^{3+} 2p$ . It is known that in XPS spectra,  $Fe^{3+} 2p$ has a higher binding energy and a larger splitting than  $Fe^{2+}$  2p. Thus, it can be deduced that Fe<sup>3+</sup> is more abundant on the SrFe<sub>12</sub>O<sub>19</sub> fiber surface than SrFe<sub>2</sub>O<sub>4</sub> since the Fe 2p of SrFe<sub>12</sub>O<sub>19</sub> has a higher binding energy and a larger splitting. For SrFeO<sub>2.86</sub>, the Fe  $2p_{3/2}$  and  $2p_{1/2}$  peaks shift to binding energies of 710.9 eV and 724.4 eV, respectively, lower than those of SrFe<sub>2</sub>O<sub>4</sub> and SrFe<sub>12</sub>O<sub>19</sub>. This can be attributed to the coexistence of  $Fe^{4+}$  and  $Fe^{3+}$  in  $SrFeO_{2.86}\text{,}$  rather than  $Fe^{2+}$  and  $Fe^{3+}\text{.}$ Peak fitting analysis of Fe spectra was carried out to qualitatively deduce the composition of Fe with different valence states. For SrFe<sub>2</sub>O<sub>4</sub> and  $SrFe_{12}O_{19}$ , the Fe  $2p_{3/2}$  peak can be fitted with two peaks at 710 eV and 712 eV, corresponding to  $Fe^{2+}$  and  $Fe^{3+}$ , respectively [25]. As for the Fe  $2p_{1/2}$  peak, the binding energies for Fe<sup>2+</sup> and Fe<sup>3+</sup> are 723 eV and 726 eV, respectively [25]. The peaks at around 716, 720, 728 and 733 eV are satellite peaks of Fe 2p [25]. For both SrFe<sub>2</sub>O<sub>4</sub> and SrFe<sub>12</sub>O<sub>19</sub>, Fe<sup>3+</sup> 2p peaks have a larger area than  $Fe^{2+} 2p$ , hence there is more  $Fe^{3+}$  on both fiber surfaces. In addition, the peak area ratio of  $Fe^{3+}$  and  $Fe^{2+}$  in

SrFe<sub>12</sub>O<sub>19</sub> is bigger than that in SrFe<sub>2</sub>O<sub>4</sub>, indicating a higher proportion of Fe<sup>3+</sup> exists on the SrFe<sub>12</sub>O<sub>19</sub> fiber surface. For SrFeO<sub>2.86</sub>, the Fe  $2p_{3/2}$  and  $2p_{1/2}$  peak can be fitted with Fe 2p peaks of SrFeO<sub>3-x</sub> and Fe<sup>3+</sup>, for which the Fe  $2p_{3/2}$  peaks are located at 710.4 eV [26–28] and 711.0 eV [26], respectively. The peaks at around 716 eV, 728 eV, and 733 eV are satellite peaks of Fe 2p [25]. The Fe 2p peaks of SrFeO<sub>3-x</sub> have a larger area than Fe<sup>3+</sup> 2p, showing that Fe atoms of SrFeO<sub>2.86</sub> contributes to most of the surface Fe atoms.

