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Journal of Alloys and Compounds 329 (2001) 31–36

Journal of
ALLOYS
AND COMPOUNDS

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Structural and magnetic properties of $\text{Lu}_2\text{Fe}_{17}\text{H}_x$ ($x=0; 3$) single crystals

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Received 18 April 2001; accepted 18 May 2001

Abstract

The structural and magnetic characterization of $\text{Lu}_2\text{Fe}_{17}\text{H}_x$ ($x=0; 3$) single crystals is presented. The host alloy crystallizes with a disordered variant of hexagonal ($\text{Th}_2\text{Ni}_{17}$ -type $P6_3/mmc$) structure. It is observed that the host alloy symmetry is retained upon hydrogenation. Hydrogen is found to be accommodated in the rare earth-rich octahedral position. The Néel temperature of the hydride $\text{Lu}_2\text{Fe}_{17}\text{H}_3$ is increased by 35 K per hydrogen atom. The magnetization and magnetic anisotropy constant remains practically unaffected in the hydride $\text{Lu}_2\text{Fe}_{17}\text{H}_x$ at $x=3$. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Rare earth–transition metal compounds; Hydrogen absorbing materials; Crystal structure; Anisotropy; Magnetic measurements

1. Introduction

The study of the magnetic properties of hexagonal rare-earth–transition metal compound with the 2:17 stoichiometry has been of interest for many years, because of the excellent permanent magnetic properties displayed by several materials in this group. The crystallographic structure of the R_2Fe_{17} compounds, the magnetization, the magnetocrystalline anisotropy and the magnetostriction have been intensively studied previously [1,2]. The investigations have shown that R_2Fe_{17} compounds can absorb some amounts of hydrogen to form the corresponding hydrides by gas–solid reaction [3]. The hydrogen atoms were found to have a remarkable effect in increasing the Curie temperature and modifying the magnetocrystalline anisotropy (MCA) [4]. It is well known that the most reliable data for the magnetic anisotropy and magnetization can be obtained from measurements of single-crystal samples.

Here we report on the structural and magnetic properties of $\text{Lu}_2\text{Fe}_{17}\text{H}_x$ ($x=0, 3$) single crystals. Note, that we have obtained the hydride without disintegration of the single crystal samples. In this paper, X-ray diffraction spectra of

$\text{Lu}_2\text{Fe}_{17}$ and $\text{Lu}_2\text{Fe}_{17}\text{H}_3$ are given and analyzed in terms of the crystallographic structure and site occupancy of both compounds. The effects of hydrogenation on magnetization, ordering temperature and magnetocrystalline anisotropy are also studied.

2. Experimental

Polycrystalline ingots of $\text{Lu}_2\text{Fe}_{17}$ compounds were prepared by induction melting of appropriate amounts of the constituent metals having a purity better than 99.95 wt.%. The ingots were remelted in an electric resistance furnace with high temperature gradient and cooled slowly through the melting point in order to increase the grain size. The single crystals of $\text{Lu}_2\text{Fe}_{17}$ were extracted from the solidified ingots.

Hydrogenation was performed in a stainless steel autoclave under a hydrogen gas pressure of 1 MPa at 150°C and it led to stable hydrides. The hydrogen uptake was determined by a volumetric method with an accuracy of 0.1 hydrogen atom per formula unit.

The X-ray intensity data were collected in a KUMA-Diffraction KM-4 four-circle single crystal diffractometer equipped with a CCD camera using graphite monochromatized $\text{MoK}\alpha$ radiation ($\lambda=0.71073$ Å). The inten-

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sities of the reflections were corrected for Lorentz and polarization effects. Analytical absorption correction was applied.

