Physics of the Solid State, 2024, Vol. 66, No. 8

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A change in the exchange integral due to the hydrogen atoms introduction into the crystal lattice of Terfenol-D

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Received June 18, 2024
Revised June 18, 2024
Accepted June 20, 2024

The effect of hydrogenation on the structural and magnetic characteristics of the compound $Tb_{0.3}Dy_{0.7}Fe_2$ (Terfenol-D) was investigated. A hydride $Tb_{0.3}Dy_{0.7}Fe_2H_3$ with a Curie temperature near room temperature and a magnetic compensation point at $T = 140$ K was obtained. The main mechanisms responsible for both the change in the Curie temperature during hydrogenation and the appear of a noticeable volume magnetostriction near the Curie temperature were established. The change in the exchange integral upon the introduction of a fixed of hydrogen atoms (3 at.H/f.u.) into the crystal lattice of the $Tb_{0.3}Dy_{0.7}Fe_2$ compound was calculated.

Keywords: rare earth compounds, Laves phases, hydrides, Curie temperature, magnetostriction.

DOI: 10.61011/PSS.2024.08.59055.156

1. Introduction

Currently compounds of type $R\mathrm{Fe}_2$ with structure of Laves phases attracts attention not only from a practical but also from a fundamental point of view [1–3]. Due to their simple crystalline and magnetic structure, these compounds are considered by theorists as model objects, able to demonstrate high Curie temperature, larger saturation magnetization, giant magnetostriction, high magnetocaloric effect [4–5]. For example, the magnetostriction deformations of compounds $TbFe_2$ and $DyFe_2$ in region of temperatures close to room temperature, reach values $\sim 2 \cdot 10^{-3}$. Both compositions coincide significantly by value of magnetocrystalline anisotropy (MCA), and, hence, maximum values of magnetostriction can be obtained only in rather strong magnetic fields. After selection of definite ratio of terbium and dysprosium it is possible to significantly decrease MCA. Such approach resulted in finding the composition $Tb_{0.3}Dy_{0.7}Fe_2$ (in publications this compound with structure of Laves phases, and clarified composition $Tb_{0.27}Dy_{0.73}Fe_{1.95}$, is called as terfenol-D) [6]. Due to giant magnetostriction ($\lambda \sim 10^{-2}$) at room temperature observed in relatively weak magnetic fields (due to small value of magnetocrystalline anisotropy) the Terfenol-D is widely used in hydroacoustics, magnetostriction power drives, microelectronics and other applications [7–9].

Note especially that compounds $R\mathrm{Fe}_2$ are easily absorbed by hydrogen [9], its maximum amount can reach 4 at.H/form.unit. As a rule, hydrogen atoms entering the lattice of these compounds and spreading in interstices, do not result in type change of crystalline structure. But, depending on amount of absorbed hydrogen change in unit cell volume, interatomic spacings between magnitoactive atoms, as well change in magnetic properties can be significant [9–12]. Hydrogenated samples acquire new properties that differ from their original compositions, which can significantly expand the scope of application of these materials, for example, as detectors and sensors in hydrogen energy.

Due to the above mentioned this paper task is synthesis of hydrogenated samples of Terfenol-D, study of their structural, magnetic and magnetostriction properties, identification of basic physical mechanisms responsible for Curie temperature change and appearance of volume magnetostriction near Curie temperature upon introduction of fixed amount of hydrogen (3 at.H/f.u.) into crystal lattice $Tb_{0.3}Dy_{0.7}Fe_2$, and evaluation of relative change in resulting exchange integral during hydrogenation.

2. Samples preparation and experiment procedure

The initial alloy $Tb_{0.3}Dy_{0.7}Fe_2$ was synthesized in induction furnace in argon atmosphere by melting of the initial components Tb (99.5%), Dy (99.3%), Fe (99.9%). All basic details of such synthesis can be found in paper [13]. Hydride of initial compound was obtained as result of direct reaction on initial sample with high pure gaseous hydrogen, obtained during decomposition of titanium dihydride TiH_2. Details of procedure of hydrogenation and amount determination of hydrogen absorbed by the initial sample, are also given earlier in paper [14].

