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Magnetic and structural properties of Lu₂(Fe, Mn)₁₇H_y hydrides

W Iwasieczko¹, A G Kuchin^{2,3} and H Drulis¹

¹ Institute of Low Temperature and Structure Research, PAS, 1410, PL-59-950 Wroclaw 2, Poland

² Institute for Metal Physics, RAS, 18 Kovalevskaya Street, Ekaterinburg 620041, Russia

E-mail: kuchin@imp.uran.ru

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Abstract

The Lu₂Fe_{17-x}Mn_xH_y, x = 0-2, y = 0-3, hydrides with the Th₂Ni₁₇-type crystal structure were prepared and their structural and magnetic parameters were studied. The lattice parameters measured at room temperature increase smoothly with H content. The magnetic measurements indicate that the Lu₂Fe₁₇H_y hydrides (with y up to 1.77) are antiferromagnets at high temperatures and ferromagnets at low temperatures. The Lu₂Fe_{16.5}Mn_{0.5} compound also has a high temperature antiferromagnetic state, but its hydrides are ferromagnets only. The Lu₂Fe_{17-x}Mn_xH_y hydrides, with x = 0.7 and 2, are ferromagnets with T_c monotonically increasing with H content. The saturation magnetization measured for Lu₂Fe_{17-x}Mn_xH_y, (x = 0, 0.5, 0.7, 2 and 0 < y < 3) hydrides is practically insensitive to hydrogen concentration changes.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

The structural and magnetic properties of the $Lu_2Fe_{17-x}Mn_x$ (x = 0-2) system have been extensively studied recently [1]. These compounds crystallize with a hexagonal Th₂Ni₁₇-type crystal structure. The Lu₂Fe₁₇ binary compound is a collinear ferromagnet below $\Theta_{T} = 135$ K and a helical antiferromagnet in the temperature range from $\Theta_{\rm T}$ up to the Néel temperature $T_{\rm N} = 278 \text{ K} [1, 2].$ The compounds $Lu_2Fe_{17-x}Mn_x$ with x = 0-0.5 are ferromagnets at low temperatures and antiferromagnets at high temperatures. They are only ferromagnets with an almost constant value of the Curie temperature, $T_{\rm C}(x)$, when $x \ge 0.7-2$. The temperature $\Theta_{\rm T}$ of the 'ferromagnet–antiferromagnet' phase transition for x = 0– 0.5 rises fast from 135 to 253 K with Mn content, whereas the Néel temperature is practically unchanged (\sim 278 K) and close to $T_{\rm C}(0.7) = 287$ K. The saturation magnetization of the $Lu_2Fe_{17-x}Mn_x$ compounds decreases with the increase in Mn concentration. This is explained by the antiparallel coupling of both Fe and Mn magnetic moments against each other [1].

The increase of $T_{\rm C}$ with Mn content increase in the Lu₂Fe_{17-x}Mn_x (x = 0-1) system is unexpected because

in the other $R_2Fe_{17-x}Mn_x$ compounds the ferromagnetic temperature, $T_{\rm C}$, decreases or is almost constant, at least for x = 0-1 (see [1]). The lattice constants a and c in the $Lu_2Fe_{17-x}Mn_x$ systems continue to rise with Mn concentration [1]. It was found that hydrogenation of the $Lu_2Fe_{17-x}Si_xH_v$ [3], $Ce_2Fe_{17}H_v$ [4] and $Ce_2Fe_{17-x}Mn_xH_y$ [5–7] systems, when the y index is in the range of $y \leq 2$, causes the $T_{\rm C}$ to increase, leaving the lattice parameter c unchanged, or even slightly diminished. This is somewhat unusual because the R_2Fe_{17} compounds were observed to have a linear relationship between the $T_{\rm C}$ and lattice parameters, as a rule. Keeping this in mind, we decided to perform magnetic and crystallographic investigations to establish the relationship between the lattice parameters and the Curie temperature in the hydrogenated $Lu_2Fe_{17-x}Mn_x$ alloys. We have taken this opportunity to also draw attention to the situations of the ferromagnetic state in the Lu₂Fe₁₇H_v hydride with y = 0.8 [3] and the antiferromagnetic ordering for the hydride y = 3 [8]. It is important to understand this because ferromagnetism of $R_2Fe_{17}H_{\nu}$ usually gets stronger when hydrogen content in these alloys increases [4]. In this report, the structural and magnetic properties of $Lu_2Fe_{17-x}Mn_xH_y$, x = 0-2, 0 < y < 03, hydrides are investigated for the first time.

