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1990 J. Phys.: Condens. Matter 2 6169

(http://iopscience.iop.org/0953-8984/2/28/007)

J. Phys.: Condens. Matter 2 (1990) 6169–6178. Printed in the UK

Electron transport properties of thermodynamically stable Al–Cu–Ru icosahedral quasicrystals

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Received 29 January 1990, in final form 27 March 1990

Abstract. The electron transport properties of the thermodynamically stable $Al_{68}Cu_{17}Ru_{15}$ quasicrystal have been studied through the measurements of the electronic specific heat coefficient and the temperature dependence of the electrical resistivity in the range 4.2–300 K. The full-width at the half maximum for the strongest x-ray diffraction line (100000) is reduced to be less than 0.15 nm^{-1} either by remelting the ingot with subsequent furnace cooling or by annealing the melt-spun ribbon at 850 °C for 24 hours. An apparent improvement in the quasi-crystallinity upon the heat-treatment caused a drastic increase in resistivity up to 1600 $\mu\Omega$ cm and accompanied a very small electronic specific heat coefficient γ lower than 0.3 mJ mol^{-1} K^{-2}. The temperature dependence of the resistivity characterised by a concave curvature with a negative TCR can be discussed in terms of the weak localisation of conduction electrons. We are also convinced that the thermodynamically stable quasicrystals like the present Al–Cu–Ru and the previously studied Al–Li–Cu always exhibit a very low γ value coupled with a high resistivity. From this we conclude that the electron density of states in thermodynamically stable quasicrystals possesses a structure-induced minimum and that the Fermi level does fall in this critical range. An increase in the resistivity upon improvement in quasi-crystallinity has been discussed in terms of the generalised Faber–Ziman theory and also in terms of a possible enhancement in the coherent multiple scattering due to locally well-developed short-range order.

1. Introduction

As a result of previous work we are now well convinced that quasicrystals form a new family in the classification of solids in terms of atomic structure. They exhibit features different from either crystalline or amorphous solids and are characterised by possessing locally well-ordered atomic arrangements but having a non-crystallographic symmetry incompatible with the translational symmetry. These newly discovered materials have attracted attention from the point of view of the precise determination of the atomic positions and also of the electron transport properties manifested by conduction electrons travelling in such a non-periodic lattice.

The sp-electron quasicrystals are defined as those having the Fermi level in the valence band dominated by sp-electrons. They are particularly suitable for studying the

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electron transport mechanism, since they are free from magnetic effects and also from d-electron conduction. Although the studies along this line have already been made by several investigators, the results are not necessarily consistent with each other due, presumably, to the difficulty in preparing the well-characterised quasicrystals. For instance, Wagner *et al* (1989) reported that the $Al_{56.1}Li_{33.7}Cu_{10.2}$ quasicrystal obtained from annealing of the as-cast ingot and also melt-spun $Mg_{32}Zn_{52}Ga_{16}$ quasicrystal yield a very low electronic specific heat coefficient below 0.4 mJ $mol^{-1} K^{-2}$ and low resistivities, less than 100 $\mu\Omega$ cm. This is surprising in view of the fact that the reduction in the electronic specific heat coefficient γ relative to the corresponding free electron value always results in a substantial increase in resistivity in the case of sp-electron amorphous alloys (Mizutani 1988a, b). Indeed, Kimura *et al* (1989) observed an extremely high resistivity reaching 800 $\mu\Omega$ cm coupled with a small γ value of 0.32 mJ $mol^{-1} K^{-2}$ for the $Al_{55.0}Li_{35.8}Cu_{9.2}$ quasicrystal, which was grown using the Bridgman method. They claimed that they could obtain a single-grained quasicrystal by carefully removing the Al second phase by chemical etching, which unavoidably precipitates in the matrix of the quasicrystalline phase as a eutectic mixture.

