Magnetoresistance of $Al_{90}Y_{10}$ and $Al_{90}La_{10}$: Strong enhancement due to small crystalline precipitates in $Al_{90}La_{10}$

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The normal-state magnetoresistance, $\Delta\rho/\rho$, was measured for $Al_{90}La_{10}$ and $Al_{90}Y_{10}$ alloys in the temperature region from four to eight times the superconducting T_c and in magnetic fields up to 12 T. These samples were amorphous in standard x-ray diffraction. An accurate description of the $Al_{90}Y_{10}$ data in terms of quantum interference effects (QIE) was obtained only when the Maki-Thompson contribution was excluded. Two samples of $Al_{90}La_{10}$ were studied. $\Delta\rho/\rho$ was anomalously enhanced in sample I, reaching above 1%, and too large to be accounted for by QIE also in sample II. Measurements of the magnetic susceptibility and further structural investigations were made, including high-resolution x-ray diffraction, analytical transmission electron microscopy, and high-resolution electron microscopy (HREM). Small crystallites of $Al_{11}La_3$ were observed in all improved structural analyses of sample I, and only in HREM for sample II. Implications for amorphous metals are discussed. [S0163-1829(96)03834-9]

I. INTRODUCTION

Melt-spun binary aluminum-rich amorphous alloys have been found with aluminum concentration of 90 at. % or above and a second component of Y, La or a 4 f metal.[1,2] These alloys have interesting properties. The resistivities, ρ, are low, about 50 $\mu\Omega$ cm for $Al_{90}La_{10}$ and $Al_{90}Y_{10}$ at room temperature.[3–5] Similar values in amorphous alloys are otherwise found only in some non-transition-metal-based systems, such as Mg-Zn.[6] This difference is strikingly illustrated by comparison with amorphous $Al_{46}Y_{54}$ where[7] ρ is 250 $\mu\Omega$ cm and the temperature coefficient of resistivity α is negative with $\rho(290)/\rho(4\text{ K})$ of about 0.95. For $Al_{90}Y_{10}$ α is positive,[3] and $\rho(300\text{ K})/\rho(4\text{ K})$ is 1.012.[5]

Also other properties of $Al_{90}La_{10}$ and $Al_{90}Y_{10}$ are remarkable. The critical magnetic field slopes at the transition temperature T_c are a factor 6–8 smaller than usually observed in amorphous superconductors and normalized critical fields at 0 K go beyond the maximum value consistent with traditional theory.[8]

Quantum interference effects (QIE) in the magnetoresistance roughly scale with ρ, and investigations in three-dimensional (3D) metals with resistivities of 50 $\mu\Omega$ cm are in the lower end of the resistivity range where such experiments can be performed. Previous examples are amorphous Mg-Cu and Mg-Zn,[9,10] and crystalline Cu-Ge.[11]

In this paper we report on the magnetoresistance, $\Delta\rho/\rho$ of amorphous $Al_{90}La_{10}$ and $Al_{90}Y_{10}$. The original idea was to further investigate the large increase in spin-orbit scattering rate which can be obtained by substituting La for Y in $Al_{90}Y_{10}$.[5] In the course of these investigations we found that $\Delta\rho/\rho$ for one sample of $Al_{90}La_{10}$ was up to 40 times larger than for another sample, and reached values in excess of 1%. This is completely inconsistent with weak localization and a sample resistivity of order 50 $\mu\Omega$ cm. To investigate the reason for this behavior we have made further studies of $Al_{90}La_{10}$ and report here on the results of different structural investigations and of magnetic susceptibility measurements. The anomalous enhancement of $\Delta\rho/\rho$ could be correlated with small precipitates of crystalline $Al_{11}La_3$, at a level escaping detection in standard x-ray diffraction.

For amorphous metals problems regularly arise when a quantitative description of $\Delta\rho/\rho$ is attempted over extended regions of fields and temperatures, particularly for alloys containing transition elements. This has led to questions about the validity of 3D quantum correction formulas and empirically to the introduction of a ''fudge factor'' to multiply the calculated $\Delta\rho/\rho$ to fit observations.[12] However, with the excellent description of $\Delta\rho/\rho$ of icosahedral Al-Cu-Fe over wide ranges of temperatures and magnetic fields, the precision of QIE in 3D has been demonstrated.[13] The present results thus suggest a possible interpretation of such discrepancies: An unexpectedly large magnetoresistance could be due to low resistivity crystalline impurities escaping detection by standard x-ray techniques.

