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High-precision resistance measurements on amorphous CuTi down to 15 mK

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We have developed a new method for making high-precision resistance measurements on amorphous-metal ribbons with very reliable thermometry down to 15 mK. In our technique the measuring current flows perpendicular to the plane of the metal ribbon and potassium metal is used to thermally anchor the ribbon to the thermometers and refrigerator. We have been able to obtain very good fits of the weak-localization and enhanced electron-electron interaction theories to the CuTi data in zero field.

For disordered conductors, it is now generally recognized that the unusual temperature and magnetic-field dependences of their resistivities $\rho(T,B)$ are explained by theories of weak localization (WL) (Ref. 1) and enhanced electron-electron interaction (EEI).[2] For simple three-dimensional free-electron-like disordered conductors that exhibit *weak* spin-orbit scattering (e.g., amorphous CaAl),[3] these theories provide rather good quantitative agreement with the $\rho(T,B)$ data. However, for disordered conductors with *strong* spin-orbit scattering (e.g., amorphous CuTi), such complete quantitative agreement has not been possible.[4,5] For example, the spin-orbit scattering times $\tau_{\mathrm{s.o.}}$, which are determined by extracting the WL contribution from the temperature dependence of the zero-field ρ ($\Delta\rho$), are significantly shorter than $\tau_{\mathrm{s.o.}}$ determined from the magnetoresistance $\rho(T,B>0)$. Thus for the zero-field data, an important step forward would be to disentangle clearly the EEI and WL terms in $\Delta\rho$. In the low-temperature limit, the EEI (Ref. 2) term in $\Delta\rho$ is predicted to vary as $-\sqrt{T}$. For the WL term in $\Delta\rho$, let us assume that below ≈ 4 K the inelastic-scattering time τ_{ie} obeys a single average power law $\tau_{ie}=\tau_{i0}T^{-p}$ ($p\approx 1.5-3$) and that the strong spin-orbit scattering occurs, $\tau_{\mathrm{s.o.}} \ll \tau_{ie}$. Given these assumptions, the WL (Ref. 6) term in $\Delta\rho$ will vary as $+T^{p/2}$. Hence, in order to quantify the relative contributions of the WL and EEI terms in zero field, $\Delta\rho$ must be measured over a wide range of temperature since the above WL and EEI terms will only dominate $\Delta\rho$ at the high and low extremes of temperature, respectively.

For extracting the EEI term, by far the best attempt at low-T measurements on a strong spin-orbit scattering alloy was made by Lindqvist and Rapp (LR).[5] Since $\Delta\rho$ was expected to be small in comparison to the residual resistivity ρ_0, LR measured $\Delta\rho/\rho_0$ for CuTi to an $\approx$10-ppm precision well below 1 K. They used a measuring current that flowed in the plane (CIP direction) of the melt-spun ribbon, and thermal contact was made with varnish between the ribbon and a Cu cold finger with its attached carbon-resistor thermometer. Because of the rapidly rising Kapitza thermal resistance between the ribbon and the cold finger as T is lowered, LR estimated that at 20 mK the maximum error in T was about 10 mK. Unfortunately, their $\Delta\rho$ became insensitive to temperature for $T\leq 150$ mK—the very region where the EEI term might be expected to dominate $\Delta\rho$.

In order to measure $\Delta\rho$ with greater precision and with more reliable thermometry at 15 mK, we have developed a new measurement scheme: the current flows perpendicular to the plane of the sample (CPP), and excellent thermal contact is made by the sample to the refrigerator and thermometers via a nonsuperconducting potassium "glue," an adaptation of an earlier K-glue technique developed by one of us for making high-precision transport measurements on the heavy fermion system $CeCu_6$ down to 14 mK.[7] Since our technique utilizes a low-electrical-resistance path from the sample to the thermometers and refrigerator, the Wiedemann-Franz law states that the corresponding metallic thermal resistance will also be low and diverge only as T^{-1} as T is lowered—in contrast to the situation of an electrically insulating contact where the Kapitza thermal resistance is much higher and has a more rapid T^{-3} divergence. For our sample geometry, the CPP resistance R of the

