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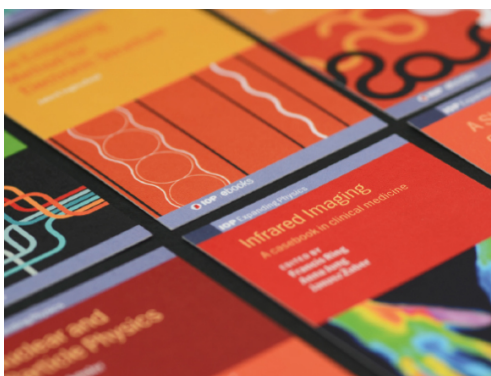
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$(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ single crystal under hydrostatic and ‘negative’ pressure induced by hydrogenation

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Abstract

An experimental study of correlations between the magnetovolume effects and the type of magnetic ordering in the $\text{Lu}_2\text{Fe}_{17}$ -based intermetallics is shown for the example of variation of the interatomic distances by means of substitution of Ce for Lu, hydrogenation and subsequent high-pressure investigation of both the initial $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ and a hydride $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ single-crystalline compounds. The magnetization study was carried out under hydrostatic pressures of up to ~ 0.8 GPa. Hydrogenation is found to totally suppress the antiferromagnetic order of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ by favoring domination of the ferromagnetic interactions which increases the magnetic ordering temperature from 247 to 300 K. The application of pressure decreases the Curie temperature and induces the non-collinearity of the magnetic moments in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$, confirming the strong influence of hydrogenation on exchange coupling.

1. Introduction

The interest in the stability of ferromagnetism against volume changes for various Fe-containing compounds has developed in the course of the study and interpretation of magnetoelastic effects in classic invar systems, Fe–Ni. In Fe-rich rare-earth (R) intermetallics, R_2Fe_{17} , the Fe atoms are located at different distances from each other, occupying four and five non-equivalent crystallographic sites within the rhombohedral or hexagonal unit cell for the light and heavy rare earths, respectively [1]. The resulting magnetic state is defined by the dependence of the exchange interaction strength on the Fe–Fe interatomic distances and by the local coordination number of Fe atoms [2].

$\text{Lu}_2\text{Fe}_{17}$ with the smallest R ($\text{Th}_2\text{Ni}_{17}$ type of structure) and $\text{Ce}_2\text{Fe}_{17}$ ($\text{Th}_2\text{Zn}_{17}$ structure) have attracted much attention due to their peculiar crystallographic and magnetic characteristics. (The unit cell volume of $\text{Ce}_2\text{Fe}_{17}$ is $\sim 4\%$ smaller than that expected from the lanthanide series contraction and, therefore, Ce is assumed to be in a mixed-

valence state [3, 4].) Both the $\text{Lu}_2\text{Fe}_{17}$ and $\text{Ce}_2\text{Fe}_{17}$ compounds studied in [5] displayed non-collinear antiferromagnetic (AF) structures with a collinear arrangement of Fe moments within the basal planes below the Néel temperature T_N with a further transformation of the helical structure into the ferromagnetic (F) one below θ_T at lowered temperatures ($T_N = 274$ K and $\theta_T = 150$ K for $\text{Lu}_2\text{Fe}_{17}$ and $T_N = 225$ K and $\theta_T = 90$ K for $\text{Ce}_2\text{Fe}_{17}$). Furthermore in some works $\text{Ce}_2\text{Fe}_{17}$ was shown to exhibit magnetic properties dissimilar from those of $\text{Lu}_2\text{Fe}_{17}$, by having a pure AF ground state [6–8]. The discrepancy in the observed results was likely to be due to the stoichiometry variations in the studied $\text{Ce}_2\text{Fe}_{17}$ compounds [8].

Extreme sensitivity of magnetic properties to the unit cell volume change has been revealed by measurements under hydrostatic (‘positive’) pressure in both $\text{Lu}_2\text{Fe}_{17}$ [9] and $\text{Ce}_2\text{Fe}_{17}$ [10, 11]. A total suppression of the F phase under pressure on account of the appearance of the new incommensurate antiferromagnetic phases was observed at low temperatures. On the contrary, previous magnetic studies on hydrogen-charged $\text{Lu}_2\text{Fe}_{17}$ [12, 13] and $\text{Ce}_2\text{Fe}_{17}$ [4, 14]

(hydrogenation may serve as a ‘negative’ pressure agent indicating an impact of a volume increase on exchange interactions) have shown that hydrogenation results in stabilization of the F phase in the whole range of the magnetically ordered state and AF is suppressed totally or partially depending on the hydrogen content. In general, hydrogenation is a powerful tool for changing both the crystal and electronic structures of compounds by narrowing the electronic bands due to the lattice expansion that decreases the degree of hybridization between the R 5d and Fe 3d states. Hydrogen intrusion can also modify the hybridized band due to the chemical bonding with the atoms contributing to the band [4, 15].

Previously studied substitutions of Y [16] and Zr [17] for Lu in $\text{Lu}_2\text{Fe}_{17}$ were shown to bring down the AF phase by strengthening the ferromagnetic states with considerably higher magnetic ordering temperatures. Nevertheless, a unique case of the antiferromagnetism stabilization in the whole temperature range of magnetic order was found recently in the case of Ce substitution for Lu [18]. The effect was explained by both the lattice c parameter decrease and by an occurrence of 3d–4f hybridization which strongly increased the Sommerfeld coefficient γ in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ as compared to $\text{Lu}_2\text{Fe}_{17}$ [19].