The magnetic ordering temperatures of the $\text{Lu}_2\text{Fe}_{17}\text{H}_x$ ($x=0; 3$) compounds were determined from the thermomagnetic analysis at low external fields (300 Oe) in the temperature range 150–500 K. Magnetization measurement along the main crystallographic directions of the hexagonal structure were performed on $\text{R}_2\text{Fe}_{17}\text{H}_x$ ($x=0; 3$) single crystals at high applied magnetic fields up to 100 kOe in the temperature range 4.2–300 K. After correcting for the demagnetizing field, the anisotropy constants K_1 and K_2 were determined by the Sucksmith–Tompson relation.

3. Results

3.1. Crystallographic structure of $\text{Lu}_2\text{Fe}_{17}\text{H}_x$ ($x=0; 3$)

Four samples, one as cast and three hydrogenated, were investigated. The crystal structure of $\text{Lu}_2\text{Fe}_{17}$ and $\text{Lu}_2\text{Fe}_{17}\text{H}_x$ ($x=0; 3$) was solved by the Patterson method [5] and refined by the full matrix least squares method using the SHELX-97 program [6]. Positional parameters of the hydrogen atoms were taken from the data of $\text{Ho}_2\text{Fe}_{17}\text{H}_4$ [7]. Small deviations from 2:17 stoichiometry, with iron excess were observed in both as cast and hydrogenated crystals. The crystal data, data collection and refinement information for two selected crystals are col-

lected in Table 1. Their final atomic positional parameters and anisotropic displacement coefficients are given in Tables 2 and 3, respectively.

Both, $\text{Lu}_2\text{Fe}_{17}$ and $\text{Lu}_2\text{Fe}_{17}\text{H}_x$ ($x=0; 3$) are isostructural with $\text{Y}_2\text{Fe}_{17.3}$ [8], a disordered variant of the hexagonal $\text{Th}_2\text{Ni}_{17}$ type of structure (space group $P6_3/mmc$) (Fig. 1) [9]. The Lu_3 , Fe_3 and Fe_5 atoms fully occupy the $2d$, $6g$ and $12k$ positions, respectively and create the ordered framework of the structure (Fig. 1). The dumbbells of Fe_1 at $4e$ (occupation factor 0.19–0.275) and Fe_2 (occupation factor 0.94–0.96) at $4f$ positions are formed around Lu_1 at $2b$ and Lu_2 at $2c$, respectively (Fig. 2a). The Fe_{41} and Fe_{42} atoms occupy, split in two, the $12j$ site in a ratio 0.83–0.86 to 0.17–0.13. Fig. 2b shows two possible configurations of the $z = 1/4$ plane in the $\text{Lu}_{1.85}\text{Fe}_{17.46}$ structure.

Hydrogenation of $\text{Lu}_2\text{Fe}_{17}$ causes an increasing of the unit cell volume and small changes of the interatomic distances. However it does not introduce significant changes in the crystal structure. A list of selected average interatomic distances is given in Table 4.

3.2. Magnetic properties of $\text{Lu}_2\text{Fe}_{17}\text{H}_x$ ($x=0; 3$) single crystals

The magnetization curves were measured in a magnetic field of 300 Oe between 150 and 450 K on a $\text{Lu}_2\text{Fe}_{17}$ single crystal and its hydride (see Fig. 3). At 267 and 372 K the temperature dependence of magnetization shows a peak characteristic of a Néel temperature for $\text{Lu}_2\text{Fe}_{17}$ and $\text{Lu}_2\text{Fe}_{17}\text{H}_3$, respectively. Our results for the host com-

Table 1
Crystal data and structure refinement for R_2Fe_{17} and $\text{R}_2\text{Fe}_{17}\text{H}_3$

