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2006 J. Phys.: Condens. Matter 18 3401

(http://iopscience.iop.org/0953-8984/18/13/008)

INSTITUTE OF PHYSICS PUBLISHING JOURNAL OF PHYSICS: CONDENSED MATTER

J. Phys.: Condens. Matter **18** (2006) 3401–3408 doi:10.1088/0953-8984/18/13/008

Size effects under a strong magnetic field: transverse magnetoresistance of thin gold films deposited on mica

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Received 8 November 2005, in final form 8 January 2006
Published 13 March 2006
Online at stacks.iop.org/JPhysCM/18/3401

Abstract

We report measurements of transverse magnetoresistance where the signal can be attributed to electron–surface scattering, together with measurements of the surface roughness of the films on an atomic scale. The measurements were performed with a scanning tunnelling microscope (STM) on four thin gold films evaporated onto mica. The magnetoresistance exhibits a marked thickness dependence: at 4 K and 9 T is about 5% for the thinner (69 nm) film, and about 14% for the thicker (185 nm) film. Sondheimer's theory provides an accurate description of the temperature dependence of the resistivity, but predicts a magnetoresistance one order of magnitude smaller than that observed at 4 K. Calecki's theory in the limit of small roughness correlation length, predicts a resistivity two orders of magnitude larger than observed at 4 K.

A fundamental question regarding thin metallic structures is how the roughness of the surface that limits the structure affects electrical transport properties when one or more of the dimensions of the structure are comparable to or smaller than the mean free path ℓ of the charge carriers in the bulk. Despite over a century of research on 'size effects' [1], the effect of electron–surface scattering on charge transport still remains an open question. In this paper we report measurements of transverse magnetoresistance performed on four gold films deposited onto mica substrates, together with measurements of the surface roughness of the samples on an atomic scale performed with a scanning tunnelling microscope (STM). The magnetoresistance signal can be attributed to electron–surface scattering. The magnetic field

is oriented perpendicular to the plane of the films. A preliminary report of the experimental results reported here has been presented at a PASI [2].

In a thin metallic film, the proximity of the upper and lower rough surfaces limiting the film should give rise to a magnetoresistance in a metal characterized by a spherical Fermi surface *for which the bulk magnetoresistance is zero*, when the distance t separating the two rough surfaces is such that $t \leqslant \ell$. The first calculation of the magnetoresistance arising from electron–surface scattering induced by a magnetic field oriented perpendicular to the plane of a thin metallic film was published by Sondheimer [3], who used the Boltzmann transport equation (BTE) to describe the motion of electrons in the sample, and introduced the specularity of the surface as an adjustable parameter (representing the fraction of electrons that are specularly reflected upon colliding with the rough surface). This calculation was improved by Calecki, who used BTE but represented electron–surface scattering in terms of a perturbation Hamiltonian (describing the perturbation induced by the rough surfaces over and above the Hamiltonian describing an electron gas confined between two parallel flat surfaces) [4]. Although several theories of size effects (in the absence of a magnetic field) have been published during the last decade (to mention a few, see [5–7] and references therein), theories of magnetomorphic effects on metallic samples published after Calecki's work are rather scarce [8, 9]. There are also only a few experimental investigations of magnetomorphic effects on non-magnetic metallic films published subsequent to Calecki's work [10–15]. Concerning theories of magnetoresistance, Calecki's work appears to be the only formalism available that predicts magnetomorphic effects in thin metallic films in terms of parameters that characterize the roughness of the surface that can be measured in an independent experiment with a STM. Since Calecki's theory has remained untested, performing both magnetoresistance as well as surface roughness measurements on the same set of samples promises to be interesting, for it should allow *comparison between theory and experiment regarding size effects without using adjustable parameters*.

To be able to observe 'size effects', the samples have to be such that the resistivity arising from electron-scattering mechanisms other than rough surfaces does not mask the effect of electron–surface scattering. Van Attekum and co-workers [16] report that the resistivity of gold films evaporated onto polished Pyrex and silicon substrates held at room temperature was found to decrease by about one order of magnitude upon annealing the samples. Transmission electron microscope (TEM) studies proved that the decrease in resistivity upon annealing was correlated to a marked increase in the lateral dimension characterizing the grains making up the sample. Based upon this work, we prepared over 50 gold films of the same thickness (70 nm), growing the films at 3 nm min^{-1} but changing the substrate and annealing temperature between -190 and 270 °C. We found the conditions of evaporation that minimize the concentration of defects (that minimize deviations of the morphology and structure of the films with respect to a thin slice of a single crystal): a minimum $\rho(295)$ is obtained when the substrate and annealing temperature are between 180 and 270 °C.