In the present work, the crystallographic and magnetic properties of single crystals of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ and the successfully prepared hydride $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ are discussed. A relevant role of variation of the unit cell volume (and consequently, of the interatomic Fe–Fe distances) on the magnetization and exchange interactions in R_2Fe_{17} compounds is demonstrated in the course of the study under extreme conditions (e.g. high hydrostatic pressure, wide temperature range).

2. Experimental details

A hexagonal $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ single crystal was grown by a modified Czochralski method (the details of sample preparation and characterization can be found elsewhere [19]). Subsequent hydrogenation of a single-crystalline sample with dimensions of 1 mm × 1 mm × 2 mm and of the polycrystalline ingot crushed into 1–2 mm particles was performed by means of direct hydrogen absorption in a vacuum glass apparatus. After a surface activation procedure by annealing the sample at 773 K for 2 h in high-vacuum conditions, the compound was cooled down to room temperature. High purity hydrogen was admitted into the reaction chamber under a pressure of 6.8 kPa and the chamber was heated up to 623 K. The reaction with hydrogen was indicated by a decreased pressure inside the chamber, but the sample was exposed to H_2 for several hours to reach equilibrium. To achieve good homogenization, the product was slowly cooled down (about 4 K h^{−1}) to room temperature. By making use of the p – V – T hydrogen state equation, the hydrogen content in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}$ was determined.

The crystal structure of the parent compound was examined by the high-resolution neutron powder diffractometer D1A operating with a take-off angle of the monochromator of 122° at the Institut Laue–Langevin (Grenoble, France).

The measurements were carried out at a wavelength of $\lambda = 1.911 \text{ \AA}$ selected by the (115) reflection of a germanium monochromator. During the neutron diffraction measurements a cylindrical vanadium sample holder of 7 mm inner diameter was used. The neutron detection was performed with a set of 6° spaced ³He counting tubes. The complete diffraction pattern was obtained by scanning over the whole 2θ range. For the hydride, powder x-ray diffraction (Cu K α radiation, Bragg–Brentano setup) was employed. The diffraction patterns were refined by means of Rietveld analysis using the FullProf/WinPLOTR software [20].

The pressure–temperature–field evolution of magnetization was measured using a miniature Cu–Be piston-cylinder cell designed at the Institute of Physics ASCR [21] for a standard SQUID magnetometer (Quantum Design, Inc.). The pressure values were determined according to a correction procedure described in [21], taking into account the temperature-induced increase of pressure inside the cell at elevated temperatures. The $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ and $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ specimens were measured under hydrostatic pressures of up to $\sim 0.8 \text{ GPa}$ at 5 K (which corresponds to $\sim 1.05 \text{ GPa}$ at room temperature). The samples were fixed in the pressure cell with the easy a -axis parallel to the field direction. All magnetization isotherms presented below were corrected for the demagnetizing field. The Curie temperatures of the ferromagnetic compound were determined using the Belov–Goryaga method [22] (also known as Arrott plots).

3. Results and discussion

3.1. Crystal structure of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_x$ ($x = 0, 0.4$)

Hexagonal crystal structure of the $\text{Th}_2\text{Ni}_{17}$ type was confirmed by powder neutron diffraction for $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ with the lattice parameters $a = 8.417(4) \text{ \AA}$ and $c = 8.268(4) \text{ \AA}$ (see the pattern at the top of figure 1 recorded at 300 K). Traces of α -Fe impurity (about 2%) were found. The refinement procedure with the use of a disordered structure model, i.e. with some of the Lu atoms substituted by Fe atoms in 4e positions as proposed by Givord *et al* [23], has led to a satisfactory fit (Bragg R factor 4.30%, R_F factor 3.38%) and allowed us to determine the atomic positions in the crystal lattice of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ (table 1, the error bars in data determination are shown in parentheses). Attempts to refine Ce/Lu on the 2c (1/3, 1/3, 1/4) site led to a nil occupancy within the experimental error. Therefore, the occupancy of the Fe 4f dumbbell site corresponding to that position was fixed to 100% in the last refinement. Three-split 12j Fe sites produce sufficient coordinations around Lu and Fe dumbbell sites and are usually observed for this type of structure. The real composition of the compound was determined as $(\text{Lu}_{0.76}\text{Ce}_{0.17})_2\text{Fe}_{17.43}$. Due to a slight decrease of the c parameter upon the substitution of Ce for Lu as compared to $\text{Lu}_2\text{Fe}_{17}$ with $a = 8.395 \text{ \AA}$ and $c = 8.294 \text{ \AA}$ [19], it was of interest to calculate the distances between the Fe atoms at different crystallographic sites of the $\text{Th}_2\text{Ni}_{17}$ -type crystal structure (see table 2).

R_2Fe_{17} compounds are known to absorb hydrogen up to 5 atoms per formula unit (at./f.u.) for the compounds with

Table 1. Details of the atomic structure of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ at 300 K derived from the powder neutron diffraction data.

Atom	Site	Atomic coordinates			Occupancy (%)
		x	y	z	
Lu ₁	2b	0	0	1/4	67.3 (3.2)
Ce ₁	2b	0	0	1/4	17.9 (3.2)
Lu ₂	2d	1/3	2/3	3/4	84.7 (1.2)
Ce ₂	2d	1/3	2/3	3/4	15.3 (1.2)
Fe ₁	4f	1/3	2/3	0.1068 (4)	100
Fe ₂	6g	1/2	0	0	100
Fe ₃₍₁₎	12j	0.3308 (10)	-0.0465 (10)	1/4	67.3 (1.2)
Fe ₃₍₂₎	12j	0.2957 (10)	-0.0078 (10)	1/4	11.9 (1.2)
Fe ₃₍₃₎	12j	0.3249 (10)	-0.0256 (10)	1/4	22.8 (1.2)
Fe ₄	12k	0.1659 (4)	0.3318 (8)	-0.0172 (2)	100
Fe ₅	4e	0	0	0.1037 (10)	15.1 (3.2)

