



Contents lists available at ScienceDirect

Journal of Science: Advanced Materials and Devices

journal homepage: www.elsevier.com/locate/jsamd

Original article

Magnetic properties of HoFe₆Al₆H hydride: A single-crystal studyA.V. Andreev^{a,*}, I.A. Pelevin^{b,c}, J. Šebek^a, E.A. Tereshina^a, D.I. Gorbunov^{a,d}, H. Drulis^e, I.S. Tereshina^{c,f}^a Institute of Physics, Czech Academy of Sciences, 182 21 Prague, Czech Republic^b Baikov Institute of Metallurgy and Materials Science, Russian Academy of Sciences, 119991 Moscow, Russia^c International Laboratory of High Magnetic Fields and Low Temperatures, 53-421 Wrocław, Poland^d Dresden High Magnetic Field Laboratory (HLD-EMFL), Helmholtz-Zentrum Dresden-Rossendorf, D-01314 Dresden, Germany^e Institute of Low Temperature and Structure Research, Polish Academy of Sciences, 50-950 Wrocław, Poland^f Lomonosov Moscow State University, Faculty of Physics, 119991 Moscow, Russia

ARTICLE INFO

Article history:

Received 19 April 2016

Received in revised form

9 May 2016

Accepted 10 May 2016

Available online 13 May 2016

Keywords:

Rare-earth intermetallics

Hydrides

Magnetic anisotropy

Ferrimagnetism

High magnetic fields

Field-induced transition

ABSTRACT

Crystal structure and magnetic properties were studied on a single crystal of HoFe₆Al₆H and compared with those of the parent HoFe₆Al₆ compound with a tetragonal crystal structure of the ThMn₁₂ type. Hydrogenation leads to a 1% volume expansion. HoFe₆Al₆ is a ferrimagnet with exact compensation of the Ho and Fe sublattices magnetizations at low temperatures. Both the hydride and the parent compound display a high magnetic anisotropy of the easy-plane type, a noticeable anisotropy exists also within the easy plane with the [110] axis as the easy magnetization direction. The hydrogenation increases slightly (from 10 to 10.45 μ_B) the magnetic moment of the Fe sublattice as a result of volume expansion. It leads to a decompensation of the Fe and Ho sublattices and HoFe₆Al₆H has a spontaneous moment 0.45 μ_B/f.u. The enhancement of the Fe–Fe intra-sublattice exchange interaction results in a higher Curie temperature (*T*_C) value, 350 K in the hydride as compared to 315 K of HoFe₆Al₆. The Ho–Fe inter-sublattice interaction is also enhanced in the hydride. The molecular field *H*_{mol} created on Ho ions by Fe sublattice is 38 T in HoFe₆Al₆ and 48 T in HoFe₆Al₆H. The inter-sublattice exchange constant *J*_{HoFe} is 3.8 T/μ_B and 4.6 T/μ_B, respectively. High-field measurements confirm the enhancement of the Ho–Fe exchange interaction in the hydride found from the temperature dependence of magnetization.

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1. Introduction

Intermetallic compounds of rare-earth metals R with 3*d* transition metals T (first of all, with T = Fe and Co) form a wide class of magnetic materials. They are very interesting not only due to their high application potential (first of all permanent-magnet materials based on the R₂Fe₁₄B compounds) but also from the scientific viewpoint since they combine two principally different groups of electrons responsible for the magnetism: localized magnetism of the 4*f* sublattice is combined with (mainly) itinerant magnetism of the 3*d* sublattice. Investigations of complicated intra- and inter-sublattice interactions of exchange and anisotropic origins are strongly desirable for a deeper understanding of fundamental problems of magnetism. The inter-sublattice interaction is

especially interesting since it “pulls” the low temperature anisotropic properties of the 4*f*-sublattice towards elevated temperatures, which is the base for applications. For this reason, the rare-earth intermetallics, especially those with a high content of a 3*d*-metal (in particular, with the RT₅, R₂T₁₇, R₂T₁₄B and RT₁₂ stoichiometries) have been extensively studied for the last several decades (see for review [1–3]).

