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REVIEWS - LETTERS - REPORTS

ANOMALOUS ELECTRICAL AND STRUCTURAL PROPERTIES OF DYSPROSIUM AND THULIUM THIN FILMS AT LOW TEMPERATURES

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Thin films of dysprosium and thulium were prepared by evaporation in high vacuum (Dy.Tm) and in ultrahigh vacuum (UHV) in the thickness range from 16 nm to 370 nm. The size-effect of the electrical resistivity of these films is without anomalies and in qualitative agreement with theory. Investigation of the crystal structure of these films by means of X-ray diffraction showed that Dy films contained a phase with hcp structure of Dy and also a small amount of DyH_2, whereas Tm films (HV and UHV) consisted only of a single phase with hcp crystal structure of Tm. The resistivity vs. temperature dependences of Dy and Tm (UHV) measured in the low temperature region from 4.2 K up to the room temperature exhibit a knee-like (in thinner films) or hump-backed (thicker films) anomalies near the magnetic phase transition from the paramagnetic state to the antiferromagnetic state which are caused by the magnetic structure. Unusual ρ vs. T anomalies were observed in two temperature regions in Tm films (HV)-below $\sim$ 60 K and near $\sim$ 170 K that could be caused by hydrogen present in the form of a solid solution in these films.

K e y w o r d s: electrical resistivity, thin films, dysprosium, thulium, low temperatures, crystal structure

1 INTRODUCTION

Transport and magnetic properties of magnetic metallic thin films exhibit a new considerable renascence since the discovery of spin polarized transport in multilayered ferromagnetic/nonmagnetic structures [1, 2]. The discovery of giant magnetoresistance (GMR) has strongly stimulated both experimentalists and theoreticians to study arrays of magnetic thin films (see *eg* [3] and citations therein). The intense activity in this area is driven, in part, by needs of magnetic recording and other applications. The effect of GMR was originally observed in Fe/Cr multilayers, later with Co and Ni as ferromagnetic thin films and with various nonmagnetic spacers like Au, Ag Cu. As for rare earth metal thin films, magnetoresistance was studied in multilayers with CoSm layers [4], GdCo films [5] and positive GMR was observed in Dy/Sc superlattices [6].

Relative importance of the resistivity contributions from the bulk and from interface spin dependent scattering, in these materials, in theory varies from one model to another but according to experiment it seems to be system dependent, the latter being dominant *eg* in Si / Py/Cu/Py/FeMn/Cu multilayers according to experiment [7].

Nevertheless, electron transport properties of thin films of individual materials are of importance for a better understanding and are studied in transition metal thin films like Ni and Cr [8]. Rare earth metal thin films attracted attention because of anomalies of their physical properties, the most studied was the size-effect of electrical resistivity.

The electrical resistivity of continuous metal thin films has been analyzed theoretically using semi-classical and quantum mechanical treatments. Semi-classical size-effect theories predict a monotonic increase of resistivity with decreasing film thickness (see *eg* [9, 10]). Such a behaviour has been observed in many transition metal films. Zhang and Butler [11] examined quantum and semi-classical approaches to the calculation of the electrical conductivity using a model of free electrons with a finite lifetime. They compared the exact evaluation of the Kubo formula with the semiclassical theories and in the case of resistivity measured in the plane of the film, when the angular dependence of the specularity parameter p is considered, "the semi-classical theories give excellent agreement with expetimental data on several thin films".

Thin films of some heavy rare earth metals (Dy, Gd, Er) prepared by evaporation in high vacuum exhibited unexpected Anomalies a maximum in the resistivity *vs* thickness dependence, a decrease of the ρ value with decreasing film thickness (below some film thickness) and even the resistivity of the thinnest films was lower than that of the bulk samples (for a survey see *eg* [12, 13]). These anomalies differ from those of other transition and noble metal films and could not be interpreted within the existing theories. Moreover, they also differ from each other in this metal group and caused some confusion in the interpretation of experimental data.

