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Magnetic and Magnetocaloric Properties of $Y(Co_{1-x}Fe_x)_2$ ($x = 0.12-0.20$) and $Lu(Co_{0.84}Fe_{0.16})_2$ Compounds

© M.S. Anikin[1], E.N. Tarasov[1], A.V. Zinin[1], N.V. Kudrevatykh[1], D.S. Neznakhin[1], M.A. Semkin[1,2], M.I. Knyazev[1], N.V. Selezneva[1], S.V. Andreev[1]

[1] Institute of Natural Sciences & Mathematics, Ural Federal University,
Ekaterinburg, Russia
[2] Mikheev Institute of Metal Physics, Ural Branch, Russian Academy of Sciences,
Ekaterinburg, Russia
E-mail: maksim.anikin@urfu.ru

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To clarify the nature of the anomalously high magnetocaloric effect at low temperatures in the RMe_2 Laves type phases with „non-magnetic" rare-earth ions ($R = \mathrm{Y}$ or Lu) and 3d elements of the Fe group ($Me = \mathrm{Fe, Co}$), the $Y(Co_{1-x}Fe_x)_2$ ($x = 0.12-0.20$) and $Lu(Co_{0.84}Fe_{0.16})_2$ compositions have been synthesized and their magnetic and magnetocaloric properties were investigated (isothermal magnetic entropy change ΔS_m and adiabatic temperature change ΔT_{ad}). It has been established that the iron concentration increases and/or Y by Lu replacement with unchanged Co:Fe ratio gives rise in the energy of the $d-d$ exchange interaction, which is followed by an increase in the Curie temperature value as well as by the low-temperature anomaly shift on the $\Delta S_m(T)$ dependence to a higher temperatures range.

Keywords: magnetocaloric effect, adiabatic temperature change, magnetic moments, Laves phase, Curie temperature, mictomagnetism.

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1. Introduction

In quasi-binary $R(Co_{1-x}Fe_x)_2$ compounds, where R are the heavy rare-earth elements, it was found that for compounds with $x = 0.12$, an increase in the R atom number from Gd to Er causes a transformation of the shape of temperature dependence parameters of magnetocaloric effect (MCE) characterizing the isothermal magnetic entropy change ΔS_m and adiabatic temperature change ΔT_{ad}, from the classic maximum at Curie temperature (T_C) for $R = \mathrm{Gd}$ to the table-like dependence observed over a wide temperature range from T_C and below (down to 50 K) for $R = \mathrm{Ho}$ and Er [1]. The table-like temperature dependences of the MCE parameters are formed by the superposition of ΔS_m (or ΔT_{ad}) maximum at T_C with the low-temperature ΔS_m (or ΔT_{ad}) maximum. The low-temperature maximum (LTM) results from the magnetic order degree change in the R sublattice under the action of the external magnetic field H, partially destroyed by thermal energy due to the weakness of the intersublattice $4f-3d$ exchange interaction [2].

On the other hand, in the $(Er_{1-x}Y_x)(Co_{0.84}Fe_{0.16})_2$ series it was found [3] that the LTM on the $\Delta S_m(T)$ dependence does not disappear even if the magnetoactive Er element is completely replaced by „non-magnetic" Y ($Y(Co_{0.84}Fe_{0.16})_2$ compound). Later in [4], it was suggested that its appearance on the $\Delta S_m(T)$ dependence in such material may be associated with partial paramagnetism of the cobalt atomic system.

It is known that in the series of $Y(Co_{1-x}Fe_x)_2$ compounds, the percolation limit is reached when the Fe content decreases below $x = 0.14$, which causes the disappearance of the far magnetic order (ferromagnetic), thus such compounds become mictomagnetic [5]. In particular, the mictomagnetic compound $Y(Co_{0.97}Fe_{0.03})_2$ includes a „non-magnetic" Y$-$Co matrix and ferromagnetic clusters based on Fe atoms surrounded by Co atoms with an induced (from Fe) magnetic moment [6]. It can be supposed that for the $Y(Co_{0.84}Fe_{0.16})_2$ compound due to its composition being close to the percolation limit, the $d-d$ energy of the Fe$-$Co exchange interaction is lacked to maintain the magnetic order in the entire subsystem of Co atoms, especially at intermediate temperatures from zero to T_C due to the strong influence of thermal fluctuations. As a result, it is possible that the exchange splitting of the $3d$ zone in the system of delocalized $3d$ Co electrons is lost. Application of a magnetic field could induce this, which means that the magnetic moment on the Co atoms is induced. Consequently, it can be assumed that the low-temperature maximum of the MCE parameters observed in $Y(Co_{0.84}Fe_{0.16})_2$ should be suppressed by the Fe$-$Co exchange interaction rise.

