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The effects of overlayer thicknesses on the electrical resistivity of polycrystalline Cu/Cr double-layered thin films

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Abstract. The total electrical resistivity of the Cu/Cr double-layered thin films, the copper base layers of which have thicknesses of 13.0–26.0 nm, is studied as a function of the temperature in the interval 100–300 K and overlayer thickness. The temperature dependence of the electrical resistivity show two types of behaviour. The temperature-dependent resistivity of the Cu/Cr films, the Cr overlayers of which have thicknesses of 1.5 and 2 nm, shows a linear variation, but the variation in the resistivity of the Cu/Cr films, the Cr overlayers of which have thicknesses greater than 7.5 nm, with the temperature exhibits an oscillatory behaviour over the whole temperature range from 100 to 300 K. Our analysis has shown that the observed resistivity increase is caused by an increased surface scattering of the Cu/Cr-I double-layered film in comparison with those of the uncovered copper base films. The interface scattering cannot be the cause of the excess resistivity of both Cu/Cr-I and Cu/Cr-II double-layered films.

1. Introduction

In recent years, there has been extensive interest in the physical properties of double-layered and multilayered films [1–3]. These multilayers or double layers of two dissimilar metals with different lattice parameters and crystal structures exhibit very novel and interesting structural, electronic and magnetic properties which can be different from those of either of the constituent metals. There has been few investigations [4] of Cu/Cr multilayered films; the dependences of resistivity and temperature coefficient of resistance (TCR) on the bilayer thickness show oscillatory behaviour.

The resistivity of double-layered thin films has been studied for the past decade. Several workers [5–8] have analysed the resistivity data of double-layered films in terms of the Lucas [9] theory based on only the Fuchs–Sondheimer formalism [10]. A more general account of electrical conduction in the multilayered or double-layered systems was given by Dimmich and Warkusz [11] and Dimmich [12], who allowed for both grain boundary and angle-independent surface scattering, formulated by Fuchs and Sondheimer. Few published results [13–15] appear for double-layered films, in which the resistivity data have been interpreted by taking the combined model (including grain boundary and angle-dependent surface scattering) formulated by Sables *et al* [16] into account. According to our best knowledge, no experimental studies on Cu/Cr double-layered films have been carried out so far.

Bulk copper has a face-centred cubic structure and exhibits a very low resistivity ($1.67 \times 10^{-8} \Omega \text{ m}$), while bulk chromium has a body-centred cubic structure and exhibits a high resistivity ($12.9 \times 10^{-8} \Omega \text{ m}$). Bulk chromium is an interesting metal because it has a unique spin-density wave (SDW) whose period is incommensurate with the lattice periodicity below the Néel temperature T_N [17–20]. The physical properties of chromium are

further complicated in thin-film form because of size effects and grain boundary scattering. In chromium films, the Néel temperature and the properties of the SDW are affected by both the tension developed in the film and grain boundaries [21–23].

The first aim of this investigation is to study the temperature dependence of the electrical resistivity of Cu/Cr double-layered films as the chromium overlayer thicknesses are varied from 1.5 to 11.0 nm. The second aim is to analyse the resistivity data of the Cu/Cr double-layered films in terms of the combined model based on the grain boundary and angle-dependent surface scattering theory, which covers the surface and interface roughness parameters r_1 and r_2 and which was formulated by Sambles *et al* [16].

2. Experimental aspects

The Cu/Cr double-layered films used in this study were prepared by thermal evaporation of pure copper (99.99%) and chromium (99.99%) in a vacuum of about 5×10^{-7} Torr using two evaporation sources in a Varian 3119 research-and-development coater system. We were not able to measure the deposition rates of both the Cu and Cr films produced since the quartz crystal thickness monitor of the equipment was out of order during the sample preparation. However, whilst the thickness of each layer was determined by the atomic absorption technique, the total thickness was measured by a Taly Step. The copper base films were deposited onto well cleaned glass substrates at 293 K and then chromium 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. Cu/Cr-I and Cu/Cr-II samples, with chromium overlayer thicknesses of 1.5–3.0 nm and 7.5–11.0 nm, respectively, were made in such a way that their copper base layers have thicknesses of 14.0–26.0 nm. 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. The substrates were covered with a mask so that a Hall-bar pattern was obtained which is suitable for resistivity measurements. The thicknesses of both the copper base and the chromium overlayers were measured using both a Taly Step (Tencor Instruments Alpha Step) and the atomic absorption technique.