A different anomalous resistivity vs. thickness behaviour has been observed in thin films of light RE metal samarium by two groups [14, 15]: although the resistivity value monotonically increased with decreasing film thickness, both research groups observed in Sm films with

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thickness $t >\sim 30$ nm the resistivity value ($\sim 50\,\mu\Omega$cm), which is lower than that of the bulk ($\sim 95\,\mu\Omega$cm), even below a half of it. Another experimental study devoted to Y, Yb and Sm thin films prepared by evaporation in high vacuum ($\sim 10^{-3}$ Pa–$\sim 10^{-4}$ Pa) is focused on the size-effect of electrical resistivity, temperature coefficient of electrical resistance and thermoelectric power of these films [16]. Despite the unexpected observed anomaly of the size-effect of electrical resistivity, which was caused by the presence of other crystal phases in their thin films (for deeper insight see *eg* [12, 13]), these authors interpreted their results according to a theory valid only for pure films [9, 10]. Moreover, these authors did not present the results of the crystal structure study of their films that could justify their interpretation. In contrast, another group [17] prepared thin Sm films by evaporation in high vacuum and the size-effect of resistivity of their films is without the above anomaly and in agreement with theory.

The observed resistivity anomalies attracted our concentration to study the transport, magnetic and crystal properties of thin films of RE metals. We summarize in this paper the experimental results obtained on Dy and Tm (no reports in the literature by other groups) by studying the size-effect of electrical resistivity, on the crystal structure of these films and also on the temperature dependences of the electrical resistivity in the low temperature region (down to 4.2 K) .We have observed resistivity anomalies of thin Dy and Tm (UHV) films at low temperatures that are caused by the magnetic structures. But we have observed unusual resistivity anomalies below ~ 50 K and near ~ 170 K in films of Tm (HV) which can not be caused by the magnetic structure.

2 EXPERIMENTAL DETAILS

Thin films of dysprosium and thulium were evaporated in high vacuum ($\sim 1\times 10^{-4}$ Pa) onto pre-cleaned glass substrates from bulk Dy and Tm samples which were studied as a reference prior to thin film studies. At first, silver electric contact pads were deposited on substrates. A liquid nitrogen trap was put between the oil diffusion pump and the vacuum bell-jar and a vessel with a large nitrogen cooled area was put inside of the bell-jar to obtain better vacuum. Thin films of thulium were also evaporated onto pre-cleaned glass substrates in ultrahigh vacuum ($\sim 10^{-7}$ Pa) in the commercial Balzers apparatus using a turbomolecular pump.

We did not allow condensation on the substrate until gettering action has finished - several outgassings were made in order to get only weakly rising pressure at evaporation of Dy and Tm. Protective SiO layers against contamination with air were deposited on Dy and Tm films before mounting them in the experimental helium cryostat.

Current leads and potential probes were cemented at appropriate positions in the contact pads of thin films using silver paint. Conventional four point dc arrangement was used to measure electrical resistance of Dy and Tm bulk and thin film samples in the temperature range from 4.2 K up to the room temperature in the helium cryostat using digital Keithley programmable current source K 220 and Keithley digital nanovoltmeter K 181.

The temperature of the bulk and of thin film samples in the helium cryostat was measured using calibrated germanium (from 4.2 K to 80 K) and platinum (from 80 K up to the room temperature) thermometers.

The thickness of thin films was estimated using the optical Tolansky method.

The crystal structure of Dy and Tm films was studied using the X-ray diffraction technique with Bragg-Brentano focusing geometry (the thin film plane making equal angles with incident and diffracted beams). A commercial horizontal diffractometer HZG 4/A has been used with the stepwise measurement method, with a step value of 0.02° for 2Θ. The cobalt or Cu (for Tm- UHV films) K_α radiation produced by a tube at 35 kV, 20 mA and monochromatized by the ROSS filter difference method has been detected with a proportional detector using the