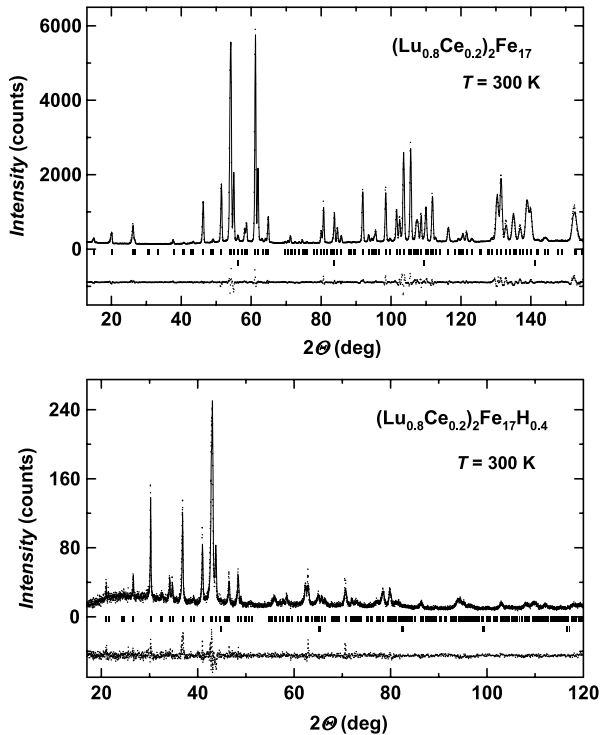


Figure 1. Top: powder neutron diffraction pattern of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ at 300 K (D1A). Bottom: x-ray diffraction pattern of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ hydride at 300 K. Observed (calculated) profiles are given by the dotted (solid) curves, and calculated positions are indicated at the bottom of each figure, including the minor α -Fe contaminant. The difference between the observed and calculated patterns is also shown at the bottom of each figure.

the light rare earths and up to 3 at./f.u. for the heavy rare earths [24]. The composition of the hydride compound in our work was determined as 0.40 ± 0.05 H at./f.u. Despite a higher background noise in the x-ray diffraction data and broadened peaks in the case of the hydrogenated compound, the line widths were well accounted for using the standard resolution functions of the x-ray diffractometer and the set of background points with refinable heights was used in the last refinements (the reliability of obtained results is confirmed by the Bragg R factor of 8.20% and R_F factor of 5.13%). The study has revealed that the pattern of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ corresponds

to that of expanded $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ (see figure 1, bottom). The metal atoms in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ form the same crystal structure as in the parent compound, i.e. the hexagonal $\text{Th}_2\text{Ni}_{17}$ -type with the lattice parameters $a = 8.446$ (4) Å, $c = 8.274$ (2) Å. The relative increase of the unit cell volume due to hydrogenation is $\Delta V/V = 0.76\%$ with the fairly anisotropic lattice expansion occurring predominantly in the basal plane. However, a small increase of the c parameter by about 0.1% was also observed.

Although x-ray diffraction does not provide reliable information regarding the positions of hydrogen atoms due to their small scattering cross-sections, it was established rather ambiguously from the neutron diffraction studies [24] that if the concentration of hydrogen does not exceed 3 at./f.u., the octahedral 6h voids are filled (for reference, the reported atomic coordinates of hydrogen in $\text{Lu}_2\text{Fe}_{17}\text{H}_3$ [12] were $x = 0.823$, $y = 0.650$, $z = 1/4$ with the 98% occupation). By comparing the data on the interatomic distances in the parent $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ and the hydride $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ compounds from tables 2 and 3, respectively, the strongest effect on the interatomic Fe–Fe bonds is observed for the Fe atoms in 12j (denoted as Fe₃) positions. In particular, an increase of 1.65% is found for the Fe₃₍₁₎–Fe₃₍₁₎ bonds, whereas the other distances increase by 0–0.3%. Such data confirm the proposed filling scheme for the octahedral 6h voids and a significant increase of the c parameter (and consequently of the interatomic distances along the c -axis) would be expected in the case of filling the tetrahedral sites by hydrogen atoms in R_2Fe_{17} [25].

3.2. $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$

The evolution of the magnetization isotherms at various pressures for $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ is shown in figure 2 for the characteristic temperatures of 5 and 160 K. In contrast to the ferromagnetic behavior at low temperatures and at ambient pressure of the parent $\text{Lu}_2\text{Fe}_{17}$ [19], $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ demonstrates a behavior typical of the antiferromagnetic compound. The $M(H)$ curves of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ display a hysteretic metamagnetic transition from the antiferromagnetic to the field-induced ferromagnetic state at 5 K. Along with a steep reduction of magnetic moment under pressure, a well-pronounced decrease of the hysteresis width ΔH_c of the

Table 2. Selected interatomic Fe–Fe distances (Å) in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ at 300 K derived from the powder neutron diffraction analysis. The multiplicity of each distance is shown in parentheses. (The last digit for some of the distances is significant.)