We obtained hydride with rather high content of hydrogen, namely 3 at.H/form.unit. Phase composition of the initial sample and its hydride was checked by X-ray diffraction analysis in diffractometer DRON-3M. Curie temperature of samples was determined using thermomagnetic analysis made on pendulum magnetometer in wide range of temperatures 80–720 K in external magnetic field 1 kOe.

The magnetostriction was measured by strain gauge techniques on set-up using electromagnet FL-1 in temperature range 80–300 K in magnetic fields up to 12 kOe [15]. The comparative studies of the magnetostriction for the initial composition and its hydride were associated with definite difficulties. Since as-cast alloy are crashed during the hydrogenation process, forming a fine powder, powder samples of both the hydride and the initial composition were used to measure magnetostriction. Powders were pressed under pressure 3 GPa in tablets with diameter of 10 mm and 3 mm thick. On pressed samples the longitudinal $\lambda_{\|}$ and transverse $\lambda_{\perp}$ magnetostriction was measured. The volume magnetostriction is calculated by equation

$$\omega = \lambda_{\|} + 2\lambda_{\perp}. \quad (1)$$

3. Results and discussion

The X-ray diffraction analysis showed that all obtained compositions, namely initial compound $Tb_{0.3}Dy_{0.7}Fe_2$ and its hydride $Tb_{0.3}Dy_{0.7}Fe_2H_3$ are practically single phase and have cubic structure of type $MgCu_2$. The lattice parameter (a) for $Tb_{0.3}Dy_{0.7}Fe_2$ and its hydride are given in Table. The obtained data agree well with literature data [13,14]. Besides, Table shows unit cell volume V and relative change in unit cell volume $\Delta V/V$ during hydrogenation. It was determined, that unit cell volume increasing $\Delta V/V$ during hydrogenation is 20.2% for $Tb_{0.3}Dy_{0.7}Fe_2H_3$ hydride (recalculated to one atom of hydrogen $\Delta V/V = 6.7\%$). Table for comparison presents parameters of binary compound YFe_2 (yttrium — non magnetic analogue of rare earth elements) and its hydride with maximum content of hydrogen YFe_2H_4 [16–18]. It is obvious that for the last $\Delta V/V = 27.2\%$, and, therefore, $\Delta V/V$ recalculated per one atom of hydrogen is 6.8%, which correlates well with data for $Tb_{0.3}Dy_{0.7}Fe_2H_3$ hydride [16,19]. Moreover, note that type of rare-earth atom has no noticeable effect on value of change in unit cell volume $\Delta V/V$ during hydrogenation.

Table. Structural and magnetic properties of compounds $Tb_{0.3}Dy_{0.7}Fe_2H_x$ (where $x = 0$ and 3) and YFe_2H_x ($x = 0$ and 4) (lattice parameters a, unit cell volume V, relative change of volume $\Delta V/V$, Curie temperature T_C, change in Curie temperature ΔT_C, magnetic compensation point T_k)

Composition	a, Å	V, Å^3	$\Delta V/V$, %	T_C, K	ΔT_C, K	T_k, K
$Tb_{0.3}Dy_{0.7}Fe_2$	7.324	392.8	–	665	–	–
$Tb_{0.3}Dy_{0.7}Fe_2H_3$	7.789	472.5	20.2	294	371	140
YFe_2	7.357	398.2	–	545	–	–
YFe_2H_4	7.973*	506.8	27.2	133	412	–

Note. * Lattice parameter calculated for cubic structure of type $MgCu_2$.

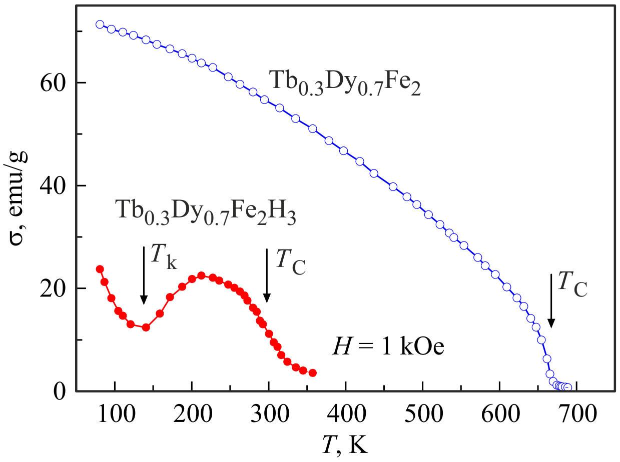

Figure 1. Thermomagnetic analysis of $Tb_{0.3}Dy_{0.7}Fe_2$ and its $Tb_{0.3}Dy_{0.7}Fe_2H_3$ hydride.