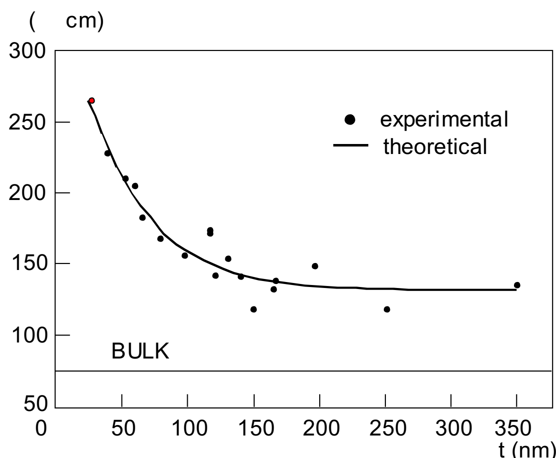

Fig. 1. Size-effect of the electrical resistivity of dysprosium thin films

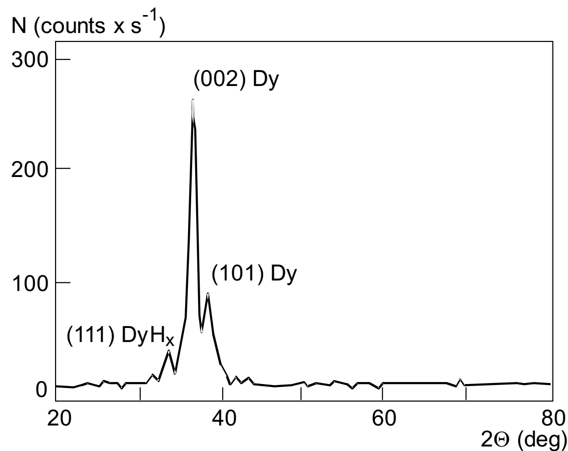

Fig. 2. X-ray diffractogram of 50 nm thin dysprosium film

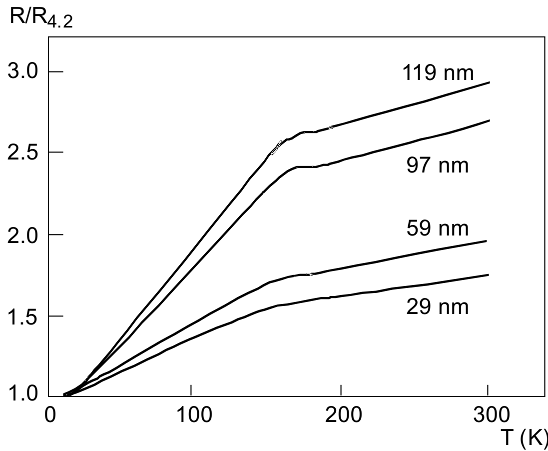

Fig. 3. Resistance ratio $R/R_{4.2}$ as a function of temperature of four Dy films: 26, 59, 97 and 119 nm

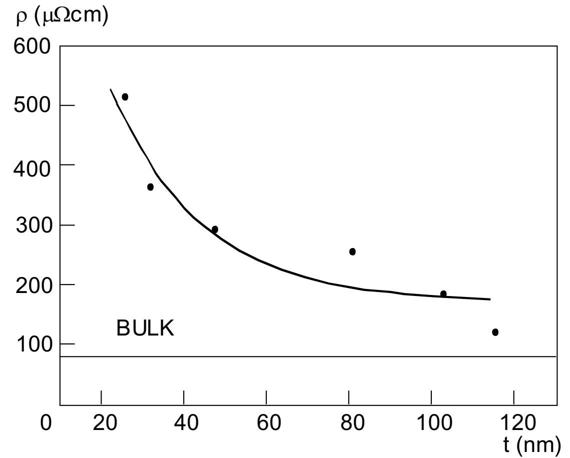

Fig. 4. Thickness dependence of the resistivity of HV thulium thin films

pulse hight selection analysis. The control of the measurements and the data processing were performed by a computer in the on-line regime. The diffraction lines were characterized by the position of the maximum intensity, and by the position of the centre of their area. The integral intensity and the integral halfwidth of the diffraction lines were determined after separation of the background and of the $K_{\alpha 2}$ radiation component.