The resistivity measurements were carried out in a vacuum of about 10^{-5} Torr, using a standard four-probe technique with a constant current of 1 mA. A constant-current source, the sensitivity of which is approximately 1 μ A and the relative error is 0.1%, was used during the resistivity measurements. The voltage on the samples was measured with a Hewlett–Packard digital multimeter (model 3466A) with an accuracy of 0.1%. Hence, the resistance of the samples was measured with an accuracy of 0.2%. The temperature of the samples was changed by a conductive heater placed in the cryostat, and measured with a copper–constantan thermocouple with an accuracy of ± 0.1 K.

3. Theory

The most widely used theory to describe the surface scattering-enhanced resistivity is the Fuchs [10] theory, based upon an angle-independent specularity parameter p , in spite of the presence of the physically more realistic Soffer [24] theory. Sambles and Elsom [25] have shown that the two theories are equivalent only when the sample thickness is greater than the bulk mean free path λ_{∞} and/or at high temperatures. Sambles *et al* [16] have used a combination of the Soffer surface scattering theory and the Mayadas–Shatzkes [26]

theory to derive an expression for the total film resistivity. Extending this combined model to the case of the films with two different surface roughness parameters, they have found a more general expression for total film resistivity, including both grain boundary and surface scattering covering two different specularly parameters r_1 and r_2 , which is given by

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

where

$$\alpha = \frac{\lambda_{\infty}(T)}{D} \frac{R}{1-R}. \quad (2)$$

Here $f(\alpha)$ is the grain boundary scattering function [27], $\rho_{\infty}(T)$ is the total resistivity of the bulk material at temperature T , R is the grain boundary reflection coefficient ($0 \leq R \leq 1$), D is the average grain diameter in the film, $\lambda_{\infty}(T)$ is the mean free path of the bulk material at temperature T without grain boundaries, $\kappa = d/\lambda_{\infty}$ is the reduced film thickness (d is the mean sample thickness), and $p(u)$ and $t(u)$ in equation (1) are the surface and interface specularities, respectively. The parameters $H(u, \phi)$, $S\{\kappa H(u, \phi), p(u), t(u), u\}$, $p(u)$ and $t(u)$ in equation (1) are defined in [15].

4. Results and discussion

4.1. Microstructure of the Cu/Cr double-layered films

The microstructure of the samples was investigated by means of transmission electron microscopy combined with electron diffraction (ED). Some of the ED patterns of Cu/Cr double-layered films are shown in figure 1. The transmission electron micrographs indicate that the grains of Cu and Cr cannot be distinguished, but the grains of Cu and Cr are more or less the same size. The ED patterns show two sets of diffraction rings for Cu and Cr. This indicates that the Cu–Cr interface is incoherent. The continuity of the rings implies a small-grained polycrystalline morphology. The following assumptions can be made from the transmission electron micrographs and ED patterns.

(a) The deposition of the chromium overlayer does not influence the grain boundary scattering or volume parameters λ_{∞} , ρ_{∞} , R and D but does affect surface scattering of the covered copper base films since interdiffusion effects are excluded. Accordingly, by depositing very thin chromium overlayers onto copper base films, at 293 K, we can assume that the grain diameters of the uncovered and covered base films are the same.

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

4.2. Temperature-dependent resistivity measurements

Temperature-dependent resistivity measurements of the Cu/Cr-I and Cu/Cr-II double-layered films are given in figures 2 and 3. As seen clearly from figure 2, the deposition of the Cr overlayers onto copper base films causes deviations from Matthiessen's rule and leads to an increase in the resistivity of Cu/Cr double-layered films with respect to those of the uncovered copper base films. This observed resistivity increase in magnitude decreases as the Cr overlayer thickness increases for a fixed thickness of the copper base film. It can also

