HIGH-MAGNETOSTRICTION LAVES-PHASE ALLOY OF THE SAMARIUM-IRON SYSTEM: THE STRUCTURE AND PHASE TRANSFORMATIONS

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In this study, the $SmFe_2$ alloy in a high-purity single-phase state has been prepared by the induction melting technique. The surface features of the alloy at room temperature were investigated using the atomic-force and magnetic-force microscopy. The forming of heterogeneous granular structure was revealed, the main structural elements were determined. The presence of the complex domain structure was shown, its description was presented and the domain sizes were found. The results of X-ray structural studies over the temperature range from 100 to 300 K were presented. The experimental data on the temperature dependences of magnetostriction in magnetic fields up to 1.2 T were obtained and analyzed in the region of the spin-reorientational phase transition. The existence of the "angular" phase was indirectly confirmed, its temperature boundaries were refined.

Keywords: rare-earth Laves phase, phase transition, magnetostriction, atomic force microscopy, structure

СТРУКТУРА И ФАЗОВЫЕ ПРЕВРАЩЕНИЯ ВЫСОКОМАГНИТОСТРИКЦИОННОГО СПЛАВА СИСТЕМЫ САМАРИЙ-ЖЕЛЕЗО СО СТРУКТУРОЙ ФАЗ ЛАВЕСА

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В работе методом индукционной плавки получен сплав $SmFe_2$ в высокочистом однофазном состоянии. Методами атомно-силовой и магнитно-силовой микроскопии исследованы особенности поверхности сплава при комнатной температуре. Выявлено наличие неоднородной зернистой структуры, определены основные структурные элементы. Показано наличие сложной доменной структуры, проведено ее описание, определены размеры доменов. Представлены результаты рентгеноструктурных исследований

в интервале температур 100 − 300 K. Получены и проанализированы экспериментальные данные по температурным зависимостям магнитострикции в магнитных полях до 1,2 Тл в области спин-переориентационного фазового перехода. Косвенно подтверждено существование «угловой» фазы, уточнены ее температурные границы.

Ключевые слова: редкоземельная фаза Лавеса, фазовый переход, магнитострикция, атомно-силовая микроскопия, магнитно-силовая микроскопия

Introduction

Recent years have seen a renewed interest in study of RFe_2 Laves-phase alloys (where R is a rare-earth metal) [1−3]. Compounds of rare-earth and $3d$ transition metals with the Laves phase structure are an important family of magnetic materials exhibiting high values of anisotropic magnetostriction (of the order of 10^{-3}) in relatively low magnetic fields (less than 1 T) below the Curie temperature. It is known [4−6] that highly magnetostrictive compounds can convert electrical energy into mechanical energy and are used in such devices as drives and sensors operating in various environments and in a wide temperature range. Some Laves phases demonstrate both high values of magnetostriction and large magnetocaloric effects around the Curie temperature, which can also find practical application [7, 8]. While the physical mechanism responsible for high values of magnetostriction in these compounds is well known [9, 10], comprehensive studies on the structure, magnetic properties and anomalies in the phase transition region were only carried out by different techniques for individual samples.

RFe_2 intermetallic compounds crystallize into a cubic structure of the $MgCu_2$ type ($Fd3m$ space group). According to theory, giant spontaneous magnetostriction is most likely to occur along the easy <111> axis of magnetization as a result of magnetic ordering. Special symmetry of cubic $C15$ Laves phase leads to large rhombohedral distortion in the <111> direction. The best-known Laves phase compounds with giant saturation magnetostriction at room temperature are $TbFe_2$ and $SmFe_2$ ($+1.7 \cdot 10^{-3}$ and $-1.5 \cdot 10^{-3}$, respectively) [11−15]. These alloys have close magnitostriction values (differing in sign) at room temperature. Both of these alloys undergo rhombohedral distortions upon transition to a magnetically ordered state; however, while the cubic lattice in $TbFe_2$ compound stretches along the <111> direction, it is slightly compressed in $SmFe_2$. These compounds also differ by type of magnetic ordering. It is known that magnetic moments of the R and Fe sublattices are parallel, with the total magnetic moments of the sublattices co-directional for light REM and antiparallel for heavy REM. Because of this, $TbFe_2$ is a ferrimagnet, and $SmFe_2$ is a ferromagnet.