Since valence states of O and Sr are the same in the three ferrites, the discrepancies of O and Sr peaks are mainly attributed to different chemical environments. The O 1s peaks around 531 eV can be resolved into peaks for surface Sr-O groups and lattice O<sup>2-</sup> ions. For SrFeO<sub>2.86</sub> and SrFe<sub>2</sub>O<sub>4</sub> nanofibers, these peaks are located at 531.2 eV and 529 eV [29,30], respectively. For SrFe<sub>12</sub>O<sub>19</sub> nanofibers, the peak for Sr-O groups remains at 531.2 eV, while the peak for lattice  $O^{2-}$  ions shifts to 529.7 eV [30], indicating that electron emission is harder for lattice  $O^{2-}$ ions in the SrFe<sub>12</sub>O<sub>19</sub> than those in SrFeO<sub>2.86</sub> and SrFe<sub>2</sub>O<sub>4</sub>. The Sr 3d peaks can be fitted with a Sr  $3d_{5/2}$  and  $3d_{3/2}$  doublet. For SrFeO<sub>2.86</sub>, the Sr  $3d_{5/2}$  and  $3d_{3/2}$  peaks are at 132.9 eV and 134.7 eV, respectively [12], with a characteristic energy gap of 1.8 eV. While for SrFe<sub>2</sub>O<sub>4</sub> and SrFe<sub>12</sub>O<sub>19</sub>, these two peaks shift to 133.1 eV and 134.9 eV, respectively [30]. Compared to SrFe<sub>2</sub>O<sub>4</sub> and SrFe<sub>12</sub>O<sub>19</sub>, there is a shift to lower binding energy for the Sr  $3d_{5/2}$  and  $3d_{3/2}$  of SrFeO<sub>2.86</sub>, which is attributed to chemical environment change induced by the oxygen vacancies in SrFeO2 86.

To further confirm the crystal structures of SrFeO<sub>2.86</sub> nanofibers, extensive BF TEM and SAED examinations were carried out. Fig. 4 presents the BF TEM images and SAED patterns of SrFeO<sub>2.86</sub> nanofibers. Fig. 4(a) shows that all the nanofibers are composed of nanograins with an average dimension of 137  $\pm$  45 nm, calculated from the histogram in Fig. 4(c). Fig. 4(b) shows the polycrystalline electron diffraction pattern obtained from the nanofibers in Fig. 4(a), in which the diffraction rings from inside to outside correspond to (220), (222), (141), (402), and (442) planes of tetragonal  $SrFeO_{2.86}$ , consistent with the XRD result. To further clarify the structure of nanograins, we carried out systematic SAED examinations of single nanograins in different nanofibers. Fig. 4 (d) shows a BF TEM image of a single fiber and Fig. 4(e) presents the SAED pattern obtained from the nanograin enclosed by a circle in Fig. 4 (d). The [001] zone-axis diffraction pattern in Fig. 4(e) can be indexed using lattice parameters of tetragonal SrFeO<sub>2.86</sub>. Fig. 4(f) demonstrates a BF TEM image of another single fiber and Fig. 4(g) presents the [010] zone-axis SAED pattern for the nanograin enclosed by a circle in Fig. 4 (f). From our extensive SAED examinations of the nanograins in the fibers, it is concluded that they possess a tetragonal structure, in good agreement with our XRD analysis results.

From our extensive HRTEM observations of SrFeO2.86 nanofibers, it is found that quite few nanofibers have perfect lattices, while most fibers have defects such as dislocations and stacking faults (SFs). Fig. 5(a) shows a [523] zone-axis HRTEM image of an individual SrFeO<sub>2.86</sub> nanofiber without lattice defects. To further demonstrate the perfect lattice, an enlarged image of the rectangular region in Fig. 5(a) is presented in Fig. 5(b). In Fig. 5(b), the interplanar spacings of two planes were measured to be 2.73 Å and 2.51 Å with an angle of 70.0°, corresponding to the (222) and (141) planes of tetragonal SrFeO<sub>2.86</sub>, respectively. In contrast to the above observation, Fig. 5(c) shows a  $[01\overline{1}]$  zone-axis HRTEM image of an individual SrFeO<sub>2.86</sub> nanofiber with crystal defects. To further highlight the defects, Fig. 5(d) and (e) show enlarged images of the rectangular region in Fig. 5(c). In Fig. 5(d), the interplanar spacings of two planes were measured both as 2.73 Å, with an angle of 59.9°, corresponding to the (222) and ( $\overline{2}22$ ) planes of tetragonal SrFeO<sub>2.86</sub>. Careful examination of Fig. 5(d) reveals three SFs (SF1, SF2 and SF3) and two edge dislocations (D1 and D2) exist in this region. Such defects have not been observed in SrFeO<sub>2.86</sub> before.