To verify this assumption experimentally, the magnetic and magnetocaloric properties of $Y(Co_{1-x}Fe_x)_2$, where $x = 0.12-0.20$, and the $Lu(Co_{0.84}Fe_{0.16})_2$ compounds both with „non-magnetic" Y and Lu ions were studied. Compound with Lu was chosen to evaluate the influence of other than Y „non-magnetic" *R* element on the Co magnetic state [7,8].

2. Experimental details

Polycrystalline alloys of $Y(Co_{1-x}Fe_x)_2$ $(x = 0.12-0.20)$ and $Lu(Co_{0.84}Fe_{0.16})_2$ compositions were melted in an electric arc furnace under a pure helium protection. To prevent the formation of 3*d*-rich phases, an excess of Y and Lu in the amount of 5 wt.% was added to the initial composition. A homogenizing annealing of alloys was made in a vacuum furnace for 24 hours, in particular for $Y(Co_{1-x}Fe_x)_2$ at 1223 K and for $Lu(Co_{0.84}Fe_{0.16})_2$ at 1273 K. Phase analysis was carried out using the *X*-ray diffraction method using the D8 Advance powder diffractometer (Bruker) with a $Cu-K_\alpha$ radiation source, $\lambda = 1.5406$ Å. FullProf Suite software was applied for lattice parameters refinement [9].

Magnetic properties were measured using the PPMS DynaCool (Quantum Design) in a temperature range of 2−370 K in a magnetic field up to 90 kOe. Direct measurement of the adiabatic temperature change at magnetic field jumps (ΔH) of 17.5 kOe was carried out using the MagEq MMS SV3 equipment in a temperature range of 80−370 K.

3. Results and discussion

X-ray diffraction patterns of $Y(Co_{1-x}Fe_x)_2$ and $Lu(Co_{0.84}Fe_{0.16})_2$ powder samples at room temperature (Figure 1) showed that the content of the main Laves phase of stoichiometric composition 1 : 2 was from 97 to 100%. The impurity phases were „non-magnetic" oxides Y_2O_3 and Lu_2O_3, but $Y(Co_{0.86}Fe_{0.14})_2$ and $Y(Co_{0.80}Fe_{0.20})_2$ samples contained ∼ 1−2% „magnetic" phases.

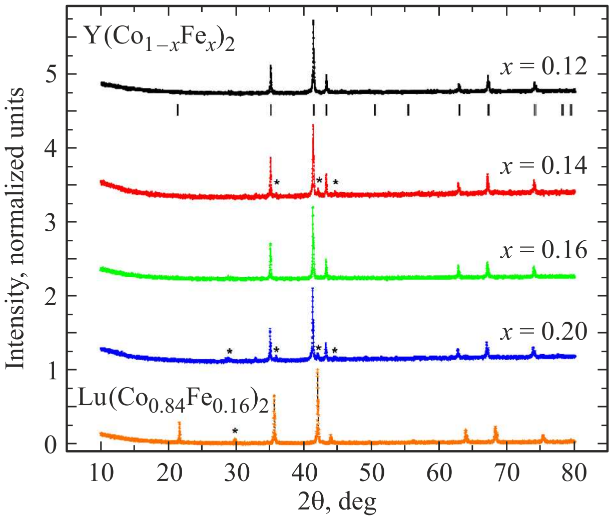

Figure 1. *X*-ray diffraction patterns of $Y(Co_{1-x}Fe_x)_2$ and $Lu(Co_{0.84}Fe_{0.16})_2$ powder samples. Positions of Miller indices for the Laves phase crystal structure are shown under the pattern for $x = 0.12$. Peaks from impurity phases are marked by asterisks.