We prepared films of different thickness, starting from 99.9999% pure gold evaporated at 3 nm min^{-1} from a tungsten basket filament onto freshly cleaved mica substrates in a HV evaporation chamber (vacuum of 1.0×10^{-5} Pa). Masks were prepared such that an evaporation run yielded four samples for each thickness. The mica was preheated to 270 °C and the films were annealed for 1 h at 270 °C after evaporation. The films exhibit a room-temperature resistivity $\rho(295)$ a few per cent in excess of the resistivity of 22.5 nΩ m expected from electron–phonon scattering, the electron scattering mechanism dominant in high-purity crystalline gold at 295 K [17]. The excess resistivity ranges from 5% for the thickest (185 nm), to 30% for the thinnest (69 nm) sample.

To rule out structural defects that could give rise to a magnetoresistance signal, we determined the morphology and structure of the samples via x-ray diffraction (XRD),

Rutherford back scattering (RBS), TEM and STM. The diffractogram of the muscovite mica substrate reveals a base-centred monoclinic structure with $a = 0.5209$ nm, $b = 0.9072$ nm and $c = 2.0063$ nm, with $\alpha = \gamma = 90°$ and $\beta = 95.715°$. The diffractogram of the gold samples reveals a fcc lattice with a lattice constant $a = 0.407\,86$ nm. The atoms in the (a, b) plane of the monoclinic structure are surrounded by six nearest neighbours arranged in an almost perfect hexagon, suggesting that gold atoms occupy the centre of gravity of the triangles that make up the basal hexagon, separated by a distance of 0.301 nm. This distance is only 4% larger than the separation between the six nearest neighbour gold atoms on the $\langle 111 \rangle$ plane of the fcc lattice. The diffractogram of each film yielded a peak at $2\theta = 38.314°$, that corresponds to the $\langle 111 \rangle$ reflection of gold. The intensity of the x-ray signal recorded by the diffractometer in the θ–2θ mode is comparable to that recorded in the rocking-curve mode. We conclude that the samples are made up of grains that grew oriented such that direction $\langle 111 \rangle$ is perpendicular to the surface of the mica [18]. The thickness of the samples was measured (to an accuracy of 5%) by recording the RBS spectra of 2 MeV alpha particles from a Van de Graaff accelerator. We measured the grain size of some 50–100 grains from samples of different thickness, using TEM. The average lateral dimensions of the grains turned out to be $167 \pm 19, 240 \pm 24, 255 \pm 28$ and 290 ± 41 nm in the 69, 93, 150 and 185 nm samples, respectively. We measured the parameters (δ, ξ) corresponding to a Gaussian representation of the roughness profile $f(x, y) = \delta^2 \exp[-(x^2 + y^2)/\xi^2]$ (where (x, y) represent the in-plane coordinates) from 25 to 30 images (10 nm $\times$ 10 nm, containing 256 $\times$ 256 pixels each) of the surface of the films recorded with a STM, following the method published [18]. The result is (0.17 nm, 10.9 nm), (0.17 nm, 10.1 nm), (0.16 nm, 12.2 nm) and (0.29 nm, 7.65 nm), in the 69, 93, 150 and 185 nm samples, respectively.