In Fig. 5(d), three SFs, SF1, SF2 and SF3, are found between regions I and II, II and III, III and IV, respectively. These SFs can be clearly



Fig. 3. XPS spectra of Fe, O, and Sr in SrFeO<sub>2.86</sub> (a, b, c), SrFe<sub>2</sub>O<sub>4</sub> (d, e, f), and SrFe<sub>12</sub>O<sub>19</sub> (g, h, i) nanofibers.

Table 1													
Binding	energies	and	splitting	(in	eV)	of	core-level	peaks	for	Fe	in	the	three
ferrite n	anofibers												

Sample	Fe 2p <sub>3/2</sub>	Fe 2p <sub>1/2</sub>	Fe 2p <sub>1/2</sub> - 2p <sub>3/2</sub>
SrFeO <sub>2.86</sub>	710.9	724.4	13.5
SrFe <sub>2</sub> O <sub>4</sub>	711.7	725.2	13.5
SrFe <sub>12</sub> O <sub>19</sub>	712.2	726.0	13.8

visualized from the slight deviation of the (222) planes labeled by three parallel white lines in regions I-IV. The formation of SFs is associated with oxygen vacancies in SrFeO<sub>2.86</sub>. Due to the non-stoichiometric nature of SrFeO<sub>2.86</sub>, lots of oxygen vacancies exist in the nanofibers. During the growth of the nanofibers, when the oxygen vacancies gather together on the (222) planes, SFs with an intrinsic rather than extrinsic characteristic will form. Usually in face-centered-cubic structures, an intrinsic SF introduces a displacement of  $R = -\frac{1}{3} \langle 111 \rangle$  between (111) lattice planes [31]. However, it is rather difficult to determine the displacement of the SFs in SrFeO<sub>2.86</sub> due to its complex structure.

To determine the Burgers vectors of the two edge dislocations, Burgers circuits are drawn in a close-up view of the region containing two edge dislocations, as shown in Fig. 5(e). For D1, the extra half atomic plane is inserted from above, whereas for D2, the half atomic plane is inserted from below. Therefore, we can regard D1 as a positive perfect dislocation, and D2 as a negative perfect dislocation. The Burgers vectors of D1 and D2 are determined to be  $b = \frac{1}{2} \langle 211 \rangle$ , but with opposite signs. If D1 and D2 are on the same gliding plane, they will attract and annihilate with each other. However, the two edge dislocations in Fig. 5 (e) did not annihilate with each other, meaning that they are in fact on different gliding planes. For two edge dislocations with opposite signs, the theoretical equilibrium configuration is that the angle between the dislocation lines is 45° [32,33]. Therefore, the dislocations D1 and D2 should form an equilibrium configuration and the angle between their dislocation lines should be  $45^\circ.$  The formation of these dislocations can release the strain due to lattice distortions induced by oxygen vacancies in the nanofibers.

To further prove the orthorhombic structure for  $SrFe_2O_4$  nanofibers, thorough TEM investigations were carried out. Fig. 6 shows BF TEM images, SAED patterns, and HRTEM images of  $SrFe_2O_4$  nanofibers. Fig. 6



Fig. 4. (a) BF TEM images of SrFeO<sub>2.86</sub> nanofibers, (b) corresponding SAED pattern, and (c) statistical analysis for the dimension of nanograins in the nanofibers. (d) [001]-oriented nanograin in a single nanofiber and (e) SAED pattern. (f) [100]-oriented nanograin in a single nanofiber and (g) SAED pattern.

(a) shows a low-magnification BF TEM image of nanofibers, whose diffraction pattern is presented in Fig. 6(b). The diffraction rings from inside to outside correspond to (002), (122), (202), (322), and (233) planes of orthorhombic  $SrFe_2O_4$ , which agrees well with XRD results.

