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Magnetic properties and magnetocaloric effect in Gd films and microwires

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An analysis of the temperature and field dependences of the magnetization of Gd films and microwires was carried out, as well as an isothermal measurement of the magnetic part of the entropy at the Curie temperature. The magnetocaloric effect (MCE), measured as an isothermal increase in the magnetic part of the entropy, in microwires shows two peaks on the temperature dependence of the magnetic part of the entropy, in contrast to one peak in films. In films and microwires, the entropy maximum at 286−293 K, which corresponds to the Curie temperature, depends on the magnetic field, shifting in the same way in films and microwires with an increase in the field at the orientation of the MgO (111) substrate, which provides the maximum mechanical stresses in Gd. In microwires, the second maximum does not change the temperature of 320 K as the field increases to 9 T, but its amplitude increases linearly with the field. This maximum can be caused by a spin-reorientation transition.

Keywords: microwires, thin films, magnetic entropy, magnetic anisotropy, spin-reorientation transition, Curie temperature.

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1. Introduction

It is known, that the energy spent for cooling in the world exceeds that spent for heating. The chemical hazard of volatile gases together with the complexity and fragility of the developed network of channels in refrigerating chambers simulates the search for more environmentally friendly technologies. For example, now the strategy of solids cooling with the use of mechanical deformation [1] or magnetic field [2] is developed extensively. The magnetocaloric effect (MCE) in ferromagnets and other magnetic-ordered structures has a special practical importance because it is able to manifest at temperatures close to the room temperature and provide a significant decrease in working medium temperature by 20−30 K [3–6]. After the discovery of anomalously high change in the magnetic part of entropy ΔS_M in gadolinium and its alloys [3–6], it became clear that these materials are suitable for the development of new technologies of magnetic machines. To improve the magnetocaloric alloys of gadolinium, their chemical modifications are mainly used by adding other transition and rare-earth metals to the to gadolinium, as well as applying heat treatment that allows achievement of the optimum phase composition. Since the search for materials with increased MCE have been going on for a long time, these two approaches are largely exhausted.

One more relatively modern approach to the improvement of such alloys consists in the search for MCE in nano- and microstructures of the alloys that demonstrate the best properties in the macroscopic state [7–10]. This is due to the following additional factors, which are able to optimize MCE in nano- or microstructures: high microstresses, the presence of long-lived nonequilibrium metastable phases that do not exist in macroscopic samples, the considerable improvement of heat exchange in a finely dispersed medium composed of micro- or nanostructures.

In addition, the considerable anisotropy of magnetization in microwires and films arising due to the texture of the material can be used to improve the refrigerating machine design. In particular, in [11,12] it is shown, that a change in the crystal-lattice orientation of the gadolinium film substrate and in appropriate internal stresses created by the substrate in the film, can result in a significant change in MCE. Bulk gadolinium samples have significantly lower crystal anisotropy of magnetization that the anisotropy observed in films and microwires. In [13] authors reported a complicated temperature dependence of magnetic anisotropy found in gadolinium microwires, which contained two maxima of entropy that indicated a complicated-stressed

state or a two-phase structure. A considerable MCE can be achieved in nano- and microstructures not only by placing the sample into a magnetic field or switching on the magnetic field, but by varying the permanent magnetic field orientation in relation to the sample. This provides for avoiding the energy-intensive procedure of switching-on the field or moving the sample, allowing simply rotate it in the magnetic field of a permanent magnet. The comparison of MCE in microwires and films presented in [11–13] shows that with constant chemical composition and temperature modes of their preparation, micro- and nanostructures can demonstrate MCE with considerably different magnitude, which is caused by variations of the phase composition and internal microstresses. Indeed, the MCE estimate should also take into account the factor of demagnetization, which depends on the sample's shape, as it is convincingly demonstrated in [14,15]. The main difference between microstructures and nanostructures in terms of their use in MCE-devices is evidently related to different mechanisms of magnetic reversal. In nanostructures, as a rule, a single-domain structure is observed, and magnetic reversal is realized by an instantaneous change in the direction of magnetization. In microstructures usually the limit of single-domain state is not achieved, and the magnetic reversal is realized by means of nucleation of magnetization nuclei or by domain wall spreading. This results in the situation that domains become involved in the magnetic reversal process, which dynamics has a significant effect on the energy balance of working medium in the refrigerating machine.

The objective of this study is to compare structures, phase and chemical compositions, magnetic properties and parameters of the magnetocaloric effect in submicron Gd films and microwires.

