Journal of Magnetism and Magnetic Materials 514 (2020) 167284

Contents lists available at ScienceDirect

Journal of Magnetism and Magnetic Materials

journal homepage: www.elsevier.com/locate/jmmm

Research articles

Effect of high magnetic field on the phase transition in Fe-24%Ga and Fe-27%Ga during isothermal annealing

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ARTICLE INFO

Keywords:
Phase transitions
Fe-Ga alloys
High magnetic field
Neutron diffraction
Metastable state

ABSTRACT

The effect of a high magnetic field (25 T) on the $D0_3$ to $L1_2$ phase transformation in two Fe-Ga alloys with 24 and 27 at.% Ga during isothermal annealing at 475 °C is investigated using neutron diffraction and magnetization measurements techniques. It is proved that annealing in the magnetic field increases the amount of fcc – derived $L1_2$ phase in both samples. This effect is stronger in the Fe-27%Ga alloy as compared with the Fe-24%Ga alloy. The reasons for enhancing the $D0_3$ to $L1_2$ phase transition in the magnetic field are discussed. We suggest that the most significant increase in the transformation rate is related to a decrease in the energy barrier between the phases involved in the transformation process.

1. Introduction

The Fe–Ga alloys is of great interest due to a high value of tetragonal magnetostriction and the still unknown nature of this phenomenon [1–3]. Unique physical properties of the Fe-Ga alloys make this material perspective to use as the basis for various electronic devices, e.g. magnetic sensors [4]. The magnetostriction dependence on Ga content has two maxima at about 19 and 27% (atomic percentage are used in this paper) [5]. These alloys may have different ordered and disordered phases depending on their composition and a heat treatment. In the as cast Fe-24%Ga and Fe-27%Ga alloys, the main phases at room temperature are A2/$D0_3$ [6] while long-term annealing leads to the formation of an equilibrium $L1_2$ phase [6,7]. Here is the

The most critical for the functional properties of FeGa alloys with the composition near the second magnetostriction peak is the transition from a metastable $D0_3$ phase to an equilibrium $L1_2$ phase. The $D0_3$ phase has positive magnetostricition; and whereas the $L1_2$ phase has negative magnetostricition [8]. It is also important that the Curie point for these phases is rather different: for the Fe-27%Ga composition, it is about 450 °C for the $D0_3$ phase, and it is above 620 °C for the $L1_2$ phase [9]. In both cases, it is practically impossible to determine the Curie temperature directly due to the phase transitions [10].

The mechanism of the $D0_3$ to $L1_2$ transition has been also discussed: a martensite-like transition was proposed in [11] and observed by a high-resolution TEM on a nanoscale level [12]. Most recent *in situ* neutron diffraction tests at both heating [13] and isothermal annealing [14] have proved the diffusion controlled character of this transition. The $L1_2$ phase nucleates mainly at the grain boundaries of a metastable $D0_3$ phase in as cast samples and grows down to the grain body. It was revealed that the transition proceeds with alternation of the first- and second-order phase transformations according to a $D0_3 \rightarrow A2 \rightarrow A1 \rightarrow L1_2$ scheme, where A1 and A2 are disordered bcc and fcc structures, receptively [14]. More information about phases and ordering processes in the FeGa alloys can be found elsewhere, for instance [6,15].

It is known that apart from temperature and time of annealing, external factors, e.g., the magnetic field applied during annealing, can affect the transition rate [16,17]. Over the past two decades, the high magnetic field (HMF) has been actively used as a tool to affect the structure and phase composition of various metallic materials [17–19]. Despite the number of studies, there are still many open questions related to the mechanisms of the HMF effect on thermally activated transformations. Qualitatively, the effect of the magnetic field can be predicted on the basis of thermodynamics. In essence, the regions of the existence of phases with a higher magnetic moment expand in the phase

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https://doi.org/10.1016/j.jmmm.2020.167284
Received 13 May 2020; Received in revised form 29 July 2020; Accepted 31 July 2020
Available online 03 August 2020
0304-8853/

diagrams, and those with a smaller or zero moments decrease. This was experimentally shown, for instance, for Fe-C [20] and Mn-Bi [21] compounds. Since the Fe-Ga alloys have a cascade of phase transformation, and the phases have different magnetic properties, the alloys of this system are ideal candidates to study the effect of the HMF effect on the kinetics of phase transformations.