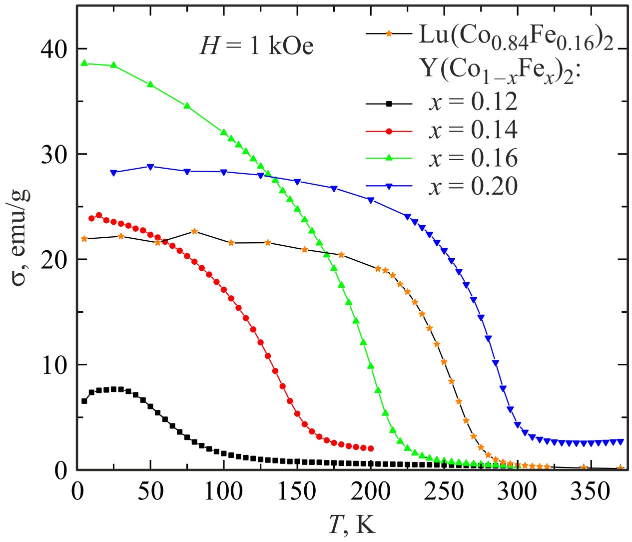

Figure 2. Temperature dependences of magnetization of $Y(Co_{1-x}Fe_x)_2$ and $Lu(Co_{0.84}Fe_{0.16})_2$ compounds in the external magnetic field of 1 kOe.

Figure 1 shows that the peaks on the *X*-ray patterns of the $Lu(Co_{0.84}Fe_{0.16})_2$ sample are shifted towards the higher angles compared to the $Y(Co_{1-x}Fe_x)_2$ series, which indicates a significant change in the value of the 1 : 2 phase lattice parameter *a*. Its crystal structure ($MgCu_2$ type) at room temperature belongs to the face-centered cubic $Fd-3m$ space group in whose unit cell the Y and Lu ions are located at nodes 8_b (3/8; 3/8; 3/8) and the 3*d* element at 16_c (0; 0; 0). The refined values of the crystal lattice parameter *a* are presented in the Table.

Figure 2 shows the temperature dependence of specific magnetisation σ of the studied alloys in an external magnetic field *H* of 1 kOe. They show the presence of impurity „magnetic" phases in $Y(Co_{0.86}Fe_{0.14})_2$ and $Y(Co_{0.80}Fe_{0.20})_2$ samples with a T_C higher than that of the 1 : 2 phase; the other samples are single-phase in a „magnetic sense".

Figure 3 shows the specific magnetization temperature dependences σ of the $Y(Co_{0.88}Fe_{0.12})_2$ and $Lu(Co_{0.84}Fe_{0.16})_2$ alloys, measured when heating a sample at $H = 100$ Oe, pre-cooled at $H = 0$ (ZFC protocol), and measured when cooling the sample at $H = 100$ Oe (FC protocol). Based on these $\sigma(T)$ dependences, the Curie temperatures of all investigated ferromagnetic samples were determined from the extremum of $d\sigma/dT$ (obtained T_C values are presented in the Table). T_C concentration dependence shows that with increasing Fe content from 14 to 20% in $Y(Co_{1-x}Fe_x)_2$ its value increases, indicating their ferromagnetic nature and $d-d$ exchange interaction energy rise. The replacement of Y by Lu in a similar compound leads to the same result.

Lattice parameter a, Curie temperature T_C, and magnetic moment $\mu_{\text{f.u.}}$ per formula unit in Bohr magnetons at 5 K and $H_{\text{ext}} = 90$ kOe, for $Y(Co_{1-x}Fe_x)_2$ and $Lu(Co_{0.84}Fe_{0.16})_2$ samples

	$Y(Co_{1-x}Fe_x)_2$				$Lu(Co_{0.84}Fe_{0.16})_2$
	$x = 0.12$	$x = 0.14$	$x = 0.16$	$x = 0.20$	
a, Å	7.2428 (4)	7.2494 (25)	7.2522 (4)	7.2602 (31)	7.1405 (2)
T_C, K	–	136	200	285	264
$\mu_{\text{f.u.}}$, μ_B	1.22	1.73	1.92	2.38	2.27