The magnetoresistance was measured using the four-point method, injecting a current of 1.3 mA and 210 Hz across terminals A–B (figure 1), and measuring the voltage drop across terminals E–G (figure 1) by means of SR-830 lock-in amplifiers. The samples were inserted into a copper block in a superconducting magnet, the temperature of which was maintained within ± 0.1 K. The resistivity at 4 K was 7.01, 4.72, 3.27 and 2.14 nΩ m on the 69, 93, 150 and 185 nm samples, respectively. At 4 K and 9 T, the Hall voltage, measured simultaneously, indicates that the product $\omega_C \tau$ (where $\omega_C = qB/m$ is the cyclotron frequency, τ is the average time between collisions) ranges between 0.14 and 0.45. Cooling to 4 K decreases the resistivity of the films by one order of magnitude, leading to a $\rho(4)$ that differs by at least a factor of three between the thinnest and thickest film, *in spite of the fact that the corresponding* $\rho(295)$ *do not differ by more than 30%.* At 4 K electron–phonon scattering is frozen out. Since the average lateral dimension of the grains is larger than the film thickness in all samples, *at 4 K the mean free path is dominated by electron–surface or electron–grain boundary scattering.*

The dependence of the magnetoresistance on the magnetic field **B** observed at different temperatures is shown in figure 1. The magnetoresistance signal exhibits a remarkable thickness dependence, that (given the small variation of the lateral grain dimensions between samples of different thickness) suggests electron–grain boundary scattering must be ruled out as an explanation. The increase of about 14% observed at 4 K and 9 T in the 185 nm sample is unexpected; it seems surprisingly large. It is reminiscent of the nonlinear dependence of the magnetoresistance that has been reported on a 110 nm film of $CoSi_2$ measured at 4.2 K and 9 T. However, the observed increase reported in $CoSi_2$ is only about 1.5%; it was attributed to the presence of two types of carriers, electrons and holes [19].

On theoretical grounds, using BTE it has been shown that a crystalline metal characterized by a spherical Fermi surface exhibits a null magnetoresistance, both when $\mathbf{E} \parallel \mathbf{B}$ and when $\mathbf{E} \perp \mathbf{B}$ [20], where **E** and **B** are the electric and magnetic fields, respectively. In crystalline noble metals such as gold, departures of the Fermi surface from a perfect sphere give rise to a

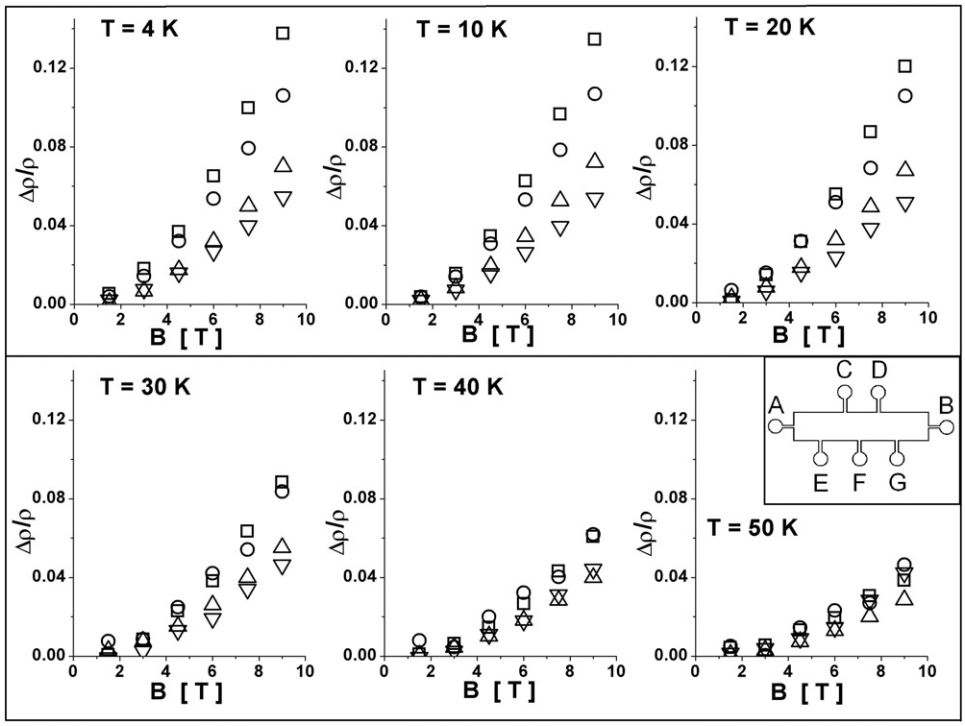

Figure 1. Dependence of the transverse magnetoresistance on the magnetic field B, at different temperatures T (4, 10, 20, 30, 40, 50 K), indicated in the figure. Squares: film 185 nm. Circles: film 150 nm. Triangles: film 93 nm. Inverted triangles: film 69 nm. The inset in the lowest rightmost panel indicates the shape of the sample. Dimensions of the rectangular centre section of the sample are 2.5 mm × 11.0 mm; terminals E and G are 7.0 mm apart.

