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Structure, spin reorientation and Mössbauer effect studies of $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys

ZHENG Xiaoping[1,2,4], ZHANG Peifeng[1,3], FAN Duowang[1], LI Fashen[3] & HAO Yuan[4]

1. Key Laboratory of Opto-Electronic Technology and Intelligent Control, Ministry of Education, Lanzhou Jiaotong University, Lanzhou 730070, China;
2. Institute of Electronic Information Science and Technology，Lanzhou Teachers College, Lanzhou 730070, China;
3. Key laboratory for Magnetism and Magnetic Materials, Ministry of Education, Lanzhou University, Lanzhou 730000, China;
4. State Key Lab. of Advanced Non-Ferrous Materials, Gansu Province, Lanzhou University of Technology, Lanzhou 730050, China

Correspondence should be addressed to Zheng Xiaoping and Zhang Peifeng (email: zxp51913@sina.com.cn, zpf519@sina.com)

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Abstract The effect of Al substitution for Fe on crystal structure, magnetostriction and spontaneous magnetostriction, anisotropy and spin reorientation of a series of polycrystalline $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys (x = 0, 0.05, 0.1, 0.15, 0.20, 0.25, 0.30, 0.35) at room temperature and 77 K was investigated systematically. It was found that the primary phase of $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ is the $MgCu_2$-type cubic Laves phase structure when $x < 0.4$ and the lattice constant a of $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ increases approximately and monotonically with the increase of x. The substitution of Al leads to the fact that the magnetostriction λ inceases slightly in a low magnetic field ($H \leqslant 40$ kA/m), but decreases sharply and is easily close to saturation in a high applied field as x increases, showing that a small amount of Al substitution is beneficial to a decrease in the magnetocrystalline anisotropy. It was also found that the spontaneous magnetostriction λ_{111} decreases greatly with x increasing. The analysis of the Mössbauer spectra indicated that the easy magnetization direction in the {110} plane deviates slightly from the main axis of symmetry with the changes of composition and temperature, namely spin reorientation. A small amount of non-magnetic phase exists for x = 0.15 in $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys and the alloys become paramagnetic for x > 0.15 at room temperture, but at 77 K the alloys still remain magnetic phase even for x = 0.2. At room temperature and 77 K, the hyperfine field decreases and the isomer shifts increase with Al concentration increasing.

Keywords: magnetostriction, cubic Laves phase, spin reorientation, Mössbauer.

1 Introduction

The pseudobinary rare-earth iron alloy $Tb_{0.3}Dy_{0.7}Fe_2$ (commercially known as Terfenol-D) is an excellent magnetostrictive material. It has relatively low magnetocrystalline anisotropy[1] and can be used as ultrasonic transducers and micro-actuators. However, there is some limitation in its application because of its low tolerance to tensile and shear forces, low electrical resistivity and relatively high saturation field. In order to modify its application properties considerable research work has been done on substituting Fe with other elements in attempt to improve the magnetic and magnetostrictive properties in the $Tb_{0.3}Dy_{0.7}(Fe_{1-x}T_x)_2$ (T = Mn, Co, Ni, Ga, Al, B, Be, etc.) alloys[2−8]. Among them Al is regarded as an ideal substitution for Fe because the addition of Al to Fe is known to possess a larger magnetostriction, furthermore, increase resistivity and ductility and decrease anisotropy[5−10]. Therefore, a kind of excellent and potential giant magnetostrictive material would be developed.

^{57}Fe Mössbauer study of the rare-earth iron alloys $R^1_{1-x}R^2_xFe_2$[11−13] has revealed that they possess several different types of spectra even though these compositions have identical crystallographic structures. Each of these spectra results from a different direction of easy magnetization relative to the crystallographic axes of the unit cell[14]. With the easy magnetization direction (EMD) along [100], all iron sites are equivalent and a simple six-line spectrum is obtained, as was observed for $HoFe_2$ and $DyFe_2$. If EMD is along the [111] axis, two magnetically inequivalent iron sites with a relative population of 3:1 exist, giving rise to a spectrum which is a superposition of two six-line patterns with a 3:1 relative intensity ratio, as observed for YFe_2, $TbFe_2$, and $ErFe_2$. If EMD is parallel to the third major cubic axis [110], two magnetically inequivalent iron sites with a relative population ratio of 2:2 occur, as observed in $SmFe_2$ at low temperature, produce a spectrum, which is a superposition of two six-line patterns with a relative intensity ratio of 1:1. U. Atzmony and M. P. Dariel[11,15] also reported the presence of unusual [uv0] and [uuv]-type EMD, explained and proved the existence of these EMD using a phenomenological treatment of a single-ion model in the $R^1_{1-y}R^2_y(Fe_{1-x}Al_x)_2$ alloys.