Empirical formula	$\text{Lu}_{1.85}\text{Fe}_{17.46}$	$\text{Lu}_{1.87}\text{Fe}_{17.26}\text{H}_3$
Formula weight	1302.41	1307.18
Temperature	293(2) K	293(2) K
Wavelength	0.71073 Å	0.71073 Å
Crystal system	Hexagonal	Hexagonal
Space group	$P6_3/mmc$ (no. 194)	$P6_3/mmc$ (no. 194)
Unit cell dimensions	$a = 8.418(1)$ Å $b = 8.418(1)$ Å $c = 8.295(2)$ Å	$a = 8.509(1)$ Å $b = 8.509(1)$ Å $c = 8.308(2)$ Å
Volume	509.05(15) Å ³	520.94(15) Å ³
Unit per cell	$Z = 2$	$Z = 2$
Density (calculated)	8.525 mg/m ³	8.334 mg/m ³
Absorption coefficient	41.922 mm ⁻¹	41.060 mm ⁻¹
$F(000)$	1178	1181
Crystal size (mm)	0.10 × 0.06 × 0.03	0.16 × 0.04 × 0.02
θ Range for data collection (°)	4.84–46.24	4.79–46.21
Index ranges	$-17 \leq h \leq 13, -10 \leq k \leq 17, -16 \leq l \leq 9$	
Reflections collected	3684	3684
Independent reflections	517 [$R_{\text{int}} = 0.086$]	524 [$R_{\text{int}} = 0.092$]
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	517/0/39	524/0/40
Goodness-of-fit on F^2	1.112	1.224
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.048, wR_2 = 0.09$	$R_1 = 0.042, wR_2 = 0.084$
R indices (all data)	$R_1 = 0.05, wR_2 = 0.10$	$R_1 = 0.05, wR_2 = 0.11$
Extinction coefficient	0.0035(6)	0.0023(5)
Largest diff. peak and hole	4.095 and $-4.631 \text{ e} \cdot \text{Å}^{-3}$	3.385 and $-2.896 \text{ e} \cdot \text{Å}^{-3}$

Table 2

Atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for $\text{Lu}_{1.85}\text{Fe}_{17.46}$ and $\text{Lu}_{1.87}\text{Fe}_{17.26}\text{H}_3$

Atom	Site	Occupancy	X/a	Y/b	Z/c	U_{eq}^a
Lu1	<i>2b</i>	0.765 <i>0.80</i>	0	0	1/4	12(1) <i>10(1)</i>
Lu2	<i>2c</i>	0.09 <i>0.06</i>	1/3	2/3	1/4	7(2) <i>4(2)</i>
Lu3	<i>2d</i>	1 <i>1</i>	1/3	2/3	3/4	8(1) <i>7(1)</i>
Fe1	<i>4e</i>	0.275 <i>0.19</i>	0	0	0.3919(10) <i>0.3939(11)</i>	12(2) <i>4(2)</i>
Fe2	<i>4f</i>	0.91 <i>0.94</i>	1/3	2/3	0.1062(2) <i>0.1053(2)</i>	10(1) <i>8(1)</i>
Fe3	<i>6g</i>	1 <i>1</i>	1/2	0	0	9(1) <i>7(1)</i>
Fe41	<i>12j</i>	0.84 <i>0.87</i>	0.3299(2) <i>0.3307(2)</i>	0.3703(2) <i>0.3777(2)</i>	1/4	12(1) <i>9(1)</i>
Fe42	<i>12j</i>	0.16 <i>0.13</i>	0.2928(10) <i>0.2938(10)</i>	0.3002(10) <i>0.3004(11)</i>	1/4	10(2) <i>13(2)</i>
Fe5	<i>12k</i>	1 <i>1</i>	0.1662(1) <i>0.1669(1)</i>	0.3323(1) <i>0.3339(1)</i>	0.5158(1) <i>0.5159(1)</i>	10(1) <i>8(1)</i>
H	<i>6h</i>		<i>0.823(19)</i>	<i>0.650(30)</i>	<i>1/4</i>	<i>98(9)</i>

Data for hydride in italics.

