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1993 J. Phys.: Condens. Matter 5 9059

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The electrical resistivity of polycrystalline Cu/Mn double-layered thin films

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Received 14 July 1993

Abstract. The dependence of electrical resistivity of Cu/Mn double-layered films, with Cu base layer thicknesses of 16.7–26.5 nm and Mn overlayer thicknesses of 1.5–7.5 nm, on the temperature from 100 to 330 K is studied. The most striking behaviour observed is that the temperature-dependent resistivity of the Cu/Mn films, the Mn overlayers of which have thicknesses greater than 5.0 nm, exhibits a well defined resistivity anomaly, while the resistivity of samples which have thicknesses smaller than 3.5 nm increases linearly with increasing temperature. According to our analysis the deposition of very thin Mn overlayers onto copper base layers causes an increase in the surface scattering of the Cu/Mn double-layered films with respect to those of the uncovered copper base films. No interface scattering could be detected in all the samples studied.

1. Introduction

It has long been known that the electrical resistivity of a single-layered thin metal film increases with decreasing film thickness because of size effects. Many studies have been published on the thickness dependence of the single-layered thin-film resistivity [1–4]. These studies have established that the resistivity of a polycrystalline single-layered film can be analysed taking only grain boundary scattering into account and that it is almost impossible to derive a reliable value for the surface scattering parameter in the presence of the dominating grain boundary scattering.

The resistivity of double-layered thin films has also been studied for the past decade [5–8]. In all studies, however, the resistivity data were interpreted in terms of the Lucas [9] and the Dimmich–Warkusz [10] models based on the Fuchs–Sondheimer [11, 12] formalism. Such models are not strictly valid for double-layered films since grain boundary scattering cannot be ignored in such systems.

In recent years, there has been extensive interest in the physical properties of double-layered and multi-layered films [13–16] since they provide a different class of materials with novel structural, electronic and magnetic properties. There have been few investigations [17–18] of Cu/Mn multilayered films; the dependences of resistivity and temperature coefficient of resistance (TCR) on the bilayer thickness show oscillatory behaviour.

Bulk copper has a face-centred cubic (FCC) structure and exhibits a very low resistivity ($1.17 \times 10^{-8} \Omega \text{ m}$). Bulk manganese has a very complex structure and exhibits a high electrical resistivity ($139 \times 10^{-8} \Omega \text{ m}$) and a low TCR. Manganese is very interesting and the least-studied transition metal in thin-film form. Thin manganese films exhibit a large resistivity and a negative TCR at small thicknesses [19]. The two metals copper and manganese are mutually insoluble.

Few published results appear for Au/Au double-layered films on a silicon substrate with $0.5 \mu\text{m}$ of SiO_2 [20] and for Cu/Ag double-layered films [21], in which the resistivity data have been interpreted by taking the combined model, including the grain boundary and surface scattering into account, formulated by Sambles *et al* [1]. According to our best knowledge, no experimental studies on Cu/Mn double-layered films have been carried out so far.

In this investigation the aim is to study the temperature dependence of the electrical resistivity of Cu/Mn double-layered films as a function of the increasing thicknesses of the manganese overlayers, from 1.5 to 7.5 nm. Another objective is to derive the values of the surface roughness parameter r_1 and the interface roughness parameter r_2 , using the combined model based on the grain boundary and angle-dependent surface scattering theory, including two specularity parameters, formulated by Sambles *et al* [1].

2. Experimental details

Two series of Cu/Mn double-layered samples were prepared by thermal evaporation of pure copper (99.99%) and manganese (99.99%) in a vacuum of about 5×10^{-7} Torr using two evaporation sources in a Varian 3119 research-and-development coater system. The Cu/Mn-I and Cu/Mn-II samples, with manganese overlayer thicknesses of 1.5–3.0 nm and 5.5–7.5 nm, respectively, were made in such a way that their copper base layers have thicknesses of 16.7–26.5 nm. The copper base films were deposited onto well cleaned glass substrates at 293 K and then manganese overlayers were deposited at 293 K onto these copper base layers without breaking the vacuum in order to avoid an interface of adsorbed gases between the two films.