Mizutani *et al* (1990b) studied the electron transport properties for a number of Mg–Al–Cu, Mg–Al–Ag, Mg–Zn–Ga quasicrystals prepared by melt-spinning and discussed the interrelationship between the observed resistivity and electronic specific heat coefficient in relation to the corresponding data for the non-magnetic amorphous alloys. They pointed out that the resistivity in the sp-electron quasicrystals increases with decreasing the electronic specific heat coefficient or the carrier density at the Fermi level in conformity with that observed in sp-electron amorphous alloys. From this it is inferred that a small value of γ ought to accompany a large resistivity, as reported by Kimura *et al* (1989). They also revealed that the value of γ for a series of the melt-spun Mg–Zn–Ga quasicrystals is not small but falls in the range 0.8–1 mJ $mol^{-1} K^{-2}$, the magnitude being comparable to or slightly lower than the corresponding free electron value.

Another interesting point to be noted is that a small value of γ has so far been observed only for the thermodynamically stable quasicrystals like Al–Li–Cu and Al–Cu–Fe (Wagner *et al* 1989, Kimura *et al* 1989). A small γ value relative to the corresponding free electron value indicates that a dip should exist in the density of states and that the Fermi level falls in this critical range. It has been suggested that the coincidence of the Fermi level with a minimum in the density of states would contribute to stabilise the atomic structure responsible for inducing such unique electronic structure. Fujiwara and Yokokawa (1989) calculated the valence band structure for the crystalline R-phase in the Al–Li–Cu alloy system, which possesses the same local atomic arrangements as in the quasicrystalline counterpart, and revealed that the Fermi level does fall at a structure-induced deep minimum.

The extension of the study of the electronic structure and the electron transport properties to other thermodynamically stable quasicrystals is, therefore, of particular interest. Tsai *et al* (1988) recently revealed that the $Al_{65}Cu_{20}Ru_{15}$ and $Al_{65}Cu_{20}Os_{15}$ quasicrystals are thermodynamically stable. They are suited for the electron transport studies because not only magnetic effects are absent but also the d-states associated with Cu and Ru (or Os) would be submerged well below the Fermi level E_F so that the sp-electrons dominate there. In the present study, we measured the low-temperature specific heat and the electrical resistivity in the range 4.2–300 K for the thermally stable Al–Cu–Ru quasicrystal and discuss the results in connection with the previously reported data for sp-electron quasicrystals (Matsuda *et al* 1989, Kimura *et al* 1989, Mizutani *et al* 1990b) as well as the sp-electron amorphous alloys (Mizutani *et al* 1990a).

2. Experimental procedure

A homogeneous alloy ingot of $Al_{68}Cu_{17}Ru_{15}$ was prepared by arc-melting of appropriate amounts of pure elements 99.99% Al, 99.99% Cu and 99.9% Ru in an Ar gas atmosphere. Melting was repeated several times to ensure a homogeneous mixing of constituent elements. The ingot thus obtained was quite porous and, hence, was remelted at 1400 °C in a carbon crucible under an Ar gas atmosphere with subsequent furnace cooling to 300 K. The remelted ingot turned out to be almost free from cracks and voids. A rectangular block (0.4 × 0.9 × 3.0 mm) cut from the ingot served as a sample for the resistivity measurements. The remaining ingot was crushed into pieces and subjected to melt-spinning, using a single-roll spinning wheel apparatus operated in Ar gas atmosphere. The resulting ribbons were also annealed at 850 °C for 24 hours. The formation of the quasi-crystalline single phase was confirmed by the ordinary x-ray diffraction with Cu Kα radiation.

The low-temperature specific heats were measured in the temperature range 1.5–6 K, using the conventional DC adiabatic method. The sample was 2.62 g in weight and had originally been a bulk one remelted at 1400 °C as described above. However, it became almost powdered during handling; hence, the measurement was carried out by compacting it into a Au-plated Cu container, whose specific heat contribution was later subtracted. The electrical resistivity was measured on the bulk and ribbon samples in the range 4.2–300 K, using the DC four-probe method. The resistivity at 300 K was determined by taking average values for the thickness and width measured at various places on the ribbon sample. Its accuracy is estimated to be ±5%.