The $Tb_{0.3}Dy_{0.7}Fe_2$ alloys generally possess a large magnetocrystalline anisotropy as well as a large magnetostrictive anisotropy $|\lambda_{111}/\lambda_{100}| \gg 1$, with a saturation magnetostriction $\lambda_s = 0.6\lambda_{111}+0.4\lambda_{100}$ in the polycrystalline alloys, therefore, the saturation magnetostriction is mainly decided by λ_{111}. In this paper, we investigated the effect of Al substitution for Fe on the macroscopic properties and microscopic characters of the polycrystalline $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys.

2 Experimental details

All the samples of the $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ (x = 0, 0.05, 0.1, 0.15, 0.20, 0.25, 0.30, 0.35) alloys were prepared by melting the appropriate amount of constituent metals in an arc-melting furnace under a high purity argon atmosphere and remelting several times to

achieve good homogeneity. The purity of the Tb and Dy was 99.98%, and the Fe and Al was 99.98%. An excess of 4 wt% Dy was added to compensate for evaporation losses. The button-shaped samples were wrapped in Ta foil, sealed in evacuated quartz tubes filled with high purity argon, homogenized at 950℃ for at least 50 hours and then cooled in air.

X-ray diffraction analysis was performed by means of a Rigaku D/max-2400 diffractometer with a pyrolytic graphite monochromator using Cu-Kα radiation ($\lambda \approx 1.5405$ Å), and the lattice constant was calculated using the software *Powdercell*. Magnetostriction measurements were carried out using a standard strain gauge in applied fields up to 125 kA/m at room temperature. The samples, which were cut from the button using a linear spark erosion cutter, were typically 5 mm × 6 mm × 2 mm.

Using a standard constant acceleration Mössbauer spectrometer, transmission spectra were recorded with a ^{57}Co source (kept at room temperature) using a multi-channel analyzer operated in the time mode. The velocity was calibrated with an α-Fe standard sample. The absorbers were prepared by powdering the sample under acetone and then mixing it with vacuum grease. The experimental spectra were fitted by a least-square fit procedure that could accommodate 13 independent parameters within an assumed theoretical spectrum.

3 Results and discussions

The powder X-ray diffraction patterns of the $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys indicate that all homogenized alloys are almost completely of the $MgCu_2$-type cubic Laves phase structure, as shown in Fig. 1. The lattice constant a of the $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys

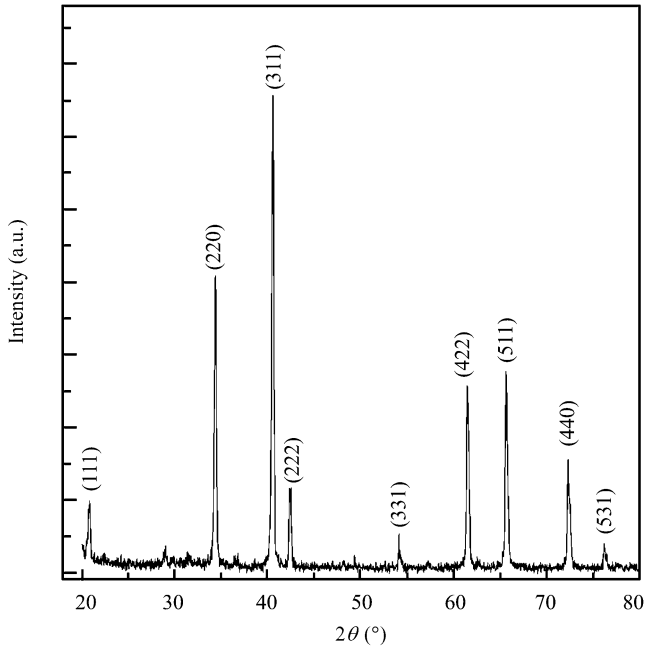

Fig. 1. X-ray diffraction patterns for the $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys.