magnetoresistance different from zero [20]. As the origin of the observed magnetoresistance, we are led to consider either:

(a) the morphology of the Fermi surface, e.g. departures of the Fermi surface of gold from a perfect sphere, or
(b) the morphology of the films, e.g. the proximity of the rough surfaces limiting the film to within a distance $t \leqslant \ell$.

Concerning the first possibility (a), let $\mathbf{E} = (E_x, E_y, 0), \mathbf{B} = (0, 0, B)$ and the current density $\mathbf{J} = (J_x, J_y, 0)$. The transverse magnetoresistance is $\Delta\rho/\rho = [\rho_{xx}(B) - \rho_{xx}(0)]/\rho_{xx}(0)$, where $\boldsymbol{\rho}$ is the tensor relating $\mathbf{E}$ and $\mathbf{J}$: $\mathbf{E} = \boldsymbol{\rho}\,\mathbf{J}$. The electron trajectory in reciprocal $\mathbf{k}$-space is defined by the intersection of a plane perpendicular to $\mathbf{B}$, and the surfaces of constant energy $\varepsilon(\mathbf{k})$ [21]. Since the Fermi surface of gold exhibits a neck around direction $\langle 111\rangle$—the direction along which the magnetic field is oriented—the electron motion in the gold films ought to involve at least two extremal orbits: (i) orbits where the plane perpendicular to $\mathbf{B}$ cuts the Fermi surface around its belly and (ii) orbits where it cuts the Fermi surface at one of its necks.

The simplest phenomenological model involving extremal orbits is a two-carrier model, characterized by $(n_1, m_1, \tau_1, \mu_1)$ and $(n_2, m_2, \tau_2, \mu_2)$, where n_i stands for the carrier density, m_i for its effective mass, τ_i for the average time between collisions, $\mu_i = q\tau_i/m_i$ for the carrier mobility and q is the electron charge. The two-carrier Drude model leads to

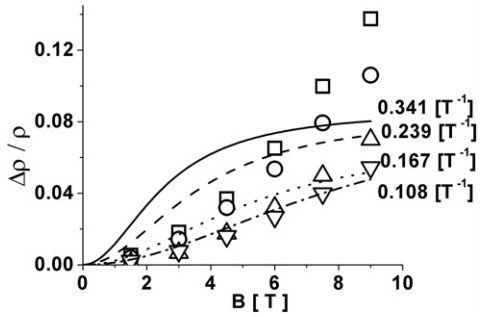

Figure 2. Dependence of the transverse magnetoresistance on the magnetic field B at 4 K, symbols as in figure 1. Fitting the two-carrier Drude model to the magnetoresistance data (corresponding to each film thickness) leads to the mobility μ_1 indicated in the figure, in units of $(\mathrm{T})^{-1}$.

$$\frac{\Delta\rho}{\rho} = \frac{\mu_1\mu_2 B^2\alpha\,(\mu_1-\mu_2)^2}{(\alpha\mu_2+\mu_1)^2+(\mu_1\mu_2 B)^2\,(\alpha+1)^2}$$

where the label 1 indicates neck orbits and the label 2 indicates belly orbits perpendicular to the direction $\langle 111\rangle$, with $\alpha = n_2/n_1$. We estimate $\alpha \approx (4\pi R^2/(2\times\pi r^2) \approx 58$, where R is the radius of the Fermi sphere of gold and r is the radius of one of its necks [21]. The expression for $\Delta\rho/\rho$ depends on μ_1, μ_2 and B. Since $\alpha \gg 1$, $\mu_2(T)$ was obtained from the Hall voltage measured on each sample at temperature T, leaving only $\mu_1(T)$ as a free parameter. We least square fitted the magnetoresistance data at 4 K using $\mu_1(4)$ as the adjustable parameter; the result of the fit is shown in figure 2. The fit is quite poor, the curvature of the magnetoresistance as a function of magnetic field B is opposite from what is observed for the 150 and 185 nm film, and the fitting parameter μ_1 turns out to increase monotonically with increasing film thickness: μ_1 for the 185 nm film turns out to be about 3.4 times larger than μ_1 for the 69 nm film.

