Journal of Magnetics 3(4), 105-111 (1998)

SWR as Tool for Determination of the Surface Magnetic Anisotropy Energy Constant

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(Received 18 August 1998)

The low energy excitations of spin waves (SWR) in thin films can be used for determination of the surface anisotropy constant and the nonhomogeneities of magnetization in the close-to-surface layer. The dispersion relation in SWR is sensitive on the geometry of experiment. We report on temperature dependence of surface magnetic anisotropy energy constant in magnetic semiconductor thin films of $CdCr_{2-2x}In_{2x}Se_4$ at spin glass state. Samples were deposited by rf sputtering technique on Corning glass substrate in controlled temperature conditions. Coexistence of the infinite ferromagnetic network (IFN) and finite spin clusters (FSC) in spin glass state (SG) is know phenomena. Some behavior typical for long range magnetic ordering is expected in samples at SG state. The spin wave resonance experiment (microwave spectrometer at X-band) with excited surface modes was applied to describe the energy state of surface spins. We determined the surface magnetic anisotropy energy constant versus temperature using the surface inhomogeneities model of magnetic thin films. It was found that two components contribute to the surface magnetic anisotropy energy. One originates from the exchange interaction term due to the lack of translation symmetry for surface spin as well as from the stray field of the surface roughness. The second one comes from the demagnetizing field of close-to surface layer with grad M. Both term linearly decrease when temperature is increased from 5 to 123 K, but dominant contribution is from the first component.

1. Introduction

Diluted by indium $CdCr_2Se_4$ spinels posses the modified energetic structure due to the nonzero density of state energy gap. Then we have some energy levels and/or narrow bands in the forbidden area. Such systems are of much interest for basic research as well as for their application as in photodetectors devices.

The $CdCr_{2-2x}In_{2x}Se_4$ thin films of magnetic semiconductors are very complex systems made up of four compounds, then some technological problems are arising to get the homogeneous and with good adhesion samples. On the basis of four technological (*rf* sputtering deposition) experiences [1] we have found that the best samples are obtained from the as-cast multilayered structure of Cr/Cd-Cr-In-Se/Cr in the amorphous state. The heat treatment finally gives the polycrystalline samples of $CdCr_{2-2x}In_{2x}Se_4$. In fact it is multilayered structure with close-to-surface layers exhibiting more amount of chromium respect to the inner uniform part of sample.

The dilution level of $CdCr_{2-2x}In_{2x}Se_4$ film controls the magnetic phase. The phase diagram predicts the with reentrant transition (REE) and spin glass (SG) state. The magnetic state with reentrant transition is characterized by high fluctuations of the exchange constant at the temperature close to REE transition, then when temperature decreases the system evolves into SG state. The spin systems with REE transition and at SG state on the microscopic scale are considered as consisting of IFN and FSC randomly distributed in sample. The ratio of IFN to FSC is sensitive on the dilution level; FSC are getting dominant over IFN when dilution level increases. For all compositions of thin films at SG state we have investigated, the amount of IFN was enough to exhibit some ferromagnetic properties. Coexistence of spin glass and ferromagnetic ordering was described in several papers [1-3]. Some evidence of IFN was confirmed in the ferromagnetic resonance (FMR) and spin waves resonance (SWR) experiments which we have performed at X-band of a microwave spectrometer [4]. The excitation of volume modes at the SWR needs long range magnetic ordering. An interaction between the IFN and FSC was also expected and alters the position and linewidth of the uniform mode in FMR experiment [5]. The state with REE and SG are characterised by typical temperature dependence of magnetization which is influenced by non-zero density of state in the energy gap.

For both type of magnetic ordering the properties have

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revealed the magnetic anisotropy field H_{an} maintaining the remanent magnetization in the direction of the applied external field. This is due to Ruderman-Kittel-Kasuya-Yosida (RKKY) interaction which is of the Dzyaloshinsky-Moriya (DM) type [6, 7]. Spin-orbit scattering of the conduction electrons by the nonmagnetic impurities is the source of this interaction [8, 9]. DM interaction between spins yields a purely unidirectional macroscopic magnetic anisotropy energy. This energy yield to the unidirectional magnetic anisotropy field H_{an}, which altered the position of resonance peak in the ferromagnetic resonance data and also the *M-H* loops measurements [10, 11].