The substrates were covered with a mask so that a Hall-bar pattern was obtained which is suitable for resistivity measurement. Resistivity measurements were carried out using a standard four-point DC technique with a constant current of 1 mA. The thicknesses of both the copper base and the manganese overlayers were measured using both a Taly Step (Tencor Instruments Alpha Step) and the atomic absorption technique in which the thickness is determined by measuring the weight of samples of known area. The microstructure of the samples was investigated by means of transmission electron microscopy combined with electron diffraction (ED). In order to determine the bulk resistivity $\rho_{\infty}(T)$ of the copper, a thick copper film of thickness 301 nm was also made at room temperature.

3. Results and discussion

3.1. Morphology of the Cu/Mn double-layered films

Some of the transmission electron micrographs and ED patterns of the Cu/Mn double-layered films are shown in figure 1. The transmission electron micrograph of the Cu/Mn-I film, with a base layer thickness of 26.5 nm and an overlayer thickness of 2.0 nm (sample 3.2) is shown in figure 1(a). This micrograph indicates that the grains of Cu and Mn cannot be distinguished, but the grains of Cu and Mn are more or less the same size. The widely differing contrasts of grains are due to diffraction conditions.

The ED patterns of two samples (3.2 and 4.7) are shown in figures 1(b) and 1(c). The continuity of the rings implies small-grained polycrystalline morphology. The ED patterns show two sets of diffraction rings for Cu and Mn. This indicates that the Cu–Mn interface is incoherent.

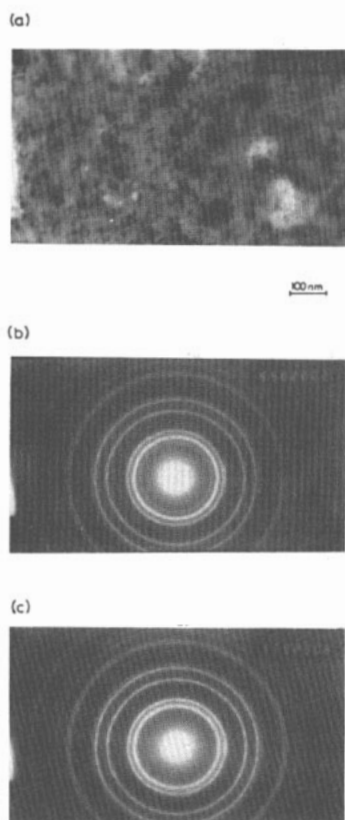


Figure 1. Transmission electron micrograph and ED patterns of the Cu/Mn double-layered films: (a) transmission electron micrograph and (b) ED pattern of covered copper base film of 26.5 nm thickness, with a Mn overlayer thickness of 2.0 nm (sample 3.2); (c) ED pattern of the covered copper base film, the base and Mn overlayer, thicknesses of which are 16.7 nm and 6.0 nm respectively (sample 4.7).

3.2. Temperature-dependent resistivity measurements

Temperature-dependent resistivity measurements of the Cu/Mn-I double-layered films are shown in figures 2 and 3 and those of Cu/Mn-II in figure 4. It can be seen from figures 2 and 4 that the temperature-dependent resistivity curves of both the Cu/Mn-I and the Cu/Mn-II double-layered films with different thicknesses are parallel to each other. This indicates that the grain boundary scattering should be the dominating contribution to the excess resistivity of the double-layered films. Sambles [22] and Dimmich [23] have shown that in the case of small-grained films the grain boundary scattering may dominate the double-layered or multi-layered film resistivity and may also reduce the effects of the surface and interface scattering. As seen from figures 3 and 4, the deposition of the Mn overlayers onto copper base films causes deviations from Matthiessen's rule and leads to an increase in the resistivity of Cu/Mn double-layered films in comparison with those of the uncovered copper base films. It has been reported in our earlier work [21] for the Cu/Ag double-layered films that this observed resistivity increase should be caused by increasing surface scattering owing to an artificial microscopic roughening of the surface by covering very thin overlayers. As can

be seen from figure 3 and table 1, this observed resistivity increase in magnitude decreases with increasing thickness of the Mn overlayer for a fixed thickness of the base film. This effect should be related to the decreasing fraction of conduction electrons (CEs) diffusely scattered at the surface [10].