Atom	Bond	Distance	Atom	Bond	Distance	Atom	Bond	Distance
Fe ₁	Fe ₁	2.367 (1)	Fe ₃₍₁₎	Fe ₃₍₁₎	2.39 (1)	Fe ₃₍₂₎	Fe ₃₍₂₎	2.42 (1)
	Fe ₂	2.58 (3)		Fe ₃₍₂₎	2.46 (1)		Fe ₃₍₃₎	2.62 (1)
	Fe ₃₍₁₎	2.69 (6)		Fe ₃₍₂₎	2.47 (1)		Fe ₃₍₃₎	2.49(1)
	Fe ₃₍₂₎	3.14 (6)		Fe ₃₍₃₎	2.93 (1)		Fe ₄	2.90 (1)
	Fe ₃₍₃₎	2.88 (6)		Fe ₃₍₃₎	2.46 (1)		Fe ₄	2.53 (2)
	Fe ₄	2.65 (3)		Fe ₄	2.60 (1)		Fe ₅	2.34 (2)
				Fe ₄	2.53 (2)		Fe ₅	2.80 (6)
				Fe ₄	2.62 (2)			
				Fe ₄	2.62 (2)			
				Fe ₅	3.10 (6)			
Fe ₂	Fe ₃₍₁₎	2.429 (4)	Fe ₃₍₃₎	Fe ₃₍₃₎	2.52 (1)	Fe ₄	Fe ₄	2.43 (2)
	Fe ₃₍₂₎	2.67 (4)		Fe ₃₍₃₎	2.73 (1)		Fe ₅	2.62 (3)
	Fe ₃₍₃₎	2.48 (4)		Fe ₄	3.16 (1)		Fe ₅	2.52 (3)
	Fe ₄	2.44 (4)		Fe ₄	2.55 (2)		Fe ₅	2.42 (1)
				Fe ₄	2.49 (2)			
		Fe ₅	3.10 (6)					

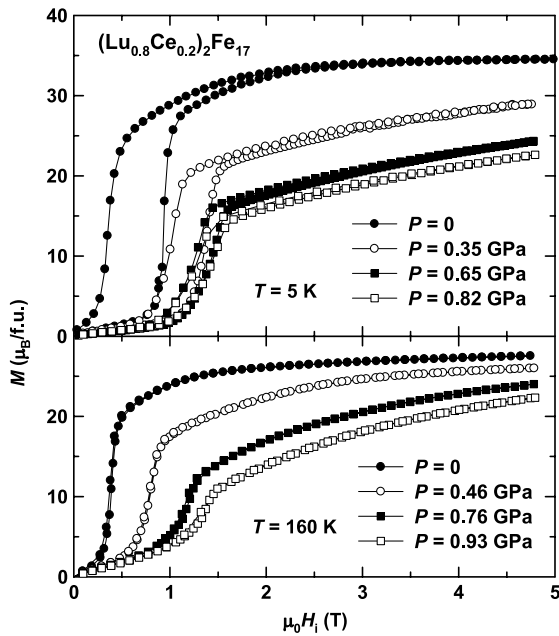


Figure 2. Magnetization isotherms at 5 and 160 K measured along the easy *a*-axis of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ single crystal at various pressures.

metamagnetic AF–F transition is observed above 0.35 GPa, i.e. $\mu_0\Delta H_c$ falls off from 0.37 T down to 0.18 T as the pressure increases from 0.35 up to 0.65 GPa, and then practically does not change upon further pressure rise. Conversely, the transition field $\mu_0 H_c$ increases up to 0.65 GPa at low temperatures (see also figure 3). The T_N value decreases with the average rate of -16 K GPa^{-1} for 0.82 GPa (see the pressure dependence of magnetic characteristics in [18]). The mixed-valence state of Ce and, therefore, the hybridization of its 4f states with the conduction band, undoubtedly play an important role in stabilization of the antiferromagnetism in $\text{Lu}_2\text{Fe}_{17}$ by means of Ce substitution for Lu, i.e. by varying both the up- and down-spin densities of states (DOS) at the Fermi level [19]. The application of pressure increases the overlap between the f and d orbitals as well as leading to the 3d bands widening.

The reduced DOS at the Fermi level and the enhanced spin fluctuations cause the instability of Fe moments, leading to an observed decrease of both the magnetization (figure 2) and T_N under pressure.

However, a rather intriguing feature is the temperature–pressure evolution of the critical field $\mu_0 H_c$ of the metamagnetic AF–F transition, taken as the average between the values in ascending and descending fields as $\mu_0(H_c\uparrow + H_c\downarrow)/2$ (figure 3). At $p < 0.8 \text{ GPa}$, the $\mu_0 H_c(T)$ curves demonstrate a peculiar wide minimum at 100–150 K. At the highest applied pressure of 0.83 GPa, $\mu_0 H_c(T)$ is fairly temperature independent. In figure 4, we have plotted the $M(T)$ dependences at ambient pressure and under 0.82–1.07 GPa pressures in various fields to observe the phenomenon in detail. At ambient pressure and in a low-field region, a non-monotonic behavior of the transition field $\mu_0 H_c(T)$ is inevitably reflected in the appearance of an additional wide cusp, apart from $T_N = 247 \text{ K}$, in the $M(T)$ curves at $p = 0$. At 0.35–0.45 T, the magnetic state of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ consists of three regions: the low-temperature antiferromagnetic state transforms into a high-magnetization phase (with a field-induced non-collinear ferromagnetic structure), which further develops into an antiferromagnetic one at higher temperatures with a characteristic peak in the vicinity of T_N . As was shown in [7, 8], the ground state of $\text{Ce}_2\text{Fe}_{17}$ is described by a modified helical spin structure with the magnetic *c*-parameter twice as large as the chemical unit cell, and above $T_t = 125 \text{ K}$ a transition into a simple helical structure occurs. Despite the fact that no additional peaks connected with a possible antiferromagnetic-to-antiferromagnetic phase transition were observed in the course of specific heat study on $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ [19], a complicated response of the magnetization to the temperature–field variation may suggest the existence of more than one type of antiferromagnetic phase (which can possibly be verified by a neutron diffraction experiment) having almost the same free energy. The intermediate field-induced F phase gradually expands down to the lowest temperatures as the field increases and the 1–2.5 T field range corresponds to a continuous transformation

Table 3. Selected interatomic Fe–Fe distances (Å) in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ at 300 K derived from the powder x-ray diffraction analysis. The multiplicity of each distance is shown in parentheses. (The last digit for some of the distances is significant.)