II. EXPERIMENTAL DETAILS

High-purity starting elements were used. In particular, for La the dominating impurity, besides a few rare-earth elements at the level below 100 ppm, was 40 ppm of Fe. Melt-spinning was used to prepare amorphous ribbons. X-ray analysis with Cu $K\alpha$ radiation was performed on both sides of the sample ribbon in a diffractometer at room temperature. The results showed that the samples were x-ray amorphous, with no crystalline minority phases detectable. The $Al_{90}La_{10}$ samples were subjected to a more thorough structural analysis as described below.

A Guildline dc current comparator bridge was used for the resistance measurements. Standard four-pole electrical contacts stabilized with silver paint and epoxy were attached to the samples. The measurements were made in a flowing gas cryostat equipped with a superconducting magnet to 12 T and an additional temperature regulated shield situated in the gas flow outside the sample holder. This arrangement facilitates a firm temperature control. A relative temperature stability within a few mK during several hours can be achieved. The temperature was obtained from carbon and platinum resistors. The magnetoresistance of the temperature sensors was compensated for separately.

The normal-state magnetic susceptibility was measured for two $Al_{90}La_{10}$ samples in order to investigate if there were any observable magnetic difference between samples showing strongly varying magnetoresistance. Samples of mass of about 10 mg were suspended in a Faraday balance (Cahn). The magnetic force was measured at eight different fields up to 15 kOe at several temperatures in the range 4.2–300 K.

III. RESULTS

The magnetoresistance of $Al_{90}Y_{10}$ and $Al_{90}La_{10}$ was measured in a temperature range above 15 K and in magnetic fields up to 12 T. Measurement temperatures were thus larger than about $4T_c$,[5] reducing the influence of superconducting fluctuations which may further complicate analyses of the magnetoresistance. In $Al_{90}Y_{10}$ the problems to be described for $Al_{90}La_{10}$ were absent and good descriptions of the observed magnetoresistance in terms of QIE could be obtained. We then discuss the problems for $Al_{90}La_{10}$.

A. Magnetoresistance of $Al_{90}Y_{10}$

The magnetoresistance results for amorphous $Al_{90}Y_{10}$ are shown in Fig. 1. $\Delta\rho/\rho$ at 15 K is positive at low fields and negative above about 2 T, reflecting intermediate values of the ratio between the spin-orbit (τ_{so}) and inelastic (τ_{ie}) scattering times. Above 20 K, τ_{ie} is small enough that $\Delta\rho/\rho$ is negative at all fields.

The magnetoresistance of $Al_{90}Y_{10}$ was analyzed by considering three contributions to QIE: (i) weak localization (WL) according to Fukuyama and Hoshino,[14] (ii) electron-electron interaction (EEI) in the Cooper channel, as given by Altshuler *et al.*[15] and (iii) the Maki-Thompson term (MT) in the superconducting fluctuations calculated by Larkin.[16] The contribution from EEI in the diffusion channel was estimated and found to be small at all temperatures and fields and was thus neglected. The Azlamasov-Larkin contribution to superconducting fluctuations can be neglected, due to the high measurement temperatures.

Different suggestions for the function $g(T,B)$ in the EEI contribution have been made. McLean and Tsuzuki[17] obtained:

$$1/g(B,T)=\Psi\left(\frac{1}{2}\right)-\Psi\left(\frac{1}{2}+\frac{DeB}{2\pi k_B T}\right)+\ln\left(\frac{T_c}{T}\right), \qquad (1)$$

while

$$1/g(B,T)=-\ln(T^*/T_c) \quad \text{where} \quad T^*=\max\{T,(4DeB/k_B)\} \qquad (2)$$

was proposed by Altshuler *et al.*[15] Both these expressions were tested.

Input parameters for the WL contribution are the resistivity ρ and diffusivity D, and for the EEI and MT terms in addition the measured T_c. Fitting parameters for the WL terms are τ_{ie}, and τ_{so}, where τ_{ie} is allowed to vary freely at each temperature, and τ_{so} is a constant. For the MT contribution one fits τ_{ie}. T_c, ρ, the density d, and the critical magnetic field slope were obtained previously,[5] and D was calculated from these results.[8] For $Al_{90}Y_{10}$ we obtained $D=2.14$ cm^2/s.