Unlike $TbFe_2$, the crystal structure of $SmFe_2$, as well as the direction of its magnetic moment change with decreasing temperature. Mцssbauer and XRD studies [15−18] found that the spontaneous magnetic moment of the $SmFe_2$ compound at room temperature is oriented along the <111> crystal direction. It was previously believed that a spin-reorientational phase transition (SRPT) is observed with a temperature decrease in the region $T = 180-200$ K, with magnetic moments oriented along the <110> direction. However, detailed analysis of the diffraction spectra obtained at low temperatures revealed [17, 18] that the structure of the alloy remains rhombohedral with a decrease in temperature to about 200 K, while the distortions increase in absolute value. An "angular" magnetic phase appears in the alloy with a further decrease in temperature (in the range of 140−240 or 106−180 K but different sources cite different ranges): the vector of spontaneous magnetic moment is in the (110) plane and does not coincide in direction with either <111> or <110>. The character that transformation of the X-ray diffraction spectrum bears at low temperatures indicates orthorhombic distortions of the crystal lattice, and the magnetic moment is directed along the <110> axis. Anomalies in the SRPT region are also manifested in the temperature dependences of magnetization [15].

The goal of this study has been to analyze the changes in the crystal structure and

magnetostrictive properties of the $SmFe_2$ alloy in the temperature range from 100 to 320 K, and also to consider the peculiarities of its surface at room temperature using atomic-force and magnetic-force microscopy.

In view of this goal, we needed to obtain a high-purity single-phase alloy of $SmFe_2$.

Experimental procedure

Sample preparation. The $SmFe_2$ alloy was synthesized by high-frequency induction melting (Donets-1 furnace) in an alundum crucible in ultra-pure argon (moisture content less than 0.02 $g \cdot m^{-3}$, nitrogen content 0.0005%, oxygen content 0.001%) at 1.1−1.2 atm. High-purity metals were taken as the initial components. We experimentally selected mixtures with higher REM contents (by 10%), which allowed to obtain single-phase compounds upon subsequent homogenizing annealing. The cooling rate of the alloys was rather slow (about 1−2 K/s), which contributed to achieving a near-steady state. The prepared samples were placed in quartz tubes pumped to high vacuum. Melted alloys were subjected to heat treatment in a specially selected mode using a Carbolite TZF 15/610 3-zone high-temperature tube furnace: the samples in tubes were heated to a temperature of 720°C at a rate of 7 K/min. Next, the samples were kept at this temperature for 72 hours and then quenched in water.

Recording the X-ray diffraction spectra. The spectra were recorded for powder samples using $CuK_{\square}$ radiation ($\square$ = 0.1540598 nm) at room temperature with a DRON-7 X-ray diffractometer modified with a rapid-response system based on a Mythen 1K linear position-sensitive strip detector (Dectris Ltd, Switzerland). The unit cell parameters were calculated from reflections in the angle range $2\square$ = 15−105°. The phase composition of the sample was studied by Rietveld refinement in the Powder Cell 2.4 program. Temperature measurements of the XRD patterns were carried out in the temperature range of 110−250 K with a Supernova X-ray diffractometer (Agilent) using filtered $MoK_{\square}$ radiation. The sample temperature was controlled by its contact with nitrogen gas stream of a given temperature, maintained by a Cobra PLus Cryosystem (Oxford Cryosystems).