Practically you can not avoid, during the deposition process producing a close-to-surface layer with grad*M*. Such nonhomogeneity creates the surface magnetic anisotropy energy being different from the magnetic anisotropy of the inner part of sample volume. These extra conditions for surface altered the magnetic and transport properties of thin films. There are not any direct experimental techniques for determination the gradient of magnetization in the close-to-surface layers as well as the surface magnetic anisotropy energy constant. The spin wave resonance technique (SWR) is a way of studying the magnetic properties of surface. For particular boundary conditions the surface modes could be excited in the SWR experiment. In the case of thin films when one of the dimensions drastically reduced the contribution of the surface magnetic anisotropy energy to the total volume magnetic anisotropy energy could be significant. The excitations of surface modes are controlled by the energy state of surface spins and the close-to-surface inhomogeneities of magnetization.

Spin wave resonance was detected with an X-band microwave spectrometer within the temperature range from 4 K to 125 K. External magnetic field was applied for directions from perpendicular to parallel to the film plane. At perpendicular case we have got the two modes, one with smaller intensity at higher external magnetic field H_s with comparison to that at lower field H_u. According to the prediction of theory [12] the mode at higher field is the surface one. The excitation of surface modes in thin films at SG state is possible because of the IFN is present. Due to the dominant amount of chromium atoms coming from buffor and top layers (see paragraph2) one can expect that the close-to-surface part of sample has higher ratio of IFN to FSC with comparison ti the inner part of film.

The boundary conditions for surface modes excitation are best described within the model of surface inhomogeneities (SI model) which account for surface magnetic anisotropy energy and gradient of magnetization in the close-to-surface layer [12, 13]. The experimental data on the position of the surface mode and the first uniform one versus the orientation of the external magnetic field with respect to the film normal could be used to determine the total value of surface magnetic anisotropy energy constant K_s^t. Two components contribute to the K_s^t constant:

- K_s originated from the lack of translation symmetry of surface spins as well as from the surface roughness (contribution of stray field energy)
- K_s^d originated from the surface demagnetizing field due to the gradient of magnetization in the close-to surface layer.

2. Experiment

2.1 Films preparation

We investigated thin $CdCr_2Se_4$ films of chromium spinel with varying amounts of In in the lattice. Films were deposited by rf sputtering technique on Corning glass substrates in controlled temperature conditions. The deposition device is equipped with three cathode system and rotatable substrate holder. We used the chromium cathode for depositing the buffer and overlayer films. The thickness of the buffer and top layer of Cr was changed from 20 Å to 100 Å when the thickness of the middle layer of Cd-Cr-In-Se was bigger than 3000 Å. The powdered CdSe and Cr_2Se_3 cathodes with uniformly spotted pieces of In2Se3 were used for sputtering the Cd-Cr-In-Se sublayers. We have found that thickness of the buffer and top Cr layers equal about 50 Å give good adhesion to the substrate and the protection against diffusion of Se and Cd, respectively.

As-deposited samples are in the amorphous state and have a form of multilayer structure of Cr/Cd-Cr-In-Se/Cr. Heat treatment provides uniform polycrystalline single films with the required composition. The composition of samples was analysed by means of an X-ray microprobe (ARL SEMQ microanalyzer). The film thickness was measured by Talysurf 4 profilometer.

Other details of the preparation technique are described in paper [1, 14].