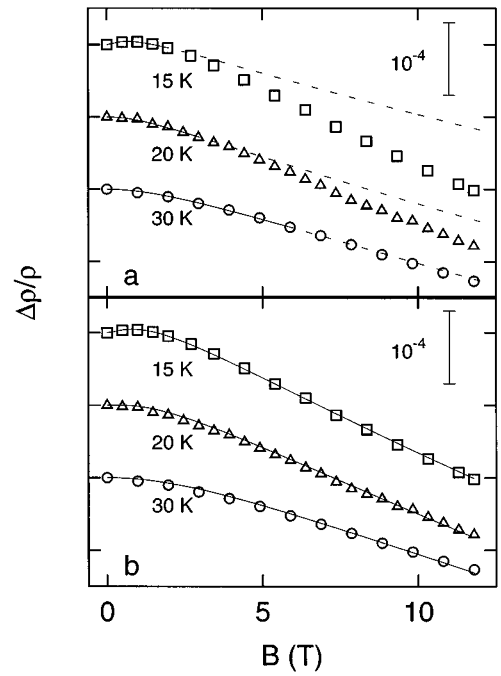

FIG. 1. The magnetoresistance of $Al_{90}Y_{10}$. The symbols are observations at the temperatures indicated. (a) the full curves are fits to WL, EEI, and MT contributions in the range $B\leqslant(k_B/4eD)T\ln(T/T_c)$ and dashed curves were calculated from these fits at higher fields. (b) the curves are fits to WL and EEI only. $\tau_{ie}(T)$ for both fits and $\beta(T)$ for the MT term are given in Fig. 2. The curves here and in Fig. 3 have been displaced vertically by an arbitrary distance for clarity.

The three contributions, (i)–(iii) were first fitted to the measured magnetoresistance. The g function of Eq. (1) was used. The results are illustrated by the curves in Fig. 1(a). The MT contribution is valid only for $B/T \ll k_B/4eD\ln(T/T_c)$, which for $Al_{90}Y_{10}$ implies $B\ll 2$, 3, and 6 T at 15, 20, and 30 K, respectively. The condition $\ll$ was relaxed to $<$, and data were analyzed up to these field limits.

For $B\leqslant(k_BT/4eD)\ln(T/T_c)$ it can be seen by the full line sections of the curves in Fig. 1(a) that excellent fits are obtained. The dashed lines are extensions to higher fields where there are increasing deviations with decreasing temperature. At 15 and 20 K the WL term is of similar magnitude as the MT term and numerically much larger than the EEI contribution. At 30 K the WL term is much larger than both the other terms and extrapolation of the low-field data describes the high-field data well. Our results indicate that deviations at high fields are due to a too slowly decreasing MT term. Further calculations are required to describe the general behavior.

An alternative analysis was performed where only the WL and one EEI term were considered. This result is shown in Fig. 1(b). Excellent fits were obtained at all fields and tem-

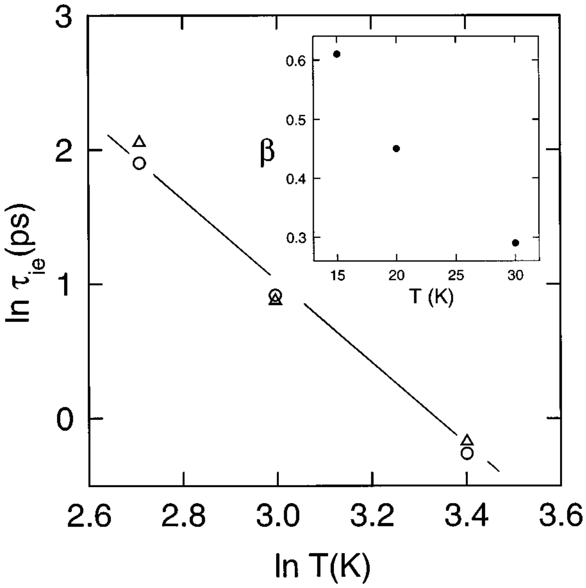

FIG. 2. $\tau_{ie}(T)$ from the fits of Fig. 1. △ (a), ○ (b). The straight line shown follows $\tau_i \approx T^{-3}$. Inset: $\beta(T)$ obtained from the fit in (a) of Fig. 1.