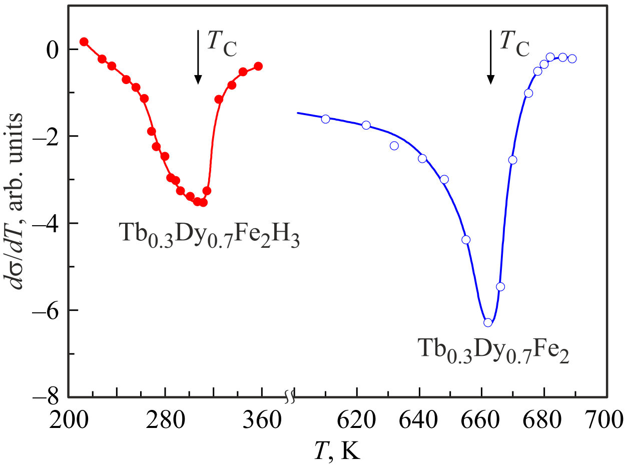

Figure 2. Temperature dependence $d\sigma/dT$ for $Tb_{0.3}Dy_{0.7}Fe_2$ and $Tb_{0.3}Dy_{0.7}Fe_2H_3$.

Figure 1 presents the curves of thermomagnetic analysis for $Tb_{0.3}Dy_{0.7}Fe_2$ and its $Tb_{0.3}Dy_{0.7}Fe_2H_3$ hydride. It is obvious that value of magnetization σ of investigated compositions during heating and approaching the Curie temperature abruptly decreases (in case of initial composition the magnetization falls practically to 0). We determined Curie temperature from the analysis of temperature dependence of magnetization derivative by temperature $d\sigma/dT$ (see Figure 2). Values T_C corresponding to minimum of derivative $d\sigma/dT$, are also presented in Table. It is seen, that for $Tb_{0.3}Dy_{0.7}Fe_2H_x$, where $x = 0$ and 3, values of Curie temperature are 665 and 294 K respectively. These values are in good agreement with known literature data for such compositions as $Tb_{0.27}Dy_{0.73}Fe_2$ and $Tb_{0.41}Dy_{0.59}Fe_2$ [13,14]. Table shows also change in Curie temperature ΔT_C, equal to 371 K when 3 atoms of hydrogen are introduced into the lattice of initial compound. Thus, $Tb_{0.3}Dy_{0.7}Fe_2H_3$ hydride has temperature of magnetic ordering near room temperature, which may be important from a practical point of view.

Moreover, for $Tb_{0.3}Dy_{0.7}Fe_2H_3$ hydride one clearly expressed minimum is observed in curve $M(T)$ in region of low temperatures (see Figure 1), near $T_k \sim 140$ K. The magnetization decreasing can be explained by compensation of the magnetic moments of rare-earth and iron sublattices in hydride. This phenomenon is not observed in the initial composition $Tb_{0.3}Dy_{0.7}Fe_2$. So, as result of hydrogenation sample with new set of magnetic characteristics can be obtained, varying hydrogen content in samples we can change Curie temperature and magnetic compensation point.

Figures 3 and 4 show the field and temperature dependences of volume magnetostriction for $Tb_{0.3}Dy_{0.7}Fe_2H_3$ hydride, respectively. Figure 3 shows that in region of temperatures below and above magnetic compensation point ($T_k = 140$ K), values of volume magnetostriction are small (do not exceed value $6 \cdot 10^{-6}$). Curves $\omega(H)$ demonstrate linear rise, trend for saturation is absent in magnetic fields below 12 kOe. At the same time, in region of temperatures $T < T_C$ ($T_C = 294$ K) the volume magnetostriction reaches values higher by order of magnitude ($42 \cdot 10^{-6}$), linearly increasing with strength increasing of magnetic field. Figure 4 shows that in region of magnetic compensation point $T_k \sim 140$ K the volume magnetostriction changes sign from positive (at $T > T_k$) to negative (at $T < T_k$).