Thus, the purpose of this paper is to study the HMF effect on the solid-state transformation in Fe-Ga alloys and to clarify the mechanism of this effect. We have chosen to study phase transitions under isothermal conditions already well known from the previous paper [13]. We have applied the magnetic field during annealing at the temperature at which a practically paramagnetic metastable $D0_3$ phase transforms to the ferromagnetic $L1_2$ phase. It is demonstrated that, indeed, the magnetic field drastically affects the transformation kinetics and, therefore, the phase ratio under the chosen conditions.

2. Materials and methods

Alloys Fe-24%Ga and Fe-27%Ga-0.08%Tb were prepared by induction melting under protective Ar atmosphere. In this paper, small additions of Tb into overstoichiometric composition Fe-27%Ga were used to increase the transformation time during isothermal annealing, because Tb decreases the $D0_3 \rightarrow L1_2$ transformation kinetic [22]. As the transformation rate in Fe-27%Ga alloy is high, it is methodological difficult to carry out careful experiment and a little amount of Tb was added to slow down the transition. Four samples with dimensions 6 × 6 × 20 mm were cut from each ingot and sealed into quartz ampoules under vacuum to prevent oxidation during annealing. High magnetic field annealing was applied in the National Laboratory of High Magnetic Fields (LNCMI-CNRS), DC high magnetic field (HMF) 25 T was used. The annealing temperature 475 °C was chosen based on the time-temperature-transition diagram recently proposed for the Fe-27%Ga alloy [15] to provoke the $D0_3 \rightarrow L1_2$ transformation. Annealing time was chosen to be 15 and 45 min for the Fe-27%Ga-0.08%Tb samples and 40 and 60 min for the Fe-24%Ga samples. All heat treatments were carried out with and without applying HMF for the same conditions. There are different stable and metastable phases in the Fe-Ga system dependently on the composition and temperature [7]. In this experiments only several phases are in play, namely A2, $D0_3$ and $L1_2$. The A2 structure[1] is disordered bcc phase where Ga atoms randomly substituting for iron atoms. The $D0_3$ phase[2] is a bcc-derivative structure because it formed by 8 bcc unit cells where Ga atoms occupying specific positions. This structure has an ideal stoichiometric composition of Fe_3Ga but exists within a wide range of Ga concentration. The A2 and $D0_3$ structures can exist coherently in the volume of alloy [6]. The $L1_2$ phase[3] is fcc equilibrium structure, where Fe atoms sitting at face-centred sites and Ga atoms at the corner sites. For the detail phase analysis, the neutron diffraction patterns were measured at a high-resolution time-of-flight Fourier diffractometer (HRFD) operating at the IBR-2 pulsed reactor at JINR (Dubna) [23]. Neutron diffraction was chosen as a very effective tool for the analysis of the ordering processes in the metallic alloys and because the high neutron penetration depth allows observing bulk effects and excluding the influence of the surface and the local inhomogeneities. High-resolution patterns were measured to study the samples structure. Due to technical limitations, these patterns were measured with a fixed sample orientation relative to the neutron beam, and the intensities of diffraction lines were strongly distorted with a crystallographic texture. To determine the phase ratio in eight studied samples, we used a high-intensity mode of neutron diffraction and a sample rotation regime to leveling the effect of the texture. To estimate magnetic properties after annealing, we measured magnetic hysteresis loops for all the samples using a vibrating sample magnetometer (VSM) LakeShore 7407. Phase distribution maps were obtained by EBSD method using scanning electron microscope FEI Quanta 200 with EBSD attachment Oxford Instruments.

[1] The A2 has an α-Fe-type structure with Fe and Ga atoms randomly distributed, sp. gr. Im3m, a ≈ 2.92 Å (at 20 °C).

[2] The D03 has a BiF3-type structure with Fe and Ga atoms partially ordered, sp. gr. Fm3m, a ≈ 5.83 Å (at 20 °C).

[3] The L12 has a Cu3Au-type structure with Fe and Ga atoms partially ordered, sp. gr. Pm3m, a ≈ 3.72 Å (at 20 °C).