Many *f*–*d* intermetallics can form so-called interstitial solutions where atoms with small atomic radii (H, C, N) do not substitute “main” atoms but rather reside in voids between them. Up to a rather high content of such doping the lattice expands without qualitative changes of the crystal structure [4,5]. This strongly influences the magnetic properties and can be in a good approximation considered as an application of negative pressure. It gives an additional tool for a study of exchange and anisotropic interactions. Since most of intermetallic compounds are very brittle and their elastic limit does not exceed strain of 10^{−3}, whereas lattice expansion as a result of hydrogenation can reach several percent, absorption of hydrogen often leads to powderization of

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Peer review under responsibility of Vietnam National University, Hanoi.

ingots. This important feature is used in the hydrogen deprecipitation desorption recombination (HDDR) process for preparation of powders of NdFeB magnets, however, it hinders preparation of single crystals which are needed for investigation of magnetic anisotropy, a very important property of the R–T compounds. Nevertheless, hydrides of some compounds can be prepared under special conditions in the single-crystalline form. Their study gives an important information of the hydrogen influence not only on the magnitude of magnetic moments and magnetic ordering temperatures but also on magnetic anisotropy and field-induced phase transitions in $R_2Fe_{14}B$ [6–9], R_2Fe_{17} [10,11] and RT_{12} [12,13].

The present work addresses the problem of the influence of hydrogen on the magnetism of $HoFe_6Al_6$. $HoFe_6Al_6$ belongs to the RT_{12} intermetallics having the tetragonal crystal structure of the $ThMn_{12}$ type. Binary compounds exist only with $T = Mn$, nevertheless, the structure can be stabilized by a third element. Compounds $R(T,M)_{12}$ (M is a stabilizing element) form a wide group of materials (see Ref. [14] for review). The Fe content x in the RFe_xM_{12-x} quasibinaries can vary in a wide interval. Compounds of this group with a high Fe content, $RFe_{11}Ti$, $RFe_{10.5}V_{1.5}$, $RFe_{10}Si_2$ and $RFe_{10}Mo_2$, are considered as promising materials for permanent magnets [15–19]. Compounds with $M = Al$, RFe_xAl_{12-x} , are characterized by competitive exchange and anisotropic interactions within their homogeneity range, $4 \leq x \leq 6$, and therefore deserve special attention from the fundamental point of view.

$HoFe_6Al_6$ is a collinear ferrimagnet with Curie temperature $T_C = 315$ K. It displays a strong magnetic anisotropy of the easy-plane type. A large anisotropy is also observed in the easy plane, the [110] axis is the easy magnetization direction [20]. The Fe sublattice, as found from the magnetization study of $LuFe_6Al_6$ with non-magnetic Lu, has a magnetic moment $M_{Fe} = 10 \mu_B$ [21]. Since the magnetic moment of single Ho^{3+} ion M_{Ho} is also $10 \mu_B$, the total spontaneous magnetic moment M_S is exactly zero at low temperatures due to antiparallel arrangement of the sublattices. Due to a faster temperature decrease of M_{Ho} as compared to M_{Fe} , M_S grows in a wide temperature interval.

In the present work, we study the influence of hydrogenation on magnetism of $HoFe_6Al_6$ by magnetization measurements on single crystals.

2. Experimental details

A single crystal of parent compound $HoFe_6Al_6$ was grown by a modified Czochralski method in a tri-arc furnace from stoichiometric mixture of pure elements (99.9% Ho, 99.98% Fe and 99.999% Al). Back-scattered Laue patterns were used to confirm a mono-crystalline state and to orient the crystals to cut samples for the magnetization measurements. Standard powder X-ray diffraction analysis performed on a part of the single crystal crushed into powder was used to check the crystal structure and to determine the lattice parameters. The analysis confirmed a single-phase state with the tetragonal $ThMn_{12}$ -type structure. The lattice parameters, $a = 864.2$ pm and $c = 503.9$ pm, are in a good agreement with literature [14].