2.2 Ferromagnetic resonance technique (FMR)

The following magnetic parameters were determined from the ferromagnetic and spin waves resonance data:

- the magnetization *M*,
- the variation of the magnetization in the close-to-surface layer,
- the surface magnetic anisotropy energy constant K_s^t,
- the exchange interaction constant *A*
- the unidirectional magnetic anisotropy field H_{an}

The microwave spectrometer at X-band was used for the FMR and SWR experiments. The spectra were recorded in the temperature range from 4.2 K to 150 K. The external magnetic field was applied at different angles θ_H with respect to the film normal, from $\theta_H=0^\circ$ to $\theta_H=90^\circ$ at the perpendicular and parallel resonance experiment, respectively. The accuracy of the magnetic field orientation with respect to the film normal was less than one degree.

3. Model

The equation of motion for magnetization in the FMR experiment leads to the dispersion relation of spin waves in the low energy excitation regime. We adopted a classical approach with the equation of motion for the magnetization. The SI model was employed with the symmetrical boundary conditions for the surface spins. In the SI model the boundary conditions included the surface anisotropy energy of the form E_S=-K_S $\cos^2\theta$=θ was the angle between magnetization and the film normal) and also the magnetization inhomogeneities of the close-to-surface layer [15].

We consider the following geometry: the film is placed in the (x, y) plane, the external magnetic field H rotates in the (y, z) plane and makes an angle θ_H with the z-axis (film normal) the microwave field h is put align x-axis, the M direction is described by angles θ and ϕ in spherical coordinates. Then we have

$$\vec{M} = M\hat{r} + m_\theta\hat{\theta} + m_\phi\hat{\phi} \tag{1}$$

where m_θ and m_ϕ are microwave components of the magnetization M with the conditions $m_\theta/M \ll 1$ and $m_\phi/M \ll 1$. We assumed that the spin waves propagate along the thin film normal. Then the microwave components of magnetization are the form:

$$\begin{aligned} m_\phi(z,t) &= m_\phi^0\, e^{(\omega t \pm kz)} \\ m_\theta(z,t) &= m_\theta^0\, e^{(\omega t \pm kz)} \end{aligned} \tag{2}$$

The equation of motion for M, with no damping term, is:

$$\frac{d\vec{M}}{dt} = r(\vec{\tau} + \vec{M}\times\frac{2A}{M^2}\nabla^2\vec{M} + \vec{M}\times\vec{h}), \tag{3}$$

where

$$\vec{\tau} = -\hat{\phi}\ \frac{\partial E}{\partial\theta} + \hat{\theta}\ (1/\sin\theta)\ \frac{\partial E}{\partial\phi},$$

is the torque given by energy density E. The second term in Eq. (3) is the torque from the exchange energy (A is the exchange interaction energy constant, M is the magnetization) and the last perturbative term the microwave energy.

Substituting Eqs. (1) and (2) into Eq. (3), taken only linear terms and within the circular precession approximation for the microwaves components $\boldsymbol{m}$ of the magnetization [15, 16] the dispersion relation has the form:

$$\left(\frac{\omega}{\gamma}\right)^2 = \left(\frac{1}{M\sin^2\theta}\frac{\partial^2 E}{\partial\phi^2} + \frac{2A}{M}k^2\right)\times\left(\frac{1}{M}\frac{\partial^2 E}{\partial\theta^2} + \frac{2A}{M}k^2\right) - \left(\frac{1}{M\sin\theta}\frac{\partial^2 E}{\partial\phi\partial\theta}\right)^2 \tag{4}$$

The energy density $E(\theta, \phi)$ is the sum of the terms:

$$E = E_H + E_d + E_{an} \tag{5}$$

where

E_H=$MH(\sin\theta\sin\theta_H\sin\phi+\cos\theta\cos\theta_H)$ is the energy of the external magnetic field H,

E_d=$2\pi M^2\cos\theta$ is the demagnetization energy,

E_{an}=-K_{an} $\cos(\theta-\theta_H)$ is the unidirectional magnetic anisotropy energy, where K_{an}=MH_{an}.

In the disordered state an unidirectional magnetic anisotropy energy manifests itself by the influence of internal field -H_{an} on the spin ordering. H_{an} depends on the field-induced remanent magnetization and keeps the direction of the cooling field. The unidirectional magnetic anisotropy filed is influenced by the dilution level and is temperature dependent [5, 17].