³ Author to whom any correspondence should be addressed.

x	у	a (Å)	c (Å)	V (Å ³)	$\Theta_{\mathrm{T}}~(\mathrm{K})^{\mathrm{a}}$	T _N (K) ^a	<i>T</i> _C (K)	$M_{ m sat.} \ (\mu_{ m B}/{ m f.u.})^{ m b}$
0	0	8.374	8.266	501.986	153	276	_	33.9
	0.5	8.405	8.271	506.016	177	279	_	33.5
	0.73	8.402	8.279	506.144	196	280	_	34.7
	0.96	8.429	8.344	513.401	206	280	_	34.4
	1.77	8.468	8.296	515.183	236	287	—	34.8
0.5	0	8.379	8.264	502.464	249	276	_	31.8
	0.15	8.382	8.267	503.007			271	36.3
	0.39	8.385	8.299	505.315			284	33.8
	0.71	8.412	8.285	507.717	_	_	285	31.8
0.7	0	8.387	8.265	503.485	_	_	287	30.6
	0.74	8.425	8.294	509.841			295	31.6
	2.72	8.463	8.295	514.512	_	_	428	30.3
2.0	0	8.387	8.271	503.851			292	26.8
	0.56	8.4	8.269	505.292			303	27.9
	1.09	8.419	8.289	508.808		_	332	26.2
	1.86	8.438	8.298	511.662	_	_	359	27.6

Table 1. Structural and magnetic properties of $Lu_2Fe_{17-x}Mn_xH_y$ hydrides.

^a $\Theta_{\rm T}$ and $T_{\rm N}$ for Lu₂Fe₁₇H_y have been measured during sample heating [9].

^b $M_{\rm sat.}$ at 1.8 K.

2. Experimental details

The studied Lu₂Fe_{17-x}Mn_x compounds (x = 0, 0.2, 0.5, 0.7, 2) were prepared by arc melting from starting materials. The ingots were homogenized at 1450 K for 9 h and then quenched in water. The x-ray diffraction measurements were carried out with a DRON diffractometer using Fe K α radiation. The Lu₂Fe_{17-x}Mn_xH_y hydride samples, with y = 0-3, were prepared in a volumetric apparatus through the direct absorption of hydrogen by metallic samples at 423 K. The hydrogen content y in the samples was determined with an accuracy of ± 0.02 by measurements of the hydrogen pressure drop in a calibrated apparatus volume. According to x-ray analysis, all the Lu₂Fe_{17-x}Mn_x compounds (x = 0-2) and their hydrides have a hexagonal crystal structure, which to a first approximation can be attributed to the Th₂Ni₁₇ type. The lattice parameters were calculated using the DICVOL program.

Magnetization measurements were performed with a SQUID magnetometer. The magnetization curves M(H) were measured on free powder in a field up to 55 kOe at T = 1.8 K. The saturation magnetization $M_{\text{sat.}}$ was determined by extrapolation of experimental dependences M(1/H) to $1/H \rightarrow 0$. Temperature dependences of magnetization M(T) in a field of 100 Oe and in the temperature range of (2–400) K were studied on fixed powders or polycrystalline samples. The temperatures of the magnetic phase transitions T_{N} , T_{C} and Θ_{T} were determined from the M(T) dependences.