From Figure 4 is also follows that temperature dependence of volume magnetostriction $\omega(T)$ keeps its maximum values in rather wide range of temperatures 260–285 K in external magnetic field 2 kOe. When strength of magnetic field increases to 12 kOe the maximum values are reached in narrower temperature range 265–280 K.

Let us recall that the increase in the unit cell volume during hydrogenation can be considered, in the first approximation, as a process inverse to the decrease in volume V under action of hydrostatic pressure, i.e. as „negative“ pressure. It is known that compressibility is determined by equation

$$\mathscr{N} = \frac{\Delta V/V}{p}. \tag{2}$$

So, we can calculate shift of Curie temperature (T_C) under action of the hydrostatic pressure using the following ratio associated with shift T_C upon change in unit cell volume ΔV:

$$\frac{d\ln T_C}{dp} = -\frac{\mathscr{N}}{T_C}\frac{dT_C}{d\ln V} = -\mathscr{N}\frac{d\ln T_C}{d\ln V}. \tag{3}$$

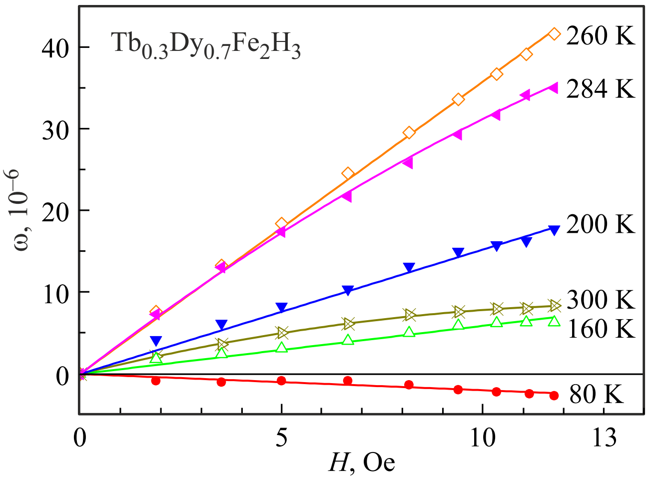

Figure 3. Field dependences of volume magnetostriction of $Tb_{0.3}Dy_{0.7}Fe_2H_3$ hydride at different temperatures 80, 160, 200, 260, 284 and 300 K.

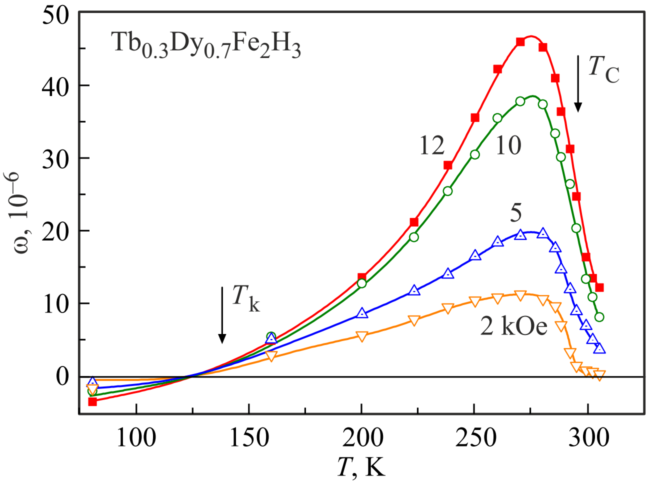

Figure 4. Temperature dependences of volume magnetostriction of $Tb_{0.3}Dy_{0.7}Fe_2H_3$ hydride in different fields 2, 5, 10 and 12 kOe.