The intensified (002) XRD peak is reflected in the polycrystalline electron diffraction pattern. From Fig. 6(c), we can see that all the nanofibers are composed of nanograins. Fig. 6(d) shows the distribution is more biased towards smaller particles, with an average dimension of



Fig. 5. (a, c) HRTEM images of individual SrFeO<sub>2.86</sub> nanofibers. (b) Enlarged image of rectangular region in (a). (d, e) Enlarged images showing lattice defects in region of interest in (c).

106 ± 43 nm. Fig. 6(f) shows a SAED pattern obtained from an individual nanograin enclosed by a circle in Fig. 6(e). The zone-axis of this SAED pattern is determined to be  $[15\overline{8}]$  using the lattice parameters of orthorhombic SrFe<sub>2</sub>O<sub>4</sub>. In addition, HRTEM examinations were performed to confirm that the nanofibers have an orthorhombic structure. Fig. 6(g) shows a  $[10\overline{2}]$  zone-axis HRTEM image of a thin edge region in an individual nanofiber. The inset in Fig. 6(g) demonstrates the corresponding electron diffraction pattern, which can be indexed using lattice parameters of orthorhombic SrFe<sub>2</sub>O<sub>4</sub>. From the enlarged HRTEM image in Fig. 6(h), the interplanar spacings of two lattice planes are measured to be 2.87 Å and 2.64 Å with an angle of 63.4°, corresponding to (231)

and  $(2\overline{4}1)$  planes of orthorhombic SrFe<sub>2</sub>O<sub>4</sub>, respectively.

Fig. 7 presents the BF TEM images and SAED patterns of  $SrFe_{12}O_{19}$  nanofibers. Fig. 7(a) shows that the  $SrFe_{12}O_{19}$  nanofibers are also made up of nanograins. The polycrystalline diffraction pattern in inset of Fig. 7 (a) can be indexed from inside to outside with ( $10\overline{1}2$ ), ( $10\overline{1}6$ ), ( $11\overline{2}4$ ), ( $20\overline{2}3$ ), and ( $20\overline{2}5$ ) planes of hexagonal  $SrFe_{12}O_{19}$ , consistent with our XRD result. From Fig. 7(b), an average dimension of  $131 \pm 43$  nm is determined for the nanograins in the nanofibers. Fig. 7(c) shows an enlarged image of an individual nanofiber labeled by a white rectangle in Fig. 7(a). To confirm the hexagonal structure, [ $1\overline{2}10$ ] zone-axis SAED pattern (Fig. 7 (d)) was obtained from a single nanograin in Fig. 7(c).



**Fig. 6.** (a) BF TEM images of SrFe<sub>2</sub>O<sub>4</sub> nanofibers, and (b) corresponding SAED pattern. (c) BF TEM images of SrFe<sub>2</sub>O<sub>4</sub> nanofibers, and (d) Statistical analysis of the dimension for the nanograins in the nanofibers. (e) [158]-oriented nanograin in a single nanofiber and (f) corresponding SAED pattern. (g) HRTEM image of a single nanofiber, inset showing its SAED pattern. (h) Enlarged HRTEM image of the region enclosed by a rectangle in (g).



**Fig. 7.** (a) BF TEM images of SrFe<sub>12</sub>O<sub>19</sub> nanofibers, inset showing polycrystalline diffraction pattern. (b) Statistical analysis of the nanograins in the fibers. (c) BF TEM image of a single nanofiber, and (d) SAED pattern obtained from a nanograin enclosed by a circle in (c).

The SAED pattern can be indexed using the lattice parameters of hexagonal  $\rm SrFe_{12}O_{19}$ , proving that the produced nanofibers possess a hexagonal structure.