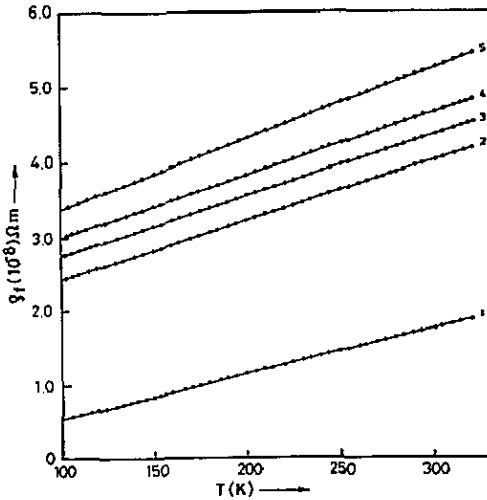


Figure 2. Total resistivity ρ_T versus temperature T for the Cu/Mn-I double-layered films: line 1, thick copper film of thickness 301 nm; line 2, covered copper film, base layer thickness of 26.5 nm, Mn overlayer thickness of 2.0 nm; line 3, covered copper film, base layer thickness of 22.5 nm, Mn overlayer thickness of 2.0 nm; line 4, covered copper film, base layer thickness of 20.2 nm, Mn overlayer thickness of 2.0 nm; line 5, covered copper film, base layer thickness of 17.5 nm, Mn overlayer thickness of 2.0 nm; —, curves fitted according to equation (1). Some of the experimental data points are not shown for clarity.

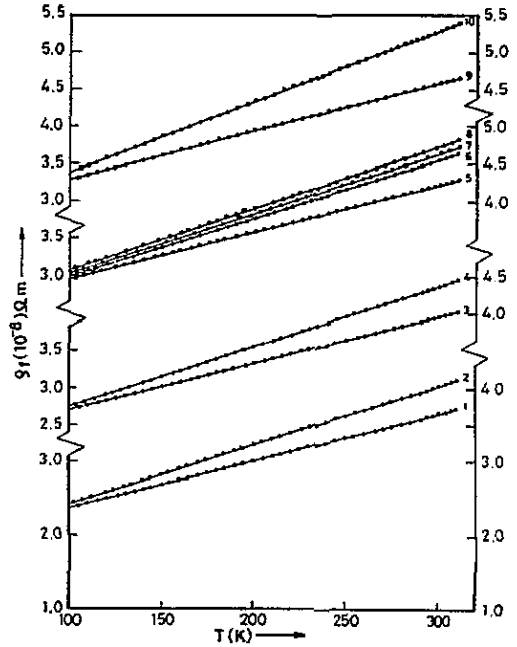


Figure 3. Total resistivity ρ_T versus temperature T for the uncovered copper base and the Cu/Mn-I double-layered films: line 1, uncovered copper base film thickness of 26.5 nm; line 2, covered copper film, base layer thickness of 26.5 nm, Mn overlayer thickness of 2.0 nm; line 3, uncovered copper base film, thickness of 22.5 nm; line 4, covered copper base film, base layer thickness of 22.5 nm, Mn overlayer thickness of 2.0 nm; line 5, uncovered copper base film, thickness of 20.2 nm; line 6, covered copper base film, base layer thickness of 20.2 nm, Mn overlayer thickness of 3.0 nm; line 7, covered copper base film, base layer thickness of 20.2 nm, Mn overlayer thickness of 2.0 nm; line 8, covered copper base film, base layer thickness of 20.2 nm, Mn overlayer thickness of 1.5 nm; line 9, uncovered copper base film, thickness of 17.5 nm; line 10, covered copper base film, base layer thickness of 17.5 nm, Mn overlayer thickness of 2.5 nm.

It is clearly seen from figures 3 and 4 that the dependence of the resistivity of the Cu/Mn-I and Cu/Mn-II double-layered films on the temperature exhibits two types of variation, depending on the thickness of the Mn overlayers, as detailed below.

(i) In the series Cu/Mn-I, with Mn overlayer thicknesses from 1.5 to 3.0 nm, the variation in the resistivity with temperature is linear in all the samples studied (figures 2 and 3).