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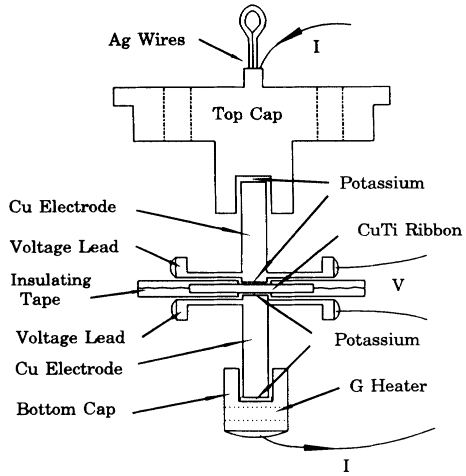

FIG. 1. Schematic diagram of sample assembly. For clarity of presentation this diagram is not drawn to scale.

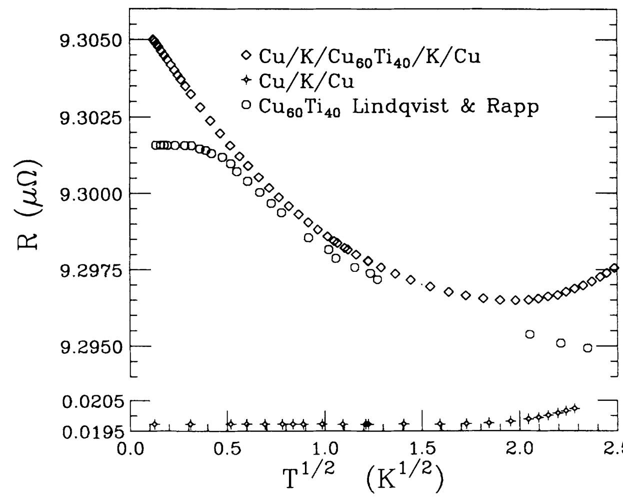

FIG. 2. R vs $T^{1/2}$ for a Cu/K/$Cu_{60}Ti_{40}$/K/Cu sample, a Cu/K/Cu sandwich, and the data of Lindqvist and Rapp (Ref. 5). The upper and lower ordinates have the same size incremental scale factor.

ribbon is a rather low 10 $\mu\Omega$; thus we can use our well-established technique[8] for making 0.1-ppm-precision R measurements below 1 K with a current comparator and SQUID null detector in a low-temperature potentiometer circuit that generates only about 1 nW of heat in the sample. With this scheme we have also made CPP magnetoresistance measurements on CuTi with 0.1-ppm precision down to 20 mK for $B \leq 0.2$ T.[9]

In Fig. 1 we show a schematic diagram of our sample assembly. Each cylindrical Cu electrode is machined from oxygen-annealed OFHC Cu and has a 3.2-mm diam, 10-mm length, and 6.4-mm diam by 0.2-mm-thick integral voltage lead. Potassium glue is used to attach the Cu electrodes to the CuTi ribbon and to the bottom and top Cu caps. Insulating tape is stuck to the ribbon in order to localize the K glue and to keep it from shorting around the edges of the $\approx$4-mm-wide ribbon. The thick outer rim of each cylindrical voltage lead is coated with a superconducting low-temperature solder, and the current and voltage wires are superconductors. Before the assembly is removed from a purified-Ar glove box, the region below the top cap is covered with paraffin wax to protect the K glue from the ambient atmosphere during mounting on the dilution refrigerator. The Ag wire, which is spot welded to the top cap, is pressed hard with a brass screw against a Ag block to which are attached the sample thermometers and the thermal link to the refrigerator. We use two calibrated Ge resistance thermometers above 50 mK and a cerrous magnesium nitrate paramagnetic thermometer with a SQUID-based ac bridge below 50 mK. For magnetoresistance measurements the top cap also serves as a rigid holder for a superconducting solenoid. The G heater is for thermopower measurements in a future development.