The equilibrium conditions $(\partial E)/(\partial\theta)$=0 and $(\partial E)/(\partial\pi)$=0 yield ϕ=$\pi/2$ and

$$H\sin(\theta-\theta_H) - 2\pi\sin 2\theta + \frac{1}{2}H_{an}\sin(\theta-\theta_H) = 0. \tag{6}$$

Taking into consideration the contributions of the energy density described above as well as the equilibrium conditions one can final form of the dispersion relation. Let us discuss the term $(2A/M)k^2$ responsible for the exchange interaction. For $k^2>0$ we get real k and so the microwave components described by Eq. (2) are a cos-like form, e.q. we have the volume modes. The case $k^2<0$ provides purely imaginary $k=ik_s$, which describes damping of the microwave m_ϕ and m_θ components of $\vec{M}$ with an increasing distance from the film surface. This case corresponds to the surface modes. The resonance field H_S, for the surface modes, is large than the resonance field H_u for a uniform mode k=0.

Finally, we have got the formula for the shift of surface mode resonance field with respect to the uniform mode [15, 18]:

$$H_s = H_u + \frac{2A}{M}\frac{k_s^2}{\cos(\theta-\theta_H)} \tag{7}$$

The wave vector of surface mode k_s results from the boundary conditions. The allowed values of k_s depend on the angle θ, there is a critical value $\theta=\theta_c$, for which the position of the surface mode coincides with uniform one; $H_s=H_u$. At this angle the surface mode ceases to exit and becomes a uniform mode.

In the equation of motion; Eq. (3) applied to the surface spins two terms have to be include:

- the surface magnetic anisotropy energy E_s=-$K_s\cos^2\theta$
- the term which accounts for the exchange of the magnetization near the film surface.

This leads to the boundary conditions arising from the coherent rotation of volume and surface spins. We have to put in Eq. (3) the term which accounts for the changes of magnetization M near the surface [13, 15].

For symmetrical boundary conditions, with the circular precession approximation of the microwave components $\boldsymbol{m}$

of the magnetization, the boundary conditions have the form [15, 18, 19, 20]:

$$\partial_n m + pm = 0$$

where

$$p = -(K_s/A)\cos 2\theta + (\partial_n M)/M \tag{8}$$

is the effective pinning parameter of surface spin, ∂_n denotes the directional derivative along the normal to the surface. The term $\partial_n M)/M$ accounts for variation of close to the surface layer magnetization [13].

For symmetrical boundary conditions the approximate solution for k_s is $k_s=p$. This allowed us rewrite the Eq. (7) in the form.

$$[(H_s H_u)\cos(\theta-\theta_H)]^{1/2} = \left(\frac{2}{AM}\right)^{1/2} \cdot |K_s|[\cos 2\theta - \frac{A}{K_s}(\partial_n M/M)] \tag{9}$$

For the known angle θ_H the magnetization direction angle is given from the equilibrium condition (see Eq. (6)). We defined the left hand side of Eq. (9) as:

$$\alpha = [(H_s - H_u)\cos(\theta-\theta_H)]^{1/2}$$

Now we can plot α against $\cos 2\theta$. The surface magnetic anisotropy energy constant K_s is found from the slope of this line; the interception with the $\cos(2\theta)$-axis gives the magnetization variation $(\partial_n M)/M$ when A and M are known parameters. Both of them can be from the FMR and SWR experiment.

4. Results

We have investigated thin films of $CdCr_{2-2x}In_{2x}Se_4$. The dilution level classifies them, due to the magnetic phase diagram, as being in spin glass state (SG). The character of temperature dependence of magnetization M and unidirectional magnetic anisotropy field H_{an} have confirmed the glass state of samples.