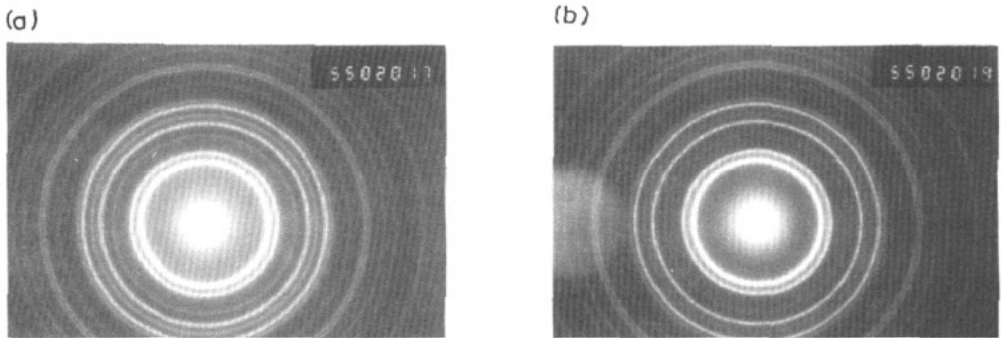


Figure 1. ED patterns of both Cu/Cr-I and Cu/Cr-II double-layered films: (a) ED pattern of covered copper base film of 17.6 nm thickness, with a Cr overlayer of 2.0 nm; (b) ED pattern of a covered copper base film of 15.0 nm, with a Cr overlayer thickness of 9.5 nm.

be seen from figure 2 that the temperature-dependent resistivity curves of the Cu/Cr films with different thicknesses are parallel to each other. This indicates that the grain boundary scattering should also dominate the excess resistivity of the double-layered films [14, 15]. It has also been reported in our earlier work [14, 15] that this observed resistivity increase is caused by increased surface scattering at the artificially roughened new free surface of the overlayer, by covering very thin overlayers but not by the interface scattering.

It can be apparently seen from figures 2 and 3 that the temperature dependence of the resistivity of Cu/Cr-I and Cu/Cr-II shows two types of behaviour as the Cr overlayer thicknesses are varied from 1.5 to 10.0 nm. In the series Cu/Cr-I, with Cr overlayer thicknesses of 1.5 and 2.0 nm, the temperature dependence of the resistivity exhibits a linear variation (figure 2), while in the series Cu/Cr-II, with Cr overlayer thicknesses from 7.5 to 10.0 nm, the temperature dependence of the resistivity shows an oscillatory behaviour (figure 3) over the whole temperature range from 100 to 300 K in all the samples studied.

It is known from our previous work [27] that in the single-layered copper films the excess resistivity can be determined only by the scattering of the conduction electrons (CEs) at the grain boundaries. In the case of the Cu/Cr-I and Cu/Cr-II double-layered films, by depositing thin Cr overlayers of thicknesses 1.5–10.0 nm the CEs can now penetrate into this overlayer and are diffusely scattered at the new surface of the double layer and in the bulk of the overlayer and also by spin disorder in the Cu/Cr-II samples.

Wang *et al* [29] have studied the electronic structure and magnetism of Ni overlayers on Cu(001). They have found that 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. Rau [30] showed that even a monolayer of Ni on a Cu(001) substrate is magnetic, but with a reduced magnetic moment. Pierce and Siegmann [31] concluded that ferromagnetism occurs when the Ni overlayer is only one to two atomic layers thick. It can be concluded from the viewpoint of the above considerations that the Cu/Cr double-layered films studied cannot be magnetically 'dead'.

Single-crystal neutron diffraction experiments [17–20] have shown that the structure of bulk chromium differs slightly from that of a classical antiferromagnet. Chromium has a sinusoidal spin order and this antiferromagnetic ordering can only be explained by the formation of a SDW whose period is incommensurate with the lattice periodicity below the Néel temperature $T_N = 310$ K, denoted ISDW. The electrical resistivity of chromium has a cusped minimum at $T_N = 310$ K. This anomaly is also ascribed to the modification of the Fermi surfaces induced by the formation of the energy gap [32]. It has been reported that