2. Procedure and samples

In MCE experiments, usually the researchers carry out the investigations in two limit modes [1–5]:

– in the adiabatic mode, when the changing in magnetic field is as fast as to make the consequent spin orientation reversal in the sample resulting in a change in its temperature ΔT, and the magnetic part of entropy is kept constant $\Delta S_M = \text{const}$;

– in the isothermal mode, when the changing in the external magnetic field is as slow as to keep the working medium temperature constant and equal to the temperature of the thermal reservoir where the ferromagnet is located, $\Delta T = \text{const}$. In this case a considerable changing in the magnetic part of entropy takes place, which is related to the changing in magnetization M by known Maxwell relations [16].

Indeed, both cases of MCE detected by the change in temperature or by the magnetic part of entropy yield characteristics that are directly proportional to each other, and an increase in one of them by modifying the working medium leads to a proportional increase in another one.

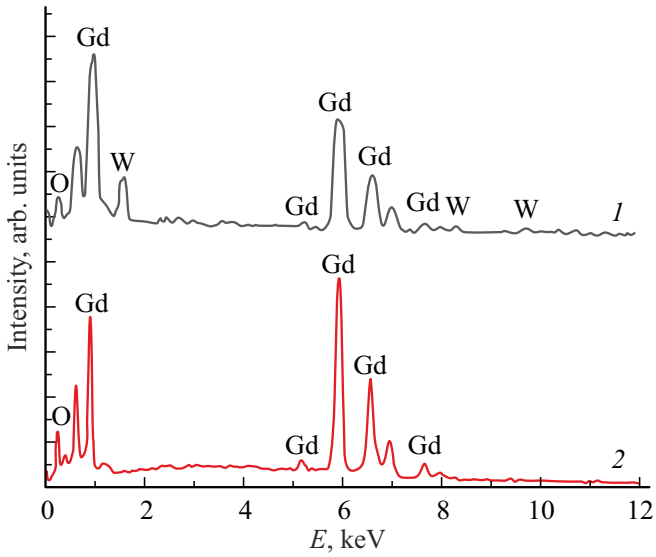

Figure 1. Energy-dispersive spectra EDX of a 300 nm gadolinium film on a MgO (100) substrate as part of a W/Gd/W/MgO layered heterostructure (*1*) and a Gd microwire (*2*).

In this study for the both cases of gadolinium films and microwires we have chosen the isothermal mode, which has been implemented in the helium atmosphere of a SQUID MPMX Quantum Design magnetometer with a thermal control function to an accuracy of 0.1 K. The magnetic field of 0−5 T was changing slowly during the transition from one value to another, so the sample temperature always succeeded to equalize when the isothermal field dependencies of magnetization $M(H)$ were recorded. In some experiments we also used a Lake Shore vibration magnetometer where maximum magnetic filed achieved a level of 9 T.

W/Gd/W/MgO film heterostructures were produced by high vacuum magnetron sputtering deposition as described in [11,12]. Thickness of the gadolinium layer was 300 nm. MgO with crystal-lattice orientation (100) was used as a substrate. The gadolinium microwires were produced by extraction of a hanging melt drop followed by superfast cooling on a water-cooled rotating disc [13].

The local chemical composition of Gd microwire and thin films at a depth of ~ 10 nm was estimated by the method of energy-dispersive analysis (EDX). EDX spectra of films and microwires are shown in Fig. 1. Gd, Mg, O and W elements are clearly distinguished in the EDX-spectrum of a thin film, that confirms the declared composition of the heterostructure samples under study (spectrum *1* in Fig. 1). In the microwire EDX-spectrum (spectrum *2* in Fig. 1) responses of gadolinium and oxygen can be distinguished. The presence of the oxygen line in the spectra of films and microwires may be indicative of the fact that part of the material turned to be oxidized.

To determine the structure od samples, the X-ray diffraction (XRD) analysis was used. XRD spectra of a thin film