3. Experimental results

The structural data for the initial non-hydrogenated Lu₂Fe_{17-x} Mn_x (x = 0-0.7) alloys have already been published [1, 9]. In table 1, the lattice parameters for the hydrogenated samples of Lu₂Fe_{17-x}Mn_xH_y (x = 0-0.7, y = 0-3) at room temperature are presented. As one can see, the crystal lattice unit is enhanced in the basal plane and elongated along



Figure 1. Temperature dependences of the magnetization of the Lu₂Fe_{16.5}Mn_{0.5} (x, O), Lu₂Fe_{16.5}Mn_{0.5}H_{0.15} (\Box) and Lu₂Fe_{16.5}Mn_{0.5}H_{0.39} (Δ) alloys measured in a field of 100 Oe. The vertical arrows indicate the $\Theta_{\rm T}$, $T_{\rm C}$ and $T_{\rm N}$ values.

the *c* axis after hydrogenation. These results are different when compared with those reported for Lu₂(Fe, Si)₁₇H_y [3], Ce₂Fe₁₇H_y [4] and Ce₂(Fe, Mn)₁₇H_y [5–7]. When $y \leq$ 2, the crystal lattice expansion has been observed only in the basal plane, whereas a small shrinkage along the *c* axis was observed with a hydrogen concentration. This effect is likely to be connected with the magnetostriction contribution to the lattice parameter magnitudes in the magnetically ordered Lu₂Fe_{17-x}Mn_xH_y compounds, similar to the initial Lu₂Fe_{17-x}Mn_x compounds [1]. It is worth stressing that a strong invar effect in the R₂Fe₁₇ system is observed [10].

In figure 1, the temperature dependence of the magnetization *M* for three Lu₂Fe_{16.5}Mn_{0.5}H_y(y = 0, 0.15, 0.39) samples taken in a field of 100 Oe are presented. The local



Figure 2. Temperature dependences of the magnetization in the Lu₂Fe₁₇ in various magnetic fields: H = 0.1 (1), 0.6 (2), 1 (3), 1.4 (4), 1.8 (5), 2.2 (6), 2.6 (7) and 3 kOe (8). The arrows indicate the $\Theta_{\rm T}$ and $T_{\rm N}$ values.

maxima on the M(T) curve of the initial Lu₂Fe_{16.5}Mn_{0.5} alloy describes the Néel temperature T_N ; the kink on the magnetization curve at temperature $\boldsymbol{\Theta}_T$ reflects the ferromagneticantiferromagnetic phase transition or para-ferromagnetic transition at Curie temperature, $T_{\rm C}$, in the hydride samples. From the figure, it appears that hydrogen destroys the antiferromagnetic ordering in Lu₂Fe_{16.5}Mn_{0.5} observed at high temperatures. As a result, the Lu₂Fe_{16.5}Mn_{0.5}H_{ν}(y = 0.15, 0.39, 0.71) hydrides are ferromagnets in the whole temperature range from room temperature to the lowest temperatures. The visible small bump on the M(T) curve in the Lu₂Fe_{16.5}Mn_{0.5}H_{0.39} sample at high temperatures is likely due to the presence of a small quantity of a second impurity phase. Similar dependences have been registered for all Lu₂Fe_{17-x}Mn_xH_y (x = 0-2, 0 < y < 03) hydride samples. The temperatures of all the magnetic phase transitions in the studied $Lu_2Fe_{17-x}Mn_xH_y$ (x = 0.5, 0.7, 2 and 0 < y < 3) hydride samples are presented in table 1.

The $T_{\rm C}(y)$ values of ferromagnetic Lu₂Fe_{17-x}Mn_xH_y hydrides increase with hydrogen content. However, for a small H concentration of up to H/f.u. = 1, the magnetic transition temperatures are very close to the Néel temperature $T_{\rm N}$ of non-hydrogenated alloys. In particular, $T_{\rm N}(0)$ practically coincides with $T_{\rm C}(y)$ for Lu₂Fe_{16.5}Mn_{0.5}H_y hydrides. From this point of view, the Lu₂Fe_{16.5}Mn_{0.5}H_y is in line with the results obtained for the Ce₂Fe₁₇H_y [4] and Ce₂Fe_{17-x}Mn_xH_y hydrides [5, 6].