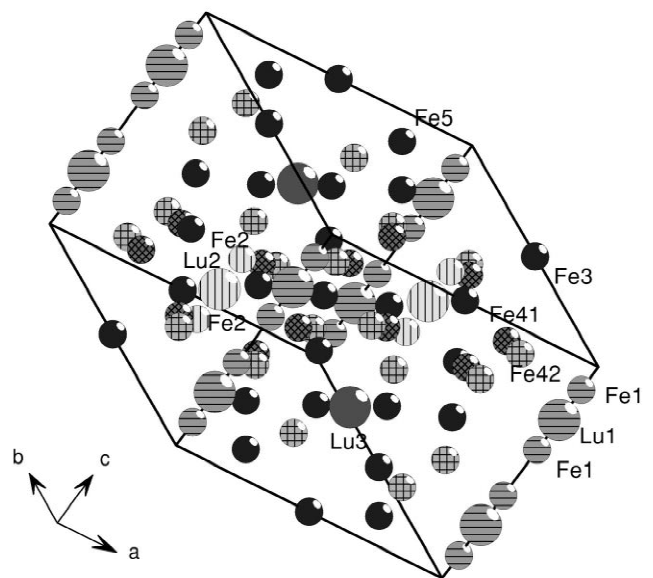
^a U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 3

Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) $\text{Lu}_{1.85}\text{Fe}_{17.46}$ and $\text{Lu}_{1.87}\text{Fe}_{17.26}\text{H}_3^a$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Lu1	9(1) <i>8(1)</i>	9(1) <i>8(1)</i>	14(1) <i>15(1)</i>	0	0	2(1) <i>4(1)</i>
Lu2	3(2) <i>2(1)</i>	3(2) <i>2(1)</i>	13(3) <i>8(3)</i>	0	0	4(1) <i>1(1)</i>
Lu3	8(1) <i>8(1)</i>	8(1) <i>8(1)</i>	10(1) <i>7(1)</i>	0	0	4(1) <i>4(1)</i>
Fe1	14(2) <i>5(2)</i>	14(2) <i>5(2)</i>	10(3) <i>4(3)</i>	0	0	7(1) <i>2(1)</i>
Fe2	11(1) <i>9(1)</i>	11(1) <i>9(1)</i>	9(1) <i>6(1)</i>	0	0	6(1) <i>5(1)</i>
Fe3	7(1) <i>7(1)</i>	7(1) <i>7(1)</i>	12(1) <i>7(1)</i>	1(1) <i>-1(1)</i>	0(1) <i>-1(1)</i>	4(1) <i>4(1)</i>
Fe41	10(1) <i>10(1)</i>	14(1) <i>15(1)</i>	8(1) <i>5(1)</i>	0	0	6(1) <i>9(1)</i>
Fe42	8(3) <i>16(3)</i>	19(3) <i>11(2)</i>	4(3) <i>3(2)</i>	0	0	10(2) <i>12(3)</i>
Fe5	7(1) <i>7(1)</i>	9(1) <i>9(1)</i>	12(1) <i>9(1)</i>	1(1) <i>2(1)</i>	0(1) <i>1(1)</i>	4(1) <i>4(1)</i>

Data for hydride in italics.

^a Anisotropic displacement factor exponent takes the form: $-2 \pi^2 [h^2 a^2 \cdot U_{11} + \dots + 2hka \cdot b \cdot U_{12}]$.Fig. 1. View of the unit cell of $\text{Lu}_{1.85}\text{Fe}_{17.46}$.

compound agree well with data obtained earlier [10,11]. We observed an extremely rapid increase of the ordering temperature of about 105 K (about 35 K per hydrogen atom). The values of the Néel temperatures T_N of $\text{Lu}_2\text{Fe}_{17}\text{H}_x$ ($x=0; 3$) are summarized in Table 5.