3. Results

Figure 1 shows the x-ray diffraction pattern for the sample remelted at 1400 °C. All diffraction lines except very weak peaks can be indexed in terms of six independent Miller indices proposed by Bancel *et al* (1985). The full-width at the half maximum (hereafter referred to as FWHM) for the very strong diffraction line (100000) turned out to be less than 0.15 nm^{-1}, which is comparable to the value of 0.13 nm^{-1} reported by Tsai *et al* (1988). The low-temperature specific heat data measured on this sample are shown in figure 2. The data can be well fitted to equation $C = \gamma T + \alpha T^3 + \delta T^5$ over the range 1.5–5 K. The resulting coefficients are summarised in table 1. The value of γ is found to be 0.28 mJ mol^{-1} K^{-2}, which is even smaller than that obtained for the $Al_{55.0}Li_{35.8}Cu_{9.2}$ quasicrystal (Kimura *et al* 1989). It is worth noting that the Debye temperature for the present Al–Cu–Ru quasicrystal is almost 600 K, being the highest among the quasicrystals so far studied.

Figure 3 shows the temperature dependence of the resistivity for the as-quenched ribbon sample, along with its diffraction profile. The diffraction lines are broad with the average FWHM of 0.29 nm^{-1}. In contrast to the remelted bulk sample, no trace of the FCC Al line was observed. The resistivity at 300 K turned out to be 760 $\mu\Omega$ cm. It can be seen that the resistivity decreases monotonically with increasing temperature over the temperature range 4.2–300 K. The temperature dependence is concave and the total variation of resistivity amounts to about 5%.

The measurement of the electrical resistivity was repeated after annealing the as-quenched ribbons at 850 °C for 24 hours. The results are shown in figure 4, together with its x-ray diffraction data. The value of FWHM for the main diffraction lines is greatly

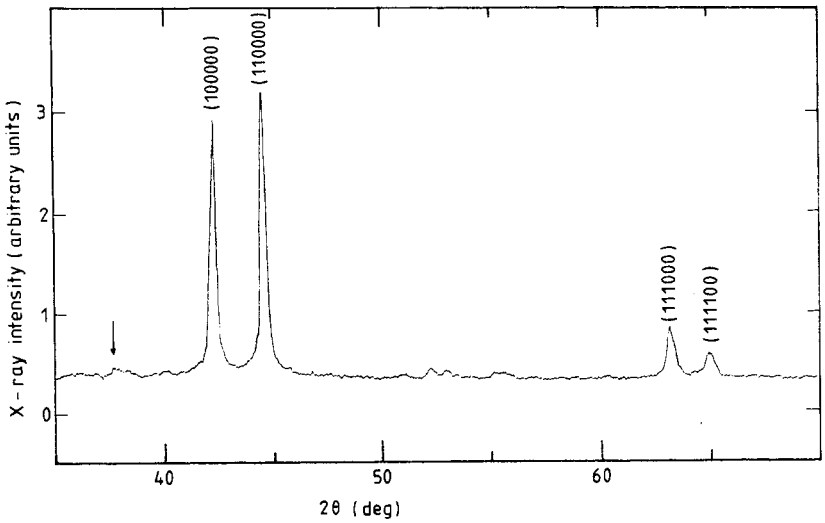

Figure 1. X-ray diffraction pattern for the $Al_{68}Cu_{17}Ru_{15}$ quasicrystal remelted at 1400 °C with subsequent furnace cooling to room temperature. The FWHM value of the (100000) line is determined to be less than 0.15 nm^{-1}. An arrow indicates a position corresponding to the FCC Al (111) line. Several very weak lines in the range $2\theta = 50$–$60°$ are also found in the original data by Tsai *et al* (1989). They have not yet been indexed.