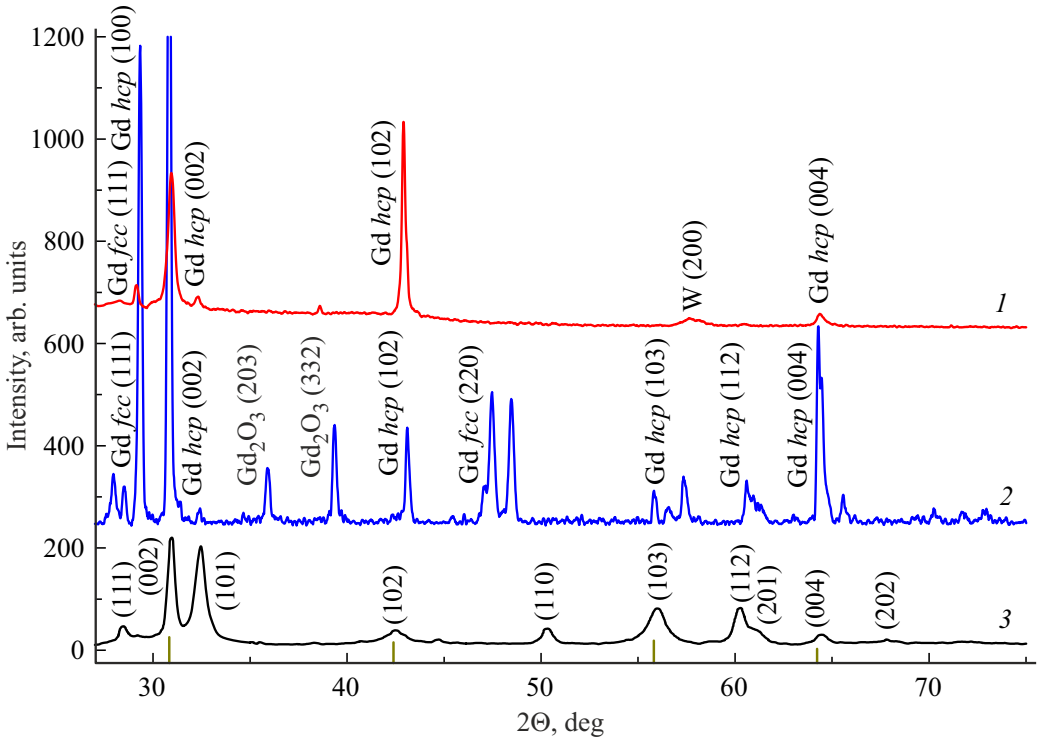

Figure 2. X-ray diffraction spectra: *1* — films with a Gd layer thickness of 300 nm and a MgO substrate orientation of (100); *2* — Gd microwires; *3* — thick film of gadolinium in [17]. Vertical dashes on the horizontal axis show positions of peaks of the polycrystal Gd from the X-ray diffraction database (PDF4+ card № 01-080-6667).

with a thickness of 300 nm (line *1*) and a microwire (line *2*) are shown in Fig. 2.

The main stable phase in Gd thin films and microwires is the hexagonal close-packed (*hcp*) phase with parameters $a = 3.606$ Å and $c = 5.786$ Å for a bulk material. The face-centered cubic phase (*fcc*) with a lattice parameter $a = 5.336$ Å, presenting in the samples, is a metastable phase and is frequently detected in thin films and microstructured samples of gadolinium [18].

In thin films of gadolinium grown on a MgO substrate, a stable *hcp* phase of Gd and a metastable *fcc* phase are formed. In the produced heterostructures internal stresses arise at the interphase boundary in the Gd layer that can result in a change of magnetic entropy. In Gd microwires, as a result of fast quenching, *hcp* and *fcc* phases of gadolinium and Gd_2O_3 gadolinium oxide are formed. The presence of Gd oxide is related to the presence of oxygen in the chamber during the fast quenching from the melt.

3. Experimental findings

Fig. 3 shows temperature dependencies of magnetization for film (curve *1*) and microwire (curve *2*) in a magnetic field of 10 kOe. The dashed line shows the saturation magnetization for the bulk gadolinium at room temperature (dashed line *3*) known from literature [1–5]. It can be seen that the magnetization of microwires is higher than the magnetization of films. Only at low temperatures the magnetizations of films and microwires become close to each other. Two critical temperatures can be seen in films: one near 290 K corresponds to the Curie temperature of gadolinium and another at 20–30 K is closer to the Curie temperature of the Gd_2O_3 oxide or the *fcc* metastable cubic phase identified by the X-ray diffraction analysis (Fig. 2). These features are not observed on the temperature dependence of magnetization in microwires. The lower magnetizations as compared with the known values can be explained by the fact that the field of 10 kOe is not a saturation field at high temperatures, but approaches it with cooling.

Fig. 4 shows dependencies of the magnetization of Gd thin film on a MgO (100) substrate and Gd microwire versus an external magnetic field applied along its axis. These series of dependencies are recorded in the isothermal mode with a step of 5 K for sample heating from 250 K followed by heating to 350 K (above the Curie temperature of 294 K). This heating procedure unifies the initial state, from which the recording of $M(T)$ is started. It can be seen, that at high temperatures the magnetization of microwires and films does not reach the saturation, and with the decrease in temperature the level of saturation magnetization in microwires and films is different, which is probably caused by different phase composition: the presence of the cubic metastable phase in films and gadolinium oxide in microwires.