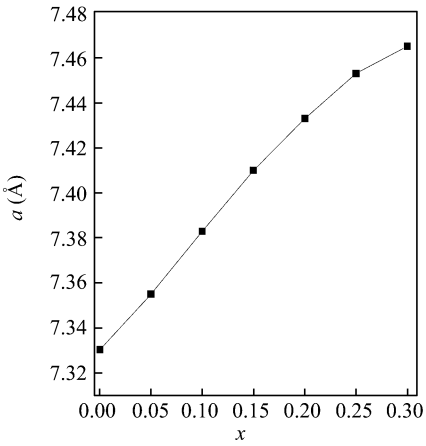

Fig. 2. The lattice constant vs. Al content for the $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys.

increases rapidly and monotonically with Al content increasing, as shown in Fig. 2, due to the larger metallic radius of Al compared to that of Fe, which is similar to the behavior of $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Co_x)_2$ and $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Be_x)_2$[8,9] and approximately follows Vegard's linear relationship.

The magnetic field dependence of the magnetostriction for the $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ ($0 \leqslant x \leqslant 0.15$) alloys is shown in Fig. 3. From the inset of Fig. 3, it is obvious that the magnetostriction is slightly enhanced in a low applied field ($H \leqslant 40$ kA/m) when x is less than 0.15. However, the magnetostriction for the Al sub-

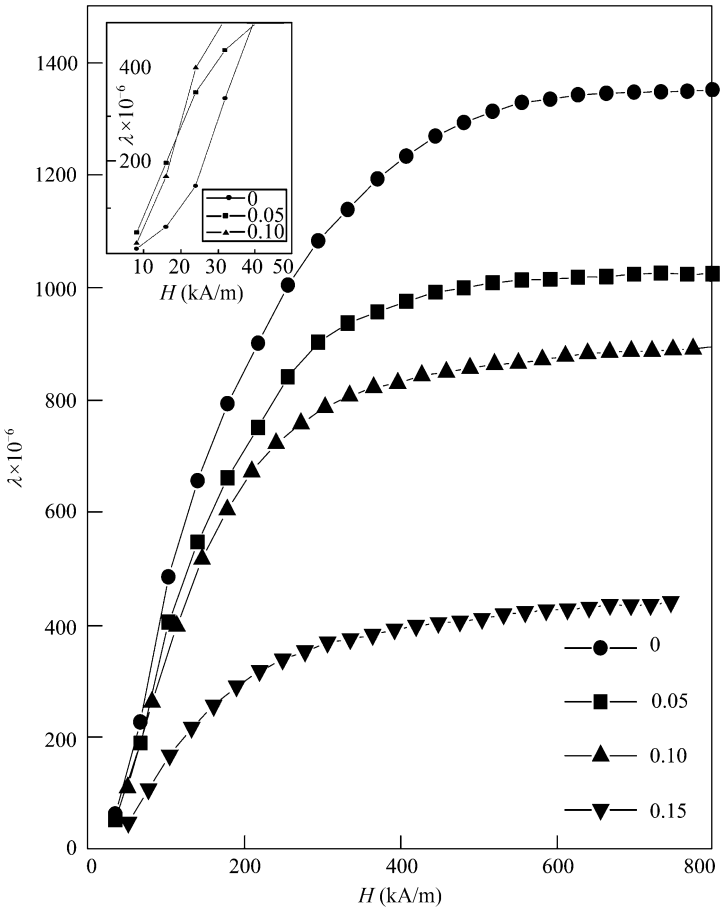

Fig. 3. Magnetostriction for the $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys as a function of the applied field. The inset shows the magnetostriction in the low applied field ($H \leqslant 40$ kA/m).

stituted samples is distinctly lower than that of the Al-free samples in a higher magnetic field. Furthermore, the magnetostriction of the Al substituted samples is more easily saturated in an applied field with the increase of x, implying that the substitution of Al may be beneficial to decreasing the magnetocrystalline anisotropy and thus leads to the transformation of EMD, i.e. the spin reorientation. Obviously, the decrease of anisotropy and the change of EMD result from the Al substitution for Fe. The giant magnetostriction is scarcely 440×10^{-6} for $x = 0.15$ and vanishes for $x > 0.15$, less than 100×10^{-6}.