4.1 Magnetization

From the FMR data (uniform mode k=0) taken for temperature range from 4 to 80 K the $M(T)$ is found, the details are presented in paper [1]. Fig. 1 presents the $M(T)$ for thin films of $CdCr_{1.7}In_{0.3}Se_4$ and $CdCr_{1.5}In_{0.5}Se_4$. The points relate to the experimental data, solid and dashed lines came from the theoretical prediction [5, 17] for SG state

$$M(T) = M(0)\left(1 - \frac{C_s}{\exp[\Delta_s/k_B T]-1}\right) \tag{10}$$

where C_s is responsible for the density of states at the energy gap of magnon dispersion relation and Δ_s is a measure of the intercluster interaction; both are fitting parameters.

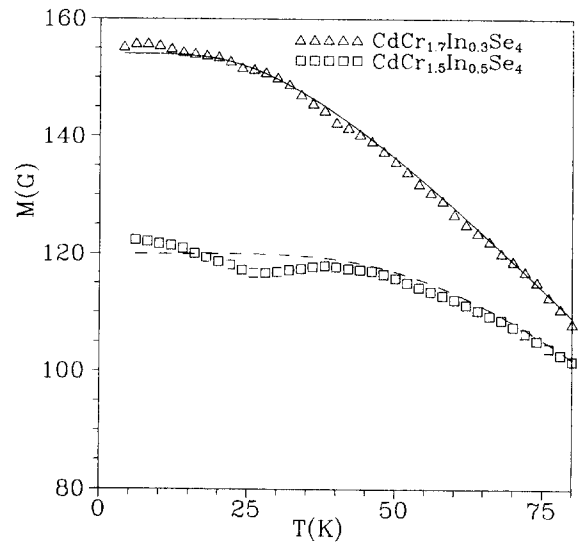

Fig. 1. Temperature dependence of magnetization for thin films of $CdCr_{1.7}In_{0.3}Se_4$ and $CdCr_{1.5}In_{0.5}Se_4$. The calculated $M(T)$ is presented by soild and dashed lines, respectively.

4.2 Unidirectional magnetic anisotropy field

The temperature dependence of H_{an} was determined from FMR data the details of the experiment are presented in paper [1.10]. The data for thin films of $CdCr_{1.7}In_{0.3}Se_4$ and $CdCr_{1.5}In_{0.5}Se_4$ are shown in Fig. 2. Only the experimental data of $H_{an}(T)$ are shown, because there are no theoretical predictions for temperature dependence of this macroscopic parameter.

4.3 Surface magnetic anisotropy energy constant

In the SWR experiment we detected two modes at the perpendicular geometry. The mode at higher field had intensity smaller then the second one; such a relation is

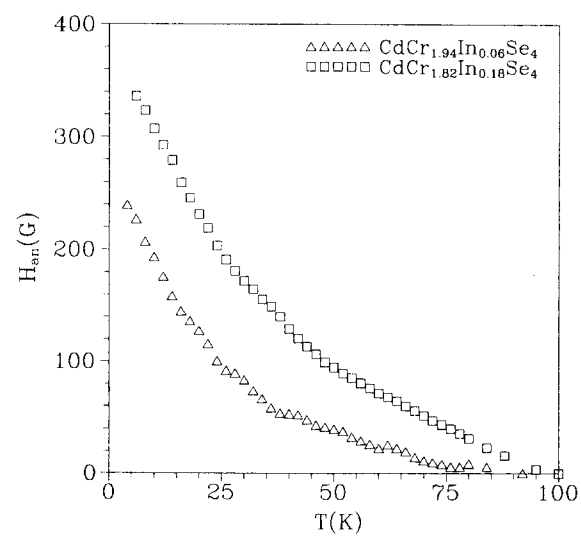

Fig. 2. Unidirectional magnetic anisotropy field as a function of temperature for $CdCr_{1.7}In_{0.3}Se_4$ and $CdCr_{1.5}In_{0.5}Se_4$ thin films.