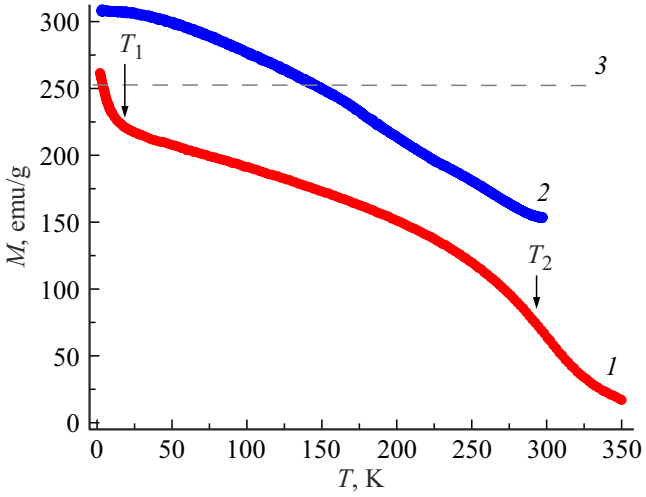

Figure 3. Temperature dependencies of magnetic moment *1* — for 300 nm Gd film on a MgO (100) substrate and *2* — for Gd microwire written in a field of 10 kOe (FC). The horizontal dashed line shows for comparison the saturation magnetization at 300 K for pure bulk gadolinium according to [1–5]. Arrows show critical temperatures of transitions.

The MCE magnitude can be detected by the change in magnetic part of entropy ΔS_M, determined by the magnetization derivative with respect to temperature added at different fields up to the current final value. This relation is established by the Maxwell relation [16]:

$$\Delta S_M(T, H) = \int_0^H \left(\frac{\partial M}{\partial T}\right)_H dH. \quad (1)$$

Since experimental points of the $M(H)$ dependence are recorded in a discrete form, usually the following formula is used instead of expression (1) [16]:

$$\Delta S_M(T, H) = \sum_i \frac{M_{i+1}, (T_{i+1}, H) - M_i(T_i, H)}{T_{i+1} - T_i} \Delta H. \quad (2)$$

Fig. 5 shows an example of one of such dependencies calculated by us from formula (2) for a series of field dependencies $\Delta S_M(T)$ in a film of gadolinium on a MgO (100) substrate. To determine parameters of $\Delta S_M(T)$ dependencies, it was necessary to select a model of approximation that made it possible to determine amplitude and width of $\Delta S_M(T)$ peaks. We used two approaches developed within the theory of molecular field, where the phonon part of entropy is written in accordance with the Debye model [19] and the mean field model, where the exchange interaction is represented in the form of internal field added to the external field in the Brillouin function [20]. Solid lines (*1*) and (*2*) in Fig. 5 show approximations by corresponding functions suggested in [19,20].

It can be seen that theoretical considerations taking into account the dispersion of exchange interaction and phonon spectrum in the above-mentioned models predict considerably narrower dependencies $\Delta S_M(T)$ that those observed in the experiments. Hence, width of the maximum of $\Delta S_M(T)$ dependence is determined not by internal physical causes related to the magnetism of the crystal lattice or phonon spectrum, but by more rough mechanisms related to the heterogeneity of samples. Therefore, to approximate the $\Delta S_M(T)$ dependencies in films and in microwires, we used the Gaussian function (Fig. 5 curve *3*). In films, after subtraction of the linear background, the $\Delta S_M(T)$ dependence was approximated by one Gaussian function (Fig. 6, *a*), and in microwires it was approximated by two such functions (Fig. 6, *b*).

It can be seen from Fig. 6, *a* that the maximum of entropy in films has almost no shift with increase in field, while its amplitude grows. It follows from Fig. 6, *b*, that the high-temperature maximum in microwires at T_{c1} has no shift, while the low-temperature maximum at T_{c2} shifts toward