The samples in powder form were step-scanned with Cu-Kα radiation by the X-ray diffractometer at a high. Bragg angle 2θ. It was observed that the (440) line was shifted to a lower Bragg angle as x increased, showing that the lattice constant increased also with the increase of x. The existence of spontaneous magneostriction leads to the crystal-structure distortion of the alloys with the cubic $MgCu_2$-typed Laves structure. The splitting of the (440) line results in the (208) and (220) lines and the spontaneous magnetostriction λ_{111} is obtained in terms of the formulation (1)

$$\lambda_{111} = \frac{\Delta d}{d} = \frac{\sin\theta_2 - \sin\theta_1}{\sin\theta_1}. \tag{1}$$

The Al content dependence of spontaneous magnetostriction λ_{111} of the $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys is shown in Fig. 4. It is apparent that λ_{111} decreases rapidly as x increases, indicating that the Al substitution for Fe leads to a great decrease of λ_{111}. The spontaneous magnetostriction is 1654×10^{-6} for $x = 0$ and 540×10^{-6} for $x = 0.2$ respectively and the decrease magnitude is up to 1100×10^{-6}, which is in good agreement with the results of Magnetostriction measurements.

The Mössbauer spectra for the $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys ($x = 0$, 0.05, 0.1, 0.15, 0.2) series at room temperature and 77 K are presented in Fig. 5, respectively, in which the

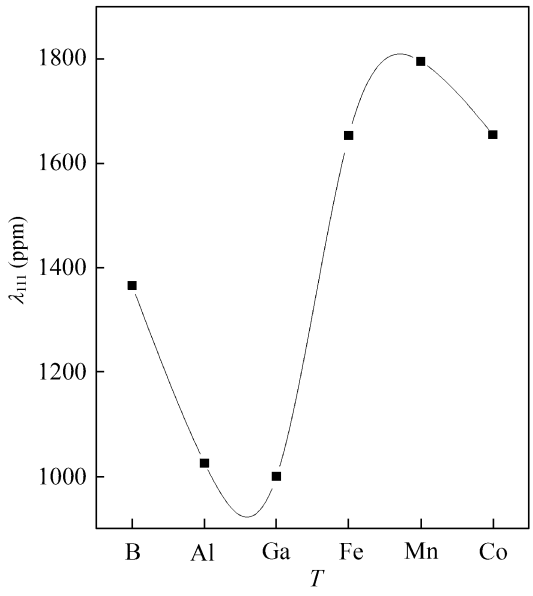

Fig. 4. The substitution dependence of spontaneous magnetostriction λ_{111} of $Tb_{0.3}Dy_{0.7}(Fe_{0.9}T_{0.1})_{1.95}$.