The starting assumption of this two-carrier Drude model is that the mobilities of the different carriers depend only on the curvature of the extremal orbits and on the average collision time that characterizes electron scattering in the bulk, and are independent of the thickness of the sample. Yet an analysis of the magnetoresistance and resistivity data leads to collision times τ_1 and τ_2 that increase monotonically with increasing film thickness. We conclude that the *collision times are somehow determined by electron–surface scattering*. We are thus led to consider possibility (b), that the magnetoresistance arises from electron–surface scattering.

The magnetoresistance predicted by Sondheimer, can be calculated from

$$\frac{\Delta\rho}{\rho} = \frac{\varphi(s,B=0)\mathrm{Re}\,\varphi(s,B\neq 0)}{|\varphi(s,B\neq 0)|^2} - 1$$

where $s = \kappa + \mathrm{i}\beta$ is a complex variable, and $\mathrm{Re}\,\varphi(s)$ stands for the real part of the complex quantity $\varphi(s)$. Here $\kappa(T) = t/\ell(T)$ (notice that $\ell(T)$ stands for the electron mean free path at temperature T *in the absence of electron–surface scattering*), and $\beta = t/r_0$ (r_0 is the radius of the cyclotron orbit) [3]. Setting $B = 0$ leads to $\rho(T) = \rho_0(T)/[\kappa(T)\varphi(s, B = 0)]$, where $\rho_0(T)$ is the bulk resistivity described by a Bloch–Grüneisen law [17]. However, Sondheimer considered a metal film limited by two rough surfaces characterized by the same specularity. Since the lower surface is cleaved mica, which is atomically flat except for cleavage steps, the gold–mica interface ought to behave as a specular surface [18], therefore the roughness of the upper gold surface is expected to dominate the resistivity induced by electron–surface scattering. Instead of adopting the form for $\varphi(s)$ proposed by Sondheimer [3], we computed

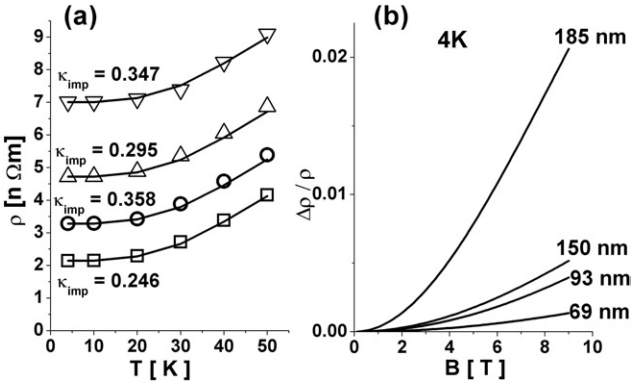

Figure 3. (a) Temperature dependence of the resistivity for films of different thickness, symbols as in figure 1. Solid line: prediction of the Sondheimer–Lucas theory, with $P = 1$ and $Q = 0$. The term $(1/\tau)_{\mathrm{IMP}}$ was adjusted to describe $\rho(4)$ for each sample; the corresponding value of $\kappa_{\mathrm{imp}} = t/\ell$ (at 4 K) is indicated in the figure. (b) Magnetic field dependence of the magnetoresistance at 4 K predicted by Sondheimer–Lucas.

$\varphi(s)$ numerically, using the form proposed by Lucas [22]:

$$\varphi(s) = \frac{1}{s} - \frac{3}{4s^2}\int_1^\infty \left(\frac{1}{t^3} - \frac{1}{t^5}\right) \times \frac{(1-\exp(-st))\left[2 - P - Q + (P + Q - 2PQ)\exp(-st)\right]}{1 - PQ\exp(-2st)}\,\mathrm{d}t$$

where we set $P = 1$ to characterize the reflectivity of the mica. The fitting parameters left in the theory are Q (the specularity of the upper gold surface) and $\kappa(T)$.