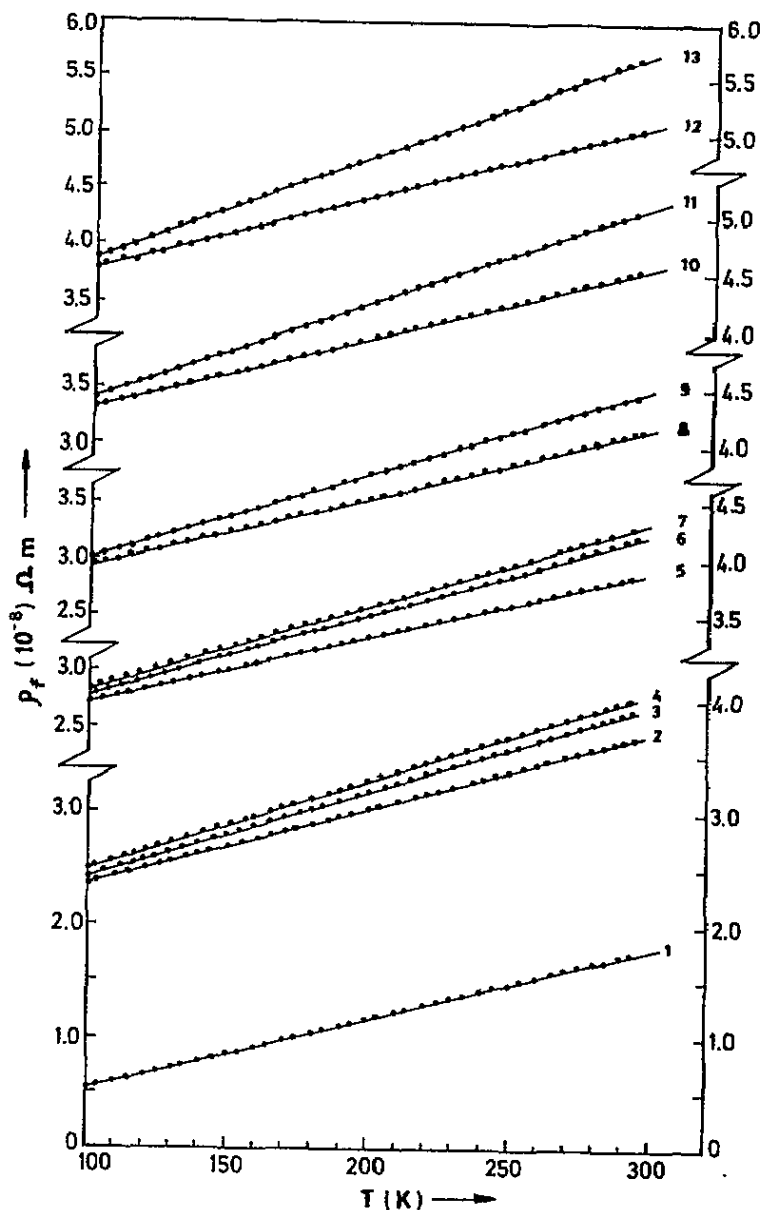


Figure 2. Total resistivity ρ_f versus temperature T for the uncovered copper base film and Cu/Cr-I double-layered films: line 1, thick copper film of thickness 301 nm; line 2, uncovered copper base film, of thickness 26.0 nm; line 3, covered copper base film of 26.0 nm, with a Cr overlayer thickness of 2.0 nm; line 4, covered copper base film of 26.0 nm, with a Cr overlayer thickness of 1.5 nm; line 5, uncovered copper base film, of thickness 23.0 nm; line 6, covered copper base film of 23.0 nm, with a Cr overlayer thickness of 2.0 nm; line 7, covered copper base film of 23.0 nm, with a Cr overlayer thickness of 1.5 nm; line 8, uncovered copper base film, of thickness 20.3 nm; line 9, covered copper base film of 20.3 nm, with a Cr overlayer thickness of 2.0 nm; line 10, uncovered copper base film, of thickness 17.6 nm; line 11, covered copper base film of 17.6 nm, with a Cr overlayer thickness of 2.0 nm; line 12, uncovered copper base film, of thickness 14.0 nm; line 13, covered copper base film of 14.0 nm, with a Cr overlayer thickness of 2.0 nm.