3. Results and discussion

The structure of both alloys in the as cast state was identified as a partly ordered $D0_3$ phase, more precisely, it is $D0_3$ clusters embedded into A2 matrix [6]. As the purpose of this paper is to study the $D0_3 \rightarrow L1_2$ transition, the details about the cluster-like structure of the $D0_3$ phase are ignored in the present paper. The $D0_3$ ordering in the Fe-Ga alloys is confirmed by the presence of the so-called "superstructure" peaks in the neutron diffraction patterns with a much lower intensity than that of the "fundamental" ones. For the $D0_3$ phase, the Miller indices of the fundamental peaks satisfy the condition (h + k + l) = 4n (for instance, 220); whereas 111, 200, 311, etc. are the superstructure peaks. For the $L1_2$ phase, the fundamental peaks have all even or all odd Miller indices (for instance, 111); whereas the superstructure peaks have mixed Miller indices (100, 110, etc.). Fig. 1 presents the medium-resolution neutron diffraction patterns of two pairs of Fe-24%Ga (annealed in 40 and 60 min with and without HMF) and two pairs of Fe-27%Ga (annealed in 15 and 45 min with and without MF) samples. It is obvious that the HMF annealing promote the $L1_2$ phase formation in all four pairs until the complete disappearance of the $D0_3$ phase (Fig. 1d). In the Fe-24%Ga alloy annealed during 40 and 60 min, the fraction of the $L1_2$ phase is increased from 3% to 8% and from 7% to 30% after as a result of applying the magnetic field. In the Fe-27%Ga alloy annealed during 15 and 45 min, the fraction of the $L1_2$ phase increased from 14% to 60% and from 71% to 100% in the presence of the magnetic field, respectively.

To estimate the influence of HMF on the transformation kinetics, we obtained *in situ* kinetic of the $D0_3 \rightarrow L1_2$ transformation during isothermal annealing without an application of HMF for the Fe-24%Ga alloy (Fig. 2a) using a neutron diffraction method. Similar data for the Fe-27%Ga were obtained earlier in [14] (Fig. 2b). The large square points correspond to the annealing under HMF and the circle points – without the magnetic field. The mismatch of this experiment points with previously obtained data connected with the slight difference in experiments conditions and alloys composition. The $D0_3 \rightarrow L1_2$ phase transformation rate in the pre-stoichiometric alloy Fe-24%Ga is much slower than in the Fe-27%Ga [21] as evidenced by the different slope of the kinetic curves, wherein the HMF practically does not affect on start of transformation. An increase in the concentration of Ga leads to the disappearance of the incubation period (Fig. 2b), which indicates the barrierless nucleation of a $L1_2$ phase.

The most significant effect of the HMF on the $L1_2$ phase content is observed for the Fe-27%Ga samples after 15 min annealing. A large difference in the phase composition leads to a significant change in the magnetic properties (Fig. 3). The saturation magnetization M_S after HMF annealing (60% $L1_2$) is 155 emu/g; whereas in the no-field annealed sample (14% $L1_2$), it is only 120 emu/g. The reason is the difference of bcc-born ($D0_3$) and fcc ($L1_2$) phase's M_S at room temperature, which is about 120 and 160 emu/g, respectively, with the average magnetic moment on Fe 1.69 and 2.27 μ_B.

These results clearly demonstrate a remarkable effect of the magnetic field on the $D0_3 \rightarrow L1_2$ transformation rate for both studied Fe-Ga alloys. The isothermal annealing temperature 475 °C is above of the Curie point T_C of the $D0_3$ phase for the Fe-27%Ga or close to Tc for the Fe-24%Ga alloy, and it is below T_C of the $L1_2$ phase for both alloys. For this reason, an increase in the volume fraction of the ferromagnetic $L1_2$ phase in the presence of HMF is expected due to energetically benefits of this phase formation. The effect of the magnetic field on the phase