Atom	Bond	Distance	Atom	Bond	Distance	Atom	Bond	Distance
Fe ₁	Fe ₁	2.369 (1)	Fe ₃₍₁₎	Fe ₃₍₁₎	2.43 (1)	Fe ₃₍₂₎	Fe ₃₍₂₎	2.43 (1)
	Fe ₂	2.59 (3)		Fe ₃₍₂₎	2.47 (1)		Fe ₃₍₃₎	2.63 (1)
	Fe ₃₍₁₎	2.72 (6)		Fe ₃₍₂₎	2.49 (1)		Fe ₃₍₃₎	2.50 (1)
	Fe ₃₍₂₎	3.15 (6)		Fe ₃₍₃₎	2.94 (1)		Fe ₄	2.91 (1)
	Fe ₃₍₃₎	2.89 (6)		Fe ₃₍₃₎	3.10 (1)		Fe ₄	2.53 (2)
	Fe ₄	2.66 (3)		Fe ₄	2.48 (1)		Fe ₅	2.35 (2)
Fe ₂	Fe ₃₍₁₎	2.430 (4)	Fe ₃₍₃₎	Fe ₃₍₃₎	2.53 (1)	Fe ₄	Fe ₄	2.45 (2)
	Fe ₃₍₂₎	2.67 (4)		Fe ₃₍₃₎	2.74 (1)		Fe ₅	2.63 (3)
	Fe ₃₍₃₎	2.49 (4)		Fe ₄	3.18 (1)		Fe ₅	2.53 (3)
	Fe ₄	2.45 (4)		Fe ₄	2.55 (2)		Fe ₅	2.42 (1)
				Fe ₅	2.50 (2)			
			Fe ₅	3.11 (6)				

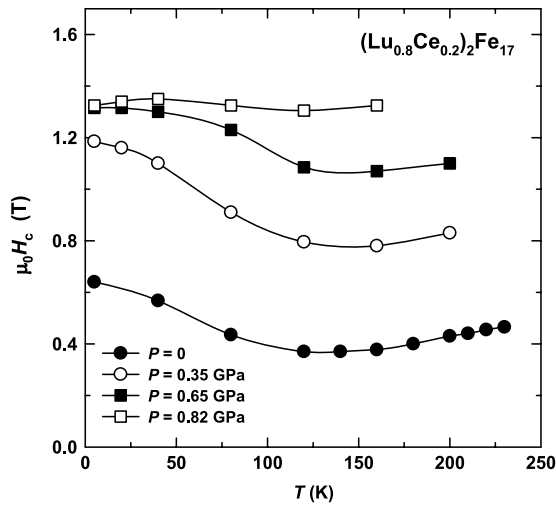


Figure 3. Temperature dependence of the critical field of metamagnetic transition $\mu_0 H_c$ in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ at various pressures.

of the non-collinear F state reached after the metamagnetic jump into the collinear F state at higher fields (figure 2). The magnetization saturates above 2.5 T.

The magnetostriction study for the parent $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ [26] has revealed the appearance of large linear strains, both in the basal plane and along the c -axis accompanying the transition from a small-volume antiferromagnetic phase to a larger-volume ferromagnetic phase. A concomitant volume increase of $\sim 0.6\%$ (at low temperatures and in a 5 T field) was shown to originate from the change of exchange interactions. The application of hydrostatic pressure should act against the lattice expansion which is reflected in the increased transition field at higher pressure (figure 3). On the other hand, a neutron diffraction study of the crystal structure of $\text{Lu}_2\text{Fe}_{17}$ performed at room temperature and under pressures of up to 1.65 GPa [27] has shown that some of the Fe–Fe distances increase with increasing pressure in spite of the overall lattice contraction. Nevertheless, the weakening of the total positive exchange appears faster than the negative one, resulting in the

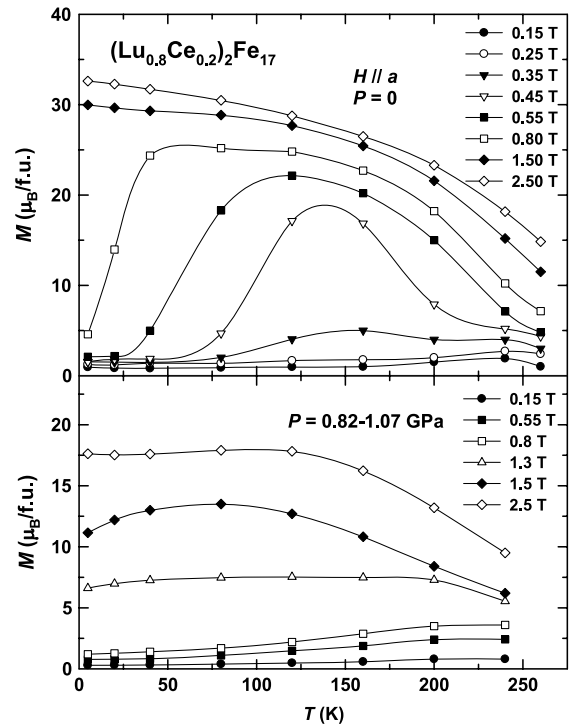


Figure 4. Temperature dependences of magnetization of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ single crystal measured along the easy a -axis in various fields at ambient pressure (top) and under 0.82–1.07 GPa (bottom).

destabilization of the ferromagnetic order in $\text{Lu}_2\text{Fe}_{17}$ (as well as in $\text{Ce}_2\text{Fe}_{17}$) under pressure. Therefore at the highest applied pressure of 0.82–1.07 GPa, the low-field (i.e. below the critical field) $M(T)$ curves in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ no longer display the intermediate field-induced F phase (see figure 4, bottom).