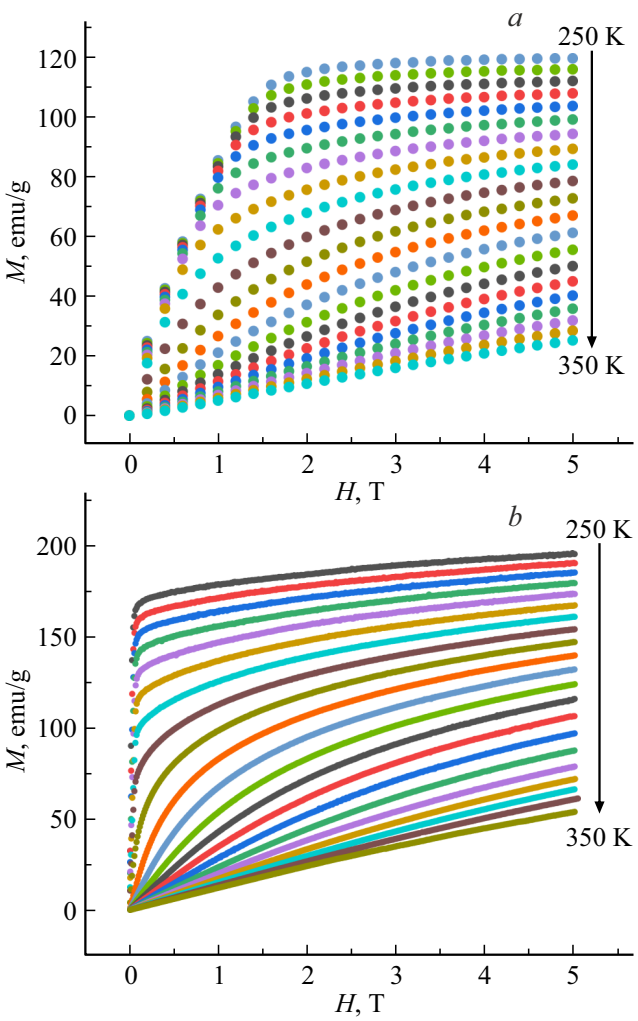

Figure 4. Dependencies of magnetization versus magnetic field for a 300 nm Gd film on a MgO (100) substrate (*a*) and a Gd microwire (*b*) recorded with a step of 5 K in a temperature range of 250–350 K. The magnetic field lays in the film plane and along the microwire.

higher temperatures with increase in field. Positions of maxima as functions of temperature are shown for both maxima in the microwire (curves *1* and *2* in Fig. 7), as well as for the Gd film with two thicknesses of 100 nm and 300 nm, deposited on MgO substrates with different crystal-lattice orientation.

Fig. 8 shows dependencies of full width at half maximum (FWHM) of curves $\Delta S_M(T)$ versus magnetic field for the first (*1*) and the second (*2*) maxima in Gd microwire and Gd films of different thicknesses on MgO substrates with different crystal-lattice orientation (curves *3*–*8*).

It can be seen, that with increase in field widths of maxima grow significantly, except for the maximum *2* in microwires. This is indicative of the growth of relative cooling power (*RCP*), which is proportional to the area of peak on the $\Delta S_M(T)$ curve. In addition, amplitudes of peaks increase with the field growth.

For example, amplitude of both maxima $\Delta S_{\max}$ in microwires increase rapidly with field growth, however in the region of weak fields these dependencies are different (Fig. 9). The amplitude of peak (*1*) grows quasi-linearly, varying in a range of 4–15 J/kg K with a coefficient of 1.83 J/kg K T (Fig. 9). Usually the magnitude of MCE, as determined by the change in entropy, follows the field dependence that has a form of the approximation by the function of $\Delta S_M(T) = a + bH^{2/3}$, where a, b are coefficients independent from the field [10]. Approximations of $\Delta S_M(T)$ dependencies by this functions for both maxima in microwires are shown in Fig. 9 by solid lines. It can be seen, that only the low-temperature maximum follows this pattern. Since the $\Delta S_M(T) \sim H^{2/3}$ dependence is a result of application of the mean field theory to the ferro-paramegnetic transition, its applicability to the low-temperature maximum *2* means that this maximum corresponds to the Curie temperature. It is evident, that the spin-flip transition is described by another field dependence, that depends on magneto-elastic constants, demagnetization field and other factors.

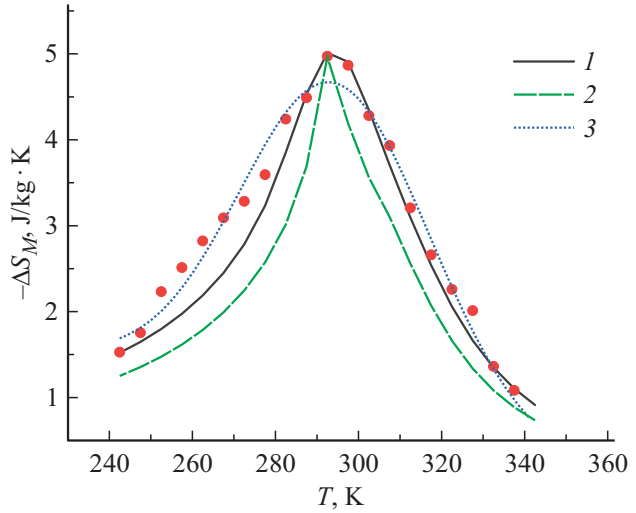

Figure 5. Change in the magnetic part of entropy for a sample of 100 nm Gd film on a MgO (100) substrate in the applied magnetic field of $H = 5$ T. Dots show changes in entropy obtained experimentally. Solid lines show approximations calculated using the mean field model (*1*), the Debye model (*2*) and the Gaussian function (*3*).