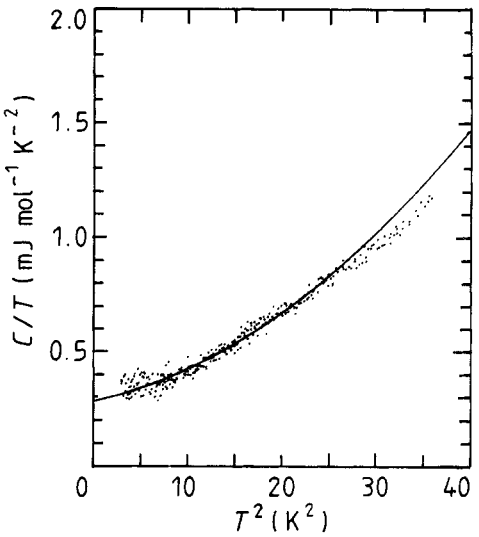

Figure 2. Low-temperature specific heat data in the form of C/T versus T^2 for the $Al_{68}Cu_{17}Ru_{15}$ quasicrystal, whose x-ray diffraction data are shown in figure 1. The data below 5 K can be fitted well to equation $C = \gamma T + \alpha T^3 + \delta T^5$.

reduced and become comparable to that for the bulk sample shown in figure 1. Although the concave feature of the ρ–T dependence is retained, its slope becomes much steeper and the total variation of resistivity exceeds 20%. More surprisingly, the resistivity at 300 K is found to be increased up to the value of 1600 $\mu\Omega$ cm due to this heat-treatment.

The electrical resistivity for the bulk sample cut from the remelted ingot was also measured, though the x-ray diffraction analysis revealed the presence of a faint trace of

Table 1. Electron transport properties of $Al_{68}Cu_{17}Ru_{15}$ icosahedral quasicrystals.

	Specific heat coefficient			
Sample condition	γ mJ mol^{-1} K^{-2}	α mJ mol^{-1} K^{-4}	θ_D K	δ mJ mol^{-1} K^{-6}
Remelted ingot	0.285 ± 0.006	0.009 ± 0.001	599 ± 20	0.0005
Melt-spun ribbon				
As-quenched				
Annealed at 850 °C				

Sample condition	Resistivity ρ_{300K} $\mu\Omega$ cm	X-ray diffraction FWHM of (100000) line nm^{-1}
Remelted ingot	730	<0.15
Melt-spun ribbon		
As-quenched	760	0.29
Annealed at 850 °C	1600	<0.15

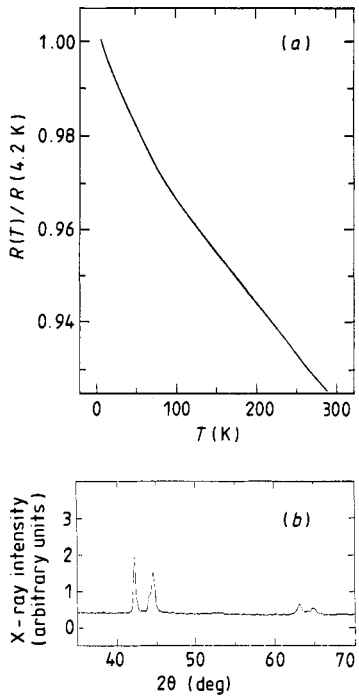

Figure 3. (*a*) Temperature dependence of electrical resistivity normalised with respect to that at 4.2 K and (*b*) x-ray diffraction data for the as-quenched $Al_{68}Cu_{17}Ru_{15}$ quasicrystal. The resistivity at 300 K is determined as 760 $\mu\Omega$ cm.