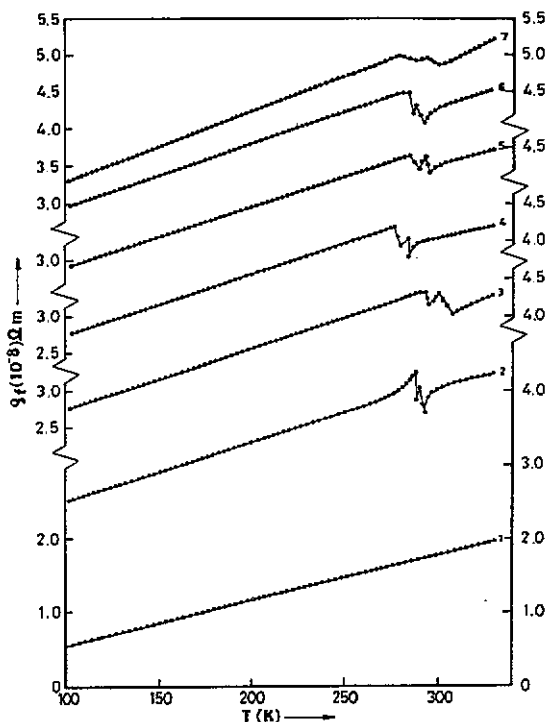


Figure 4. Total resistivity ρ_f versus temperature T for the Cu/Mn-II double-layered films: line 1, thick copper film of thickness 301 nm; line 2, covered copper film, base layer thickness of 23.0 nm, Mn overlayer thickness of 7.5 nm; line 3, covered copper film, base layer thickness of 21.5 nm, Mn overlayer thickness of 5.5 nm; line 4, covered copper film, base layer thickness of 21.2 nm, Mn overlayer thickness of 6.5 nm; line 5, covered copper film, base layer thickness of 20.3 nm, Mn overlayer thickness of 6.0 nm; line 6, covered copper film, base layer thickness of 19.1 nm, Mn overlayer thickness of 6.5 nm; line 7, covered copper film, base layer thickness of 16.7 nm, Mn overlayer thickness of 6.0 nm.

Table 1. The results of the analysis of the uncovered copper base and Cu/Mn-I double-layered films. The sample number $x.y$ in the first column corresponds to figure x and curve y ; d_{Cu}/d_{Mn} is the ratio of the thickness of the copper base layer to the thickness of the Mn overlayer; d and $\rho_f(273\text{ K})$ are the total thickness and total measured resistivity, respectively, of the double-layered films.

Sample	d_{Cu}/d_{Mn} (nm)	d (nm)	$\rho_f(273\text{ K})$ ($10^{-8}\ \Omega\ \text{m}$)	$r_1(273\text{ K})$	$r_2(273\text{ K})$
3.1	26.5 (base)	—	3.50	$r_0 = 0.000$ (0.0011)	—
3.2	26.5/2.0	28.5	3.82	0.093 (0.0011)	0.000 (0.0011)
3.3	22.5 (base)	—	3.80	$r_0 = 0.000$ (0.0011)	—
3.4	22.5/2.0	24.5	4.19	0.095 (0.0011)	0.000 (0.0011)
3.5	20.2 (base)	—	4.07	$r_0 = 0.000$ (0.0011)	—
3.6	20.2/3.0	23.2	4.39	0.092 (0.0011)	0.000 (0.0011)
3.7	20.2/2.0	24.2	4.43	0.105 (0.0011)	0.000 (0.0011)
3.8	20.3/1.5	21.8	4.54	0.107 (0.0011)	0.000 (0.0011)
3.9	17.5 (base)	—	4.42	$r_0 = 0.000$ (0.0011)	—
3.10	17.5/2.5	20.0	5.05	0.117 (0.0011)	0.000 (0.0011)

(ii) In the series Cu/Mn-II, with Mn overlayer thicknesses from 5.0 to 7.5 nm (figure 4), the temperature dependence of the resistivity of all the samples exhibits a well defined resistivity anomaly. The observed resistivity anomaly in the thickest sample, with a base layer thickness of 23.0 nm and a top layer thickness of 7.5 nm is much sharper than those observed in the other samples. The resistivity anomaly of the thinnest sample, with a base layer thickness of 16.7 nm and a top layer thickness of 6.0 nm is smeared out over a wider temperature range.