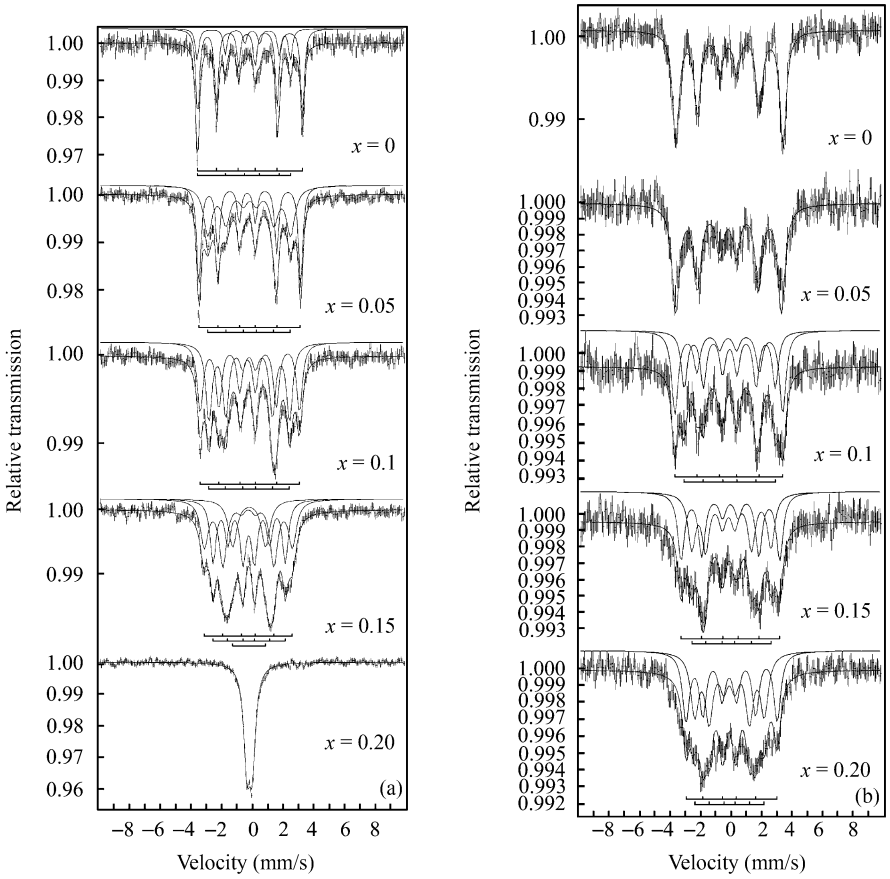

Fig. 5. Mössbauer spectra of the compounds. (a) At room temperature; (b) at 77 K.

solid line is the theoretical results of spectra fitted by a least-square fit procedure and the broken line is the experimental datum. It can be seen that the spectra are quite complex and the character of spectra is distinct with various Al content. As shown in Fig. 5(a), the spectra of $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ alloys can be analyzed as a superposition of two six-line patterns with relative intensities 3: 1, indicating that EMD is along [111]. For the alloys with $0 \leqslant x \leqslant 0.15$, the Mössbauer spectra indicate the existence of a wide hyperfine-field distribution, $p(H)$, resulting from the random distribution of the Al atoms within the transition-metal sub-lattice. With Al content x increasing, the shape of spectra has very great changes because of spin reorientation, and the relative intensity ratio of the spectra is 3:1 (x = 0), 2:2 (x = 0.05), 2:2 (x = 0.1), and 2:2 (x = 0.15) respectively, indicating that EMD rotates and deviates from the major axis [111] in the plane {110} with the substitution of Al for Fe and thus the magnetostriction correspondingly decreases. For the spectrum of x = 0.15, there is also a two-line pattern with a relative intensity of 15%, indicating that the sample contains a small amount of paramagnetic phase. It can be seen that for x = 0.2 the spectrum of the alloys is no longer a six-line pattern, which suggests that this sample is paramagnetic. The Mössbauer measurements at room temperature are consistent with the

magnetostriction measurements.

From Figs. 5(a) and (b), we can see that there is a great deal of difference between them, which shows that EMD rotates with temperature. As seen in Fig. 5(b), at 77 K, the spectra for the alloys of $x \leqslant 0.5$ can be fitted as a set of six-line pattern, which indicates that EMD could be along [100] and become greatly complicated with the increase of x, showing the transformation of EMD. Above of all, EMD rotates slightly and deviates from the major axis [111] in the plane {110} with the changes of composition and temperature, i.e. the spin reorientation, which has effects on the magnetostriction and macroscopic properties for $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys.

Although the anisotropy is determined mainly by the rare-earth sublattice, the M (M: Fe or Al) ions influence the crystal-field interaction and lattice parameters and therefore also provide a small additional contribution to the anisotropy[16]. So the change of EMD in the present system may be due to changes in the crystal-field interaction and exchange splitting introduced by the Al atom.

By fitting the spectra, the hyperfine-field, H_{hf}, the isomer shift, *IS,* the quadruple splitting, *QS*, and the average values of these parameters for the $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys at room temperature and 77 K were obtained and listed in Table 1. It is obvious that the average hyperfine-field decreases with Al content increasing. Usually it is considered that the hyperfine-field is mainly correlated with the local environment of nuclei, which has three sources[17]

$$H_{hf}=H_C+H_L+H_D, \tag{2}$$

where H_C comes from Fermi contact interaction, namely Fermi field; H_L is the couple interaction between electronic orbit and nuclear spin moment, called orbit field;. H_D results from the interation between nuclear spin and electronic spin density moment, named couple field. Usually Fermi field is regarded as the most important component in H_{hf}. It has been shown, by combining NMR and Mössbauer investigations[18], that the average iron hyperfine-field and average iron moment are proportional

$$H_{hf}=\alpha\cdot\mu_{Fe}, \tag{3}$$

where α is the proportionality constant and μ_{Fe} is the iron moment. The decrease of μ_{Fe} with Al content increasing is evident in the range of ferrite compositions, which means that the Fe moment is influenced by Al substitution. Due to the small addition of Al metals in $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys, the H_{hf} and μ_{Fe} change slightly. At the same time, we notice that the μ_{Fe} at 77 K is bigger than at room temperature. It is not understandable that the increase of μ_{Fe} in the alloys leads to the increases of H_{hf} due to the enhancement of the exchange interaction in the Fe sublattice at low temperature.