3 RESULTS AND DISCUSSION

3.1 Dysprosium thin films

The electrical resistance of bulk and thin film samples has been measured first at room temperature. The results of this measurement are illustrated in Fig. 1 as thickness dependence of the film resistivity together with ρ value of our Dy bulk sample. As could be seen in Fig. 1, the resistivity value of all Dy films decreases with increasing film thickness in the studied thickness range from 26 nm to 350 nm, and ρ value of all films is higher than that of the bulk sample. Thus, the size-effect of the electrical resistivity of our Dy films does not exhibit the above anomalies and is in qualitative agreement with the theory (see *eg* [9, 10]).

We have studied the crystal structure of our Dy films by X-ray diffraction. The result of this study is illustrated in Fig. 2 for 50 nm thin Dy film as 2Θ dependence of the detected counts number per second N. Here, Θ is the Bragg angle.

The set of diffraction lines in this figure confirms the idea of the preferential orientation of crystallites in Dy films. Three maxima are clearly resolved in this figure. The diffraction peaks at 38.349° and 37.062° are caused by diffraction at (101) and (002) planes of the hexagonal closed packed (hcp) structure of Dy. The peak at the angle of 34.69° is caused by diffraction at (111) plane of the fcc crystal structure with lattice parameter $a = 0.5187$ nm. This value is in accordance with the lattice parameter of fcc dysprosium dihydride DyH_x ($x \sim 2$) [18]. Therefore, we assume that some amount of dysprosium dihydride is present in our Dy films but this amount did not cause anomalies of the ρ vs. t curve as by other groups (see the survey, *eg* in [12, 13]).

No other contamination in the form of a crystalline phase was detected by X-ray diffraction in our Dy films.

The results of an X-ray diffraction study of films with thicknesses of 31 nm, 66 nm and 78 nm are in accordance with those of the 78 nm thin film for the preferential orientation of crystallites as well as for the detection of DyH_x .

A different preferential orientation of crystallites was found in thicker films with thickness 234 nm and of 348 nm [19]. The peaks corresponding to dysprosium dihydride were also found in thicker Dy films.

In the next step, the electrical resistivity of Dy films has been investigated in the low temperature range from 4.2 K up to the room temperature and the results of this study are illustrated in Fig. 3 .

We have observed a knee-like anomaly in thinner Dy films or hump-backed anomalies in thicker films. Such resistivity anomalies were observed in the basal plane or in the c-axis direction of the Dy single crystal in the vicinity of the Néel temperature [20]. Thus, resistivity vs. temperature anomalies in our Dy films are caused by the magnetic phase transition from the paramagnetic state to the antiferromagnetic one.

3.2 Thulium thin films — HV

First, we have prepared thulium thin films by evaporation in high vacuum, their thickness ranging from 25 nm to 115 nm. Their resistivity has been measured at room temperature and the results of this measurement are shown in Fig. 4, including the resistivity value of our Tm bulk sample. It is clearly seen in Fig. 4 that resistivity increases with decreasing film thickness and the resistivity value of all films is higher than that of the bulk. The