To test Sondheimer's theory quantitatively we need to determine $\kappa(T)$, and to do so we must evaluate $\ell(T)$ at each temperature T. $\ell(T)$ is determined by the relaxation time $\tau(T)$ in the bulk, that varies with temperature according to $1/\tau = (1/\tau)_{\mathrm{IMP}} + (1/\tau)_{\mathrm{PHON}}$, where the first (temperature-independent) term accounts for electron scattering by impurities and the second (temperature-dependent) term accounts for electron–phonon scattering [17]. For each sample we selected a value for the parameter Q, and adjusted $(1/\tau)_{\mathrm{IMP}}$ to describe either $\rho(4)$ or $(\Delta\rho/\rho)(4)$, neglecting $(1/\tau)_{\mathrm{PHON}}$ at 4 K. To compute $1/\tau$ at $T > 4$ K, we added to $(1/\tau)_{\mathrm{IMP}}$ the corresponding $(1/\tau)_{\mathrm{PHON}}$ computed from the Bloch–Grüneisen intrinsic resistivity listed on page 1209 of [17]. We repeated this procedure for different values of Q. The best fit to the temperature dependence of the resistivity data was obtained for $Q = 0$, as shown in figure 3. The unexpected result is that, if $(1/\tau)_{\mathrm{IMP}}$ is adjusted to fit $\rho(4)$, then the Sondheimer–Lucas theory provides a fair description of the temperature dependence of the resistivity of each sample as shown in figure 3(a), but the predicted magnetoresistance at 4 K turns out to be *an order of magnitude too small* (figure 3(b)). If $(1/\tau)_{\mathrm{IMP}}$ is adjusted to fit $(\Delta\rho/\rho)(4)$ rather than $\rho(4)$, then theory does not describe appropriately either the magnetic field dependence of the magnetoresistance for the two thicker films at 4 K or the temperature dependence of the resistivity in any of the films.

In the case of Calecki's theory, the author introduced an electron distribution function $f_\nu(\mathbf{k}) = f_0(\varepsilon_{\nu\mathbf{k}}) + \phi_\nu(\mathbf{k})$ (equation (11) in [4]) for electrons occupying each subband with an energy $\varepsilon_{\nu\mathbf{k}} = \hbar^2(\mathbf{k}^2 + k_\nu^2)/2m$, where $\mathbf{k} = (k_x, k_y)$ represents the in-plane momentum, $k_\nu = \nu\pi/t$ represents the quantized momentum along z, $\phi_\nu(\mathbf{k})$ represents a linear function in $\mathbf{E}$ and $f_0(\varepsilon_{\nu\mathbf{k}})$ represents the equilibrium Fermi–Dirac distribution function. In this work, Calecki set up a BTE for $f_\nu(\mathbf{k})$, and proved that, in the presence of a magnetic field, the Boltzmann

collision operator describing electron–rough surface scattering cannot be characterized by a relaxation time τ unless $\nu_F = n$ (where ν_F is the number of occupied subbands). For this reason the author introduced the matrix $T(\varepsilon)_{\nu\nu'}$ with dimensions of time, defined by equation (22) from [4]. The magnetoresistance predicted by theory can be calculated in terms of $T(\varepsilon)_{\nu\nu'}$ from

$$\frac{\Delta\rho}{\rho} = \frac{(\sigma_0 - \sigma_2)\sigma_0}{(\sigma_0 - \sigma_2)^2 + \sigma_1^2} - 1$$

where σ_0, σ_1 and σ_2 are given by

$$\sigma_0 = \sum_\nu \frac{n_\nu q^2}{m} \sum_{\nu'} \langle T(\varepsilon)_{\nu\nu'} \rangle_\nu$$

$$\sigma_1 = \omega_C \sum_\nu \frac{n_\nu q^2}{m} \sum_{\nu'} \left\langle \{[1 + \omega_C^2 T^2(\varepsilon)]^{-1} T^2(\varepsilon)\}_{\nu\nu'} \right\rangle_\nu$$

$$\sigma_2 = \omega_C^2 \sum_\nu \frac{n_\nu q^2}{m} \sum_{\nu'} \left\langle \{[1 + \omega_C^2 T^2(\varepsilon)]^{-1} T^3(\varepsilon)\}_{\nu\nu'} \right\rangle_\nu$$

(equations (32)–(34) from [4]), where n_ν is the density of states of subband ν, and $\langle\psi\rangle$ indicates the average of ψ over the occupied subbands.