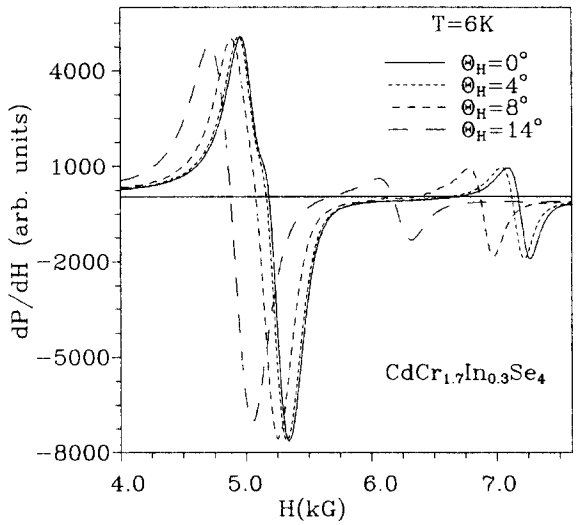

Fig. 3. The SWR spectra of $CdCr_{1.7}In_{0.3}Se_4$ thin film (T=6 K), taken for θ_H=0, 4, 8, 14 degree.

typical for surface mode. The second mode corresponds to the uniform one. Fig. 3 shows the SWR spectra of $CdCr_{1.7}In_{0.3}Se_4$, for some θ_H, taken for T=6 K There is visible the shift of resonance field H_s of surface mode with respect to uniform one H_u when θ_H is increased.

The angular dependence of resonance fields for surface H_s and uniform H_u modes taken at T=6 K for the thin films of the same composition is presented on Fig. 4. It could be noticed that at a particular value of $\Theta_H=\Theta_c$ (critical angle) both modes overlap; since then they have same resonance field up to Θ_H=90°. On the basis of this data, for the temperature range from 4 K to 125 K, we have calculated α vs cos(2θ). The angle θ was found from the equilibrium condition described by Eq. (6). The results obtained for several temperatures are presented on Fig. 5. for thin film of $CdCr_{1.7}In_{0.3}Se_4$. The experimental points follow straight lines in agreement with theoretical predic-

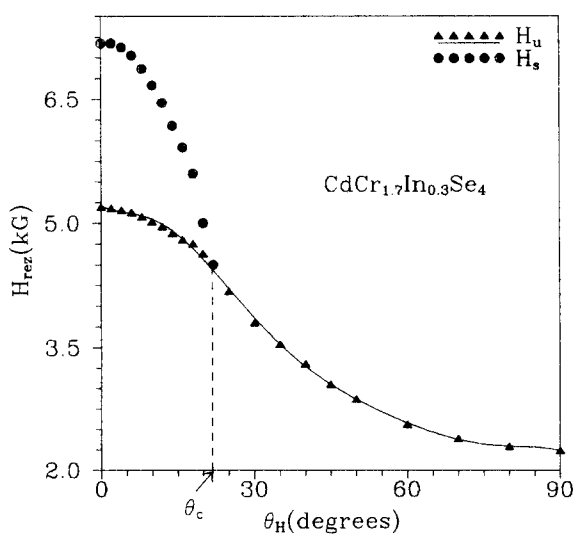

Fig. 4. The angular dependence of resonance field for uniform and surface modes for $CdCr_{1.7}IN_{0.3}Se_4$ thin film.

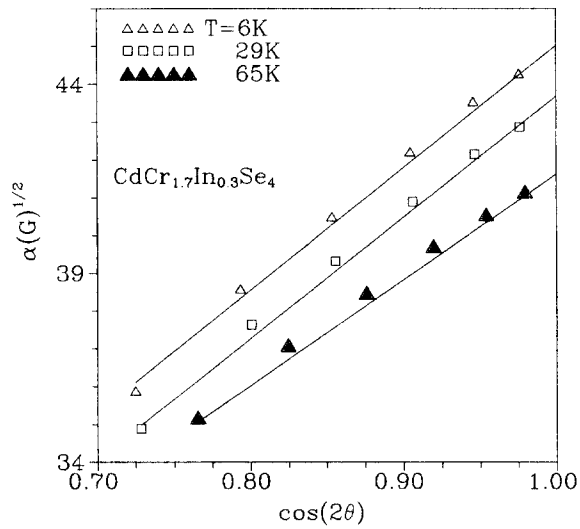

Fig. 5. Dependence of $\alpha=[(H_s-H_u)\cos(\theta-\theta_H)]^{1/2}$ on cos(2θ) for $CdCr_{1.7}In_{0.3}Se_4$ thin film.