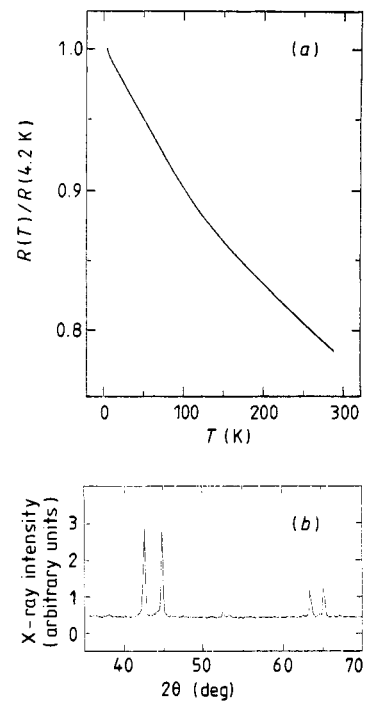

Figure 4. (*a*) Temperature dependence of electrical resistivity normalised with respect to that at 4.2 K and (*b*) x-ray diffraction data for the annealed $Al_{68}Cu_{17}Ru_{15}$ quasicrystal. The resistivity at 300 K is determined as 1600 $\mu\Omega$ cm.

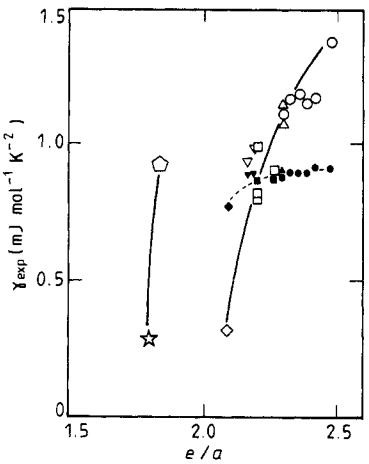

Figure 5. The measured electronic specific heat coefficient γ_{exp} as a function of the average electrons per atom e/a for various sp-electron quasicrystals. (▽) Mg–Zn–Al (Matsuda *et al* 1989), (○) Mg–Al–Cu, (△) Mg–Al–Ag, (□) Mg–Zn–Ga (Mizutani *et al* 1990b), (◇) Al–Li–Cu (Kimura *et al* 1989), (⬠) Al–Cu–V (Matsuda *et al* 1990) and (☆) Al–Cu–Ru (present data). Corresponding small full symbols refer to the value of γ in the free electron model.

the FCC Al second phase (see figure 1). Its resistivity value at 300 K was found to be 730 $\mu\Omega$ cm and the temperature coefficient to be weakly positive over a whole temperature range. The occurrence of a positive TCR may be due to the presence of the crystalline second phase. As mentioned above in connection with figure 3, the ribbon sample is apparently free from the second phase, but we are well aware that a complete elimination of the second phase and its experimental confirmation is indeed difficult. The effect of the second Al phase on the value of γ and the electron transport in the Al–Li–Cu quasicrystal has been discussed (Kimura *et al* 1989). Judging from the close agreement in the FWHM value of the x-ray diffraction lines and the minute amount of the second phase in the specific heat sample, we believe that the small γ value may well be correlated with the resistivity of 1600 $\mu\Omega$ cm for the annealed ribbon sample shown in figure 4. All relevant data are summarised in table 1.

4. Discussion

4.1. Electronic structure

Tsai *et al* (1989) evaluated the average electron concentration e/a for the transition metal-bearing quasicrystals by assigning each transition metal element to the e/a value deduced from the charge transfer model originally introduced by Pauling (1938). A choice of this simple e/a rule was very successful, since they could discover many new quasicrystals by using this rule as a guide to predict a composition range in favour of the formation of a quasicrystal. They pointed out that the thermodynamically stable quasicrystals are always achieved at the average e/a value of 1.75 or when the Fermi wavenumber $2k_F$, which can be derived from the e/a value, coincides with the wavenumber K_p corresponding to the strongest diffraction line of (100000) or (110000).