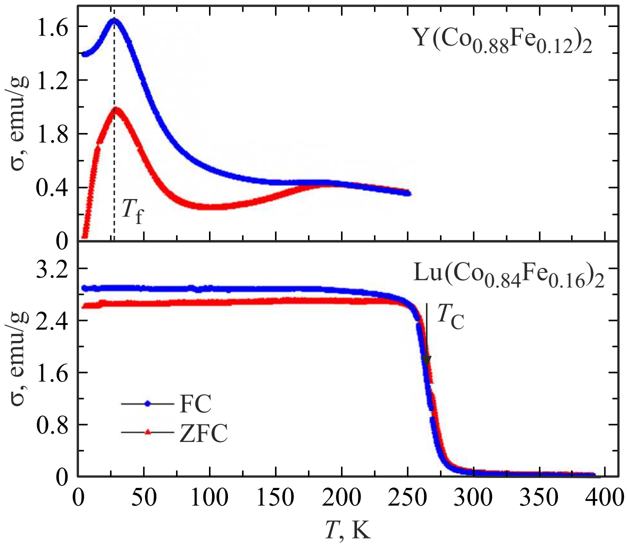

Figure 3. Temperature dependences of magnetization of $Y(Co_{0.88}Fe_{0.12})_2$ and $Lu(Co_{0.84}Fe_{0.16})_2$ samples in external magnetic field of 100 Oe, measured using ZFC and FC protocols. The vertical dashed line corresponds to the freezing temperature $T_f = 28$ K.

At $H = 100$ Oe, the $\sigma(T)$ dependence of $Y(Co_{0.88}Fe_{0.12})_2$ compound essentially differs from the similar dependences of ferromagnetic ones. In particular, this compound shows a difference in $\sigma(T)$ courses measured using ZFC and FC protocols. For ZFC, the magnetization value at $T = 5$ K is practically close to zero and increases with increasing temperature, reaching a maximum at $T = 28$ K, while for FC its value is significantly higher and decreases slightly with temperature lowering from 28 to 5 K. This behavior is inherent in the mictomagnetics [10], and the temperature, below which the differences in ZFC and FC $\sigma(T)$ dependences are observed, is called the freezing temperature (T_f). Apparently, at $T = 5$ K in the ZFC case the antiferromagnetic coupling occurs between the Fe–Co clusters, and upon the T increasing to T_f it transforms into ferromagnetic one by the action of H. At the same time, when cooling down in the magnetic field (FC curve) below T_f, the Fe–Co clusters remain ordered ferromagnetically with the magnetic moment orientation towards the external magnetic field. The decrease of $\sigma(T)$ in the interval from T_f to 5 K is probably related to the growth of magnetocrystalline anisotropy.

Figure 4 shows magnetization isotherms at $T = 5$ K of $Y(Co_{0.88}Fe_{0.12})_2$, $Y(Co_{0.84}Fe_{0.16})_2$, and $Lu(Co_{0.84}Fe_{0.16})_2$ compounds calculated in Boron magnetons per the formulae unit ($\mu_{\text{f.u.}}$). As it is seen, $\mu_{\text{f.u.}}(H)$ of $Y(Co_{0.84}Fe_{0.16})_2$ indicates the presence of a strong paraprocess in it. In particular, in a magnetic fields range of 20–90 kOe the paraprocess susceptibility χ_{para} is determined as 0.050 emu/gOe for the $Y(Co_{0.84}Fe_{0.16})_2$, and in the sample with $R = $ Lu a weaker paraprocess is observed where $\chi_{\text{para}} = 0.004$ emu/gOe. This fact testifies the strengthening of d–d exchange interaction in case of Y replacement by Lu. A similar weak paraprocess was observed in the $Y(Co_{0.50}Fe_{0.50})_2$ compound [6] where the energy of the d–d exchange interaction is essentially higher due to greater Fe percentage in it. The values of $\mu_{\text{f.u.}}$ at 5 K and 90 kOe for all synthesized compounds are given in the Table.

From the analysis of the Table data, it follows that $Y(Co_{0.80}Fe_{0.20})_2$ and $Lu(Co_{0.84}Fe_{0.16})_2$ phases have the similar values of T_C and $\mu_{\text{f.u.}}$ Thus, comparing them with the $Y(Co_{0.84}Fe_{0.16})_2$ it can be argued that the Fe