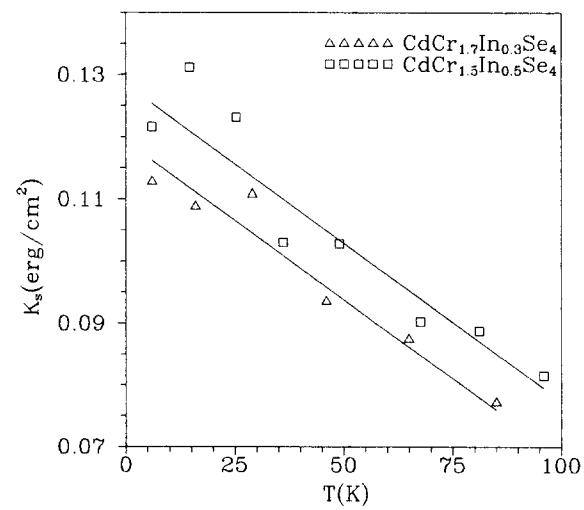

Fig. 6. The temperature dependence of the surface magnetic anisotropy energy constant K_s for $CdCr_{1.7}In_{0.3}Se_4$ and $CdCr_{1.5}In_{0.5}Se_4$ thin films.

tion. The temperature dependence of the surface magnetic anisotropy energy constant K_s was determined from the slopes ($(2/AM)^{1/2}K_s$) of the straight lines. The needed values of $M(T)$ were found from the data presented on Fig. 1. We use the value of $A=1.6\times10^{-7}$ erg/cm which was found from our previous experimental data presented in paper [4]. Temperature dependence of K_s for thin films $CdCr_{1.7}In_{0.3}Se_4$ and $CdCr_{1.5}In_{0.5}Se_4$ is presented in Fig. 6. It is seen that K_s decreases with the temperature increment.

In the boundary conditions for surface spins, we have also considered the gradient of magnetization in the close-to-surface layers (nonhomogeneities of magnetization). This also contributes to the total surface magnetic anisotropy energy and originated from the surface demagnetizing field. The value of this component of surface magnetic anisotropy constant K_s^d is given by the formula [18, 21]:

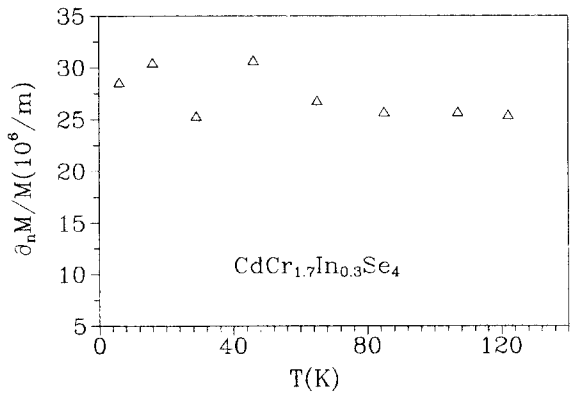

Fig. 7. $\partial_n M/M$ as a function of temperature for $CdCr_{1.7}In_{0.3}Se_4$ thin film.