Microscopy. The microstructure of the samples was studied with a Neophot-30 metallographic microscope. The samples were observed and photographed under a xenon lamp in either bright field or polarized light modes. Microstructure patterns were fed through the optical channel of the microscope to a Levenhuk M800 Plus high-resolution digital camera; a Smena-A scanning probe microscope (Solver platform, NT-MDT, Russia) was used.

Analysis of surface morphology. The morphology was studied by atomic force microscopy (AFM) of Laves phases in the samples in both tapping and contact modes at room temperature using standard HA_NC Etalon silicon cantilevers. Thin sections of the samples were etched in a 5% solution of nitric acid in alcohol to reveal the nanostructural features of the surface. Magnetic force microscopy (MFM) studies were carried out with fully demagnetized thin sections of the samples using special magnetic cantilevers MFM01 with a cobalt chromium coating, at a resonant frequency of about 70 kHz and a force constant of 1−5 N/m. MFM in non-contact vibration mode yields greater sensitivity, allowing to collect high-quality MFM images of the sample surface via a two-pass quasistatic technique (the maximum sensitivity of the method is achieved when the cantilever excitation frequency coincides with the resonant frequency of the system comprising the probe and the sample).

Deformation measurements. Deformation of the polycrystalline sample made of $SmFe_2$ alloy was studied by the strain-gauge method in the temperature range from 100 to 320 K and in magnetic fields up to 1.2 T, with measurements carried out both along the direction of the magnetic field (longitudinal magnetostriction) and perpendicular to it (transverse magnetostriction).

Experimental results and discussion

$SmFe_2$ samples were synthesized by high-frequency induction melting. The problem with synthesizing RFe_2 compounds is that the eutectic points of RFe_2 and RFe_3 are close and, as a result, it is the 1:3 phase that crystallizes in the alloy if there is insufficient REM content in the mixture or if the perictetic reaction does not go to completion, since it takes fewer REM atoms for this phase to form. Analysis of the XRD data obtained at room temperature (Fig. 1) established that the alloy is single-phase and the atomic and crystal structure is isotypic to the structure of the cubic Laves phase $C15$ ($MgCu_2$). The lattice parameter found was 7.4239 E.

The temperature dependences of the lattice parameters (Fig. *2, a*) were obtained by fitting the XRD spectra to the cubic lattice model. Evidently, the slope of the temperature

dependence of the lattice parameter changes in the temperature range of 180−190 K; this indicates a change in the crystal lattice associated with the spin-reorientational phase transition. The results obtained are in good agreement with the data given in literature [15−18], describing a structural transition from the high-temperature rhombohedral phase to the angular phase, which is then transformed into the low-temperature rhombic phase, in this temperature range. Temperature dependences of the lattice parameters *a b c*, obtained by fitting the XRD spectra in accordance with the models of rhombohedral (above 190 K) and rhombic (orthorhombic) (below 180 K) crystal lattices are shown in Fig. 2, *b*. For easy comparison in a single graph, the parameters were transformed (factors √2, √3) and reduced to a pseudocubic cell.

Analysis of the microstructure by the optical method revealed the second phase in the samples, which was not detected by the X-ray method. Quantitative analysis of the

micrographs was carried out with a computer program calculating the relative content of the phases by pixel shades, confirming that the volume content of the secondary phases did not exceed 1−2%, suggesting that the synthesized compounds are almost single-phase.