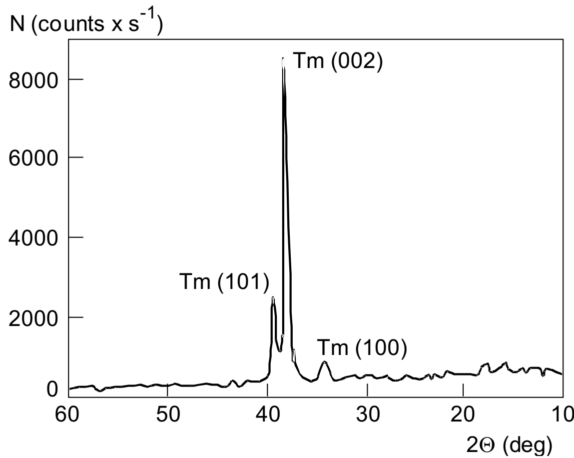

Fig. 5. Diffraction pattern of the 115 nm HV Tm thin film

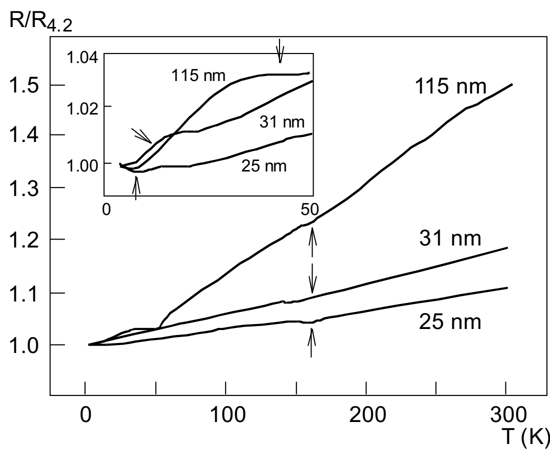

Fig. 6. Temperature dependence of the resistivity ratio of three Tm films with thicknesses of 25 nm, 31 nm and 115 nm. The resistivity anomalies near $\sim$ 170 K are indicated by arrows and resistivity anomalies below 50 K are more clearly seen in the insert.

size-effect of the electrical resistivity of our HV Tm thin films does not exhibit the above anomalies and is in qualitative agreement with theory.

As in the case of our Dy films, we expected some amounts of thulium dihydride in our Tm films. We have examined their crystal structure using X-ray diffraction. The positions of the expected diffraction peaks of TmH_2 were evaluated by means of the Bragg law using X-rays with a wave length of $\lambda = 0.17902$ nm and the lattice parameter of its fcc crystal structure $a = (0.509 \pm 0.001)$ nm is known from the literature [18]. Diffraction peaks should appear at 2Θ positions: 35.467° (111) plane, 41.184° (200) plane, 59.655° (220) plane, 71.358° (311) plane, 75.061° (222) plane , *etc.*

The result of the experimental X-ray diffraction study is shown in Fig. 5 for 115 nm thin film [21]. The highest peak at 37.80° is caused by (002) plane of the Tm hcp crystal structure and the peaks with lower values of intensity at 34.40° and 39.20° represent diffractions at (100) and (102) planes, respectively, of Tm hcp structure. We have observed two more peaks with lower intensities at 74.90° and 80.80° positions caused by diffraction at (201) and (004) planes of Tm hcp crystal structure (not shown in Fig. 5).

The set of diffraction lines in Fig. 5 represents the preferential orientation of crystallites.

There is no evidence about diffraction peaks at any plane of thulium dihydride crystal structure in this figure. Therefore, we assume, this Tm film does not contain TmH_2 .

The results of the X-ray diffraction study of films with thicknesses of 25 nm, 31 nm and 102 nm are in accordance with those of 115 nm thin film as for the preferential orientation of crystallites as well as for the non detection of thulium dihydride.

This is in contrast to Curzon and Chlebek [22] who observed fcc phase in their Tm films evaporated in high vacuum $\sim 10^{-4}$ Pa.

Electrical resistivity of thulium bulk sample has been investigated in the temperature range from 4.2 K up to room temperature prior to the thin film study an it has been measured with the step $\Delta T = 0.1$ K near the Néel temperature region, with $\Delta T = 0.2$ K outside and with the step ~ 1 K–~ 2 K in the paramagnetic region. The high residual resistivity ratio $RRR = 146$ (ratio of resistance at room temperature to that at 4.2 K) means a high purity of our Tm bulk sample. A "hump-backed" resistivity anomaly has been observed near ~ 60 K [21] and this is typical for the magnetic phase transition from the paramagnetic state to the antiferromagnetic one with the CAM spin structure [20]. Numerical analysis of the experimental data yielded a sharp deep minimum of the $\mathrm{d}\rho/\mathrm{d}?T$ vs. T curve corresponding to the Néel temperature $T_N = 57.5$ K [23].