From our HRTEM examinations of  $SrFe_{12}O_{19}$  nanofibers, some nanofibers are found without lattice defects, while the others contain lattice defects such as edge dislocations. Fig. 8(a) shows a [1210] zoneaxis HRTEM image of an individual  $SrFe_{12}O_{19}$  nanofiber without lattice defects. Fig. 8(b) shows an enlarged image of the rectangular region in Fig. 8(a). It can be clearly seen from Fig. 8(b) that a modulated structure exists in this nanofiber, showing periodical dark and bright stripes along the [0001] direction, which might be caused by the cation ordering of Sr and Fe [34]. The interplanar spacings of two planes were measured to be 2.49 Å and 2.30 Å with an angle of 102.5°, corresponding to the (2022) and (00010) planes of  $SrFe_{12}O_{19}$ , respectively. To our knowledge, such modulated structure has not been reported in  $SrFe_{12}O_{19}$  before.

Contrary to the perfect fringes without defects, edge dislocations are observed in Fig. 8(c) and (d), viewed from  $[1\overline{1}00]$  direction. The interplanar spacing of two planes was measured both as 2.62 Å with an angle of 54.1°, corresponding to  $(11\overline{2}4)$  and  $(11\overline{2}4)$  planes of SrFe<sub>12</sub>O<sub>19</sub>. In Fig. 8(c), an area with brighter contrast is enclosed by a rectangle. To clarify this contrast discrepancy, Fig. 8(d) shows an enlarged image of this rectangular region. In Fig. 8(d), two edge dislocations, labeled as D1 and D2, were found above and below the bright spot, respectively. An analysis of the plotted Burgers circuits shows that Burgers vectors of D1 and D2 are both  $b = \frac{1}{3} < 22\overline{4}3$ , with the same signs. For dislocations with the same sign, the theoretical equilibrium configuration is that one is located just above the other. [32] Here, D1 does not locate just above D2, meaning that they cannot form an equilibrium configuration. In addition, the interaction between them should be a repulsive force.

Thus, the brighter contrast originates from the tensile strain field around the two edge dislocations.

Fig. 9 shows the hysteresis loops of the randomly oriented SrFeO<sub>2.86</sub>, SrFe<sub>2</sub>O<sub>4</sub> and SrFe<sub>12</sub>O<sub>19</sub> nanofibers. The loops of the three ferrite nanofibers exhibit typical hard magnetic characteristics. Table 2 summarizes the magnetic parameters of randomly oriented nanofibers with different stoichiometries. From Table 2, the coercive forces (H<sub>c</sub>) of the three ferrite nanofibers are close, with values around 6000 Oe. However, the saturation and remanent magnetisms (M<sub>s</sub> and M<sub>r</sub>) are considerably different. The M<sub>s</sub> values for SrFeO<sub>2.86</sub>, SrFe<sub>2</sub>O<sub>4</sub> and SrFe<sub>12</sub>O<sub>19</sub> nanofibers are 3.18, 19.00, and 58.43 emu/g, and the M<sub>r</sub> values are 1.19, 9.22, and 29.75 emu/g, respectively. Among the three ferrite nanofibers, SrFe<sub>12</sub>O<sub>19</sub> fibers have the largest M<sub>r</sub>/M<sub>s</sub>, suggesting they possess the strongest capability to retain magnetization. Taking into account H<sub>c</sub>, M<sub>s</sub>, M<sub>r</sub> and M<sub>r</sub>/M<sub>s</sub>, SrFe<sub>12</sub>O<sub>19</sub> nanofibers appear to be the best candidate for permanent magnets.

 $H_c$  and  $M_s$  are correlated with the anisotropy constant K, which is described by Neel's two sub-lattices model of ferrimagnetism [35]:

$$H_c = \frac{0.96 \times K}{M_s} \tag{1}$$

Using the data in Table 2 and Eq. (1), the K values of  $SrFeO_{2.86}$ ,  $SrFe_2O_4$  and  $SrFe_{12}O_{19}$  nanofibers are calculated to be 19,785, 119,581, and 349,667 Oe•emu/g, respectively. The drastic discrepancy in the anisotropy constant is mainly attributed to the different crystal structures of the three ferrites.