The melt-spun $Cu_{50}Ti_{50}$ and $Cu_{60}Ti_{40}$ ribbons were made at the University of Leeds. Two different techniques were used to overcome the insulating oxide layer on these ribbons. (1) After the surfaces of the Cu electrodes and the ribbon were scraped in the glove box, K was worked into the two surfaces of the CuTi with a file. (2) On each side, the CuTi ribbons were lightly ion milled in an UHV sputtering system and about 300 nm of Cu was sputtered down. This second technique turned out to be much more reliable since only very light scraping in the glove box was needed to make the K glue stick to the Cu-coated surfaces.

In Fig. 2 we plot R vs $T^{1/2}$ for a Cu-coated $Cu_{60}Ti_{40}$ sample and a simple Cu/K/Cu sandwich where the 0.3-mm thickness of the K layer is about equal to the sum of K-layer thicknesses for the CuTi sample. Having $R_{\mathrm{CPP}}=9.3$ $\mu\Omega$ is consistent with an independent CIP measurement of $\rho_0=1.93$ $\mu\Omega$ m for this ribbon, given that the potassium contact area and ribbon thickness are about 6 mm^2 and 27 μm, respectively. For this CuTi sample, no evidence of a superconductivity onset is seen near 15 mK. Note that the magnitude of R and its temperature dependence below 2.7 K for the Cu/K/Cu sandwich are negligible in comparison to those of the CuTi sample. Above 2.7 K the increase in R of the sandwich is due to umklapp electron-phonon scattering in the potassium that has the form $\rho(T)\simeq T\exp(-\Theta^*/T)$, where $\Theta^*=20$ K.[10] Above 2.7 K, R for the CuTi sample is also affected by this umklapp contribution of potassium. Thus below about 2.7 K, it would seem that no corrections to $R(T)$ are necessary due to the presence of the Cu electrodes and the K layers.

The open circles in Fig. 2 represent the $\Delta\rho/\rho_0$ CIP data for a $Cu_{60}Ti_{40}$ sample of LR,[5] suitably renormalized for direct comparison with our CPP sample and displaced downward slightly for clarity. For $0.25 \leq T \leq 1.6$ K, their data agree *beautifully* with ours; and most of the disagreement between our two sets of data above 4.0 K is due to the previously mentioned umklapp contribution in our samples. For $T \leq 0.25$ K, however, there is a significant disagreement which implies that the electron temperature in LR's samples did not cool much below 0.25 K. It is possible that this disagreement might also

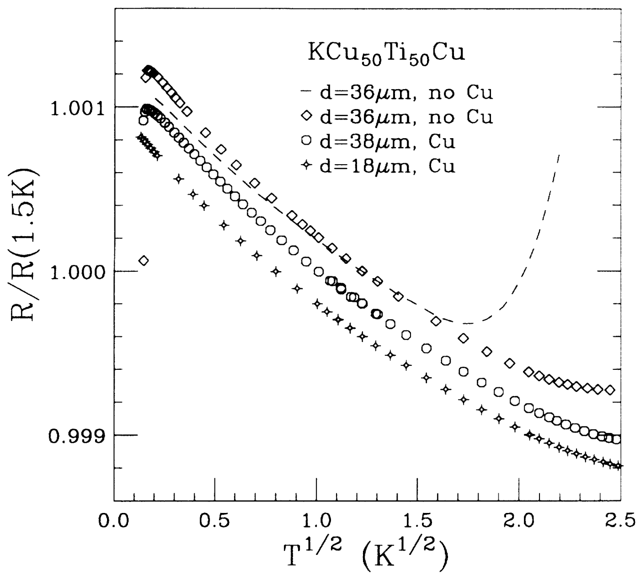

FIG. 3. $R(T)/R(1.5\text{ K})$ vs $T^{1/2}$ for $Cu_{50}Ti_{50}$ samples. For clarity, the middle and lower data sets were displaced downward by one and two small-scale divisions, respectively. See the text for further details.