Atomic-force and magnetic-force microscopy are getting increasingly popular in materials science, providing additional data on homogeneity of phase composition, graininess of synthesized phases, size and morphology of individual grains, domain structure [19−21]. The studies were carried out for micron (scan size of 90 × 90 μm, see Fig. 3, *d*), nanometer (scan size of 4 × 4 μm, see Fig. 3, *a, b*) and smaller scales. It was established by analysis of AFM images of the surface topology that the cells) ranging from 500 to 700 nm in one direction and up to 1 μm in the perpendicular direction. The cells were filled with small grains (these are other structural elements) with an average size of 80 nm, which can be clearly seen in Fig. 3, *a*.
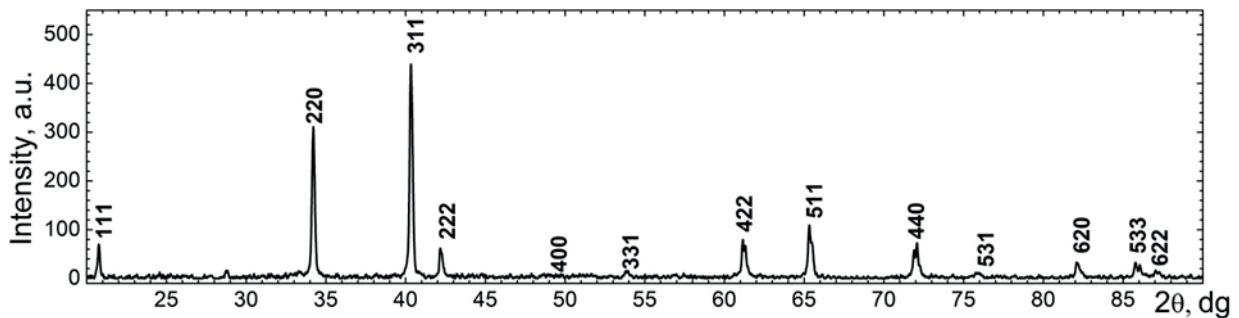

Fig. 1. Experimental XRD pattern of $SmFe_2$ alloy;
numbers above the peaks correspond to the indices hkl; the data are for room temperature
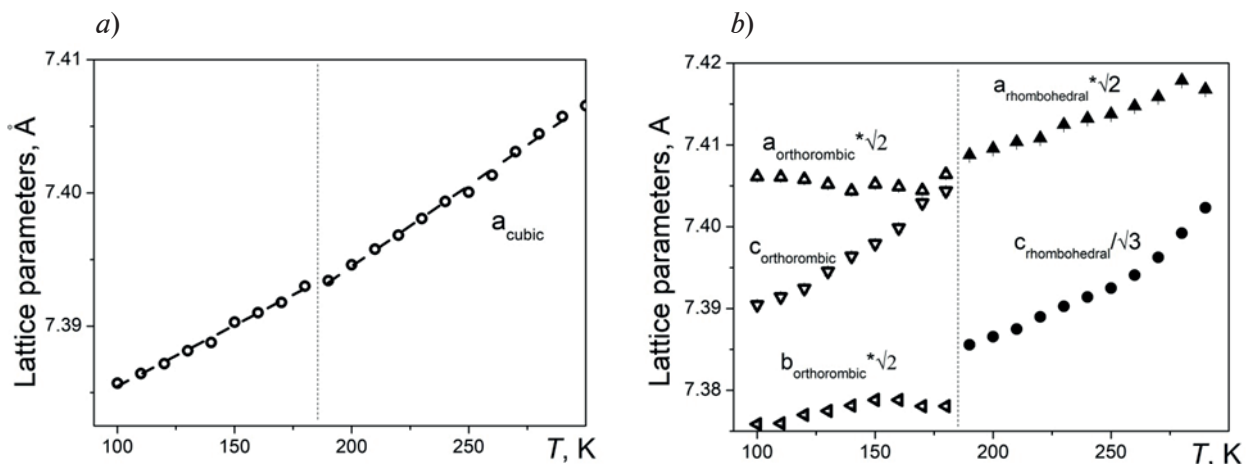

a)

b)