Unit cell volumes of initial compound $Tb_{0.3}Dy_{0.7}Fe_2$ and its $Tb_{0.3}Dy_{0.7}Fe_2H_3$ hydride differ by value

$$\frac{\Delta V}{V} = \frac{V_{Tb_{0.3}Dy_{0.7}Fe_2H_3} - V_{Tb_{0.3}Dy_{0.7}Fe_2}}{V_{Tb_{0.3}Dy_{0.7}Fe_2}} = 0.202. \tag{4}$$

Shift in Curie temperature upon change in unit cell volume is determined by equation

$$\Delta T_C = -\frac{T_C}{\mathscr{N}}\frac{\Delta V}{V}\frac{d\ln T_C}{dp}. \tag{5}$$

Then value $d\ln T_C/dp$ can be expressed as:

$$\frac{d\ln T_C}{dp} = \Delta T_C \frac{1}{\Delta V/V}\frac{\mathscr{N}}{T_C}. \tag{6}$$

Let's determine value $d\ln T_C/dp$ based on the fact that we know Curie temperature of $Tb_{0.3}Dy_{0.7}Fe_2H_3$ hydride (see Table), and compressibility $\mathscr{N} = 1.06 \cdot 10^{-3}\,\text{kbar}^{-1}$ ($1.06 \cdot 10^{-9}$ cm/din) [20]:

$$\frac{d \ln T_C}{dp} = 2.9 \cdot 10^{-3}\,\text{kbar}^{-1}. \tag{7}$$

Same value $d \ln T_C/dp$ can be calculated based on the known thermodynamic relations linking such values as volume magnetostriction ω and magnetization M, and assuming that in region of forced magnetization (above Curie temperature) the magnetization is $M = \rho\sigma$ (where ρ — density) is function of reduced temperature (T/T_C) and external magnetic field H

$$\left(\frac{\partial \omega}{\partial H}\right)_p = -\left(\frac{\partial M}{\partial p}\right)_H = T\left(\frac{\partial M}{\partial T}\right)_H \frac{\partial \ln T_C}{\partial p}. \tag{8}$$

Hence, we find

$$\frac{\partial \ln T_C}{\partial p} = \frac{1}{T\left(\frac{\partial M}{\partial T}\right)_H}\left(\frac{\partial \omega}{\partial H}\right)_p. \tag{9}$$

From experimental data of field dependences of volume magnetostriction $\omega(H)$ (see Figure 3) the value of magnetostriction susceptibility was determined

$$\left(\frac{\partial \omega}{\partial H}\right)_p = 3.4\,\text{kOe}^{-1}. \tag{10}$$

Using the experimental data on magnetization the derivative was calculated

$$\left(\frac{\partial M}{\partial T}\right)_H = 2.2\,\text{G/K}$$

(density of Terfenol-D $\rho = 9.25\,\text{g/cm}^3$)

$$\frac{d \ln T_C}{dp} = 3.8 \cdot 10^{-3}\,\text{kbar}^{-1}. \tag{11}$$

Comparing values $d \ln T_C/dp$, obtained with methods using different experimental data, we confirmed that they have the same order of magnitude and close values.

Hence, further special interest was associated with determination of the relative change in resulting exchange integral A for Terfenol-D with change in unit cell volume. Value of such change α can be evaluated by equation

$$\alpha = \frac{d \ln A}{d \ln V} = \frac{d \ln T_C}{d \ln V} = -\frac{1}{\mathscr{N}}\frac{d \ln T_C}{dp}. \tag{12}$$

We found that α is 2.7. Same calculations were made also for compound YFe_2, they give $\alpha = 2.8$. Obtained values for Terfenol-D $Tb_{0.3}Dy_{0.7}Fe_2$ and YFe_2 coincide within accuracy of our calculations. Thus, it was determined that in compounds RFe$_2$ type of rare-earth atom has no noticeable effect on the relative change of resulting exchange integral A with change in unit cell volume. Note that for pure rare-earth metals, such as terbium or gadolinium, value α is lower. For Tb $\alpha = 1.8$, for Gd $\alpha = 2$ [21]. At same time, for the compound with high content of iron Y_2Fe_{17} value α by many times ($\alpha = 12$) exceed values obtained by us for terfenol $Tb_{0.3}Dy_{0.7}Fe_2$ and YFe_2.