The non-monotonic temperature behavior of the critical field $\mu_0 H_c$ in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ and a high-magnetization fan structure emerging in weak magnetic fields point to the competing antiferromagnetic and ferromagnetic exchange interactions having a strong dependence on the temperature. The detailed refinement of the crystal structure of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$

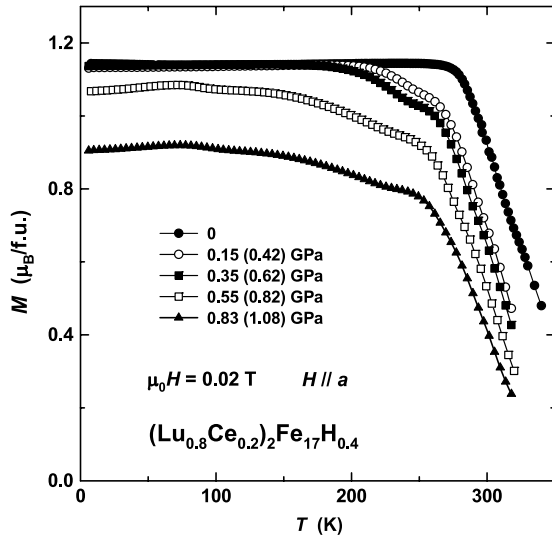


Figure 5. Temperature dependence of magnetization of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ single crystal measured in a field of 0.02 T applied along the a -axis at different pressures (the pressure values within and without parentheses correspond to 300 K and 5 K, respectively).

obtained at room temperature and at ambient pressure has revealed the decreased Fe–Fe distances at particular sites as compared to $\text{Lu}_2\text{Fe}_{17}$ [23]. Surprisingly, the crucial distance for R_2Fe_{17} compounds between the Fe atoms in 4f positions (so-called dumbbells) located along the c -axis and characterized by a very strong local negative exchange interaction decreased from 2.387 Å in $\text{Lu}_2\text{Fe}_{17}$ down to 2.367 Å in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ (table 2). Therefore, the observed effects on the hydrostatic pressure acting together with the uniaxial ‘chemical’ pressure induced by Ce substitution (contraction of the lattice along the c -axis) are also in agreement with a simple semi-phenomenological Néel–Bethe–Slater [28] model for the dependence of the local exchange integrals on the interatomic distances. This model predicts an increase of the strength of negative exchange interactions for decrease of the Fe–Fe distances that is reflected in stabilization of the long period oscillating magnetic structure in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$.

Finally, it should be borne in mind that a pressure increase and the temperature decrease produce a transition to the higher valency of Ce [1] with a corresponding decrease of the Ce atomic volume. Unfortunately, it was not possible to check whether the pressure dependence of the unit cell volume of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ shows any peculiarities at lowered temperatures, but a low Ce content in the sample may have ensured an insignificant volume change.

3.3. $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$

In order to check the stability of a sustained antiferromagnetic order in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ by inducing the lattice expansion (i.e. under the ‘negative’ chemical pressure), we have obtained and investigated the hydrogen-charged single-crystalline $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}$ compound. In our work, we limited the hydrogen uptake to a rather low value of 0.4 H at./f.u. With

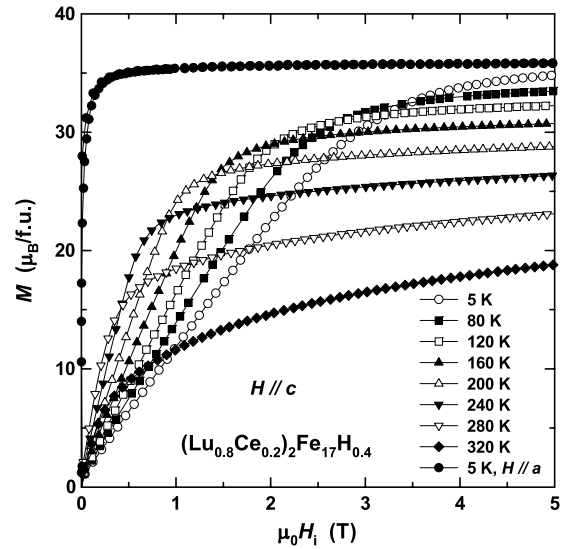


Figure 6. Magnetization isotherms in fields applied along the c -axis of the $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ single crystal. For $T = 5$ K, the curve measured along the a -axis is shown as well.

this provision, the sample retained the single-crystalline state and did not disintegrate during the experiments under extreme conditions. The temperature evolution of magnetization of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ single crystal measured in a magnetic field of 0.02 T applied along the a -axis is presented in figure 5. A striking feature of the obtained new compound is a total suppression of the antiferromagnetic state despite a rather low hydrogen content (for comparison, in [14], introduction of twice as high an amount of hydrogen in $\text{Ce}_2\text{Fe}_{17}$ led to only a shift of the Curie point T_C toward the higher temperatures, whereas the T_N remained unchanged). $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ shows typical ferromagnetic behavior with $T_C = 300$ K.