As seen from TEM images, the nanofibers are composed of nanograins. To demonstrate the advantages of nanofiber morphology over nanoparticle, we compare the magnetic parameters of the three



Fig. 8. (a, c) HRTEM images of individual SrFe<sub>12</sub>O<sub>19</sub> nanofibers. (b) Enlarged image of rectangular region in (a). (d) Enlarged image showing lattice defects in region of interest in (c).



Fig. 9. Hysteresis loops of randomly oriented SrFeO<sub>2.86</sub> (blue curve), SrFe<sub>2</sub>O<sub>4</sub> (black curve) and SrFe<sub>12</sub>O<sub>19</sub> (red curve) nanofibers. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

 Table 2

 Magnetic parameters of randomly oriented nanofibers with different stoichiometry.

	5				
Ferrite	H <sub>c</sub> (Oe)	M <sub>s</sub> (emu/g)	M <sub>r</sub> (emu/g)	$M_r  /  M_s$	K (Oe∙emu∕g)
SrFeO <sub>2.86</sub>	5973	3.18	1.19	0.37	19,785
SrFe <sub>2</sub> O <sub>4</sub>	6042	19.00	9.22	0.49	119,581
SrFe <sub>12</sub> O <sub>19</sub>	5745	58.43	29.75	0.51	349,667

nanofibers in our work and corresponding nanoparticles in previous studies. For SrFeO<sub>2.86</sub>, no magnetic parameters for nanoparticles can be found in the literature for comparison. However, SrFeO<sub>3-x</sub> nanoparticles are reported to have  $H_c,\,M_s$  and  $M_r$  values of 410.47 Oe, 1.166 emu/g and 0.225 emu/g, respectively [6], which are all much lower than those of SrFeO<sub>2.86</sub> nanofibers in the present work. In addition, the cubic SrFeO<sub>3-x</sub> nanoparticles do not possess hard magnetic characteristic [6], while the tetragonal SrFeO<sub>2.86</sub> nanofibers in present work do. For SrFe<sub>2</sub>O<sub>4</sub>, several reports can be found on magnetic parameters of nanoparticles. Ateia et al. obtained H<sub>c</sub>, M<sub>s</sub> and M<sub>r</sub> values of 5396 Oe, 15.1 emu/g and 7.9 emu/g for nanoparticles produced by citrate autocombustion technique [11], which are all less than the corresponding values in the present work. Mousavi Ghahfarokhi et al. achieved H<sub>c</sub>, M<sub>s</sub> and Mr values of 3069 Oe, 22.99 emu/g and 9.91 emu/g for SrFe2O4 nanoparticles prepared by a sol-gel method [9], whose H<sub>c</sub> is much lower than our SrFe<sub>2</sub>O<sub>4</sub> nanofibers. For SrFe<sub>12</sub>O<sub>19</sub>, two previous studies measured H<sub>c</sub> and M<sub>s</sub> for nanoparticles: 4783 Oe and 56.97 emu/g reported by García-Cerda et al. [14]; 1643 Oe and 24.7 emu/g reported by

Jean et al. [15]. Comparing with our  $SrFe_{12}O_{19}$  nanofibers, it can be clearly seen that the  $H_c$  and  $M_s$  values are larger in our present study.