The magnetic properties (including Curie temperature) of the listed above materials are determined by a few types of exchange interactions. Thus, for two-sublattice magnetics of type R–Fe (Terfenol-D is such material) the properties are determined by three types of exchange interactions: R–R, R–Fe and Fe–Fe [22]. But in pure REM there is only one type of exchange interaction R–R, this corresponds to minimum value of parameter α [21]. In compounds Y_2Fe_{17} and YFe_2 there also one type of exchange interaction Fe–Fe, as trivalent T^{3+} ion has no noticeable magnetic moment. Nevertheless, value α for these compounds greatly differs (by ~ 4 times). Such difference in rare-earth intermetallic compounds Y_2Fe_{17} and YFe_2 can be associated with ratio Fe/R. Actually, in case of compounds YFe_2 and Y_2Fe_{17} this ratio will be 2 and 8.5 respectively, i. e. difference is 4.25 times and correlates with values α. The compound Y_2Fe_{17} is known as compound, which structure has „dumbbell“, formed by Fe atoms in crystallographic cites $4f$. Spacings between Fe atoms in „dumbbell“ are lower than critical (as per Bette-Sleter curve), and exchange integral for these Fe atoms has negative sign. Taken as a whole, the compound Y_2T_{17} has mixed type of exchange interactions (both negative, and positive) in pairs Fe–Fe depending on their crystallographic cites [23–24]. The mixed type has extraordinary effect on the magnetic properties (remember that Curie temperature of compound Y_2Fe_{17} with high content of Fe slightly exceeds the room temperature, $T_C = 341$ K).

In Curie temperature region the noticeable volume magnetostriction usually occurs due to the sharp dependence of the exchange integral A on distance between the magnetoactive ions (Fe–Fe and R–Fe), and is, in essence, the exchange magnetostriction. Refs. [23,25] have showed that compound Y_2Fe_{17} can demonstrate very high values of exchange magnetostriction, as for it $\alpha = 12$.

Our studies showed that in $Tb_{0.3}Dy_{0.7}Fe_2H_3$ hydride the volume magnetostriction also demonstrates large values near temperature of magnetic phase transition from ferromagnetic into paramagnetic state (Curie temperature), due to high value of $\alpha \sim 3$. In this region of temperatures close to room temperatures the contribution of exchange forced magnetostriction will be prevailing in hydride, while other contributions (from processes of magnetization vector rotation and shift of domain boundaries) are low. Such compositions, where we can vary hydrogen content to obtain new materials with specified set of properties, are rather attractive for application, in particular, in hydrogen energy.

4. Conclusion

The paper evaluates relative change in exchange integral A upon change in unit cell volume of Terfenol-D

$Tb_{0.3}Dy_{0.7}Fe_2$ and YFe_2. The obtained value $\alpha \sim 3$ exceeds magnitudes that are demonstrated by pure rare-earth metals $\alpha \sim 2$, such as, terbium and gadolinium, but is below the value for compound Y_2Fe_{17} $\alpha \sim 12$. In $Tb_{0.3}Dy_{0.7}Fe_2$ the unit cell volume increasing $\Delta V/V$ is observed upon introduction of hydrogen atoms in interstices of crystal structure of Laves phase C15 type and reaches values 20.2% for $Tb_{0.3}Dy_{0.7}Fe_2H_3$ hydride. It was shown that increase in unit cell volume is also possible under effect of the external magnetic field. This is why in the Curie temperature region the noticeable by value volume magnetostriction is observed, which is actually the exchange forced magnetostriction.

It is found that during hydrogenation of the Terfenol-D the shift of Curie temperature T_C is $d\ln T_C/dp = 2.9 \cdot 10^{-3}\,\text{kbar}^{-1}$. In $Tb_{0.3}Dy_{0.7}Fe_2H_3$ hydride the same value obtained from data analysis of volume magnetostriction in region of Curie temperature is $3.8 \cdot 10^{-3}\,\text{kbar}^{-1}$. The close values confirm that the noticeable by value magnetostriction in $Tb_{0.3}Dy_{0.7}Fe_2H_3$ hydride and shift of Curie temperature in the initial compound $Tb_{0.3}Dy_{0.7}Fe_2$ upon introduction of hydrogen atoms are due to the same mechanism, based on the dependence of resulting exchange integral A on unit cell volume, therefore, on interatomic spacings between magnetoactive ions.

Conflict of interest

The authors declare that they have no conflict of interest.

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Translated by I.Mazurov