The resistivity of thulium thin films has also been investigated in the temperature range from 4.2 K up to the room temperature [21]. The result of this investigation is illustrated as resistivity ratio vs. temperature dependence for three of them with thicknesses of 25 nm, 31 nm and of 115 nm in Fig. 6. We have observed resistivity vs. temperature anomalies in two temperature ranges - below ~ 60 K and near ~ 170 K. A sudden increase of the ρ value with increasing temperature at ~ 20 K in 31 nm thin film, a resistivity plateau in the temperature interval $\Delta T = 16$ K in 115 nm thin film and even resistivity minimum in 25 nm thin film are clearly seen below ~ 60 K in the insert in Fig. 6. All observed resistivity anomalies in Tm thin films are quite different in shape from that of Tm bulk sample, thus, these anomalies are caused by another mechanism than in the Tm bulk sample. Another type of resistivity anomaly has been observed near ~ 170 K as slope change of the ρ vs. T curves. This kind of anomaly has not been observed on Tm bulk sample, therefore it is not caused by magnetic spins of Tm.

Thulium is one of several RE metals which possess a high solubility of hydrogen (α-phase), retaining H in the form of a solid solution down to 0 K without any

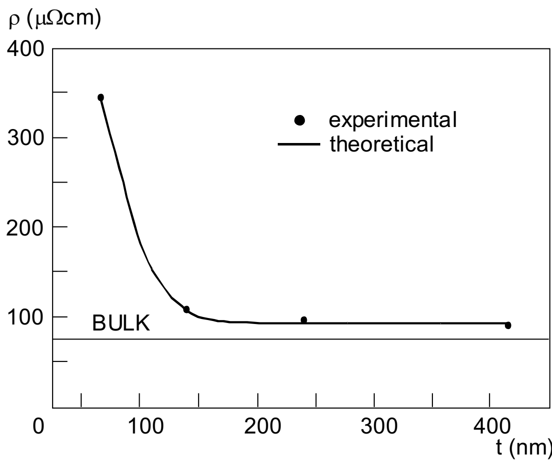

Fig. 7. The size-effect of the electrical resistivity of UHV Tm thin films

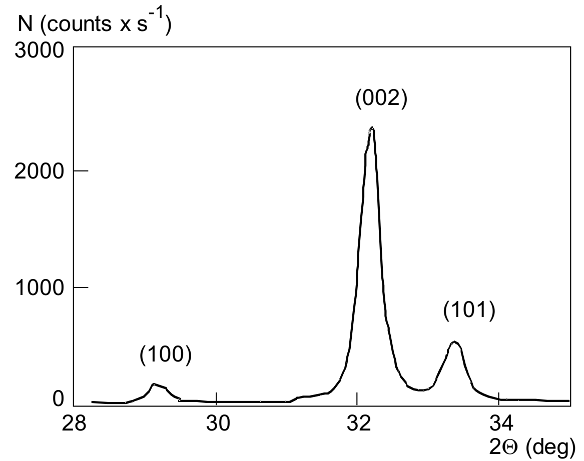

Fig. 8. Diffraction pattern for the 95 nm thin UHV thulium film

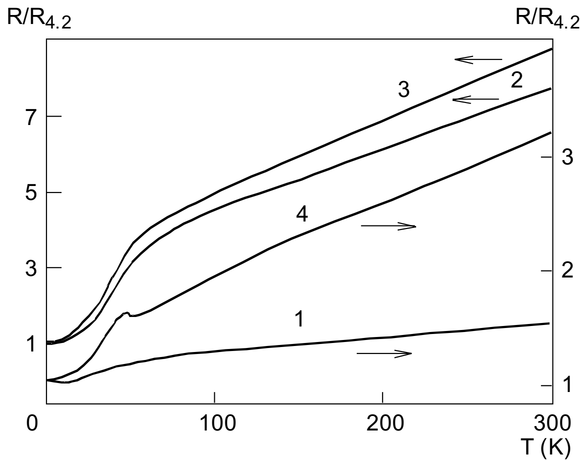

Fig. 9. Resistance ratio as a function of temperature of four UHV Tm films: 1 – 16 nm, 2 – 89 nm, 3 – 189 nm, 4 – 364 nm.