Considering the parallel connection of two conductors, an increase in the resistivity of a double-layered film is possible only if the scatterings of the CEs in the uncovered base and overlayer are not independent of each other. In that case, scattering processes are assumed to occur not only at grain boundaries but also at the new free surface or in the bulk of the overlayer [10, 24, 25].

It is known from our previous work [4] that in the single-layered copper films the excess resistivity can be determined only by the scattering of the CEs at the grain boundaries. For the series Cu/Mn-I and Cu/Mn-II, when very thin film Mn overlayers of thicknesses 1.5–7.5 nm is deposited onto the copper base layer, apart from the scattering at the grain boundaries, the CEs can now penetrate into this top layer and are diffusely scattered back from highly concentrated lattice defects at the new surface of the double layer and in the bulk of the overlayer. On the contrary the manganese atoms at the surface of the covered copper base film act as localized scattering centres of atomic dimensions which increase the surface scattering.

Wang *et al* [26] have studied the electronic structure and magnetism of Ni overlayers on Cu(001). They have found that a monolayer or two-layer Ni-on-Cu film is not magnetically 'dead' and that the effects of surface and interface on the magnetization are important and different. Bergmann [27] confirmed the existence of a dead magnetic layer of Ni on Cu when the Ni overlayer is less than about 2.5 times the atomic layer. We can accept from the viewpoint of the above considerations that the Cu/Mn double-layered films studied cannot be magnetically 'dead'. The resistivity anomaly observed in the Cu/Mn-II double-layered films, the Mn overlayers of which have thicknesses greater than 5.0 nm, should be related to antiferromagnetic-to-paramagnetic phase transition of the manganese. These observed resistivity anomalies appear to be sharper than those observed in bulk manganese [29] because of the size effects [30]. A similar well defined resistivity anomaly associated with antiferromagnetic-to-paramagnetic transition has been reported for single-layered Cr films [30].

In the series Cu/Mn-I, no resistivity anomaly has been observed in the vicinity of the Néel temperature. This behaviour may be due to the resistivity anomaly (or the Néel temperature) shifts above 330 K because of the size effects (such as dimensional effects, surface effects and interface effects) [26,31] and internal stress developed in the film [30, 31], which have important and different effects on the spin-density wave and magnetic properties.

3.3. Analysis of the resistivity measurements

Sambles and Elsom [32] have shown that the Fuchs theory [12, 13] may be used only as an approximation for thick samples or with very rough surface limits and that the Soffer [33] theory which involves an angle-dependent specular parameter $p(u)$ is a physically more realistic theory for thin samples. In this study, the more realistic theory of Soffer related to surface scattering has been employed since very thin films have been used for investigation.

The most widely used theory to describe the grain-boundary-enhanced resistivity is that of Mayadas and Shatzkes (MS) [34].

Sambles *et al* [1] have used a combination of the Soffer angle-dependent surface scattering and the MS grain boundary scattering theory to derive an expression for the total film resistivity. Recently, they have extended this combined model to the case of the films with two different surface roughness parameters. Their more general expression for the total resistivity, including both grain boundary and surface scattering covering two different specularly parameters r_1 and r_2 takes the form

$$\frac{\rho_{\infty}}{\rho_f} = f(\alpha) - \frac{4}{\pi} \int_0^{\pi/2} d\phi \int_0^1 du \frac{\cos^2 \phi}{H(u, \phi)} S\{\kappa H(u, \phi), p(u), t(u), u\} \quad (1)$$

where

$$f(\alpha) = 1 - \frac{3}{2}\alpha + 3\alpha^2 - 3\alpha^3 \ln(1 + 1/\alpha) \quad (2)$$

$$\alpha = [\lambda_{\infty}(T)/D][R/(1 - R)] \quad (3)$$

$$H(u, \phi) = 1 + \alpha / [(1 - u^2)^{1/2} \cos \phi] \quad (4)$$

$$S\{\kappa H(u, \phi), p(u), t(u), u\} = 3(u - u^3)[1 - \exp(-\kappa H/u)] \\ \times [1 - \bar{p} + (\bar{p} - pt) \exp(-\kappa H/u)] / 2\kappa H [1 - pt \exp(-2\kappa H/u)] \quad (5)$$

$$\bar{p} = \frac{1}{2}(p + t)$$

and the specularly parameters p and t are functions of u of the form

$$p(u) = \exp[(-4\pi r_1 u)^2] \quad (6)$$

$$t(u) = \exp[(-4\pi r_2 u)^2]. \quad (7)$$

The parameters $p(u)$ and $t(u)$ in equations (6) and (7) represent the surface and interface specularities respectively.