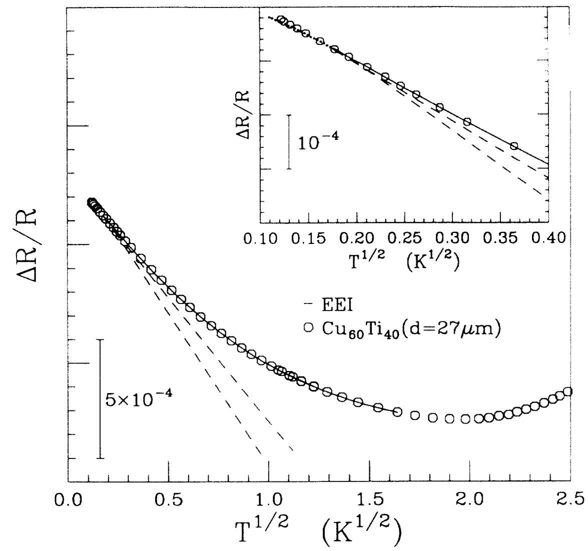

FIG. 4. The relative change in resistance, $\Delta R/R$, vs $T^{1/2}$ for a $Cu_{60}Ti_{40}$ sample. The placement of the data along the ordinate is arbitrary. The inset shows an expanded view of the low-T region. See the text for explanation of solid, dotted (only in the inset), and dashed curves.

be due to a T-dependent boundary resistance at each interface between our CuTi ribbon and the Cu (or K) coating.

In Fig. 3 we explore this possibility by comparing data for $Cu_{50}Ti_{50}$ samples prepared with and without sputtered-Cu coatings and with different ribbon thicknesses d. For the two 36-μm-thick uncoated ribbons, we see that $R(T)$ is well behaved when the contact between CuTi and K is "good" but that $R(T)$ exhibits a very large e-p umklapp contribution above 2 K when the contact is "poor" (dashed curve). The 38-μm sample has a sputtered-Cu coating, and $R(T)$ for it and the good-contact uncoated 36-μm sample agree very well over the whole temperature range. Thus $R(T)$ is not affected by the Cu-coating process. Indeed we have found that this very large umklapp contribution rarely occurs if we use the Cu-coated ribbons. To address the possibility of an intrinsic contribution of the CuTi-Cu interface to $R(T)$, we show the data for a Cu-coated 18-μm ribbon that was prepared by gently sanding down a thicker ribbon. If there were a significant interface contribution to $R(T)$, then $R(T)/R(1.5\text{ K})$ for the thinner sample would show a different T dependence. Instead, the data for the 18- and 38-μm samples agree rather well, especially above 40 mK. Similar experiments on our $Cu_{60}Ti_{40}$ samples lead to the same conclusion. The sudden downturn in $R(T)$ near 20 mK is due to the onset of superconductivity in the thicker $Cu_{50}Ti_{50}$ samples.

Since our CPP-direction technique is free of serious systematic errors, we can proceed with a fit of the WL and EEI theories to our data. However, for our $Cu_{50}Ti_{50}$ samples with their higher T_c, a complete analysis will require the inclusion of the Aslamasov-Larkin (AL) and Maki-Thompson (MT) superconducting-fluctuation contributions to $\Delta\rho$ (Ref. 11) at the lowest temperatures. Since our $Cu_{60}Ti_{40}$ samples show no evidence of this onset, it should be possible to simplify the analysis by neglecting these AL and MT contributions. For $T \geq 30$ mK, we estimate for our $Cu_{60}Ti_{40}$ samples with $T_c \lesssim 6$ mK that the AL and MT terms in $\Delta\rho$ will be negligible in comparison to the WL and EEI terms.