DFT calculations were carried out to explore the origin of magnetism in these three ferrites. Fig. 10 shows unit cell configurations of the three ferrites. In Fig. 10, sets of equivalent positions are denoted using Wyckoff notation. Fig. 10(a) shows the unit cell of hexagonal  $SrFe_{12}O_{19}$ with space group  $P6_3/mmc$ . The Fe atoms in SrFe<sub>12</sub>O<sub>19</sub> occupy five Wyckoff sites: three octahedral sites (12 k, 4f<sub>1</sub>, 2a), a tetrahedral site  $(4f_2)$ , and a trigonal bipyramidal site (2b). Fig. 10(b) demonstrates the unit cell of orthorhombic SrFe<sub>2</sub>O<sub>4</sub> with space group *Pbc2*<sub>1</sub>. The Fe atoms occupy four distinct tetrahedral sites:  $4a_1$ ,  $4a_2$ ,  $4a_3$  and  $4a_4$ . To our knowledge, no unit-cell model of orthorhombic  $SrFe_2O_4$  (a = 8.031 Å, b= 18.208 Å, c = 5.454 Å) can be found in previous studies. Considering the huge calculation load due to the large unit cell of SrFeO<sub>2.86</sub>, we adopt the unit cell of SrFeO<sub>2.875</sub> instead, which has similar amount of oxygen vacancies and a tetragonal structure. Fig. 10(c) presents the unit cell of tetragonal SrFeO<sub>2.875</sub> with space group I4/mmm. The Fe atoms are located at octahedral (8f), tetrahedral (4e), and octahedral (4d) lattice

#### Table 3

Magnetic moments of all atoms in the unit cell and total magnetization of  $\ensuremath{\text{SrFe}_{12}}\ensuremath{\text{O}_{19}}\xspace.$ 

Site	Atom	Μ (μ <sub>B</sub> )
12k	12Fe	14.668
$4f_1$	4Fe	-2.442
$4f_2$	4Fe	13.042
2a	2Fe	2.221
2b	2Fe	0.886
2d	2Sr	0.008
12k	120	1.218
4f	40	0.256
12k	120	0.802
6h	60	0.074
4e	40	0.140
	30.873	
	81.20	

4d

Sr



**Fig. 10.** Unit cells of SrFe<sub>12</sub>O<sub>19</sub> (a), SrFe<sub>2</sub>O<sub>4</sub> (b) and SrFeO<sub>2.875</sub> (c). The small red balls denote O atoms, and the green balls represent Sr atoms. The Fe atoms are at non-equivalent lattice sites. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

#### Table 4

Magnetic moments of all atoms in the unit cell and the total magnetization of  $\mathrm{SrFe_2O_4}$ .

Site	Atom	Μ (μ <sub>B</sub> )
4a <sub>1</sub>	4Fe	8.769
$4a_2$	4Fe	-8.362
$4a_3$	4Fe	3.941
$4a_4$	4Fe	8.209
4a	4Sr	0.016
4a	4Sr	0.027
4a	40	0.500
4a	40	-0.068
4a	40	0.507
4a	40	0.175
4a	40	0.057
4a	40	0.164
4a	40	0.240
4a	40	0.045
	Σ Μ (μ <sub>B</sub> )	14.220
$\Sigma$ M (emu/g) per formula unit		37.70

## Table 5

Magnetic moment of all atoms in the unit cell and the total magnetization of  $\rm SrFeO_{2.875.}$ 

Site	Atom	Μ (μ <sub>B</sub> )
8f	8Fe	22.298
4e	4Fe	-9.592
4d	4Fe	-10.736
8i	8Sr	0.032
8j	8Sr	-0.02
16k	160	0.076
16m	160	-0.08
8h	80	0.944
4c	40	-0.432
2a	20	-0.354
	2.136	
	$\Sigma$ M (emu/g) per formula unit	3.92

sites.

Tables 3, 4 and 5 summarize the calculated magnetic moments of all atoms in the unit cells in Fig. 10, and the total magnetization obtained from the self-consistent calculations. For the magnetic moment calculations, all three ferrites were assumed to be ferrimagnetic. It can be seen from the tables that the magnetism of the three ferrites mainly comes from the Fe atoms at non-equivalent lattice sites. For SrFe<sub>12</sub>O<sub>19</sub>, the magnetic moments of Fe atoms at 4f<sub>1</sub> sites are anti-parallel to those of Fe at the other sites after structure optimization. For SrFe<sub>2</sub>O<sub>4</sub>, the Fe atoms at 4a<sub>2</sub> sites have opposite magnetic moments to those at the other three sites. For SrFeO<sub>2.875</sub>, different to previous studies, we chose a ferrimagnetic state instead of ferromagnetic state to replace the helical magnetic ordering. The magnetic moments of Fe atoms at 8f sites were set as positive, while the Fe atoms at 4e and 4d sites had negative magnetic moments.