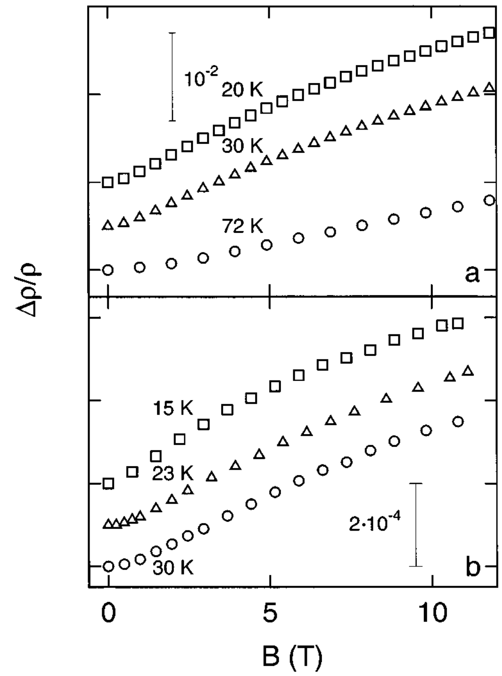

FIG. 3. (a) $\Delta\rho/\rho$ for $Al_{90}La_{10}$-I. Improved x-ray analysis showed a fraction of precipitates of $Al_{11}La_3$. (b) $\Delta\rho/\rho$ for $Al_{90}La_{10}$-II. $\Delta\rho/\rho$ is somewhat too large to be ascribed solely to quantum corrections. Some crystalline inclusions were detected only in HREM. Note the difference in ordinate scale by about a factor of 50 between (a) and (b).

peratures within our measurement range. The main change of parameters is a decrease of τ_{so} by a factor of 5 from 14 ps in Fig. 1(a) to 2.6 ps in Fig. 1(b), while the results for τ_{ie} are remarkably similar as shown in Fig. 2. $\tau_{ie} \approx T^{-3}$ is consistent with a dominant contribution from electron-phonon interaction. The alternative g function of Eq. (2) was then tested. Also in this case it was found that the observed magnetoresistance could be well described by weak localization and electron-electron interaction contributions only.

From these results the validity of the Maki-Thompson contribution at temperatures well above T_c may be questioned. Superconducting fluctuations are usually not observable above about[18] 2–3 T_c. Our analyses are consistent with the contention that superconducting fluctuations do not contribute to the magnetoresistance at temperatures above $4T_c$. Similar conclusions were obtained previously and the discrepancy between theory and experiment was attributed to strong pair breaking from thermal phonons.[19] QIE were not considered at that time.

It was recently claimed that the MT and WL terms could adequately describe the observed magnetoresistance of crystalline alloys of Al-Ti including Sn or Co doping with resistivities in the range 150 $\mu\Omega$ cm.[20] The prefactor $\beta(T/T_c)$ of the MT term was used as an adjustable parameter. Although β was found to have a reasonable temperature dependence, T_c was not measured. With an additional free parameter the significance of the result is less clear. The range of analysis in Ref. 20 of $B/T < 0.5$ T/K is too large for the validity of the MT term at the lowest measuring temperatures.

B. $Al_{90}La_{10}$

1. Magnetoresistance

The magnetoresistance of $Al_{90}La_{10}$, sample I, is shown in Fig. 3(a). It can be seen that $\Delta\rho/\rho$ is positive at all fields and temperatures and remarkably large. The magnitude is by far too large to be consistent with QIE in a metal with $\rho \approx 50$ $\mu\Omega$ cm and more in line with observations for quasicrystals with $\rho > 1000$ μQ cm.[21,22]

Therefore a new sample of the same composition was also studied, $Al_{90}La_{10}$-II. Starting materials were the same as for sample I, and processing parameters, such as melt temperature and wheel speed, were similar. $\Delta\rho(B)/\rho$ of this sample is shown in Fig. 3(b). The magnitude is a factor of 40 smaller than for sample I and qualitatively more in line with what can be expected for a strong spin-orbit scattering, disordered metal of low resistivity.

Nevertheless, nor was it possible to account for the data of sample II by QIE with acceptable precision for any combination of parameters. Excluding contributions from superconducting fluctuations, the observed $\Delta\rho/\rho$ was too large at all temperatures. The WL contribution had to be multiplied by a factor of 3 to obtain an acceptable description. This factor is larger than those employed by Bieri, Fert, and Schul.[9] When the fluctuation contribution was included, only a partial fit was obtained with τ_i almost temperature independent, which was discarded as unphysical.