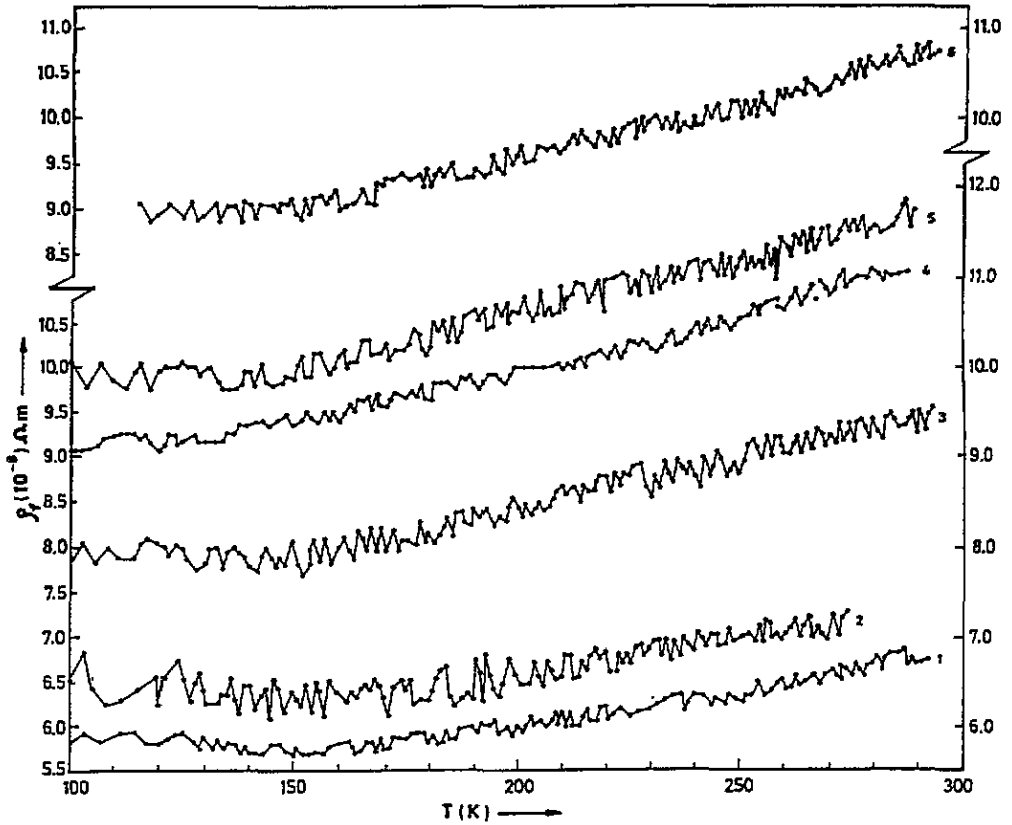


Figure 3. Total resistivity ρ_f versus temperature T for the Cu/Cr-II double-layered films: line 1, covered copper base film of 21.0 nm, with a Cr overlayer thickness of 8.5 nm; line 2, covered copper base film of 18.0 nm, with a Cr overlayer thickness of 10.0 nm; line 3, covered copper base film of 15.0 nm, with a Cr overlayer thickness of 9.5 nm; line 4, covered copper base film of 14.0 nm, with a Cr overlayer thickness of 8.5 nm; line 5, covered copper base film of 13.0 nm, with a Cr overlayer thickness of 9.5 nm; line 6, covered copper base film of 13.5 nm, with a Cr overlayer thickness of 9.0 nm.

the Cr-Fe alloys containing more than 3 at.% do not have an ISDW but a commensurate antiferromagnetic SDW (CSDW) and show a negative TCR over the whole temperature range below T_N [33].

Hara and Sakata [23] have observed a similar extremely large resistivity anomaly (or negative TCR) in the resistivity of both Cr-Fe alloy films containing 6.4–8.5 at.% Fe, with a CSDW and pure Cr films, deposited at 20°C over the whole temperature range 100–370 K. They have concluded from these experimental results that the magnetic structure of 20°C pure Cr films, which consist of very fine subgrains with an average diameter smaller than the period of the SDW, has changed into the SDW commensurate with lattice periodicity.