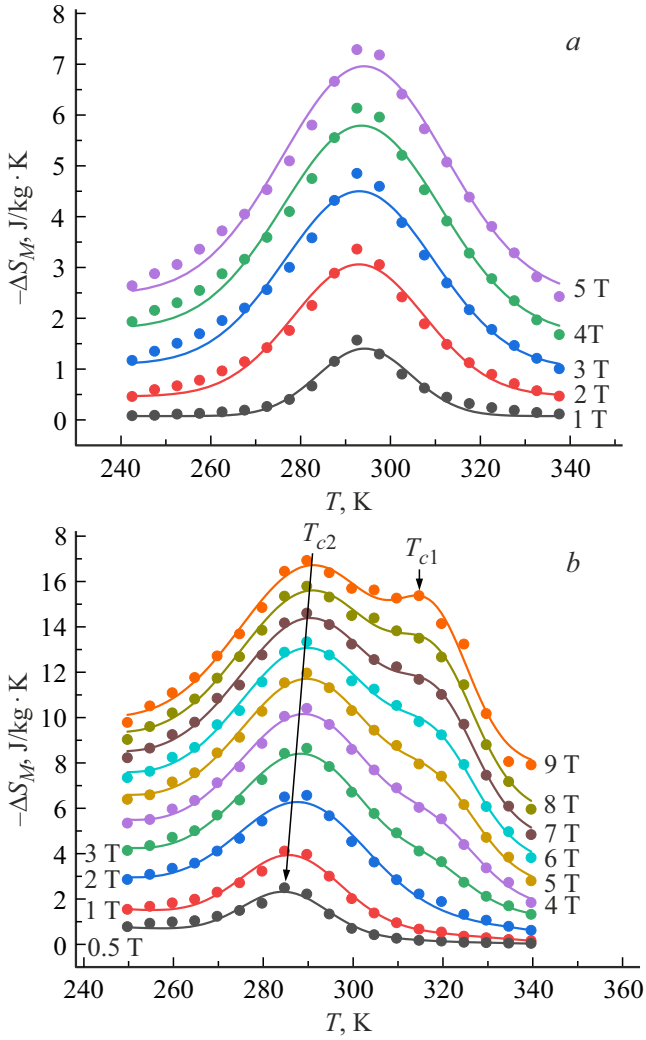

Figure 6. Temperature dependencies of the change in magnetic part of entropy *a* — for 300 nm Gd film on a MgO (100) substrate in magnetic fields specified rightward to curves, *b* — for Gd microwire. Dots show changes in entropy obtained experimentally. Solid lines show approximations by Gaussian functions.

4. Discussion

Double maxima of the increment of magnetic part of entropy in alloys with gadolinium are not uncommon in the literature. For example, in [21] authors reported the presence of a double peak in the $Gd_{1-x}Ho_xNi$ alloy that was produced by the rapid cooling melt-spinning process. The authors have found, that an additional maximum of entropy arises as Ho is added, and this maximum is absent at a zero concentration of Ho. Since Ho has a symmetry of single-

ion anisotropy different from that of Gd, a competition of magnetic anisotropies of two rare-earth sublattices takes place. With a change in temperature, the prevalence of Gd symmetry is changed to the symmetry of Ho ions and

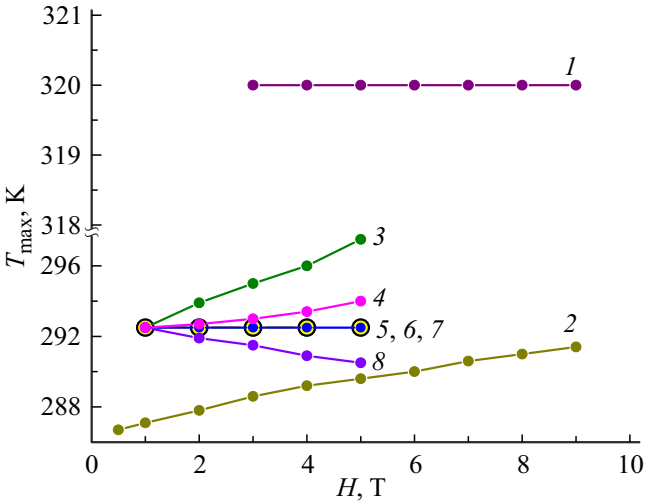

Figure 7. Dependencies of maxima temperatures of the magnetic part of entropy T_{max} versus magnetic field for Gd microwire (*1, 2*) and Gd films of different thicknesses on different substrates (*3–8*): *3* — film with a thickness of 100 nm on a MgO (111) substrate; *4* — film with a thickness of 300 nm on a MgO (111) substrate; *5* — film with a thickness of 100 nm on a MgO (100) substrate; *6* — 100 nm film on a MgO (110) substrate; *7* — 300 nm film on a MgO (100) substrate; *8* — 300 nm film on a MgO (110) substrate.