2. Magnetic susceptibility

To investigate the reason for these different results for $\Delta\rho/\rho$, we first studied the magnetic susceptibility. The measured magnetic susceptibility χ_m may contain contributions from minor ferromagnetic spins and clusters in addition to the matrix susceptibility χ. Assuming such clusters to be saturated, of magnetization σ, and of weight fraction ω, one

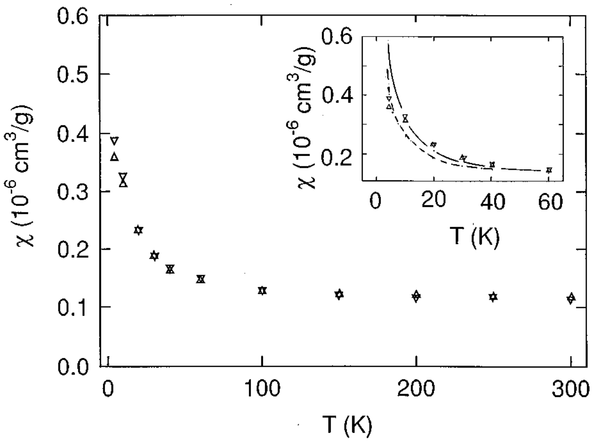

FIG. 4. The temperature dependence of the magnetic susceptibility for $Al_{90}La_{10}$. ∇ $Al_{90}La_{10}$-I, $\triangle$ $Al_{90}La_{10}$-II. Inset: extrapolation to low temperatures of a high-temperature fit to the data. Dashed curve: $Al_{90}La_{10}$-I, full curve: $Al_{90}La_{10}$-II.

can decompose χ_m as $\chi_m = \chi + \omega\sigma/H$. From this relation χ is obtained from χ_m by plotting vs $1/H$ and extrapolating to infinite field strength.

$\omega\sigma$ was found to be closely constant above 10 K, and about 10^{-6} kOe cm^3/g for both samples. If Fe impurities in the constituent elements, of $\sigma = 2.22\mu_B$ per atom are responsible for this moment, there would be 5 ppm of Fe in the sample corresponding to 50 ppm of Fe in the La used. Most of these moments thus originate from Fe impurities in La, where the nominal concentration was 40 ppm of Fe.

χ is seen in Fig. 4 to be small and increase weakly with decreasing temperature down to 60 K, while at lower temperatures a somewhat stronger temperature dependence is observed. This may be due, e.g., to the presence of clusters which cannot be saturated at our measuring fields. A simple expression, $\chi = A + B/T$, was fitted to the data in the temperature region 100–300 K, where the results for χ are most accurate. Extrapolation of those fits, inset of Fig. 4, show that the calculated curves fall below data at $T \leqslant 30$ K, as would be the case if some unsaturated spins are present. At 10 K for $Al_{90}La_{10}$-II, and at 4.4 K for both samples, data fall below the calculated curves, indicating fluctuations above the superconducting transitions.

The magnetic susceptibility of the two $Al_{90}La_{10}$ samples is thus quite similar. In particular, there is no larger difference in Stoner enhancement factors which, by an enhancement of the effective Landé factor,[23] could enhance the magnetoresistance. Thus the reason for the widely different magnetoresistances of samples I and II is not likely due to any difference in magnetic properties.

3. Structural investigations

The first x-ray investigation, referred to above, was performed in a diffractometer with Cu $K\alpha$ radiation, a monochromator in the diffracted beam and a computer controlling the experiment and storing and analyzing data (Siemens D 5000). As mentioned all samples were x-ray amorphous on both sides of the ribbons. The first peak in the structure factor, $k_p = (4\pi \sin\theta)/\lambda$, and the width, Δk_p, of this peak at half maximum were determined for the two $Al_{90}La_{10}$ samples. The results were for sample I: $k_p = 25.41 \pm 0.03$ nm^{-1}, $\Delta k_p = 4.58$ nm^{-1}, and for sample II: $k_p = 25.23 \pm 0.03$ nm^{-1}, $\Delta k_p = 4.64$ nm^{-1}. The differences in k_p is within the range of variations often observed for different amorphous alloys of similar chemical composition.[24]

A second more sensitive x-ray experiment was then performed. A Guinier-Hägg focusing camera was used with Fe $K\alpha$ radiation and Si as an internal standard. The evaluation of the photographs was performed with a microdensitometer system.[25] For sample II only the amorphous phase was observed. For the anomalous sample I, a few weak diffraction lines were detected, which could be indexed on the orthorhombic lattice of $Al_{11}La_3$. From the two x-ray experiments one can roughly estimate the amount of crystalline impurities in sample I to be in the range 1–5 %. Different degrees of absorption in the samples make this estimate uncertain.