It is well known that the R_2Fe_{17} compounds have anomalously low ordering temperature for alloys containing so much iron. The increase of the iron–iron

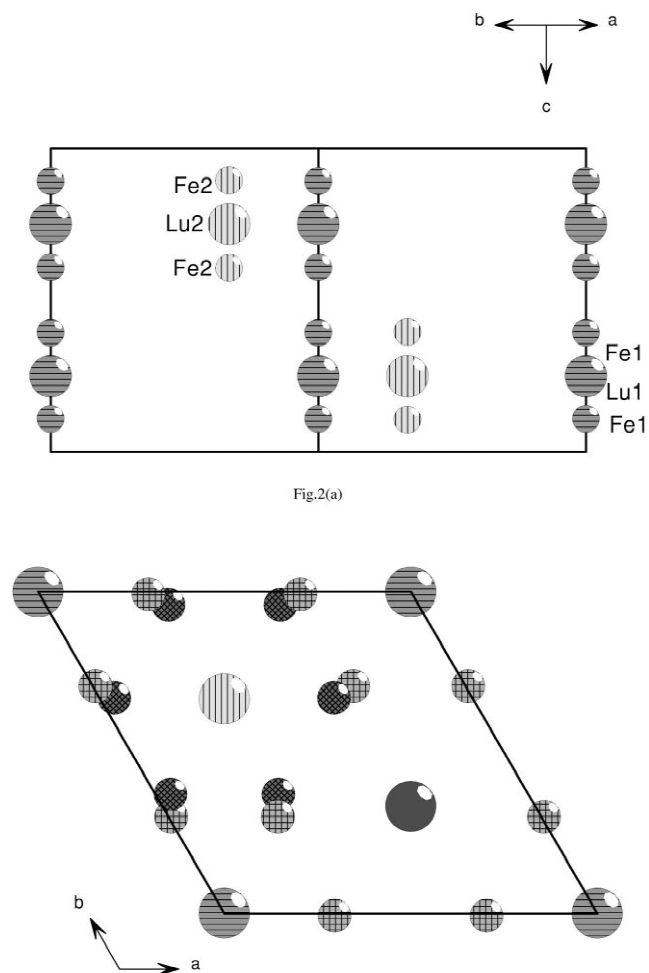


Fig. 2. (a) Projection of the Fe1 and Fe2 dumbbells on the (110) plane. (b) Possible configurations of the $z = 1/4$ plane.

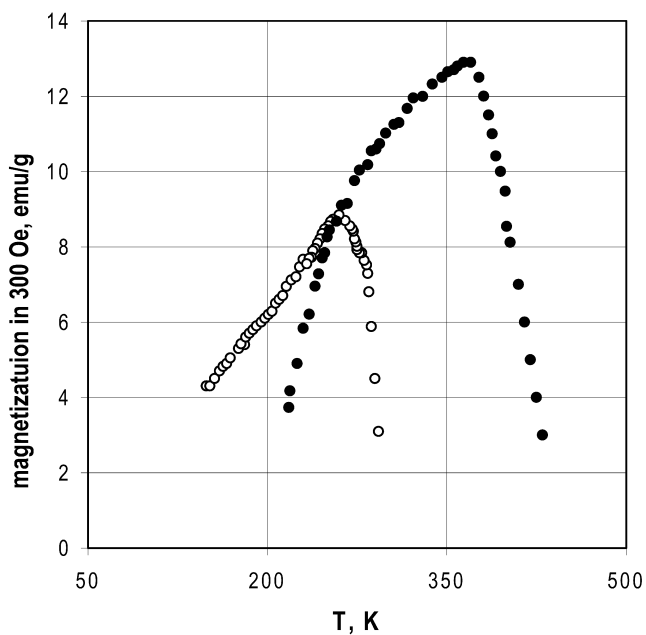


Fig. 3. Temperature dependence of magnetization of $\text{Lu}_2\text{Fe}_{17}$ (open circles) and its hydride (full circles) in a field of 300 Oe.