Figure 6 demonstrates the magnetization isotherms along the a -axis at 5 K and in fields applied along the c -axis of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ at various temperatures. Hydrogenation is found to not influence the type of magnetocrystalline anisotropy (MCA). Similar to both $\text{Lu}_2\text{Fe}_{17}$ and $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ [19], the easy magnetization direction (EMD) of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ lies within the basal plane, and the c -axis is the hard magnetization direction (HMD). The EMD curve of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ at 5 K shows a spontaneous ferromagnetism with the moment $M_s = 35.2 \mu_B/\text{f.u.}$ as derived from extrapolation of experimental $M(H)$ curves onto zero field. On the other hand, the absence of any spontaneous magnetization component and a good linearity of the HMD curves indicate the uniformity of the hydride crystal.

Figure 7 displays the temperature dependences of the MCA constants K_1 and K_2 derived by the Sucksmith–Thompson method [29] from the HMD data presented in figure 6. The values of $K_1 = 2.2 \text{ MJ m}^{-3}$ and $K_2 < 0.2 \text{ MJ m}^{-3}$ at the lowest temperatures agree with those obtained previously for the other $\text{Lu}_2\text{Fe}_{17}$ -based compounds [30] and are a common feature of the R–Fe intermetallics with non-magnetic R elements. Quantitatively the same anisotropy of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ compared to that

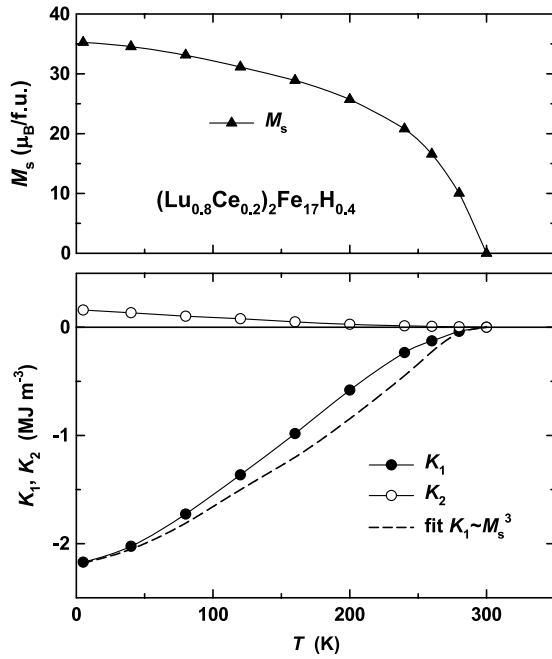


Figure 7. Temperature dependence of spontaneous magnetization M_s and magnetocrystalline anisotropy constants K_1 and K_2 for $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$. The dashed line represents the fit $K_1(T) = K_1(0) [M_s(T)/M_s(0)]^3$.

of $\text{Lu}_2\text{Fe}_{17}$ [31, 32] is not due to a low H content, e.g. in the related $\text{Gd}_2\text{Fe}_{17}\text{H}_x$ compounds, the decrease of MCA of the Fe sublattice was found to be rather small even for a much larger H content [33]. An attempt to describe $K_1(T)/K_1(0)$ versus the reduced temperature T/T_C as the third power of reduced magnetization $m = M_s(T)/M_s(0)$ (such a procedure provides the evidence for the single-ion mechanism of MCA in the case of $|K_1| \gg |K_2|$ [34]) has shown that the experimental $K_1(T)$ dependence agrees roughly with the fit used (see figure 7). However, a faster decrease of $K_1(T)$ than that predicted by the ' $l(l+1)/2$ power law' similar to that observed in $\text{Lu}_2\text{Fe}_{17}$ [32] may be attributed to a band-electron contribution in addition to a single-ion main part of magnetocrystalline anisotropy. (It is interesting to note the following trait. In the case of, e.g., non-collinear (helical) ferromagnets, $\text{Ce}_2\text{Fe}_{17-x}\text{Mn}_x$ ($x = 1.7, 2$), a slower decrease of the absolute value of K_1 was observed with increasing temperature [35]. This fact was attributed to the presence of an additional contribution to the anisotropy arising from the change of exchange energy involved in the magnetization process of the non-collinear ferromagnets.)

The effect of hydrogenation on the magnetic properties, such as an increase of the average Fe magnetic moment and the Curie temperatures in the R-Fe systems with a high Fe content, is to a large extent a consequence of the increased volume of the hydrogen-charged materials as compared to the precursors [15]. However, owing to a highly anisotropic lattice expansion of the unit cell of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ upon hydrogenation (the c/a ratio decreases from 0.9823 in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ down to 0.9796 in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$) and due to the presence of the very short Fe-Fe dumbbells along the c -axis it was of interest to examine the data on the

interatomic 4f-4f distance changes upon hydrogenation. Although hydrogen does not occupy the interstitial sites involving the iron atoms in the 4f positions [24, 25], it usually produces the effect by increasing the interatomic dumbbell distances and relaxing the stressed bonds. Due to the small hydrogen concentration in the compound investigated, the observed increase is rather small, i.e. about 0.08%. Nevertheless, the comparison of tables 2 and 3 shows that the majority of the interatomic Fe-Fe bonds have slightly increased in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ as compared to $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$, which probably leads to reinforcing the positive character of exchange interactions resulting in the rise of the Curie temperature by favoring the ferromagnetic alignment of the iron magnetic moments. Furthermore, taking into account a rather anisotropic lattice expansion of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ under applied magnetic field [26] ($\sim 0.3\%$ along the easy a -axis in a 5 T field and in the field-induced ferromagnetic state), it can be deduced that both the lattice expansion induced by the hydrogen insertion in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ and the very strong field-induced magnetostrictive strains in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ may have produced somewhat similar effects on the exchange interactions in the compounds studied.