The following assumptions can be made from the transmission electron micrograph and ED patterns (figure 1).

(a) The deposition of the manganese overlayer does not influence the grain boundary scattering or volume parameters λ_{∞} , ρ_{∞} , R and D , but the surface scattering of the covered copper base films.

(b) By depositing very thin manganese overlayers onto the copper base films at 293 K, we can assume that the grain diameters of the uncovered and covered base film are the same since interdiffusion effects are excluded.

(c) The effect of the manganese overlayer on the excess resistivity of the Cu/Mn double-layered film is included in equation (1) through κ .

The grain diameter D and grain boundary reflection coefficient R of the single-layered copper films have been derived in our previous work [4], where we found that $R = 0.38$ and $D/d = 1.12$ (D is the average grain diameter and d is the film thickness). Accordingly, the values of R and D/d can also be used for the Cu/Mn-I and Cu/Mn-II double-layered films. The resistivity data for the copper film with a thickness of 301 nm are used to determine the bulk resistivity $\rho_{\infty}(T)$ of copper.

The combination of temperature-dependent resistivity measurements (figures 2–4) from 100 to 330 K with transmission electron micrographs and ED patterns enable us to apply a model that combines grain boundary scattering and angle-dependent surface scattering

theory. Therefore, equation (1) of the combined model can be used irrespective of whether only surface scattering or both surface and interface scattering are employed together for the analysis of the resistivity data of the Cu/Mn double-layered films, where grain boundary scattering is the main contribution.

In our earlier work [4], the values of surface roughness parameters r_0 of the single-layered copper films were found to be 0.00 for all the films studied, at 293 K, except for the thinnest (16.6 nm) samples which had $r_0 = 0.025$. We have again fitted the equation (7) of the combined model (see [4]) to the resistivity data of the uncovered copper base films of the series Cu/Mn-I and Cu/Mn-II, at 273 K, using the values $R = 0.38$, $D/d = 1.12$, $(\rho\lambda)_\infty = 0.66 \times 10^{-5} \Omega \text{ m}^2$ [34], $\rho_f(273 \text{ K})$ and $\rho_\infty(273 \text{ K})$. The values obtained for the surface roughness parameters r_0 , which are given in tables 1 and 2, show that the scattering of the CEs at the surfaces of the uncovered copper films is almost specular in all the base films studied. The results of the fitting are indicated in figure 3 as full curves together with experimental data for the uncovered base films. It is clearly seen that there is a good agreement between the theory and experiments.

Table 2. The results of the analysis of the uncovered copper base and Cu/Mn-II double-layered films. The meanings of the symbols are the same as in table 1.

Sample	$d_{\text{Cu}}/d_{\text{Mn}}$ (nm)	d (nm)	$\rho_f(273 \text{ K})$ ($10^{-8} \Omega \text{ m}$)	$r_1(273 \text{ K})$	$r_2(273 \text{ K})$
	23.0 (base)	—	3.65	$r_0 = 0.000 (0.0011)$	—
4.2	23.0/7.5	30.5	3.92	0.050 (0.0011)	0.000 (0.0011)
	21.5 (base)	—	3.90	$r_0 = 0.000 (0.0011)$	—
4.3	21.5/5.5	27.0	4.16	0.052 (0.0011)	0.000 (0.0011)
	21.2 (base)	—	3.95	$r_0 = 0.000 (0.0011)$	—
4.4	21.2/6.5	27.7	4.15	0.052 (0.0011)	0.000 (0.0011)
	20.3 (base)	—	4.05	$r_0 = 0.000 (0.0011)$	—
4.5	20.3/6.0	26.3	4.31	0.053 (0.0011)	0.000 (0.0011)
	19.1 (base)	—	4.15	$r_0 = 0.000 (0.0011)$	—
4.6	19.1/6.5	25.6	4.41	0.053 (0.0011)	0.000 (0.0011)
	16.7 (base)	—	4.55	$r_0 = 0.014 (0.0011)$	—
4.7	16.7/6.0	22.7	4.93	0.079 (0.0011)	0.014 (0.0011)