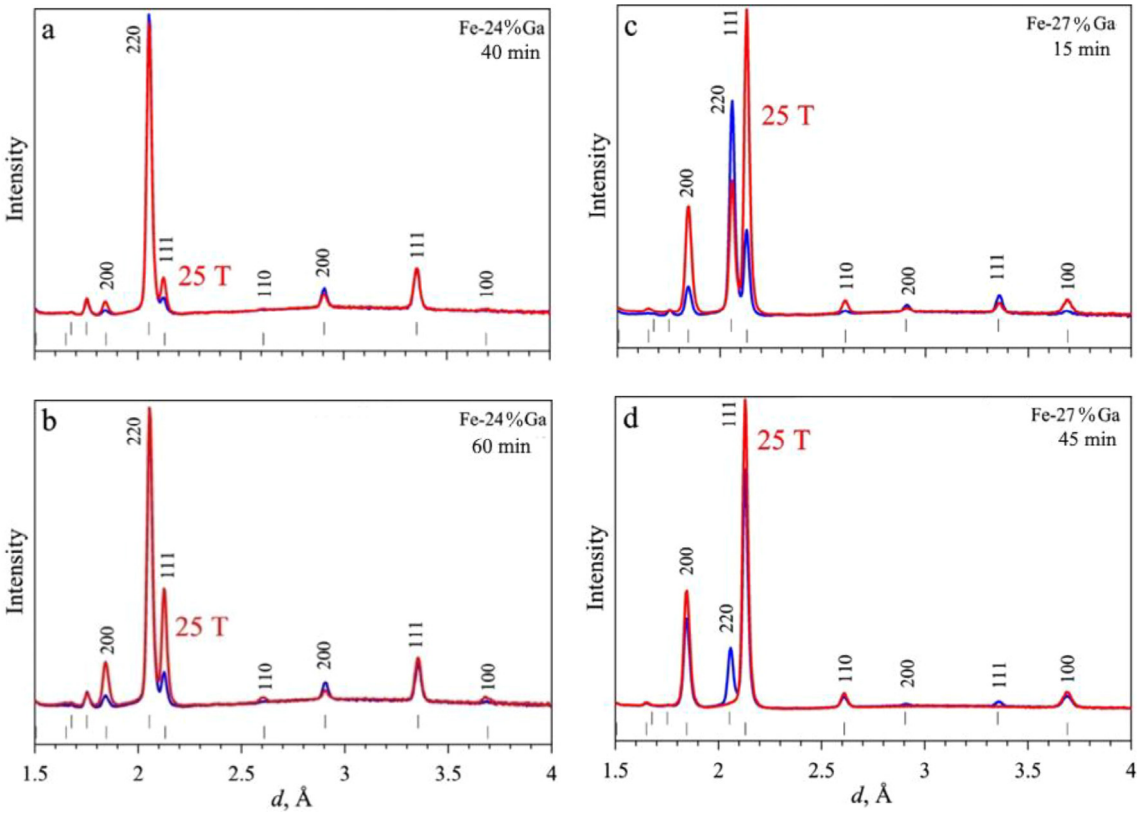

Fig. 1. Neutron diffraction patterns of the Fe-24%Ga (a, b) and Fe-27%Ga (c, d) annealed without (blue line) and with the magnetic field 25 T (red line) at 475 °C, 40 min (a); 60 min (b); 15 min (c) and 45 min (d). The vertical bars indicate the calculated peak positions for the $D0_3$ (upper row) and $L1_2$ (lower row) phases. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