In the above tables, the theoretical total magnetic moment per formula unit corresponds to  $M_r$  values obtained from the hysteresis loops. To compare these values,  $\Sigma$  M per formula unit was converted into emu/g using the following equation.

$$\Sigma M_{f.u.}(\mu_B) = \frac{M_w \times \Sigma M_{f.u.}(emu/g)}{5585}$$
(2)

Where  $M_w$  is the molecular weight of each ferrite, and  $\Sigma M_{f.u.}$  is the total magnetization per formula unit. The  $\Sigma M_{f.u.}$  (emu/g) for SrFe<sub>12</sub>O<sub>19</sub>, SrFe<sub>2</sub>O<sub>4</sub> and SrFeO<sub>2.875</sub> are calculated to be 81.20 emu/g, 37.70 emu/g and 3.92 emu/g, respectively, as summarized in Tables 3, 4 and 5. For each ferrite, the theoretical  $\Sigma M_{f.u.}$  value and experimental  $M_r$  value for nanofibers are of the same order of magnitude, but all  $\Sigma M_{f.u.}$  values are higher. This difference might be caused by some Fe ions with different valence states on the nanofiber surfaces, microstructural defects, and the

spin-glass structure at grain boundaries in the nanofibers [36,37].

## 4. Conclusions

In summary, three ferrite nanofibers with Sr:Fe ratios of 1:1, 1:2, and 1:12 have been fabricated using electrospinning followed by annealing at 850 °C in air. The ferrite nanofibers with Sr:Fe ratios of 1:1, 1:2, and 1:12 are identified as tetragonal SrFeO<sub>2.86</sub>, orthorhombic SrFe<sub>2</sub>O<sub>4</sub> and hexagonal SrFe<sub>12</sub>O<sub>19</sub>, respectively. The nanofibers have average diameters of 130-140 nm and are made of nanograins with average dimensions of 105-140 nm. HRTEM examinations reveal that stacking faults and edge dislocations exist in SrFeO<sub>2.86</sub> nanofibers while edge dislocations exist in SrFe12O19 nanofibers. Hysteresis loop measurements show that the three nanofibers have a similar  $H_c$  of ~6000 Oe, but different Ms and Mr. SrFe12O19 nanofibers have Ms of 58.43 emu/g and Mr of 29.75 emu/g; SrFe<sub>2</sub>O<sub>4</sub> nanofibers possess Ms of 19.00 emu/g and  $M_r$  of 9.22 emu/g; SrFeO<sub>2.86</sub> nanofibers show the lowest  $M_s$  and  $M_r$ values of 3.18 emu/g and 1.19 emu/g. Among the three ferrite nanofibers, SrFe12O19 nanofibers are the best candidate for permanent magnet applications. The magnetic moments obtained from DFT calculations are of the same order of magnitude but all higher than M<sub>r</sub> values acquired experimentally. This work sheds light on the crystal structure of Sr-Fe-O nanofibers, and provides a good alternative to replace rare earth alloys as permanent magnets. Future work will be carried out on formation mechanisms of these nanofibers, and further improvement on their magnetic properties.

## CRediT authorship contribution statement

**Jiaju Wang:** Data curation, Formal analysis, Investigation, Writing – original draft. **Xuyan Xue:** Methodology, Resources, Software, Supervision. **Yiqian Wang:** Funding acquisition, Conceptualization, Project administration, Writing – review & editing.

#### **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

#### Data availability

Data will be made available on request.

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