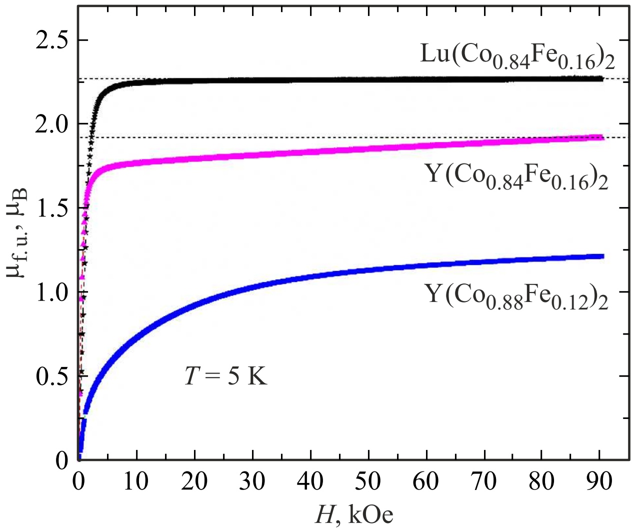

Figure 4. Magnetization curves of $Y(Co_{0.88}Fe_{0.12})_2$, $Y(Co_{0.84}Fe_{0.16})_2$ and $Lu(Co_{0.84}Fe_{0.16})_2$ compounds in Boron magnetons per formula unit ($\mu_{\text{f.u.}}$), measured at $T = 5$ K.

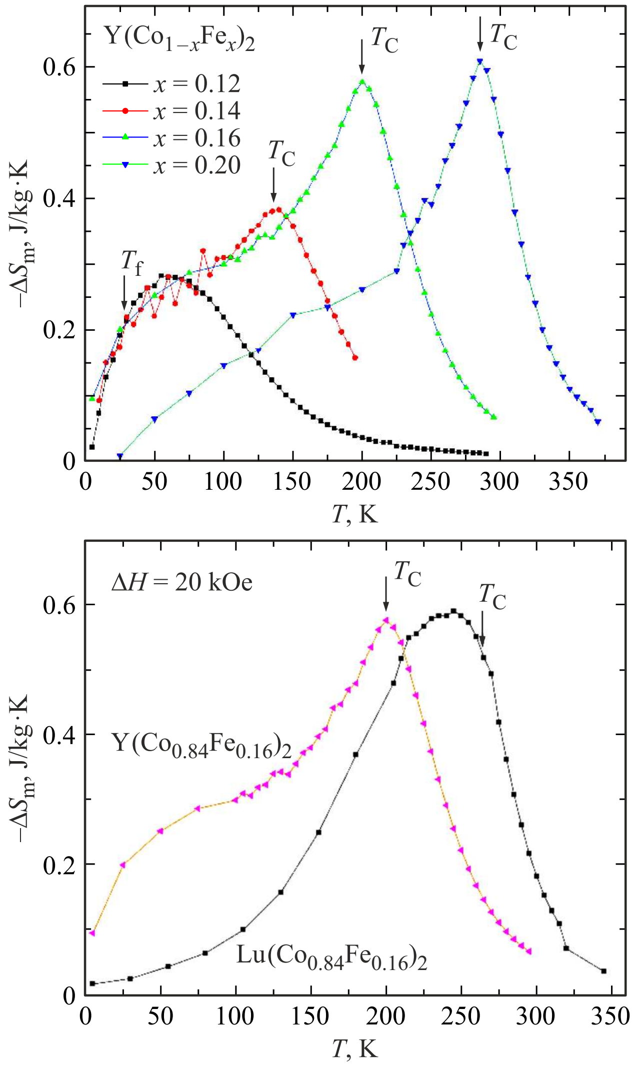

Figure 5. Temperature dependences of magnetic entropy change $\Delta S_m(T)$ of Y(Co$_{1-x}$Fe$_x$)$_2$ and Lu(Co$_{0.84}$Fe$_{0.16}$)$_2$ compounds at the external magnetic field changing from 0 to 20 kOe.

content increase or lattice parameters decrease (replacement of Y by Lu) leads to equivalent $d-d$ exchange interaction strengthening.