Further investigations were performed in an analytical transmission electron microscope. Representative pieces of sample I and II of $Al_{90}La_{10}$ were electrolytically thinned in a mixture of perchloric acid and ethanol at -30 °C. In sample I a dispersion of crystallites was observed, of diameters of 50–200 Å, and at distances from several hundreds to one thousand Å. This structure was absent in sample II. Analysis of the inclusions indicated that they were somewhat richer in La than the matrix. One obtains only a lower limit of the La concentration of the precipitates since, when illuminating them, one inevitably also investigates some of the matrix material behind them. However, the lower limit obtained, of 15–20 % La, is consistent with the results from the x-ray investigation.

High-resolution transmission electron microscope (HREM) images were obtained in a JEM 2110-F at 200 kV. For sample I, areas of crystalline inclusions were readily detected. More surprisingly, smaller such areas could be found also in sample II, Fig. 5, x-ray diffraction from a grain showed a rectangular pattern as expected for a projection of an orthorhombic lattice. Diffraction from the background only displayed one diffuse ring, characteristic for an amorphous material.

We thus found crystalline inclusions of $Al_{11}La_3$ in sample I at a level which is not observed in standard x-ray diffraction. In HREM some crystallites of $Al_{11}La_3$ were found also in sample II, at a level escaping detection in high sensitivity x-ray diffraction as well as in analytical TEM.

4. Resistivity of a mixture of $Al_{11}La_3$ and amorphous $Al_{90}La_{10}$

Can crystalline impurities at the level of a few percent explain an anomalously enhanced magnetoresistance? This would seem to require that the impurity phase has quite low resistivity. $\Delta\rho/\rho$ can reach large values for pure materials at low temperatures; e.g., above 10 at 10 T for Cu of resistivity ≈ 3 nΩ cm.[26]

Resistivity measurements of $Al_{11}La_3$ at low temperatures have been made.[27] With the limited accuracy from a graph, ρ(20 K) is only about 0.5 $\mu\Omega$ cm and decreasing for decreasing temperature. Without information on the geometry of the second phase the best lower and upper bounds of the conductivity σ^* of an isotropic two phase system were obtained in a variational calculation by Hashin and Shtrikman.[28] With conductivity σ_2 and volume fraction v_2 for the minority

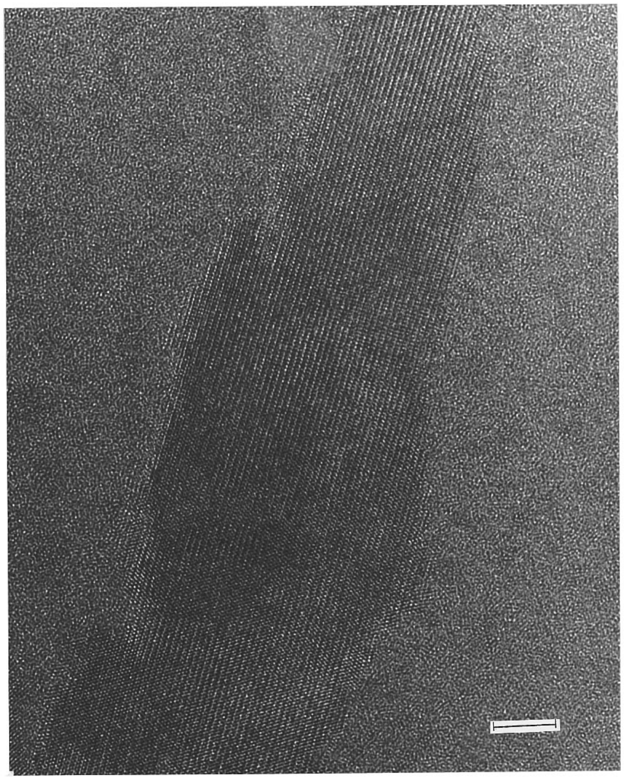

FIG. 5. High-resolution image of $Al_{90}La_{10}$, sample II. A grain of $Al_{11}La_3$, with a diameter of about 200 Å, is seen in the amorphous matrix. The unit length shown is 50 Å.