For several samples of Lu₂Fe_{17-x}Mn_xH_y, the magnetization, M(T), measurements in different magnetic fields have been carried out. In figures 2–4, the magnetization of M(T)for Lu₂Fe₁₇, Lu₂Fe_{16.5}Mn_{0.5} and Lu₂Fe₁₇H_{0.5} are presented. It is seen that both the kink at Θ_T and the maximum at T_N are smeared out in a magnetic field, whereas T_N completely disappears, beginning with some critical value of the magnetic field above which the induced ferromagnetism exists.

In comparing figures 1 and 3, it is obvious that the $Lu_2Fe_{16.5}Mn_{0.5}$ alloy can be transferred to the ferromagnetic state in two different ways: (i) using a magnetic field of 0.7 kOe



Figure 3. Temperature dependences of the magnetization in the Lu₂Fe_{16.5}Mn_{0.5} in various magnetic fields: H = 0.1 (1), 0.2 (2), 0.3 (3), 0.4 (4), 0.5 (5), 0.6 (6) and 0.7 kOe (7). The arrows indicate the Θ_{T} and T_{N} values.



Figure 4. Temperature dependences of the magnetization in the Lu₂Fe₁₇H_{0.5} in various magnetic fields: H = 0.2 (1), 0.4 (2), 0.6 (3), 0.8 (4), 1 (5), 1.4 (6), 1.8 (7), 2.2 (8) and 2.6 (9) kOe. The arrows indicate the $\Theta_{\rm T}$ and $T_{\rm N}$ values.

or (ii) through the introduction of at least 0.15 H/f.u. into the lattice. The Curie temperatures for both samples are close to each other—266 K and 271 K, respectively. This indicates that the enhancement of the positive exchange interaction energy in the $Lu_2Fe_{16.5}Mn_{0.5}$ lattice by 0.15 hydrogen atoms per formula unit is approximately equivalent to a magnetic energy of the 3d magnetic moments interacting with an external magnetic field of 0.7 kOe.

From figures 2–4, it appears that the Néel temperature $T_{\rm N}$ diminishes, whereas $\Theta_{\rm T}$ increases with the magnetic field increasing. The dependence of $\Theta_{\rm T}$ and $T_{\rm N}$ on the magnetic field *H* is shown in figure 5. The data have been taken from the M(T) curves measured in several magnetic fields. The dashed lines on the diagrams show the extrapolated border



Figure 5. The *H*–*T* magnetic phase diagrams for Lu₂Fe₁₇ (1), Lu₂Fe₁₇H_{0.5} (2), Lu₂Fe₁₇H_{0.73} (3), Lu₂Fe₁₇H_{0.96} (4), Lu₂Fe_{16.8}Mn_{0.2} (5) and Lu₂Fe_{16.5}Mn_{0.5} (6). The thick dotted line describes the extrapolation of the critical values $(\Theta_T)_{\text{crit.}}$ to H = 0.

of the antiferromagnetic state existence in the high magnetic fields. In figure 5, the Néel temperature T_N only for the binary Lu₂Fe₁₇ compound is shown. For the other alloys, the $T_N(H)$ dependence has the same character. In the compounds with relatively high Θ_T , the critical field necessary to induce the ferromagnetic state is much lower. It is understandable that the interaction energy between magnetic field and magnetic moments of 3d ions gains the exchange interaction energy between magnetic state alone.

The critical $(\Theta_T)_{crit.}$ values for Lu₂Fe_{17-x}Mn_xH_y hydrides (see the thick dotted line in figure 5) transform into T_N when H = 0. This result clearly indicates that the exchange interactions in the field inducing the ferromagnetic state have an energy value comparable with those measured in the initial spontaneous antiferromagnetic state.

The dependence of $\Theta_{T}(H)$ is somewhat different for various alloys (see figure 5). For Lu₂Fe₁₇, the slope of $\Theta_{T}(H)$ is the largest, whereas for the Lu₂Fe_{17-x}Mn_xH_y hydrides the change of Θ_{T} (with *H*) decreases as Θ_{T} becomes larger.

In figure 6, the field dependence of the magnetization M(H) for the powder samples of Lu₂Fe_{16.5}Mn_{0.5}H_y (y = 0–0.71 at T = 1.8 K) is presented. All M(H) curves at 1.8 K for Lu₂Fe_{17-x}Mn_xH_y (x = 0–2, y = 0–3) have the same shape typical for ferromagnetic substances.