Table 5
Néel temperature, saturation magnetization, magnetic anisotropy constant K_1 of $\text{LuFe}_{11}\text{Ti}$ and its hydride

Compound	T_N (K)	σ_s (emu/g) $T=4.2$ K	K_1 (10^7 erg/cm 3) $T=4.2$ K
$\text{Lu}_2\text{Fe}_{17}$	267	156.7	-2.3
$\text{Lu}_2\text{Fe}_{17}\text{H}_3$	372	158.8	-2.2

Table 4

Selected interatomic distances (\AA) in $\text{Lu}_{1.85}\text{Fe}_{17.46}$ and $\text{Lu}_{1.87}\text{Fe}_{17.26}\text{H}_3$ ^a

Lu1	Fe1	1.1777(8)	<i>1.195(9)</i>	Fe3	Fe5	2.4373(6)	<i>2.4579(6)</i>
	Fe42	2.497(7)	<i>2.529(11)</i>		Fe41	2.4451(8)	<i>2.4440(8)</i>
Lu2	Fe1	2.971(8)	<i>2.959(11)</i>	Fe41	Fe42	2.690(4)	<i>2.701(7)</i>
	Fe41	2.9622(12)	<i>3.0335(15)</i>		Fe42	0.511(7)	<i>0.569(10)</i>
	Fe2	1.1927(19)	<i>1.201(2)</i>		Fe5	2.5340(13)	<i>2.5377(13)</i>
Lu3	Fe41	2.4803(16)	<i>2.4481(14)</i>	Fe41	Fe41	2.437(3)	<i>2.414(3)</i>
	Fe42	2.929(8)	<i>2.962(11)</i>		Fe42	2.473(7)	<i>2.494(12)</i>
	Fe2	2.955(2)	<i>2.953(2)</i>		Fe41	2.523(2)	<i>2.482(3)</i>
Fe1	Fe42	3.018(8)	<i>3.043(13)</i>	Fe42	Fe5	2.6010(14)	<i>2.6549(15)</i>
	Fe41	3.0025(17)	<i>3.0647(1)</i>		Fe5	2.521(4)	<i>2.536(6)</i>
	Fe1	1.794(16)	<i>1.763(19)</i>		Fe42	2.402(15)	<i>2.44(2)</i>
Fe2	Fe1	2.354(16)	<i>2.391(19)</i>	Fe5	Fe42	2.590(16)	<i>2.61(2)</i>
	Fe5	2.631(3)	<i>2.661(4)</i>		Fe5	2.353(5)	<i>2.362(7)</i>
	Fe5	2.541(3)	<i>2.572(3)</i>		Fe5	2.4366(10)	<i>2.4744(1)</i>
	Fe42	2.760(7)	<i>2.797(11)</i>				
	Fe2	2.385(4)	<i>2.401(4)</i>				
	Fe5	2.6392(13)	<i>2.6514(13)</i>				
	Fe3	2.5849(7)	<i>2.6080(8)</i>				
	Fe41	2.7522(16)	<i>2.7267(18)</i>				

^a In italic.

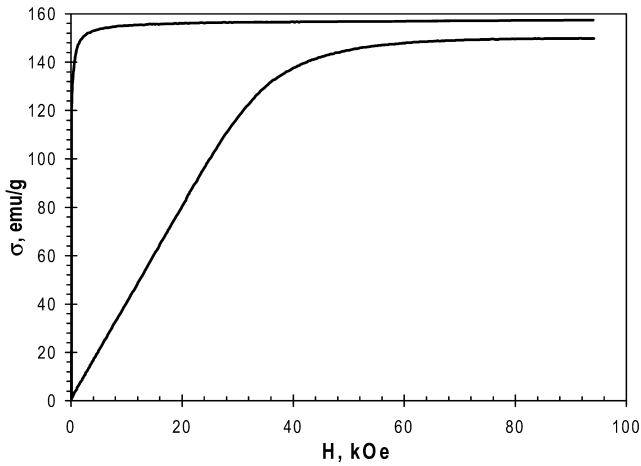


Fig. 4. Magnetization measurements made on $\text{Lu}_2\text{Fe}_{17}$, at 4.2 K. The magnetic field is applied parallel (lower curve) and perpendicular (upper curve) to the c axis.

distances upon hydrogenation lead to enhancement of the exchange interaction and a concomitant rise of the ordering temperature is observed in these compounds [12,13].