Although the formulae for calculating σ_0, σ_1 and σ_2 requires computing the matrix T (involving hundreds of elements $T(\varepsilon)_{\nu\nu'}$ for each sample), in the limit of small correlation lengths (e.g. $k\xi < 1$, where ξ is the roughness correlation length), $T(\varepsilon)_{\nu\nu'}$ becomes diagonal, for in this case electron–surface scattering causes the distribution function $f_\nu(\mathbf{k})$ to relax towards the Fermi–Dirac distribution function $f_0(\varepsilon_{\nu\mathbf{k}})$ with a 'relaxation time' τ_ν associated with subband ν, given by

$$T_{\nu\nu'}(\varepsilon_F) = \delta_{\nu\nu'} \frac{m}{\pi^5\hbar} \frac{6t^6}{\nu_F(\nu_F + 1)(2\nu_F + 1)} \frac{1}{\delta^2\xi^2\nu^2} = \tau_\nu(\varepsilon_F) \tag{1}$$

(equation (64) from [4]) and δ is the r.m.s. roughness amplitude. Using this notation, the effect of the Boltzmann collision operator describing electron–phonon and electron–impurity scattering can be characterized by a single relaxation time $(\tau)_{\mathrm{PHON}}$ and $(\tau)_{\mathrm{IMP}}$ that are independent of the subband label ν, since the scattering rates corresponding to these scattering processes (present in the bulk) are independent of the thickness of the sample. In the presence of electron–phonon and electron–impurity scattering, the total scattering rate corresponding to subband ν, should be computed by adding the scattering rates corresponding to each of the three processes acting separately, electron–impurity + electron–phonon + electron–surface scattering, $(1/\tau)_{\mathrm{IMP}} + (1/\tau)_{\mathrm{PHON}} + (1/\tau_\nu)$.

However, the resistivity $\rho_0 = (\sigma_0)^{-1}$ predicted at 4 K arising solely from electron–surface scattering, characterized by the 'relaxation time' $\tau_\nu(\varepsilon_F)$ given by equation (1) above, turns out to be about two orders of magnitude larger than observed. It is not clear whether such discrepancy arises from an overestimation of the effect of electron–surface scattering within the theory, or is rather a consequence of the approximation $k\xi < 1$ used to derive the diagonal form for the matrix $T_{\nu\nu'}(\varepsilon_F) = \tau_\nu(\varepsilon_F)$ given by equation (1), an approximation which is far from being fulfilled in our samples. To elucidate the origin of this discrepancy we have to solve the transport equations numerically, computing the matrix $T(\varepsilon)_{\nu\nu'}$ for each sample. Such work is in progress.

Summarizing, we have measured the magnetoresistance $\Delta\rho$ of gold films evaporated onto mica substrates at low temperatures and high magnetic fields, and have measured the surface roughness of the samples with an STM having atomic resolution. The magnetoresistance

exhibits a marked thickness dependence, which points to the dominant role played by electron–rough surface scattering. A model consisting of an electron gas composed of two types of carriers that ignores electron–surface scattering does not explain the thickness dependence of the data. Such a model constitutes a gross oversimplification, since the importance of electron–surface scattering is completely neglected. The magnetoresistance data qualitatively confirm Sondheimer's predictions made over 50 years ago. Sondheimer's theory predicts accurately the temperature dependence of the resistivity $\rho(T)$, but predicts at 4 K a magnetoresistance which is an order of magnitude smaller than that observed. Calecki's theory (under the approximation $k\xi < 1$) predicts a resistivity arising from electron–surface scattering that is two orders of magnitude larger than observed at 4 K. The question remains open of whether such discrepancy arises as a consequence of the small roughness correlation length approximation ($k\xi < 1$, certainly not valid in our samples) used by the author to derive the diagonal form for the matrix $T(\varepsilon)_{\nu\nu'}$ or arises because theory overestimates electron–surface scattering.

Acknowledgment

This work was funded by FONDECYT under contract 1040723.

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