They have also shown that the Néel temperature and the properties of the SDW are affected both by the tension developed in the films and by grain boundaries, which have an effect like that of localized moments. The former increases the Néel temperature, while the latter disturbs the periodicity of the SDW; the incommensurate-commensurate (I-C) transition occurs when the diameter of a grain becomes comparable with or shorter than the

period of the ISDW.

To the best of our knowledge, the temperature dependence of the resistivity of the Cu/Cr double-layered films showing an oscillatory behaviour, depending on the Cr overlayer thicknesses, over the temperature range from 100 to 300 K, has not been reported so far.

In this study, the extremely large anomaly (or oscillatory behaviour) observed in the resistivity of the Cu/Cr-II double-layered films, with Cr overlayer thicknesses from 8.5 to 10.0 nm, may indicate that the I-C transition of the SDW has occurred in all the Cu/Cr-II films studied since, in the Cr films with ISDW, a resistivity anomaly occurs only just below T_N , but not over such a wide temperature range [22, 23]. The appearance of the anomalies observed in Cu/Cr-II double-layered films is considerably different from the appearance of those observed in the 20°C single-layered Cr films used by Hara and Sakata, because of different experimental conditions, grain diameters and size effects (such as surface and interface effects).

Accordingly, we can conclude that the extremely large resistivity anomaly may be explained in terms of the change in the magnetic structure of the Cu/Cr-II double-layered films into the CSDW. This explanation which is based on the mechanism of the I-C transition seems to be plausible since such a extremely large anomaly can never be expected to occur in Cr films with the ISDW.

In the series Cu/Cr-I, the Cr overlayers of which have thicknesses smaller than 3.0 nm, no resistivity anomaly has been observed over the whole temperature range studied. This behaviour indicates that the Cr has a SDW, whose period is incommensurate with the lattice periodicity. This linear variation in the temperature-dependent resistivity of Cu/Cr-I double-layered films should be explained in terms of the shift of Néel temperature of the Cu/Cr double-layered system above 300 K because of the tension developed in the film and the size effects (such as dimensional effects and surface and interface effects) [21–23].

It is reported in our previous work [15] that for Cu/Mn double-layered films with an ISDW, the Mn overlayers of which have thicknesses 5.0–7.5 nm, the temperature dependence of the resistivity exhibits a well defined resistivity anomaly, while the temperature-dependent resistivity of the samples, the Mn overlayers of which have thicknesses smaller than 3.5 nm, increases linearly with increasing temperature. Furthermore, in this study we have prepared Cu/Mn double-layered films, the Mn overlayers of which have thicknesses greater than 7.5 nm. These Cu/Mn films also show a similar oscillatory behaviour, this should be due to the change in the magnetic structure in the grain which changes into the CSDW (see figure 4).

4.3. Analysis of the resistivity measurements

As pointed out in our results and discussion (section 4.1), by depositing very thin Cr overlayers onto the copper base films, at 293 K, it can be assumed that volume parameters λ_∞ , ρ_∞ , R and D of the uncovered and covered base films are the same. We have found in our previous work [27] that the grain boundary reflection coefficient $R = 0.38$ and the ratio $D/d = 1.12$ (D is the average grain diameter and d is the film thickness). Accordingly, these values of R and D/d can also be used for the Cu/Cr double-layered films. The resistivity data of the copper film with a thickness of 301 nm are used to determine the bulk resistivity $\rho_\infty(T)$ of copper.

As described extensively in our earlier work [27], firstly we have fitted equation (7) of the combined model in [27], including both grain boundary and surface scattering covering only one specularly parameter $p(u)$, to the resistivity data of the uncovered copper base films of the Cu/Cr-I series, at 293 K. The obtained values of surface roughness parameters r_0 are listed in table 1. These values show that the scattering of CEs at the surfaces of