In contrast to the substantial change in the magnetic ordering temperature, the magnetization remains practically unaffected by the increasing hydrogen concentration. The magnetization curves obtained at 4.2 K for $\text{Lu}_2\text{Fe}_{17}$ and its hydride, when the magnetic field is applied along or perpendicular to the c axis are shown in Figs. 4 and 5, respectively. The spontaneous magnetization lies in the basal plane in both cases. When the field is applied along the c axis in $\text{Lu}_2\text{Fe}_{17}$ and $\text{Lu}_2\text{Fe}_{17}\text{H}_3$, the dependence of magnetization on the field is linear up to 35 kOe and then tends to saturate. However, the saturation value remains inferior to that measured in the easy direction; the deduced magnetization anisotropy is 4% of the spontaneous magnetization.

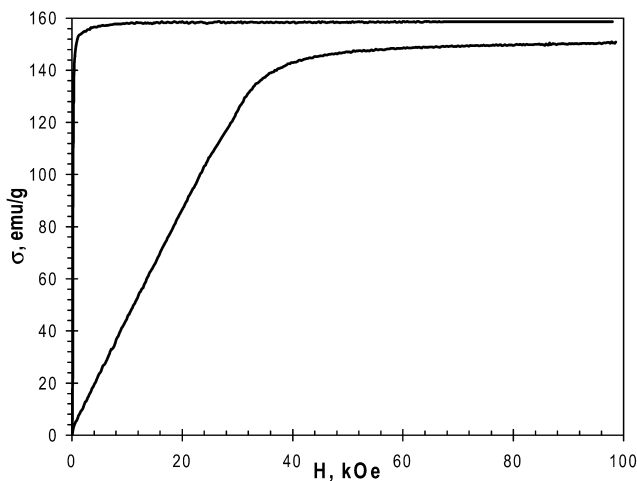


Fig. 5. Magnetization measurements made on $\text{Lu}_2\text{Fe}_{17}\text{H}_3$ at 4.2 K. The magnetic field is applied parallel (lower curve) and perpendicular (upper curve) to the c axis.

For a quantitative analysis we used the Sucksmith–Tompson relation. The fit of the magnetization curve leads to the values of the uniaxial anisotropy constant at 4.2 K, $K_1 = -2.3 \times 10^7 \text{ erg/cm}^3$ for $\text{Lu}_2\text{Fe}_{17}$ and $K_1 = -2.2 \times 10^7 \text{ erg/cm}^3$ for $\text{Lu}_2\text{Fe}_{17}\text{H}_3$. K_2 appears to be negligible compared to K_1 . The hydrogen occupies octahedral positions for $x \leq 3$. Therefore, it is supposed that the hydrogen at the octahedral positions do not influence the magneto-crystalline anisotropy of the 3d sublattice in R_2Fe_{17} .

4. Discussion

Our experimental results show that the magnetic ordering temperature increases strongly upon hydrogenation (see Fig. 3 and Table 5). In order to clarify the physical mechanism responsible for this effect we will use the dependence of the change of the temperature of magnetic ordering ΔT_N under pressure. The value of $d \ln T_N / dp$ in $\text{Lu}_2\text{Fe}_{17}$ have been determined from the change of the maximum of magnetic susceptibility under pressure in a weak magnetic field, $d \ln T_N / dp = -8.4 \cdot 10^{-3} \text{ kbar}^{-1}$ [14]. Besides our value of $d \ln T_N / dp$ have been calculated from the paraprocess magnetostriction in a magnetic field $H = 10 \text{ kOe}$ and it is equal to $6.75 \cdot 10^{-3} \text{ kbar}^{-1}$ [15].