The magnetic entropy change was calculated from a series of magnetization isotherms using the formula [11]:

$$\Delta S_m(\Delta H, T) = \int_{H_s}^{H_f} \left(\frac{\partial \sigma}{\partial T}\right)_H dH, \tag{1}$$

where H_s is the minimum magnetic field value, H_f is the maximum magnetic field value, $\Delta H = H_f - H_s$, and T is the absolute temperature. Figure 5 shows obtained ΔS_m temperature dependences at the external magnetic field strength ΔH changes from zero to 20 kOe. It is clearly seen that low-temperature maximum $\Delta S_m(T)$ is present in all investigated samples, both in mictomagnetic Y(Co$_{0.88}$Fe$_{0.12}$)$_2$ compound and in ferromagnetic ones. An increase in the $d-d$ exchange interaction occurs both with an increase in the Fe content and with the substitution of Lu for Y, but at the same time leads to various changes of $\Delta S_m(T)$ curves shape. As x increases from 0.14 to 0.20, ΔS_m values increase at $T = T_C$ and ΔS_m values decrease in the vicinity of LTM. A different situation is observed in the compound with Lu. Taking the compound Y(Co$_{0.84}$Fe$_{0.16}$)$_2$ as a starting point, in comparison with it, the Y(Co$_{0.80}$Fe$_{0.20}$)$_2$ and Lu(Co$_{0.84}$Fe$_{0.16}$)$_2$ samples have large and, at the same time, close values of T_C. In Y(Co$_{0.80}$Fe$_{0.20}$)$_2$, an explicit maximum of ΔS_m at $T = T_C$ is observed, and at 100 K below T_C there is a plateau corresponding to LTM contribution to the MCE, at which the ΔS_m values are nearly 3 times lower than those at $T = T_C$. In the sample with Lu, LTM ΔS_m located at 25 K below T_C and ΔS_m maximum at $T = T_C$ merged to form a widened plateau. It follows that with decreasing lattice parameter, the Fe−Co exchange interaction is stronger and a higher temperature corresponding to a higher thermal fluctuation energy is necessary for appearance of LTM.

Taking Y(Co$_{0.84}$Fe$_{0.16}$)$_2$ and Lu(Co$_{0.84}$Fe$_{0.16}$)$_2$ compounds with non-magnetic Y and Lu elements and fixed Co : Fe ratio, let's consider in detail a situation with their MCE. Figure 6 gives the magnetic entropy change vs temperature dependences of these compounds normalized to T_C value ($\Delta S_m/\Delta S_{Tc}$) in a varying magnetic field. For the sample with $R = \mathrm{Y}$, the LTM on the $\Delta S_m(T)$ dependence is observed at $T \approx 100$ K, while in the sample with Lu at 240 K. The ΔS_m field dependences in the proximity of LTM differ in these samples. In the case of $R = \mathrm{Y}$, the $\Delta S_m/\Delta S_{Tc}$ at 100 K increases continuously with ΔH value rise. In Lu(Co$_{0.84}$Fe$_{0.16}$)$_2$, normalized ΔS_m value at $T = 240$ K increases only up to $\Delta H = 30$ kOe, then at higher ΔH it decreases, which may be due to the reduced susceptibility value lowering of the Co atoms magnetic sublattice at $H > 30$ kOe. This effect is clearly illustrated by insets in Figure 6, where the dependence $\Delta S_m(T)$ is calculated at $\Delta H = 20$ kOe, but with different values of H_f and H_s (triangles for $H_s = 0$ and $H_f = 20$ kOe, circles for $H_s = 70$ and $H_f = 90$ kOe). The figure shows that at $T = T_C$, ΔS_m values of both samples decrease with H_s value rise. At the same time, for Y(Co$_{0.84}$Fe$_{0.16}$)$_2$ sample ΔS_m remains practically constant at $T = 100$ K with increasing H_s value. This may indicate that the nature of LTM at $T = 100$ K is similar to that in the mictomagnetic Y(Co$_{0.88}$Fe$_{0.12}$)$_2$ compound, where a similar dependence of ΔS_m on H_s is observed, as shown in the inset of Figure 7.

From the comparison of $\Delta S_m(T)$ Y(Co$_{0.88}$Fe$_{0.12}$)$_2$ dependence with ones for compounds where $x = 0.14$ and 0.16 (given in Figure 5), it is seen that all these LTM are close in absolute values. From this, it may be supposed that the dependence of $\Delta S_m(T)$ for compounds with $x \geq 0.14$ is a superposition of the maximum at T_C due to the ferromagnetic−paramagnetic transition similar to the $\Delta S_m(T)$ peak for YFe$_2$ [12] and LTM due to the presence of mictomagnetic clusters and/or the appearance of magnetic moments induced by a magnetic field in some Co atoms,