The value of the saturation magnetic moment $M_{\text{sat.}}$ at 1.8 K for Lu₂Fe_{17-x}Mn_xH_y (x = 0-2, y = 0-3) as a function of hydrogen concentration is shown in table 1. It can be seen that $M_{\text{sat.}}(y)$ is practically constant with H/f.u. content increasing when x = 0, 0.7, 2.

4. Discussion

The magnetic properties of $Lu_2Fe_{17}H_y$ hydrides found in our research are essentially different than those published



H(kOe)

Figure 6. Demagnetization curves of the Lu₂Fe_{16.5}Mn_{0.5} (O), Lu₂Fe_{16.5}Mn_{0.5}H_{0.15} (Δ), Lu₂Fe_{16.5}Mn_{0.5}H_{0.39} (\Box) and Lu₂Fe_{16.5}Mn_{0.5}H_{0.71} (×) compounds measured on free powder samples at T = 1.8 K.

According to their study, the high temperature in [3]. antiferromagnetic state disappears in binary Lu₂Fe₁₇H_v hydride at y = 0.8. Our results, however, are in accordance with the data reported in [8], where it is stated that the high temperature antiferromagnet state with $T_{\rm N} = 372$ K survives up to y = 3. We think that the different results on the magnetic properties of Lu₂Fe₁₇H_v hydrides reported are due to the different magnetic and structural properties of the initial binary Lu_2Fe_{17} compounds [1, 3, 8]. For example, the lattice parameters of Lu_2Fe_{17} studied in [1] (see table 1) are smaller than reported in [3] (a = 8.3946(8) Å, c = 8.2926(9) Å, $V = 506.08(12) \text{ Å}^3$). These in turn are smaller than those found in [8] $(a = 8.418 \text{ Å}, c = 8.295 \text{ Å}, V = 509.05 \text{ Å}^3)$. In [1] and [3], the $\Theta_{\rm T}$ and $T_{\rm N}$ values for Lu₂Fe₁₇ are practically the same, and $T_{\rm N}$ = 276 K is somewhat larger than reported in [8] where $T_{\rm N} = 267$ K.

The temperature hysteresis of M for Lu₂Fe₁₇ in [3] is about two times wider than in [1]. This type of hysteresis loop is observed only in Lu₂Fe₁₇, among the other R₂Fe₁₇ compounds. Both magnetic phases (antiferromagnetic and ferromagnetic) coexist over the temperature range 130-165 K [2]. This is probably due to some non-homogeneous distribution of magnetic ions in the sample, which has been confirmed by neutron diffraction studies [2, 8, 11]. It has been clearly shown that the crystal structure of Lu₂Fe₁₇ is not of the ideal Th₂Ni₁₇ structure type but it is a disordered structure as proposed by Givord et al [12]. The equilibrium composition of the compound is shifted from the 2:17 stoichiometry to approximately 2:19, and the real structure of the compound, usually denoted as 'Lu₂Fe₁₇', differs in detail from the Th₂Ni₁₇ structure type due to this composition shift [2, 8, 11-13]. The determined stoichiometry of the sample studied in the current paper is close to 2:19. The shift of the composition from the stoichiometry ratio 2:17 in the direction of the 2:19 ratio is very characteristic for the R₂Fe₁₇ compound with R elements from the end of the lanthanide row and for the Y. The composition ratio 2:17 and the Th₂Zn₁₇-type structure is preferred in the iron compounds with light R lanthanide elements [7, 14]. In an ideal unit cell of the Th₂Ni₁₇ type, Lu atoms should occupy two different sites, whereas Fe atoms are located in the four nonequivalent positions including 'dumbbell' 4f sites. Unfortunately, the Th₂Ni₁₇ structure with a ratio of 2:17 appears to be unstable for iron compounds with a heavy R. Part of the Fe atoms in the dumbbell positions are substituted by R atoms, whereas some dumbbell Fe pairs occupy the vacant 4e positions. Simultaneously, the 12j (Fe) and 12k (Fe) positions are split for other nonequivalent positions [8, 12, 13]. The disorder degree in the Lu and Fe-Fe pair locations depends on the initial elements' ratio and the synthesis conditions. Finally, the composition ratio 2:19 appears to be more stable. This atomic disorder and the presence of defects are likely to be the origin of different magnetic ($\Theta_{\rm T}$ and $T_{\rm N}$) results reported for Lu₂Fe₁₇ and its hydride system, as was mentioned at the beginning of this section.