Fig. 2. Temperature dependences of $SmFe_2$ unit cell parameters:
obtained by fitting XRD spectra within models for cubic (*a*), rhombic (< 180 K)
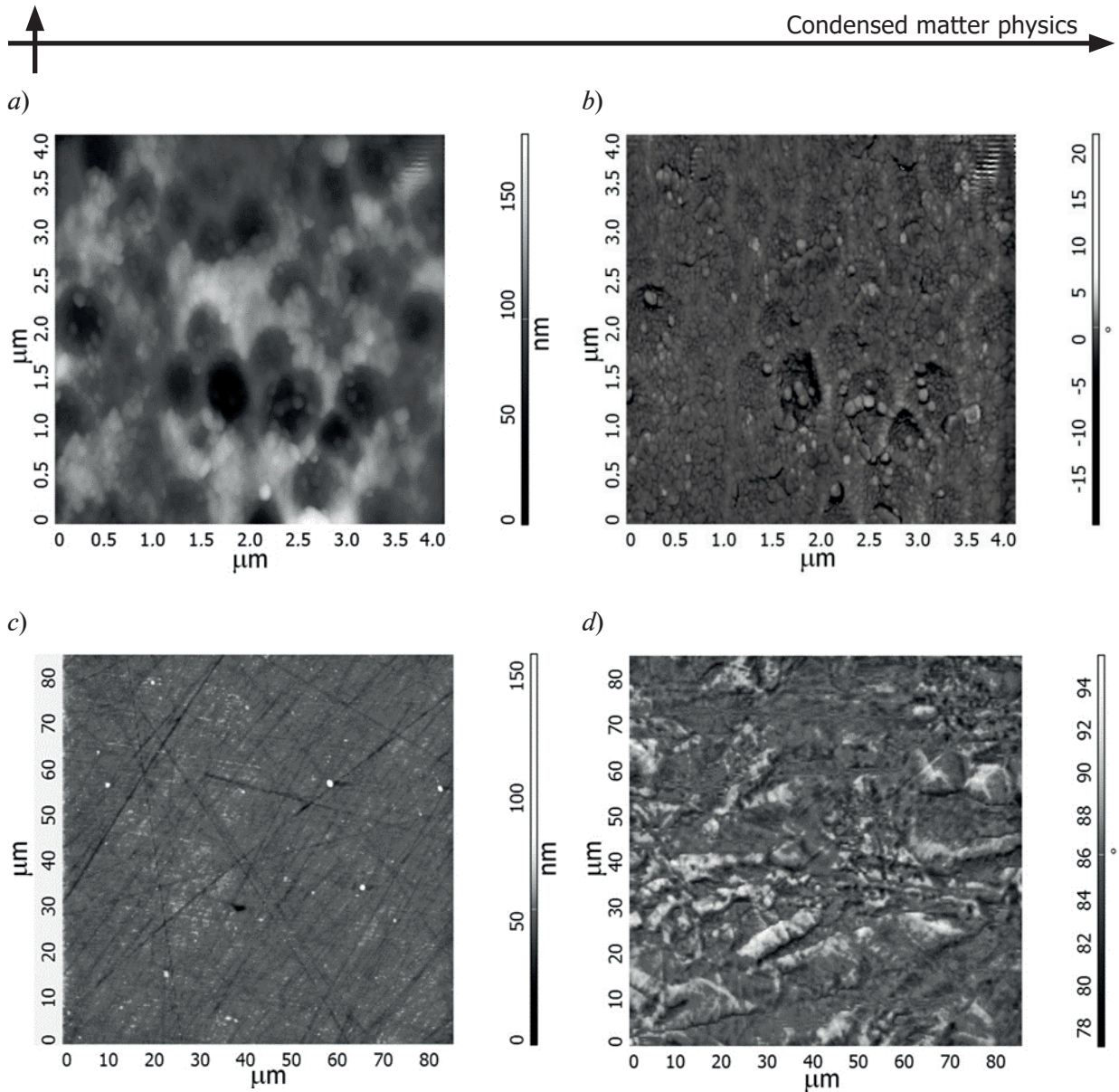and rhombohedral (> 190 K) (*b*) lattices

a)

b)

c)

d)

Fig. 3. AFM (*a*, *b*, *c*) and MFM (*d*) images of SmFe2 alloy surface, obtained with scan sizes of 4 × 4 μm (*a*, *b*) and 90 × 90 μm (*c*, *d*); phase contrast method was used in *b*