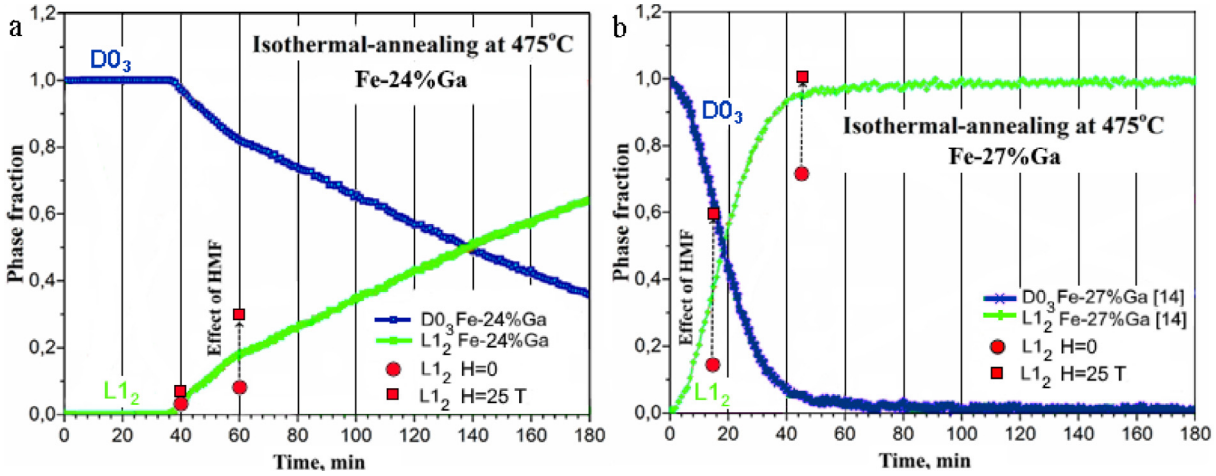

Fig 2. *In situ* transformation kinetics for Fe24%Ga (a) and Fe-27%Ga (b) alloys during isothermal annealing at 475 °C. The experimental point (this paper): the circles and squares correspond to the annealing without and with the magnetic field of 25 T. The black arrows show the value of the effect of the high magnetic field.

transformation is usually considered regarding a change of the thermodynamic potential and a shift of the transformation temperature [20,21,24,25]. From this viewpoint, the effect of the magnetic field H is similar to the external pressure, and the shift of the transformation temperature is determined by the Clausius–Clapeyron type relation $\Delta T \sim \Delta MH$ [26] (here ΔM is a change in magnetization during transformation). On the other hand, the HMF can affect the transformation kinetics by a decrease of the energy barrier of a new phase nucleation and/or an increase in the growth rate. However, considering the polymorphic $\gamma \rightarrow \alpha$ transformations in iron under cooling, as an example, it was found that the magnetic field weakly affects the kinetics of the new phase growth [27].

At the same time, as it is shown in [28], the effect of HMF on the martensitic transformations in steel can be described by taking into account a decrease of the transformation barrier due to the ordering of magnetic moments under the applied magnetic field. Thus the influence of the magnetic field on the transformation kinetics from a metastable paramagnetic $D0_3$ phase to an equilibrium ferromagnetic $L1_2$ phase is proved, however, the mechanism of this effect is rather complex and remains under discussion. We believe that both the change in the thermodynamic conditions and the transformation kinetics under HMF can be important and their relative role depends on the alloy

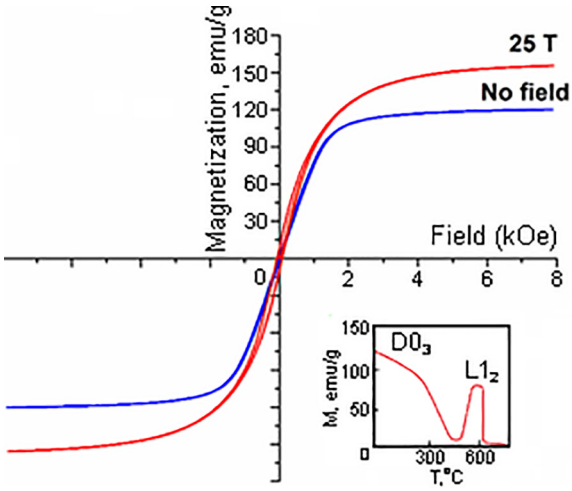

Fig.3. Magnetization hysteresis loop Fe-27%Ga samples annealed at 475 °C 15 min with and without the magnetic field. The insert is the corresponding temperature dependence of magnetization during 6 °C/min heating.

composition and temperature. As it is seen from Fig. 2a, in the Fe-24%Ga alloy, the HMF practically does not affect the nucleation (incubation period) and results in a remarkable increase of a new phase growth rate; this behavior is, most probably, due to an increase in the new phase energy gain under HMF. At the same time, for the over-stoichiometric Fe-27%Ga alloy, the transformation kinetics is quite different and the applied HMF increases significantly the nucleation rate (see Fig. 2b). This conclusion is also confirmed by phase's distribution maps (Fig. 4). After 15 min annealing there are a small amount of fcc phase grains which are localized mainly along grain boundaries of initial bcc phase (Fig. 4a). Application of HMF lead to significant increase of fcc nucleus amount. At the same time, size of fcc grains remains approximately the same as after annealing without HMF (Fig. 4b).