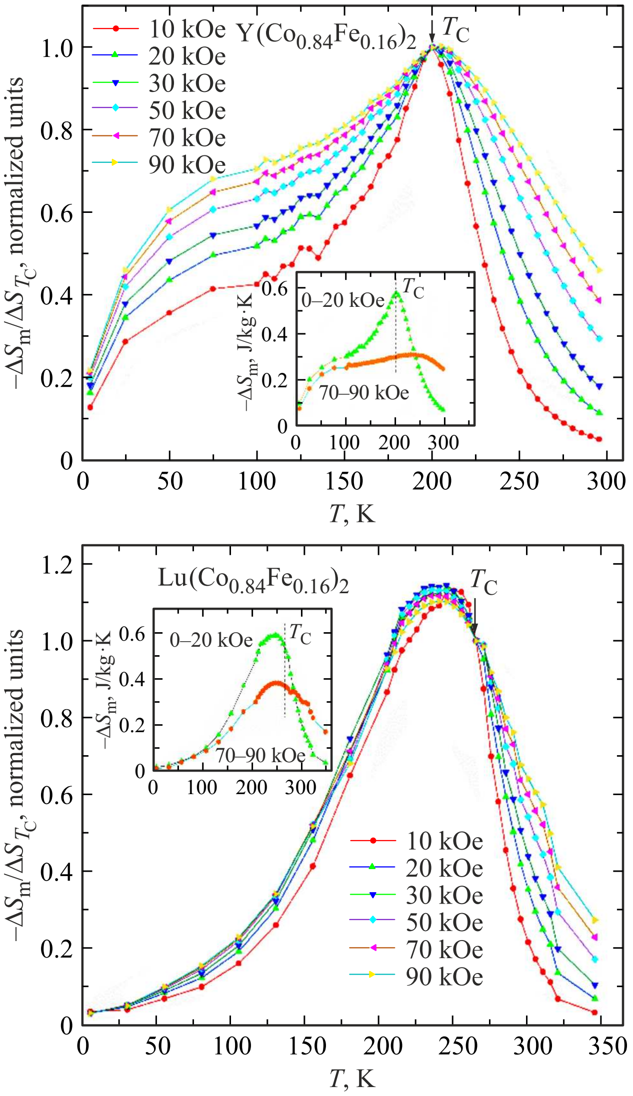

Figure 6. Temperature dependences of the magnetic entropy change in normalized units (respectively to that in T_C point) of $Y(Co_{0.84}Fe_{0.16})_2$ and $Lu(Co_{0.84}Fe_{0.16})_2$ alloys at different external magnetic fields. The insets show the dependences of $\Delta S_m(T)$ at $\Delta H = 20$ kOe calculated at different values of H_s.

disordered due to the weakness of the $d-d$ exchange interaction.

Let us consider in detail the MCE in $Y(Co_{0.88}Fe_{0.12})_2$ sample. In this sample, the long-range magnetic order is absent, and for explaining the nature of the MCE here it is necessary to consider that the sample in „magnetic sense“ is a paramagnetic matrix with ferromagnetic clusters included in it, in which the magnetic moments of the Co atoms are induced by the exchange field from the Fe atoms, which are known to retain their magnetic moment almost regardless of their surroundings [13]. Without an external magnetic field, the clusters are ordered antiferromagnetically. At $T > 0$ K and when H is applied, several processes are realized: the magnetic moment orientations of the cluster atoms are ordered along the external magnetic field, disorder caused by thermal fluctuations is suppressed, and the exchange (or molecular) field acting from the Fe atoms to the Co atoms is increased; the resulting magnetization increases and consequently, the ΔS_m values grow. Thus, the $\Delta S_m(T)$ dependence has a maximum value at $T > T_f$, as can be seen in Figure 7, which shows the temperature dependence of ΔS_m, normalized to the value of ΔS_m, at $T = 55$ K.