phase, $Al_{11}La_3$, and conductivity $\sigma_1 < \sigma_2$ for the amorphous phase one has

$$\sigma_1 + \frac{v_2}{1/(\sigma_2-\sigma_1)+(1-v_2)/3\sigma_1} < \sigma^* < \sigma_2 + \frac{1-v_2}{1/(\sigma_1-\sigma_2)+v_2/3\sigma_2}. \qquad (3)$$

$1/\sigma_1 \approx 50$ $\mu\Omega$ cm,[5] $1/\sigma_2$ is taken to be[27] 0.5 $\mu\Omega$ cm, and $v_2 = 0.03$ (± 0.02). One then finds $16(^{+14}_{-5}) < 1/\sigma^* < 46(\pm 2.5)$ $\mu\Omega$ cm. The observed $1/\sigma^*$ at low temperatures of[5] 41 $\mu\Omega$ cm$\pm$10% is within the limits of Eq. (3) for all reasonable values of the conductivities and with v_2 in the range 0.03 ± 0.02. $\Delta\rho/\rho$ for the amorphous phase at 10 T and 15 K was approximated by the value for sample II of $\Delta\rho/\rho$ of about 4×10^{-4}. This overestimates $\Delta\rho/\rho$ of the amorphous phase. σ^* was allowed to vary in the large range given above, and the calculation of $\Delta\rho/\rho$ of sample I was iterated by adjusting $\Delta\rho/\rho$ of $Al_{11}La_3$ at 10 T until the observed value was obtained. The range for σ^* above gave a large range for $\Delta\rho(B)/\rho$ of $Al_{11}La_3$ from several percent to a factor of 5. This rough calculation illustrates that reasonable values of the magnetoresistance of the impurity phase can explain the observations.

The range of results for σ^* could be narrowed by considering a realistic structural model for the $Al_{11}La_3$ inclusions. The HREM studies showed that none of standard geometrical approximations such as spheres, fibers, or disks, have any strong preference. The range given above for the magnetoresistance of $Al_{11}La_3$ at 10 T encompasses these special cases. Although these calculations are thus quite qualitative, they nevertheless clearly illustrate our main point: Crystalline impurities of low resistivity at the level of a few percent can drastically enhance the magnetoresistance of an amorphous metal.

IV. SUMMARY AND CONCLUDING REMARKS

The magnetoresistance of amorphous $Al_{90}Y_{10}$ was found to be well accounted for by reasonable parameters and with the weak localization and Cooper channel EEI contributions only, disregarding superconducting fluctuations at temperatures above $4T_c$. This result suggests, but does not prove, that superconducting fluctuations are not observed at these temperatures. On the other hand, when the accepted theory for the Maki-Thompson fluctuation contribution is included we find good agreement at low fields only and increasing discrepancies at higher fields, which increase with decreasing temperature. Thus, in both scenarios, further theoretical work on the fluctuation contribution to the magnetoresistance far above T_c is necessary.

The magnetoresistance for $Al_{90}La_{10}$ was found to be strongly enhanced for one sample, which appeared to be amorphous on both sample sides with standard x-ray techniques. However, inclusions of small crystallites of $Al_{11}La_3$, at the level of a few percent, could be verified by a more sensitive x-ray technique, and analytical TEM and HREM investigations. A second sample of $Al_{90}La_{10}$ showed a magnetoresistance which was smaller by a factor of 40 and qualitatively of the order expected for amorphous metals. This sample appeared to be amorphous both in a Guinier-Hägg camera and in an analytical TEM. However, $\Delta\rho(B)/\rho$ was too large to be quantitatively described by QIE. Small amounts of crystallites below the detection limit of the other methods were observed in HREM.

These results are interesting in view of the frequently reported discrepancies between observations and theories for quantum interference effects of amorphous metals. When data could not be explained by theory, the number of different contributions and the complex formalism of QIE have often led experimentalists to assume that theory must be remedied, or that some parameters should have unusual values. With the quantitative precision obtained[13] for icosahedral Al-Cu-Fe over a much larger range of temperatures than feasible for amorphous metals, it appears less likely that WL and EEI theories in 3D should be questioned. Our results indicate another possibility. Small crystalline precipitates in the amorphous matrix of a low resistivity crystalline phase can lead to strong enhancement of the magnetoresistance. Such crystallites could escape also the majority of careful structural investigations of amorphous alloys.

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