This allows us to conclude that increase in the volume fraction of $L1_2$ phase by HMF application is a result of higher nucleation rate. The latter observation indicates a decrease transformation barrier, which may be caused by the ordering of the magnetic moments under HMF, as it discussed in Refs. [28,29].

4. Conclusions

A noticeable increase of the $L1_2$ phase was observed upon isothermal annealing at 475 °C of Fe-24%Ga and Fe-27%Ga samples under the DC magnetic field of 25 T as compared with annealing without field. The effect is observed for all the studied samples and at any holding time until the complete disappearance of the $D0_3$ phase. The most considerable difference in the phase composition of the samples annealed with and without magnetic field is found in the sample Fe-27%Ga after annealing for 15 min. This indicates an acceleration of the formation of new phase nuclei by the magnetic field during the $D0_3 \rightarrow L1_2$ transformation.

CRediT authorship contribution statement

V.A. Milyutin: Conceptualization, Investigation, Writing - original draft. **I.V. Gervasyeva:** Conceptualization, Supervision, Writing - review & editing. **D.A. Shishkin:** Methodology, Data curation. **Yu. N. Gornostyrev:** Writing - review & editing. **E. Beaugnon:** Supervision. **I.A. Bobrikov:** Methodology, Data curation. **A.M. Balagurov:** Methodology, Data curation, Writing - review & editing. **A.K. Mohamed:** Data curation. **I.S. Golovin:** Conceptualization, 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.

Acknowledgements

The research was carried out within the state assignment of the Ministry of Education and Science, Russia (topics "Magnet" No. AAAA-A18-118020290129-5 and "Structure" No. AAAA-A18-118020190116-6) partly supported by the Russian Foundation for Basic Research (RFBR) No. 18-03-00623 and Russian Science Foundation (RSF) No. 19-72-20080 (neutron diffraction study). The authors are grateful to

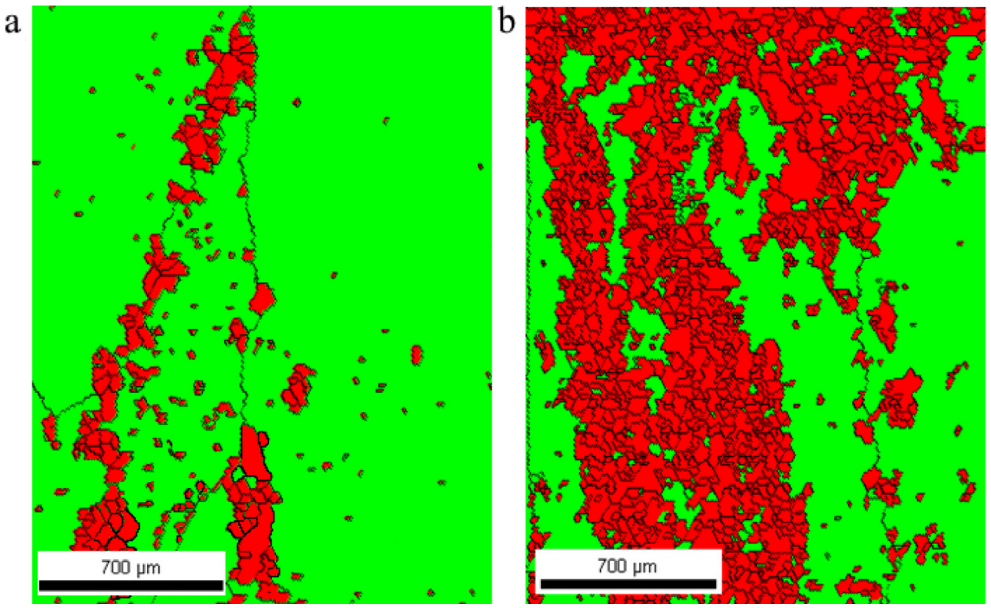

Fig.4. Maps of the phases in Fe-27%Ga samples annealed at 475 °C 15 min without (a) and with the magnetic field (b). Green color is bcc phase, red color is fcc phase. Field direction is vertical. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

LNCMI-CNRS, a member of the European Magnetic Field Laboratories, for their support. The authors thank Dr. E. Bazanova for her critical reading of the manuscript and Dr. V.V. Palacheva for her help with preparing the alloys for the investigation.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jmmm.2020.167284.

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