The hydride was prepared by direct reaction of gaseous hydrogen with small (20–30 mg) pieces of single crystal of the parent compound $HoFe_6Al_6$ using a glass Sieverts-type apparatus. The hydrogenation was carried out using pure hydrogen gas under pressures of up to 0.1 MPa obtained from TiH_2 hydrogen storage. In order to prevent the sample from powderization, hydrogenation was performed rather slowly. A short (1 h) thermal activation process at 400 °C and at high vacuum was carried out to initiate the hydrogen absorption process and was completed by 1-h cooling to room temperature. After that, reaction chamber was filled with hydrogen gas under 1 MPa pressure and the system was heated to

650 °C. The $HoFe_6Al_6$ sample was annealed in the hydrogen atmosphere for 3 h, cooled slowly (48 h) down to room temperature and then left at these conditions. The amount of absorbed hydrogen was determined from the hydrogen pressure change in the calibrated reactor chamber. We realized that at the conditions used hydrogen absorption is rather slow. After 3 days the calculated hydrogen content y in $HoFe_6Al_6H_y$ was only 0.56, whereas in other $RFe_xM_{12-x}H_y$ compounds it reached the value $y = 1$ [12,13,22–24]. So, the temperature annealing program was repeated and the sample was left in a hydrogen atmosphere for 18 days. During this time, sample slowly absorbed hydrogen. The final hydrogen concentration close to $y = 3.1$ was determined. However, we found that a large portion of hydrogen was soon released by the crystals. The concentration of hydrogen remaining in the samples was determined to be about $y = 1$ (see below).

The field and temperature dependences of the magnetization in fields up to 7 T were measured between 2 and 350 K for fields applied along the principal crystallographic directions [100], [110] and [001] of 20–30 mg samples of hydride and parent compound using a MPMS magnetometer (Quantum Design).

High-field magnetization curves were measured along the principal axes at 2 K in pulsed magnetic fields up to 58 T (pulse duration 20 ms). The magnetization was measured by the induction method using a coaxial pick-up coil system. A detailed description of the set-up is given in Ref. [25]. Absolute magnetization values of were calibrated using data obtained by measuring the crystals in static magnetic fields.

3. Results and discussion

Owing to the slow hydrogenation procedure, the single-crystalline structure was preserved in hydride (confirmed by back-scattered Laue patterns). Powder X-ray analysis (Fig. 1) showed a single-phase state with the tetragonal $ThMn_{12}$ -type structure (Fig. 2). The obtained lattice parameters are $a = 868.3$ pm

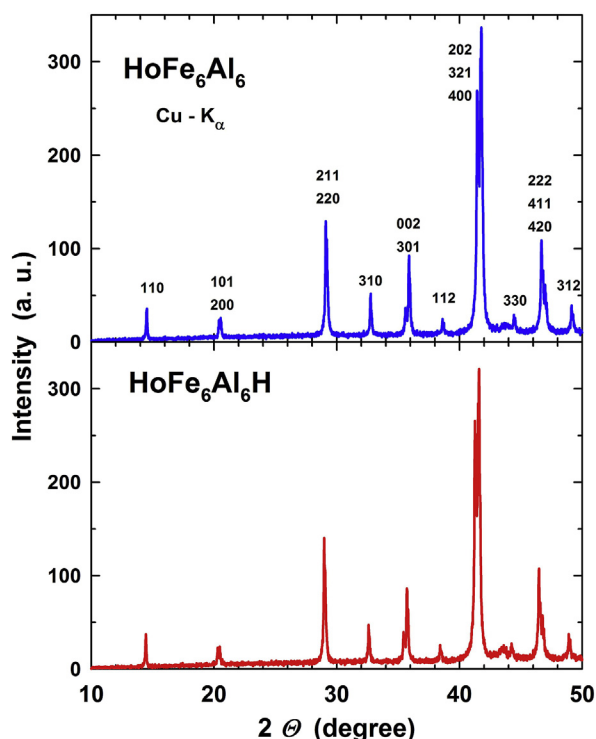


Fig. 1. X-ray powder diffraction patterns of $HoFe_6Al_6$ and $HoFe_6Al_6H$.

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