Figure 8 shows the averaged experimental data of temperature dependences of the adiabatic temperature change. The variation of experimental data with respect to the averaged values does not exceed ±0.05 K. Comparison of $\Delta T_{ad}(T)$ and $\Delta S_m(T)$ shows their qualitative similarity. For the $Lu(Co_{0.84}Fe_{0.16})_2$ sample, the dependence $\Delta T_{ad}(T)$ can

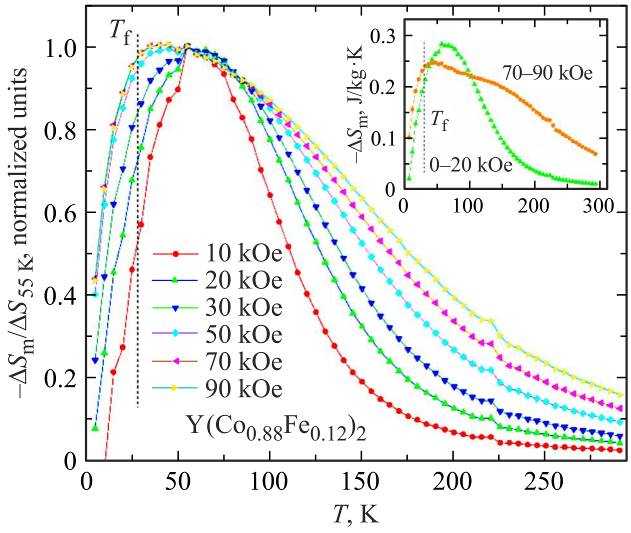

Figure 7. Temperature dependences of the magnetic entropy change in normalized units (respectively to that in $T = 55$ K) of $Y(Co_{0.88}Fe_{0.12})_2$ sample at different external magnetic fields. The insets show the dependences of $\Delta S_m(T)$ at $\Delta H = 20$ kOe calculated at different values of H_s.

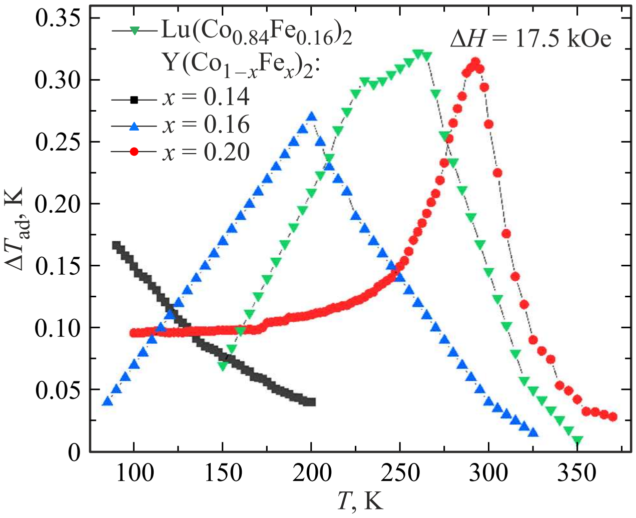

Figure 8. Temperature dependence of the ΔT_{ad} of $Y(Co_{1-x}Fe_x)_2$ and $Lu(Co_{0.84}Fe_{0.16})_2$ samples at $\Delta H = 17.5$ kOe.

clearly identify T_{C}, at which the maximum values of ΔT_{ad} is observed.

4. Conclusions

In the study of magnetic and magnetocaloric properties of $\text{Y}(\text{Co}_{1-x}\text{Fe}_x)_2$, $x = 0.12$, 0.14, 0.16, 0.20, and $\text{Lu}(\text{Co}_{0.84}\text{Fe}_{0.16})_2$ compounds, it was found that Fe concentration increasing and/or Y replacing by Lu with unchanged proportion of Co and Fe atoms increases the energy of $d{-}d$ exchange interaction, accompanied by Curie temperature rise. This leads to the suppression and shift to higher temperatures of a low-temperature maximum of $\Delta S_m(T)$ dependence. This phenomenon is similar to that observed in $R(\text{Co}_{0.88}\text{Fe}_{0.12})_2$ ferrimagnetics with magnetoactive rare-earth elements [1]. The $\Delta S_m(T)$ dependence evolved from a table-like to a classical peak when the energy of $4f{-}3d$ exchange interaction is increased, and a such R magnetic sublattice was termed as „weak". By analogy with this, in $\text{Y}(\text{Co}_{1-x}\text{Fe}_x)_2$ compounds at $x = 0.12{-}0.20$ as well as in $\text{Lu}(\text{Co}_{0.84}\text{Fe}_{0.16})_2$ one a magnetic subsystem of cobalt atoms acts as a „weak" sublattice.

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Conflict of interest

The authors declare that they have no conflicts of interest.

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