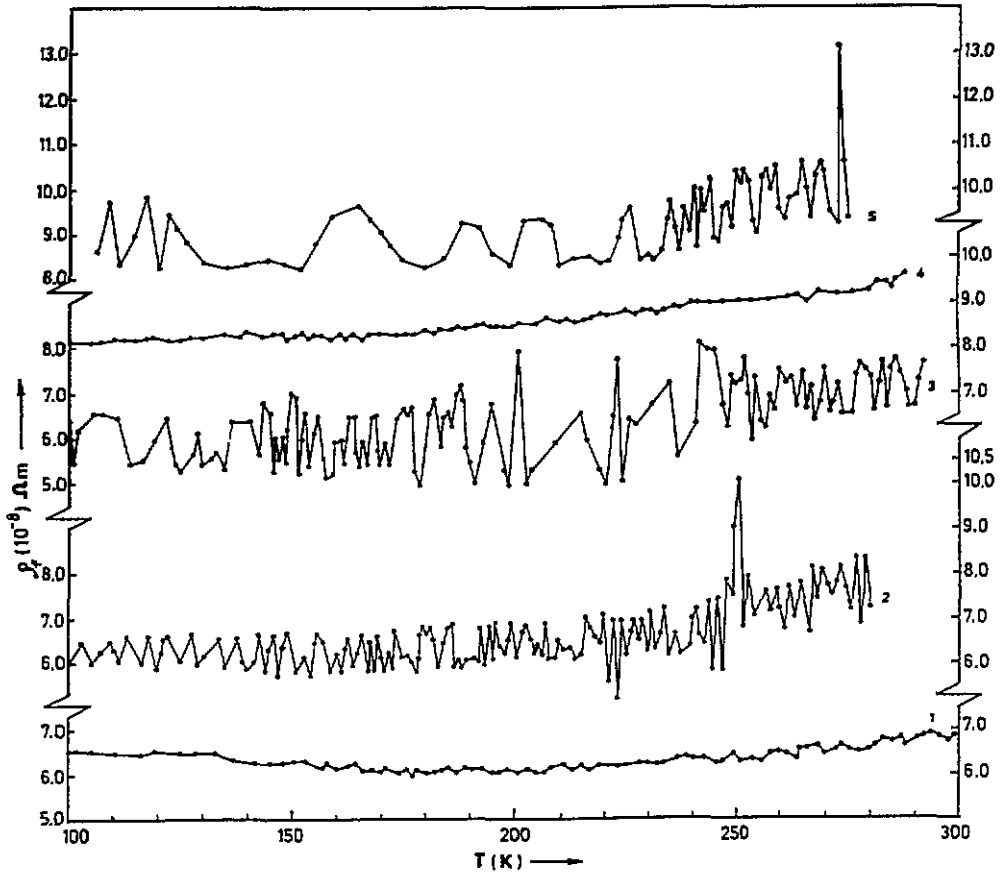


Figure 4. Total resistivity ρ_f versus temperature T for the Cu/Mn double-layered films: line 1, covered copper base film of 21.5 nm, with a Mn overlayer thickness of 9.0 nm; line 2, covered copper base film of 20.0 nm, with a Mn overlayer thickness of 11.0 nm; line 3, covered copper base film of 19.5 nm, with a Mn overlayer thickness of 11.0 nm; line 4, covered copper base film of 15.0 nm, with a Mn overlayer thickness of 8.0 nm; line 5, covered copper base film of 13.5 nm, with a Mn overlayer thickness of 10.0 nm.

the uncovered copper films is almost specular in all the base films studied. The results of fitting are shown in figure 2 as full curves together with experimental data for uncovered base films.

Secondly, we have fitted equation (1) of the combined model to the resistivity data of Cu/Cr-I samples, in which the grain boundary scattering dominates, using the values $R = 0.38$, $D/d = 1.12$, $(\rho\lambda)_\infty = 0.66 \times 10^{-15} \Omega \text{ m}^2$ [26], ρ_∞ (293 K) and ρ_f (293 K) in order to derive values for the surface roughness and interface roughness parameters r_1 and r_2 of the Cu/Cr-I films. As can be seen in table 1, the surface and interface roughness parameters were found to be $r_1 = 0.102\text{--}0.129$ and $r_2 = 0.000$, respectively, for all the samples studied. If we transform these values of r_1 and r_2 into the well known Fuchs specularity parameters, in the high-temperature limit, at 293 K [25], we then find the surface and interface specularity parameters $p = 0.65\text{--}0.46$ and $t = 1.00$ for all the samples studied. The interface scattering parameter $t = 0.99$ (or $r_2 = 0.015$) observed for the sample with a base layer thickness of 14.0 nm and an overlayer thickness of 2.0 nm is too small. It is

Table 1. The results of the analysis of the uncovered copper base and Cu/Cr-I double-layered films. The sample number $x.y$ in the first column corresponds figure x and curve y ; d_{Cu} corresponds to the thickness of the copper base layer and d_{Cr} to the thickness of the Cr overlayer; d and ρ_f (293 K) are the total thickness and measured resistivity of the Cu/Cr-I films, respectively.