The observed strong enhancement of $T_{\rm C}(y)$ of the ferromagnetic Lu₂Fe_{17-x}Mn_x (x = 0.7, 2) alloys under hydrogen absorption [1] is very similar to that reported for the R₂Fe₁₇H_y [4] and Ce₂Fe_{17-x}Mn_xH_y systems [5, 6]. They are explained as a result of the increase of the crystallographic unit cell volume. The enhancement of $T_{\rm C}$ observed by us in the Lu₂Fe_{17-x}Mn_xH_y (x = 0-2, y = 0-3) samples can also be explained by the increase of the unit cell volume. The straight relationship between $T_{\rm C}$ and the unit cell volume is a well-known phenomenon in R₂Fe₁₇. Every time the distance between 3d ions increases, an increase in their magnetic moments value takes place [10].

An almost constant value of spontaneous magnetization $M_{\text{sat.}}(y)$ is a characteristic behavior for the hydrogenated collinear $R_2Fe_{17}H_y$ ferri- and ferromagnetics [8, 15]. In the case of the helical ferromagnet-like $Ce_2Fe_{17-x}Mn_x$ [5, 6], for instance, the hydrogenation visibly increases the spontaneous magnetization due to a transformation of a non-collinear magnetic structure to a collinear one by a gradual strengthening of the positive magnetic exchange interactions. The constant $M_{\text{sat.}}(y)$ value for $Lu_2Fe_{17-x}Mn_xH_y$ hydrides at 1.8 K indicates that the collinear ferromagnet is indeed a magnetic ground state of this system in the range of x = 0-2 and y = 0-3.

The increase of $\Theta_{T}(H)$ with magnetic field in Lu₂Fe_{17-x} Mn_xH_y hydride samples (see figure 5) is a result of a gradual transformation of the helical antiferromagnetic structure into a collinear type in the manner observed in the Ce₂(Fe, Mn)₁₇H_y hydride system [6, 7, 16]. In this light, a practically constant value of $\Theta_{T}(H)$ found for Lu₂Fe_{16.5}Mn_{0.5} appears as non-typical for those alloys where the 'helical antiferromagnetic–ferromagnetic' phase transition takes place. In these circumstances, we suggest that the helical antiferromagnetic structure in Lu₂Fe_{16.5}Mn_{0.5} is essentially different from that in the Lu₂Fe₁₇ and Lu₂Fe₁₇H_y systems in which more sophisticated magnetic structures are formed. An almost constant $\Theta_{T}(H)$ value in Lu₂Fe_{16.5}Mn_{0.5} suggests that a mutual orientation of 3d magnetic moments is close to be collinear. It seems reasonable to make the neutron diffraction experiments for $Lu_2Fe_{16.5}Mn_{0.5}$ in the temperature range in which the antiferromagnetic state is realized.

5. Conclusions

The hydrogenation process of the $Lu_2Fe_{17-x}Mn_x$ compounds causes an increase in the Curie temperature. The $Lu_2Fe_{16.5}Mn_{0.5}H_y$ (with $y \ge 0.15$) and $Lu_2Fe_{17-x}Mn_xH_y$ (with x = 0.7 and 2) hydrides appear to be ferromagnetic systems. It may be due to the increase of the interatomic distances, which accompanied the hydrogen absorption; the larger the interatomic distances, the more positive the exchange interactions between 3d magnetic moments. We think that the different results of the magnetic properties of the $Lu_2Fe_{17}H_y$ hydrides reported are due to the tendency of the initial binary Lu_2Fe_{17} compound to form the crystallographically not sufficiently homogeneous phases.

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