Surface studies of the polished thin section of $SmFe_2$ by magnetic force microscopy (Fig, 3, *d)* revealed a pronounced and rather complicated domain structure consisting of islands with the sizes from 6 to 12 μm and strips with the widths from 3 to 5 μm. The smallest domains are 0.8−1.0 μm wide. There may be different reasons for such a complex domain structure on the sample surface, for example, scattering fields (associated with stresses in the sample), as well as microinclusions of another phase. The presence of stresses is most likely due to high magnetostrictive properties of the $SmFe_2$ compound.

We studied linear deformation of $SmFe_2$ samples under the action of an external magnetic field depending on temperature (thermal expansion). Fig. 4 shows the temperature dependences of elongation at break and the coefficient of thermal expansion of the sample. Two anomalies can be observed at temperatures of 126 and 188 K, associated with phase transitions: a transition from the rhombohedral to the "angular" phase occurs at $T_2 = 188$ K, and a transition from the "angular" to the rhombic phase at $T_2 = 126$ K. These assertions are supported by the data found in literature [11, 12], as well as by the results of our own XRD studies. Notably, while there are only slight differences in the data given in literature for the value of the temperature T_2, the values cited for T_1 differ greatly. Thus, we have refined the values of the given temperature points by measuring the thermal expansion coefficient of $SmFe_2$ samples as a function of temperature.
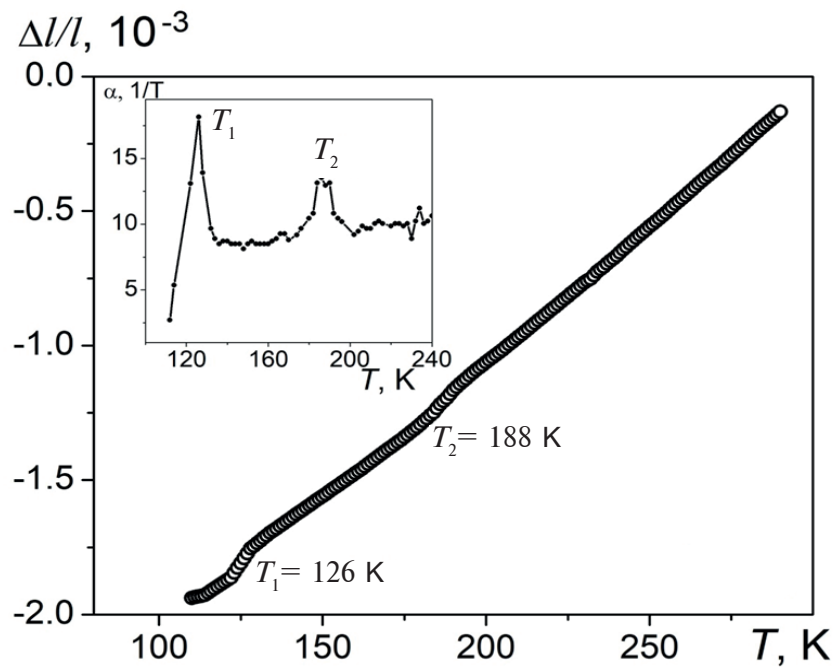
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Fig. 4. Temperature dependences of elongation at break in SmFe$_2$ sample (thermal expansion) and its coefficient of thermal expansion (inset);
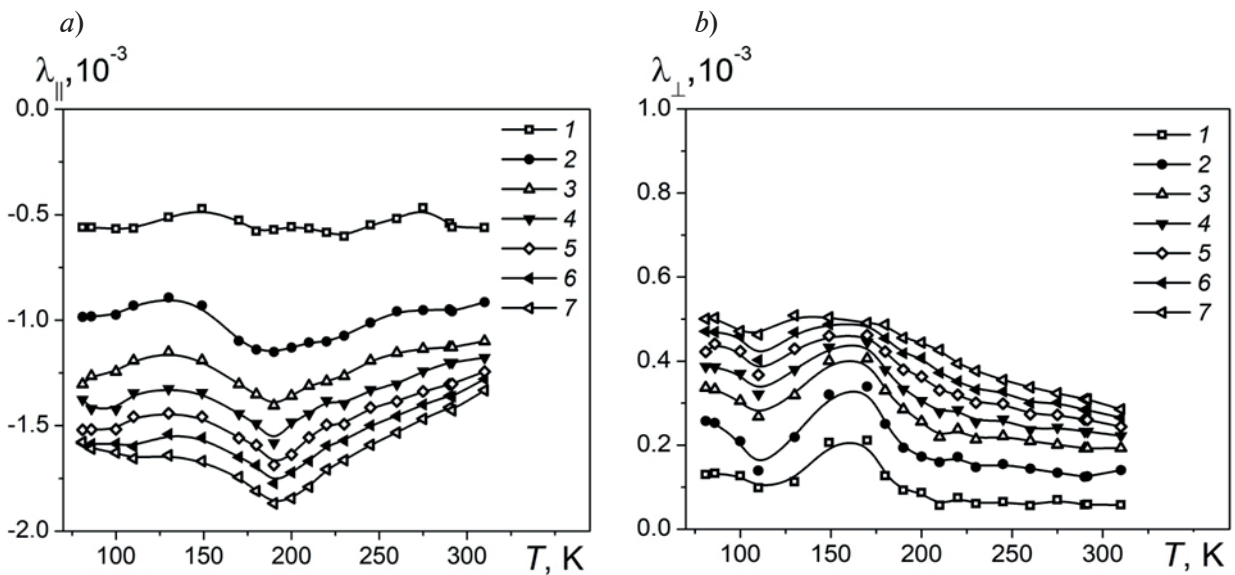T_1, T_2 correspond to phase transition points