A partial withdrawing of the d states (or even the f states for the valence-fluctuating rare earths) due to the H-bonding effect may also play a significant role in varying the magnetic properties as a result of hydrogenation. In principle, both the volume effect of hydrogen insertion with the main impact on exchange interactions and the electronic effect can be 'separated' in the course of the pressure study on hydrides. For $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$, the estimated negative pressure induced by hydrogenation and equivalent to 0.76% volume expansion is -0.79 GPa (here we used the experimental value of low-temperature volume compressibility $\kappa = -1/V(\partial V/\partial p) = 1.2 \times 10^{-2} \text{ GPa}^{-1}$ of $\text{Lu}_2\text{Fe}_{17}$ [36, 37]), which is similar to that found from the pressure dependences of T_C in another $\text{R}_2\text{Fe}_{17}\text{H}_x$ with a larger H content (about -0.8 GPa/H at [38]). Therefore, if the H-bonding effect is negligible, the maximum hydrostatic pressure of 0.83 GPa (1.1 GPa at 300 K) available in our work should compensate fully the lattice expansion and restore the magnetic properties of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$.

An unexpectedly weak pressure effect on the hydrogenation-induced ferromagnetic states was found in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ (figures 5 and 8). The application of as much as $p = 0.15$ GPa (corresponding to 0.42 GPa at room temperature) has resulted in the appearance of a small high-temperature anomaly on the $M(T)$ curve. With a further pressure increase, the Curie temperature T_C of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ was found to shift downwards with a rate of -27 K GPa^{-1} under a pressure change of ~ 0.7 GPa. Despite the fact that the value obtained is close to the pressure sensitivity of the ordering temperature T_N reported earlier for the Ce-richer phase [14] ($dT_N/dp = -25 \text{ K GPa}^{-1}$), the transition temperature T_C between the AF and the low-temperature ferromagnetic phase of $\text{Ce}_2\text{Fe}_{17}\text{H}_{0.8}$ showed a considerably faster decrease. Nevertheless, an induced non-collinearity of the magnetic moments under pressure in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ was revealed both in the decrease of spontaneous magnetic moment ($d \ln M/dp = -0.11 \text{ GPa}^{-1}$ at 5 K) and in the appearance of hysteresis in

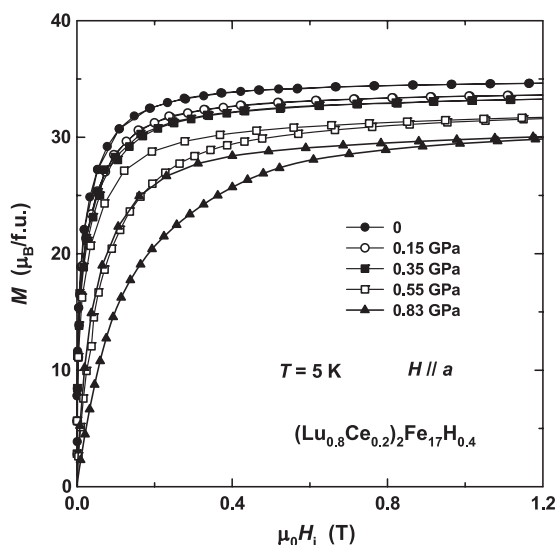


Figure 8. Magnetization isotherms of the $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ single crystal in fields applied along the a -axis at 5 K under different pressures.

low fields (figure 8). Therefore, the maximum applied pressure in the course of our study has only led to the formation of a fan-like structure in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$. The effect is probably due to a highly anisotropic expansion of the lattice caused by the hydrogen insertion and to the strong anisotropy of the elastic moduli in R_2Fe_{17} compounds [37], whereas the hydrostatic pressure is assumed to act uniformly on a sample surface. As was shown in [13], considerably higher values of hydrostatic pressure as compared to the ‘negative’ pressure induced by hydrogen insertion were required in order to fully suppress the hydrogen-induced ferromagnetic state in $\text{Lu}_2\text{Fe}_{17}\text{H}$. Therefore, it is expected that total suppression of the ferromagnetic state in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ may be achieved at pressures about twice as large that those available in the course of the present study.

4. Conclusions

In this work, a combined investigation of the interplay between the magnetovolume effects and the resulting magnetic state of the $\text{Lu}_2\text{Fe}_{17}$ -based intermetallics was performed by means of structural and magnetization (under ambient/high pressure) studies on $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ compound and its hydride. The magnetic behavior of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ under pressure arises from the increased overlap between the hybridized states f states of Ce with the conduction band and from the 3d band widening, that lead to a decrease of both the magnetization and the Néel temperature T_N . On the other hand, the revealed strong dependence of the exchange interactions on the variation of external conditions (by means of applying high pressure, and varying the temperature and applied magnetic field strength) in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ is in agreement with a simple Néel–Bethe–Slater model.

In order to study the relevant role of the change of unit cell dimensions on the exchange interactions, we have successfully obtained and investigated the hydrogen-charged single crystal

of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}$. Even a very low hydrogen content of 0.4 H at./f.u. in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ was found to suppress completely the antiferromagnetic arrangement of magnetic moments observed in the parent compound. Despite the tiny increase of the 4f–4f interatomic bond distances upon hydrogenation, the overall increase of the majority of the bonds may have strengthened the positive exchange interactions, leading to a considerably higher magnetic ordering temperature in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$ as compared to $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$. The volume effect on the magnetism of $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}$ was found to be qualitatively reversible, i.e. an application of hydrostatic pressure induced the non-collinearity of the magnetic moments in $(\text{Lu}_{0.8}\text{Ce}_{0.2})_2\text{Fe}_{17}\text{H}_{0.4}$. However, the anisotropic lattice expansion and a different anisotropic pressure driven lattice compression may have caused a misfit between the estimated ‘negative’ and the required hydrostatic pressures that did not allow the observation of the quantitative effects.

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