Sample	d_{Cu}/d_{Cr} (nm/nm)	d (nm)	ρ_f (293 K) ($10^{-8} \Omega m$)	r_1 (293 K)	r_2 (293 K)
2.2	26.0 (base)	26.0	3.65	$r_0 = 0.000$ (0.0011)	—
2.3	26.0/2.0	28.0	3.90	0.102 (0.0011)	0.000 (0.0011)
2.4	26.0/1.5	27.5	3.97	0.114 (0.0011)	0.000 (0.0011)
2.5	23.0 (base)	23.0	3.85	$r_0 = 0.000$ (0.0011)	—
2.6	23.0/2.0	25.0	4.17	0.108 (0.0011)	0.000 (0.0011)
2.7	23.0/1.5	24.5	4.28	0.118 (0.0011)	0.000 (0.0011)
2.8	20.3 (base)	20.3	4.09	$r_0 = 0.000$ (0.0011)	—
2.9	20.3/2.0	22.3	4.46	0.110 (0.0011)	0.000 (0.0011)
2.10	17.6 (base)	17.6	4.50	$r_0 = 0.000$ (0.0011)	—
2.11	17.6/2.0	19.6	5.03	0.126 (0.0011)	0.000 (0.0011)
2.12	14.0 (base)	14.0	5.10	$r_0 = 0.015$ (0.0011)	—
2.13	14.0/2.0	16.0	5.66	0.129 (0.0011)	0.015 (0.0011)

apparent from table 1 that the deposition of the chromium overlayer causes an increase in the surface roughness parameters of the covered copper base films with respect to those of the uncovered base films but no interface scattering in all samples studied.

In the series Cu/Cr-II, which show an oscillatory behaviour over the whole temperature range, we have not fitted equation (1) to the resistivity data since it is rather difficult to derive a reliable value for the surface scattering parameter in the presence of the magnetic contribution resulted from spin-disorder scattering. However, it should be noted that the CEs are not scattered by interface but by the covered copper film surface which is artificially roughened.

The derived values of surface scattering parameters $r_1 = 0.102$ – 0.129 (or $p = 0.65$ – 0.46) at 293 K, for Cu/Cr-I double-layered films, with Cu base layer thicknesses of 14.0–26.0 nm and Cr overlayer thicknesses of 1.5–3.0 nm, are nearly the same as those given elsewhere [15] for Cu/Mn double-layered films but are greater than those obtained in our earlier work [14] 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 $r_1 = 0.0025$ – 0.07 (or $p = 0.98$ – 0.84). de Vries [13] has also found that the increase in surface scattering when Au films are covered with one of the transition elements Fe, Co or Ni is much larger than when Au films are covered with Au.

5. Conclusion

In conclusion, the most striking behaviour observed in this study is that the temperature-dependent resistivity of Cu/Cr films, the Cr overlayers of which have thicknesses smaller than 2.0 nm, increases linearly with increasing temperature, while the temperature-dependent resistivity of the Cu/Cr films, the Cr overlayers of which have thicknesses greater than 7.5 nm, exhibits an oscillatory behaviour over the whole temperature range from 100 to 300 K. The change observed in the temperature-dependent resistivity from linear to oscillatory behaviour as the chromium overlayer thicknesses are varied from 3.0 to 10.0 nm may be explained in terms of the I–C transition of the SDW, resulting from the disturbance of its periodicity by the grain boundaries.

The resistivity data of Cu/Cr-I double-layered films are analysed using equation (1) of the combined model, formulated by Sambles *et al* [16]. According to our analysis the deposition of very thin Cr overlayers onto copper base layers causes an increase in the surface roughness parameters of the covered copper base films in comparison with those of the uncovered copper base films. The interface scattering cannot be responsible for this resistivity increase observed in both the Cu/Cr-I and the Cu/Cr-II double-layered films.

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