Fig. 5. Temperature dependence of longitudinal (*a*) and transverse (*b*) magnetostriction in SmFe$_2$ with magnetic field strengths, T:
0.15 (*1*), 0.35 (*2*), 0.50 (*3*), 0.70 (*4*) 0.80 (*5*), 1.0 (*6*), 1.2 (*7*)

Magnetostrictive deformations turn out to be even more sensitive to magnetic and structural changes in the sample; therefore, it is convenient to use magnetostriction measurements as a method for detecting magnetic and structural phase transitions. Fig. 5 shows the temperature dependences of magnetostriction in the given alloy. Both longitudinal (Fig. 5, *a),* and transverse (Fig. 5, b) magnetostriction anomalies are observed at temperatures of 126 and 188 K for all values of the applied external field, expressed in terms of minimum and maximum absolute magnetostriction, respectively. Longitudinal magnetostriction in $SmFe_2$ has a negative sign and the maximum absolute value of $-1.9 \cdot 10^{-3}$, while transverse magnetostriction is positive and takes a maximum value of $+0.5 \cdot 10^{-3}$. The absolute values of magnetostriction decrease at room temperature, with the values of the longitudinal and transverse components amounting, respectively, to $-1.4 \cdot 10^{-3}$ and $+0.3 \cdot 10^{-3}$.

Conclusion

We have used induction melting to synthesize a $SmFe_2$ alloy; comprehensive study of the alloy included optical metallography and X-ray diffraction analysis to confirm that the alloy was homogeneous and single-phase. In addition, we have specifically established that a phase with 1:3 stoichiometry (most frequently observed in these compounds) was absent in our case.

We have used atomic force and magnetic force microscopy to verify that the sample surface had a heterogeneous cellular microstructure and a complex domain pattern. We have determined the sizes of the main structural elements.

We have found the specific temperature range where the angular phase exists during transition from rhombohedral to rhombic phase. We have considered the behavior of magnetostriction in the region of these anomalies.

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