The isomer shift is given by

$$IS=\alpha\cdot(\rho_A(0)-\rho_S(0)), \tag{4}$$

where $\rho_A(0)$ and $\rho_S(0)$ are the electron densities (contact densities) at the nuclear sites of absorber and source. The nuclear calibration constant α depends solely on nuclear properties. The average isomer shifts of the compounds obtained from Mössbauer measurements are also listed in Table 1. Research work[19,20] finds that for RFe_2 alloys the isomer shift can be affected by the electron concentration at the Fe nucleus shift and volume

change of the alloys. The effect of the volume change on *IS* could be approximately calculated by $\partial(IS)/(\partial \ln V)$, where $V = a^3$ and a is the lattice constant of the alloys. It is clear from Table 1 that the isomer shift increases with x-value increasing and $\partial(IS)/(\partial \ln V) > 3$ is obtained from these experiment data. However, the largest value $\partial(IS)/(\partial \ln V)$ so far found in high pressure experiment is only 1.8[19,20]. This would imply $\partial(IS)/(\partial \ln V) > 3$, nearly twice the largest value found in pressure experiments. The increase of isomer shift can result not only from the volume increase, but also mainly from s-electron density decrease due to Al substitution. We suggest that the decrease in s-electron density at iron nuclei, which results in the increase of the shielding effect of 3d electrons and therefore the increase of isomer shift, is mainly due to a filling up of the 3d iron band by the charge transfer of 2p electrons of Al atoms into the 3d band of Fe.

As shown in Table 1, the Quadrupole splitting, *QS*, for the alloys does not display concentration dependence.

Table 1 Hyperfine parameters H_{hf}(±0.7 kOe), *IS*(±0.022 mm/s) (rel. to α-Fe), *QS*(±0.022 mm/s) and average values of these parameters for the $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys at room temperature and 77 K

Room temperature	H_{hf}			*IS*			*QS*		
x	(Ⅰ)	(Ⅱ)	ave	(Ⅰ)	(Ⅱ)	ave	(Ⅰ)	(Ⅱ)	ave
0.00	189.4	212.5	206.7	–0.263	–0.293	–0.286	–0.275	0.105	0.013
0.05	168.0	206.3	187.2	–0.226	–0.254	–0.240	–0.021	0.08	0.030
0.10	163.7	201.3	182.5	–0.211	–0.245	–0.228	–0.003	0.072	0.035
0.15	146.4	178.7	162.6	–0.191	–0.205	–0.198	0.016	–0.000	0.008
77 K	H_{hf}			*IS*			*QS*		
x	(Ⅰ)	(Ⅱ)	ave.	(Ⅰ)	(Ⅱ)	ave.	(Ⅰ)	(Ⅱ)	ave.
0.00			219.3			–0.133			0.038
0.05			216.0			–0.127			0.014
0.10	208.2	202.9	205.6	–0.320	0.077	–0.121	–0.014	0.123	0.055
0.15	201.8	162.1	181.9	–0.037	–0.065	–0.051	0.002	0.092	0.047
0.20	186.7	142.5	164.6	–0.034	–0.103	–0.068	0.078	0.007	0.043

* The columns labelled (I) and (II) represent the two inequivalent Fe sites.

4 Conclusion

Aluminum substituted Terfenol-D $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys almost possess the $MgCu_2$-type cubic Laves phase when $x < 0.4$, and the lattice constant increases monotonically with x increasing. The magnetostriction of the alloys increases slightly in a low magnetic field ($H \leqslant 40$ kA/m) for $x < 0.15$ but decreases sharply and simultaneously is easily close to saturation in a large applied field with Al content x increasing and vanishes for $x > 0.15$. The spontaneous magnetostriction decreases greatly with the increase of x. The analysis of the Mössbauer spectra indicates that the easy magnetization direction in the {110} plane deviates slightly from the main axis of symmetry with the changes of composition and temperature. A small amount of non-magnetic phase exists for $x = 0.15$ in $Tb_{0.3}Dy_{0.7}(Fe_{1-x}Al_x)_{1.95}$ alloys and the alloys become paramagnetic for $x > 0.15$ at room temperature, but at 77 K the alloys still remain magnetic phase even for $x = 0.2$. At room temperature and 77 K, the hyperfine field decreases and the isomer shifts

increase with Al content increasing.

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