In order to derive values for the surface roughness parameter r_1 and the interface roughness parameter r_2 of the Cu/Mn double-layered films, at 273 K, we have fitted equation (1) of the combined model to the resistivity data for both Cu/Mn-I and Cu/Mn-II samples in which the grain boundary scattering dominates, using the known values of R , D/d , $(\rho\lambda)_\infty$, $\rho_\infty(273 \text{ K})$ and $\rho_f(273 \text{ K})$. From this fitting procedure, the values obtained for r_1 and r_2 are listed in tables 1 and 2. As can be seen in tables 1 and 2, we have found that the values of surface roughness parameters are $r_1 = 0.092\text{--}0.117$ and $r_1 = 0.050\text{--}0.079$ for Cu/Mn-I and Cu/Mn-II double-layered films, respectively. The interface scattering parameters have been found to be $r_2 = 0.00$ for both series for all the samples studied except for the sample with a base layer thickness of 16.7 nm and a Mn overlayer thickness of 6.0 nm which had $r_2 = 0.014$. If we transform these values of r_1 and r_2 into the well known Fuchs specularly parameters, in the high-temperature limit, at 273 K [32], we then find the surface specularly parameters $p = 0.68\text{--}0.52$ and $p = 0.92\text{--}0.78$ for Cu/Mn-I and Cu/Mn-II double-layered films, respectively, and the interface specularly parameter $t = 1$ for both series. The interface scattering $t = 0.99$ observed for the sample with a base layer thickness of 16.7 nm and an overlayer thickness of 6.0 nm is too small.

It can be seen from tables 1 and 2 that the deposition of the manganese overlayer causes an increase in the surface roughness parameters of the covered copper base films in comparison with those of the uncovered base layers, but no interface scattering in all the samples studied. The observed surface scattering increasing decreases as the thicknesses of the Mn overlayers increase for a fixed copper base film thickness. This effect should be related to decreasing fraction of the CES, diffusely scattered at the surfaces of the Cu/Mn films.

The derived values of the surface scattering parameters, $r_1 = 0.100$ – 0.125 (or $p = 0.65$ – 0.50), at 293 K, for the Cu/Mn-I double-layered films, with Cu base layer thicknesses of 16.7–26.5 nm and Mn overlayer thicknesses of 1.5–3.0 nm are greater than those obtained in our earlier work [21] for Cu/Ag double-layered films, with Cu base layer thicknesses of 16.6–26.3 nm and top layer thicknesses of 1.5–3.5 nm, where $r_1 = 0.025$ – 0.07 (or $p = 0.98$ – 0.84). de Vries [20] has also found much larger increase in surface scattering when Au films are covered with one of the transition elements Fe, Co or Ni than when Au films are covered with Au.

4. Conclusion

The temperature dependence of the electrical resistivity of Cu/Mn double-layered films show two types of behaviour, depending on the thicknesses of the manganese overlayers. The variation in the resistivity of the Cu/Mn films, the manganese overlayers of which have thicknesses greater than 5.0 nm, with the temperature exhibits a well defined resistivity anomaly which should be related to the antiferromagnetic-to-paramagnetic transition, but the temperature-dependent resistivity of the Cu/Mn films, the manganese overlayers of which have thicknesses smaller than 3.0 nm, show a linear variation in the temperature range 100–330 K.

The resistivity data of both series Cu/Mn double-layered films are analysed using equation (1) of the combined model based on the MS theory for grain boundary scattering and the Soffer theory for angle-dependent surface scattering, including two specularly parameters r_1 and r_2 , formulated by Sambles *et al* [1]. Our analysis has shown that the deposition of the manganese overlayers, with thicknesses from 1.5 to 7.5 nm, leads to an increase in the surface roughness parameters of the covered copper base films in comparison with those of the uncovered copper base films. The CES are scattered by the covered copper film surface which is artificially roughened by covering with the Mn overlayers but not by the interface.

Acknowledgments

We would like to thank Z Z Öztürk and A Altındal for preparing the Cu/Mn double-layered films at Tübitak, Marmara Research Centre, and Professor Ş Bor and K Aydınoglu for taking the electron micrographs at the Metallurgical Engineering Department, Middle East Technical University.

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