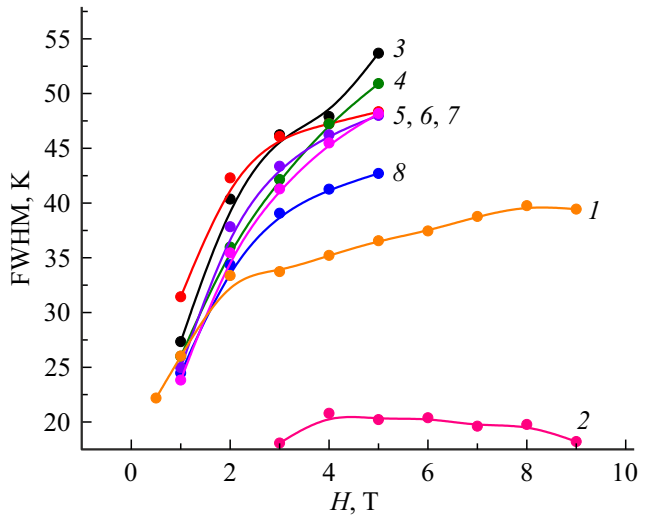

Figure 8. Dependencies of full width at half maximum (FWHM) of curves $\Delta S_M(T)$ versus magnetic field for the first (*1*) and the second (*2*) maxima in Gd microwire and Gd films of different thicknesses on different substrates (*3–8*): *3* — film with a thickness of 100 nm on a MgO (100) substrate; *4* — film with a thickness of 100 nm on a MgO (111) substrate; *5* — film with a thickness of 100 nm on a MgO (110) substrate; *6* — 300 nm film on a MgO (110) substrate; *7* — 300 nm film on a MgO (111) substrate; *8* — 300 nm film on a MgO (100) substrate.

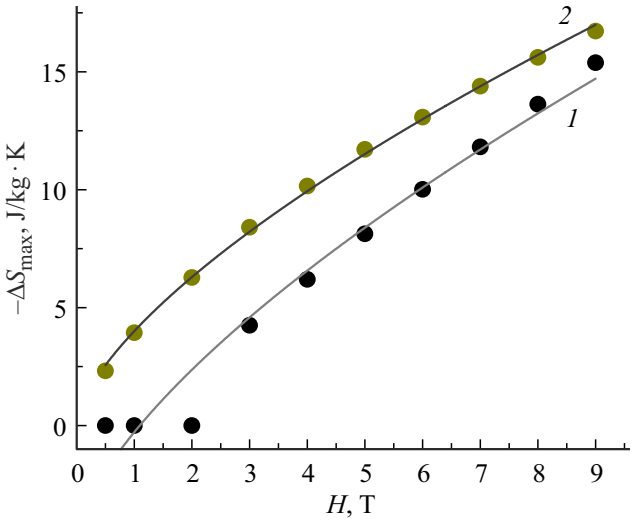

Figure 9. Dependence of amplitudes of maxima (*1*) and (*2*) of the magnetic entropy in the Gd microwire (Fig. 6, *b*) versus magnetic field as a result of expansion to two Gaussian functions. Solid lines show approximations by the function of $\Delta S_M(T) = a + bH^{2/3}$.

a spin-flip transition takes place, which, as well as the transition at Curie temperature, gives an increase in the magnetic part of entropy.

In a zero magnetic field the Curie temperature of bulk gadolinium monocrystal T_c is 294 K, and the spin-flip temperature T_r is 227 K. Authors of this study have found that there are two closely located maxima of anisotropy, one of which corresponds to the Curie temperature of gadolinium, and another corresponds to the spin-flip in gadolinium. At the same time, in a range of 2–7.5 T the T_c in Gd increases linearly with a coefficient of 6 K/T, the spin-flip transition is extinguished completely by magnetic fields higher than 2–2.5 T.

In [4] it is noted, that chemical purity of gadolinium plays an important role in the MCE and has a great effect on both the T_c and the T_r. Usually, there is a large number of impurities even in samples with a commercial grade of 99.9 wt.%. Impurities decreases the Curie temperature and broaden the temperature interval of paramagnetic-ferromagnetic transition, masking the spin-flip transition, which can be easier detected in monocrystal samples of high quality. Therefore, the critical temperatures found by us for microwires and films can be shifted in relation to their values in perfect crystals of gadolinium.

It can be expected that peak *1* at 320 K corresponds to the spin-flip transition shifted from 235 K as a result of microstresses and high percentage of impurity in microwires (Fig. 6, *b*). The low-temperature peak *2* in microwires changes its position monotonously from 286 to 293 K in a field range of 3–9 T (curve *2* in Fig. 6, *b*), i.e. approximately with a coefficient of $dT_c/dH = 1.17$ K/T, which is considerably lower than the coefficient of $dT_c/dH = 6$ K/T determined in [4], where the entropy maximum corresponded

to the Curie temperature. In our study also peak *2* at 286−293 K probably corresponds to the Curie temperature shifted to toward higher temperatures due to high anisotropy of shape and strong microstresses arising at a high rate of melt cooling.