evidence of an $\alpha - \beta$?transition [24]. The influence of hydrogen in solid solution on magnetism of monocrystalline Tm is reported in [25]. Varying concentrations in TmH_x up to $x = 0.1$ the authors observed a variety of the ρ vs. T anomalies below $\sim$ 60 K, including that we have observed in our Tm films. They also observed another anomaly near $\sim$ 170 K as slopes change in the ρ vs. T dependences. The same anomaly has been observed in our Tm films

Our Tm films were prepared by evaporation in high vacuum and some amount of hydrogen is always present in such vacuum in the vacuum chamber [26]. Therefore, we assume, the temperature anomalies of the electrical resistivity below $\sim$ 60 K and near $\sim$ 170 K observed in our Tm films could be caused by hydrogen present in solid solution in such films. This interpretation was confirmed by [27].

3.3 Thulium thin films — UHV

In the next step we have prepared Tm thin films by evaporation in ultrahigh vacuum ($\sim$ 10^{-7} Pa) in the thickness range from 16 nm to 370 nm. The size-effect of the resistivity of these films is illustrated in Fig. 7.

The resistivity value of all films is higher than that of the bulk and ρ value increases with decreasing film thickness, thus, the size-effect of resistivity is without anomalies and in qualitative agreement with theory.

The results of the crystal structure study by means of X-ray diffraction are illustrated in Fig. 8 for 95 nm thin film of Tm. The set of diffraction lines represent the preferential orientation of crystallites in this film. The diffraction peak at 32.201° is caused by the (002) plane of the hcp Tm crystal structure. The peaks at 29.152° and 33.357° represent diffraction at (100) and (101) planes of hcp Tm structure.

The results of the X-ray diffraction work in films with thicknesses of 15 nm, 21 nm and 79 nm are in accordance with those of 95 nm thin film [28].

Electrical resistivity of these films has been investigated in the temperature range from 4.2 K up to room temperature [28]. The results of this investigation are illustrated in Fig. 9 as resistance ratio vs. temperature dependences for four of them.

In the difference to the ρ vs. T results obtained in our HV Tm thin films, we have observed temperature anomalies of resistance only in one temperature region - below $\sim$ 60 K. These include a hump-backed anomaly in thicker films as has been observed in the c-axis of Tm single crystal near the Néel temperature [20], and a knee-like anomaly in thinner films as observed in the basal plane of Tm single crystal near T_N. Both kinds of resistance anomalies are caused by the transition from the paramagnetic state to the antiferromagnetic one with the c-axis modulated spin structure [20].

4 CONCLUSIONS

We have prepared thin films of dysprosium and thulium by evaporation in high vacuum (Dy, Tm) and in ultrahigh vacuum (Tm) and studied their electrical resistivity in the low temperature region from 4.2 K up to room temperature and their crystal structure by means of X-ray diffraction.

It follows from this study:

1) The size-effect of the electrical resistivity of Dy and Tm films (HV and UHV) is without anomalies and in qualitative agreement with theory.
2) The Crystal structure of Dy and Tm films (HV and UHV) shows preferential orientation of crystallites.
3) Thin films of Tm (HV and UHV) are formed only with a single hcp phase of Tm.
4) Thin films of Dy contain a small amount of dysprosium dihydride that qualitatively influenced neither the size-effect of electrical resistivity nor the temperature dependences of the resistivity.
5)

Thin films of Dy and Tm (UHV) exhibit two kinds of resistivity anomalies in the low temperature region - hump-backed ρ vs. T anomalies in thicker films and knee-like anomalies in thinner films as it has been observed in the c-axis and in the basal plane of the single crystals of dysprosium and thulium near the Néel temperature.

6) Thin films of thulium prepared by evaporation in high vacuum exhibit different resistivity anomalies below ~ 60 K, including a sudden increase of the ρ value and a resistivity plateau, and a slopes change of the ρ vs. T curve near ~ 170 K. The observed resistivity anomalies could be caused by the hydrogen present in solid solution in such films.

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