In Fig. 4 we present a plot of the relative dependence of R upon $T^{1/2}$ for our $Cu_{60}Ti_{40}$ sample in Fig. 2. We assume that $\Delta\rho/\rho_0$ is a simple summation of the WL (Ref. 6) and EEI (Ref. 2) terms for the three-dimensional case and that $\tau_{ie} = \tau_{i0}T^{-p}$. We obtain

$$\Delta\rho/\rho_0 = \rho_0 \left[\frac{e^2}{4\pi^2\hbar}\right] \left[\frac{2(\sqrt{t} - 3\sqrt{t+1})}{\sqrt{D\tau_{\text{s.o.}}}} - 0.92\left[\tfrac{4}{3} + \tfrac{3}{2}F^* + \frac{2}{\ln(T/T_c)}\right] \times \left[\frac{k_B T}{\hbar D}\right]^{1/2}\right], \quad (1)$$

where $t = \tau_{\text{s.o.}}/4\tau_{ie}$, $F^* = \lambda - \tilde{F}_\sigma$, λ is the e-p mass-enhancement factor, $\tilde{F}_\sigma$ is the effective e-e screening parameter, and D is the diffusion constant. The solid curve in Fig. 4 represents the best fit of Eq. (1) to the data for 30 mK $\leq T \leq 2.7$ K, where the only fixed parameters of the fit are $\rho_0 = 1.93\ \mu\Omega$ m and $D = 0.28\ \text{cm}^2/\text{s}$; the latter is calculated directly from heat capacity data.[12] This fit is excellent even well below 160 mK (see inset).

Our preliminary values for the free parameters of the fit are as follows: $p = (1.8, \underline{2.2}, 2.4)$, $\tau_{i0} = (24, \underline{52}, 78)$ ps K^p, $\tau_{\text{s.o.}} = (0.02, \underline{0.17}, 0.36)$ ps, $T_c = (3.7, \underline{2.2}, 0.8)$ mK, and $F^* = (+0.10, \underline{-0.05}, -0.10)$. The underlined numbers correspond to the best fit. The left and right numbers are a generous estimate of the maximum range for these parameters and were obtained by noting what fixed values of parameter p caused a significant increase in the

χ^2 of the fit as the remaining parameters were allowed to attain the indicated best-fit values. Over this range of parameter values, the solid curve in Fig. 4 shows no significant change. As expected, we have $\tau_{s.o.} \ll \tau_{ie}$ for $T \leq 2.7$ K. Using these values of T_c and the McMillan formula,[13] we obtain $\lambda \approx 0.25$ and $\tilde{F}_\sigma = (0.15, \underline{0.30}, 0.35)$. Our parameters compare well with those of LR's $Cu_{65}Ti_{35}$ sample,[5] for which $p = 2.0-2.5$, $\tau_{s.o.} = 0.08-0.25$ ps, and $\tilde{F}_\sigma = 0.0-0.4$ with $0.3 \leq T \leq 18$ K. Inclusion of our other $Cu_{60}Ti_{40}$ samples in the analysis gives parameter values close to the above underlined numbers.[9]

The dashed curves in Fig. 4 indicate the EEI term only [right-hand term in Eq. (1)] for the extreme values of p: 1.8 and 2.4. A comparison of the data and dashed curves in the inset shows clearly that the WL contribution to $\Delta\rho$ is significant even below 160 mK, which implies that τ_{ie} has not reached saturation by 160 mK. The dotted curve is an extrapolation of the best-fit solid curve for $T \leq 30$ mK. It is perhaps not surprising that this extrapolation deviates from the data since the Cooper-channel $\ln(T/T_c)$ term in Eq. (1) is only valid[2] in the limit that $\ln(T/T_c) \gg 1$. If we require the fit to have $T \geq 60$ mK, the WL contribution to $\Delta\rho$ below 160 mK increases and thus remains significant.

In conclusion, we have developed a new method of making 0.1-ppm-precision resistance measurements on amorphous metals down to 15 mK that are free of systematic errors that might arise from this technique. We have obtained very good fits of the weak-localization and enhanced electron-electron interaction theories to the $Cu_{60}Ti_{40}$ data and have evaluated the parameters of the fits. We have also observed that the zero-field weak-localization term continues to make a significant contribution to $\Delta\rho$ even below 160 mK. Finally, we note that our K-glue technique is quite general and could be applied to resistance and thermopower measurements on a variety of thin-film metallic samples at ultralow temperatures.

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