It is well known that the $d \ln T_N / dp$ value includes information about the dependence of the exchange integral A on unit cell volume V

$$\frac{d \ln T_N}{dp} = -\chi \frac{d \ln T_N}{d \ln V} = -\chi \frac{d \ln A}{d \ln V} \quad (1)$$

The change of T_N connected with the change of atomic volume in $\text{Lu}_2\text{Fe}_{17}$ upon hydrogenation was calculated using the relative increase of the unit cell volume $\Delta V/V$. Table 1 shows the values of the unit cell volume $V = 509.05$ and 520.94 \AA^3 for $\text{Lu}_2\text{Fe}_{17}$ and $\text{Lu}_2\text{Fe}_{17}\text{H}_3$, respectively. Hence, we can immediately obtain the unit cell volume expansion upon hydrogenation $\Delta V/V = 2.33 \cdot 10^{-2}$. Assuming that increase of T_N upon hydrogenation originates only from the increase of the exchange integrals due to the expansion of the unit cell volume, the value of ΔT_N can be found from the equation

$$\Delta T_N = -\frac{T_N}{\chi} \frac{\Delta V}{V} \frac{d \ln T_N}{dp} \quad (2)$$

where χ is the compressibility. By using the experimental data for the values of $\Delta V/V$ and $d \ln T_N / dp$ the increase of T_N was determined as $\Delta T_N = 56 \text{ K}$ from the data of [14] and $\Delta T_N = 45 \text{ K}$ from the data of [15]. We take the value of the compressibility χ equal to that in the similar Y_2Fe_{17} compound, $\chi = 1.05 \cdot 10^{-3} \text{ kbar}^{-1}$ [16].

It can be seen that the experimental value of ΔT_N is 105 K and it is higher than the calculated values. This result is explained by the fact that the insertion of hydrogen atoms into the $\text{Lu}_2\text{Fe}_{17}$ crystal lattice leads not only to an

isotropic expansion of the crystal lattice, but also to a perturbation of the exchange interactions along certain interatomic distances. This is a consequence of a change in local anisotropy due to the occupancy of hydrogen atoms which prefer to occupy predominantly the octahedron positions for hydrogen concentrations $x \leq 3$.

This conclusion is consistent with the crystallographic data in Table 1. It can be seen from the experimental results that the interatomic distances for the different atomic pairs (Fe–Fe, Fe–Lu) do not change equally upon hydrogenation, i.e. the expansion of the crystalline lattice is not isotropic along the different crystallographic directions.

The strength of the exchange interactions between the iron atoms is very sensitive to the bonding distances. Although the Fe–Fe exchange is positive for $\text{Lu}_2\text{Fe}_{17}$, there are very short interatomic distance between the Fe–Fe atoms in the $4f$ sites which form a dumbbell, perpendicular to a hexagon of nearest neighbor Fe atoms in the $12j$ sites and the exchange interactions are negative for the $4f$ site Fe atoms [17]. According to the theory [17] the direct electron hopping between the $4f$ site Fe atoms is giving the main contribution to the two-ion-exchange magnetostriction. Thus it can be assumed that hydrogenation not only expands the unit cell but also modifies the two-ion exchange interactions in the dumbbell of the $4f$ site Fe atoms.

5. Conclusion

X-ray diffraction and bulk magnetic measurements were applied in order to elucidate the influence of hydrogen on the structural and magnetic properties of the ternary hydride $\text{Lu}_2\text{Fe}_{17}\text{H}_x$ ($x = 0; 3$). The hydrogenation results in an expansion of the crystal lattice and in a modification of the exchange interactions. The expansion of the crystal lattice is not isotropic along the different crystallographic directions. The modification of the exchange interactions is related in turn to the change of bonding distances which is not uniform. As concerns the magnetic properties, the hydrogenation increases the magnetic transition tempera-

ture of $\text{Lu}_2\text{Fe}_{17}$ by 35 K/H atom, whereas other magnetic characteristics remain practically unaffected for $x \leq 3$.

Acknowledgements

We are indebted to K.P. Skokov for the preparation of the single crystals. The work has been supported by RFBR Grant no. 99-02-17821.

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