In films with MgO substrate orientation (111) that provides maximum Gd stretching, a growth of the Curie temperature is observed in a field range of 1−5 T with a coefficient of $dT_c/dH = 1.25$ K/T close to that observed in microwires (Fig. 7, curve *3*). Changes in the substrate orientation to (110) or (100) or its thickness from 100 to 300 nm decrease the stresses in gadolinium, thus lead to a zero coefficient or even to a negative coefficient (Fig. 7, curves *4*−*8*). The temperature of peak *1* does not depend on field and remains equal to 320 K at field up to 9 T.

Spin-flip in bulk samples of Gd is well-known and reliably verified by the neutron diffractometry method (see, for example, [22]). The easy magnetization axis in Gd is parallel to the hexagonal axis between the Curie temperature ($T_c = 293$ K) and the spin-flip temperature ($T_r = 235$ K). The easy magnetization axis moves away of the c axis below T_r and lays on the surface of the axis cone at an angle, which is equal to 30−40° at low temperatures, increases approximately up to 70−90° in the region of 150−200 K, then decreases abruptly down to 0° at T_r.

The effect of the spin-flip transition on the MCE was investigated in detail in [4] in gadolinium samples of different purity and different structural perfection (from poly- to monocrystal). The spin-flip transition temperature in different sources is specified as 227 K [4], 232 K [22], etc., while its position may be significantly shifted under the effect of stresses [23]. The theoretical coefficient of temperature shift T_r under an applied hydrostatic pressure P is $dT_r/dP = -4.9\,\mathrm{K}\cdot\mathrm{kbar}^{-1}$, which is close to the experimental value of $dT_r/dP = -4.3\,\mathrm{K}\cdot\mathrm{kbar}^{-1}$ [23]. Negative value of this coefficient does not allow for explanation of the T_r temperature shift to 320 K in our study under a hydrostatic pressure, however the stresses in microwire are not of hydrostatic nature and significantly vary probably creating a mosaic of areas with stresses of different directions. Gradients of these stresses can cause a significant shift toward higher temperatures, as it is found in our experiments. It is worth noting that below the Curie temperature in gadolinium a significant effect of magnetic field on the Young modulus is observed [24], which can result in significant changes in the magneto-elastic anisotropy, changing the constant of anisotropy K_1. When a field is applied, a change in the elastic constant c_{33} is observed in gadolinium, which corresponds to softening of the crystal lattice, that is also confirmed by calculations within the theory of molecular field and indicates that this effect is related to magneto-elastic constants [24].

It must be noted that the balance of different kinds of magnetic anisotropy includes not only the crystalline and magneto-elastic anisotropy, but also the anisotropy of shape, which can, for example, result in a significant shift of the Curie temperature and a change in MCE magnitude [25]. In microwires this anisotropy of shape can considerably shift critical temperatures T_c and T_r.

5. Conclusions

1. Two peaks (low-temperature and high-temperature) are observed on the temperature dependence of the magnetic part of entropy microwires in contrast to one peak in Gd films.

2. In gadolinium films and microwires a low-temperature maximum of the change in the magnetic part of entropy is observed near the Curie temperature of 286−293 K reported in the literature. This maximum in microwires is shifted toward higher Curie temperatures with a coefficient of 1.17 K/T, which is considerably less than the coefficient of 6 K/T determined for bulk gadolinium. In films a differently directed shift of the maximum is observed with an increase in field depending on the crystal-lattice orientation of the substrate and the magnitude of stresses caused by it. This shift is caused by the absence of magnetization saturation up to the highest field strengths and growth of the demagnetization field as the external field increases.

3. A significant effect of magnetic field on the position of maximum of the entropy dependence on temperature is found in films in contrast to microwires, where this dependence is only observed for one of peaks. The highest effect of magnetic field on the position of entropy maximum in films is observed for the MgO substrate orientation of (111) at the lowest thickness of the Gd film, i.e. in the conditions of maximum stresses created by the substrate. At the same time, the coefficients of linear shift of the entropy maximum with field growth coincide in films and in microwires within the accuracy of the experiment.

4. The high-temperature peak in Gd microwires does not change its position and is located at 320 K, however its amplitude grows linearly with the field and varies in a range of 4−15 J/kg K with a coefficient of 1.83 J/kg K T. The probable mechanism of the high-temperature peak arising is the spin-flip, which is considerably shifted from low temperatures to high temperatures under the effect of high internal stresses and with a growth of the demagnetization field in microwires up to 9 T.

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Conflict of interest

The authors declare that they have no conflict of interest.

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