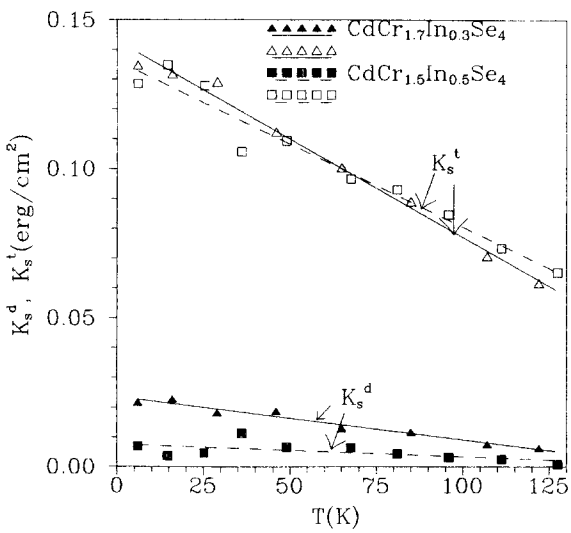

Fig. 8. The temperature dependence of surface magnetic anisotropy energy constants K_s^d and K_s^t for $CdCr_{1.7}In_{0.3}Se_4$ and $CdCr_{1.5}In_{0.5}Se_4$ thin films.

$$K_s^d = 4\pi M^2(\partial_n M/M)d^2 \qquad (11)$$

where d is the thickness of the close-to-surface layer. In the Eq. (9) the term with $\partial_n M/M$ is included and can be calculated on the basis of the relation between α and cos 2θ (see Fig. 5). The interception with the cos(2θ)-axis gives the magnetization variation ($\partial_n M/M$). Fig. 7 presents the temperature dependence of $\partial_n M/M$ for thin film of $CdCr_{1.7}In_{0.3}Se_4$. To calculate K_s^d, we applied the Eq. (11), put d=50 Å, used the temperature dependence of magnetization as presented in Fig. 1 and we have taken ∂_nM/M from data presented in Fig. 7. The value of d=50 Å is related to the technology procedure and corresponds to the Cr thickness of buffer and overlayer. We can expect that this extra amount of Cr alters the distribution of magnetization and the magnetic properties of close-to-surface layer.

Fig. 8 shows the temperature dependence of K_s^d and the total value of surface magnetic anisotropy energy constant: $K_s^t=K_s+K_s^d$ for thin films of $CdCr_{1.7}In_{0.3}Se_4$ and $CdCr_{1.5}In_{0.5}Se_4$. As it is seen from Fig. 8 the dominant contribution to K_s^t comes from the K_s component. The common general feature of K_s^t and K_s^d as well as K_s (see Fig. 5) is linear relationship with respect to temperature. Within the temperature range from 4.5 K to 125 K there is no significant quantitative difference between K_s^n's values of the investigated samples at the SG state.

5. Conclusions

In conclusion, we have studied the temperature dependence of the surface magnetic anisotropy energy constant of $CdCr_{2-2x}In_{2x}Se_4$ magnetic semiconductor thin films. We are classifying the type of magnetic phase by determination of temperature dependence of magnetization $M(T)$ and the unidirectional magnetic anisotropy field $H_{an}(T)$.

The buffer and top layers of chromium in the as-deposited thin film could be responsible for the ($\partial_n M/M$) term at the boundary conditions for surface spins in the finally obtain polycrystalline thin film.

We performed the resonance experiment at microwave spectrometer (X-band) for temperature changing from 4, 5 to 125 K and the surface mode was observed at the external magnetic field higher than the uniform one. The SI model with nonhomogeneities of magnetization at the close-to-surface layer was applied for the determination of the surface magnetic anisotropy constant K_s. From the angular dependence of the resonance field for uniform and surface modes we have calculated the value of K_s as well as $\partial_n M$ (gradient M) of close-to-surface layers. The gradM contributes to the total magnetic anisotropy energy, the source of which originated from the surface demagnetizing field. This extra surface magnetic anisotropy energy constant is described by the formula $K_s^d=4\pi M^2(\partial_n M/M)d^2$, where d is the thickness of the close-to-surface layer. The total value of surface magnetic anisotropy constant $K_s^t=K_s+K_s^d$ was found to be a linear function of temperature (decreasing when temperature is increased) and the main contribution comes from K_s.

The work was partly supported by KBN Grant No 249. 620.05 and KBN Grant No 8 T11B 054 09.

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