Figure 5 shows the measured electronic specific heat coefficient against the average electron concentration for quasicrystals so far studied. Included are the data for Mg–Zn–Al (Matsuda *et al* 1989), Mg–Al–Cu, Mg–Al–Ag, Mg–Zn–Ga (Mizutani *et al* 1990b), Al–Li–Cu (Kimura *et al* 1989), Al–Cu–V (Matsuda *et al* 1990) and the present

Al–Cu–Ru quasicrystals. The data are divided into two groups: one based on triacontahedron-type icosahedral quasicrystals such as Al–Li–Cu and Mg based ones and the other based on the Mackay icosahedron such as Al–Cu–V and Al–Cu–Ru. The electron concentration for the former can be designated without difficulty, since the nominal valence electrons per atom for Al, Mg, Zn, Cu, Ag and Li are simply given by 3, 2, 2, 1, 1 and 1, respectively. For the latter we followed the procedure employed by Tsai *et al* and assigned the value of e/a for Ru and V to be -2.66 and -5.66, respectively. It is seen that the data are grouped into two, depending on the triacontahedron-type or the Mackay icosahedron-type quasicrystals, and that the value of γ in both cases decreases sharply with decreasing e/a. More important is that the thermodynamically stable quasicrystals Al–Li–Cu and Al–Cu–Ru indeed take the smallest value of γ among the quasicrystals in respective groups. We take this as evidence that the valence band in these thermally stable quasicrystals definitely possesses a structure-induced deep minimum and that the Fermi level meets with this minimum.

4.2. Electron transport properties

As mentioned in the preceding section, an extremely high resistivity of 1600 $\mu\Omega$ cm was observed, coupled with a very small value of γ, for the Al–Cu–Ru quasicrystal characterised by sharp diffraction lines. A possession of a high-resistivity with a low γ value is consistent with the previously reported data for the Al–Li–Cu (Kimura *et al* 1989). Figure 6 illustrates the interrelationship between the measured value of ρ at 300 K and the electronic specific heat coefficient for sp-electron quasicrystals so far studied (Mizutani *et al* 1990b, Kimura *et al* 1989) and also for non-magnetic amorphous alloys (Mizutani *et al* 1990a, Mizutani *et al* 1990c). The data for the amorphous alloys can be grouped into three families depending on the degree of the d-character at the Fermi level. The data for the sp-electron amorphous alloys fall on a steeply declining curve, regardless of the alloy system. Here it is seen that the value of ρ sharply increases with decreasing the value of γ and that an increase in ρ accompanies a systematic change in the ρ–T types from (*a*) to (*e*), which is schematically illustrated in the inset.

It can be seen that the ρ–γ data for the sp-electron quasicrystals fall on a curve almost identical to that found for the sp-electron amorphous alloys (Mizutani *et al* 1990a). Kimura *et al* (1989) pointed out that the data for the Al–Li–Cu quasicrystal, whose ρ–T is characterised by type (*d*), were located at the high-resistivity end of this master curve. Here the type (*d*) is referred to as the ρ–T, in which the resistivity decreases almost linearly with increasing temperature over a wide temperature range, 2–300 K. In contrast, the concave curvature is clearly visible in the present Al–Cu–Ru quasicrystal and hence, its ρ–T is designated to type (*e*). Note here that type (*e*) appears after type (*d*) at the high-resistivity limit in the case of the sp-electron amorphous alloys (Mizutani 1988a, b, Mizutani *et al* 1990a). Indeed, the present ρ–γ data for the Al–Cu–Ru are found to be located at the further end of the master curve. Therefore, we are led to conclude that, in sp-electron quasicrystals, the value of ρ increases with decreasing the value of γ and changes its ρ–T character from type (*a*) to (*e*) in the same manner as observed in the non-magnetic amorphous alloys.

A dashed curve drawn in figure 6 represents a possible high-resistivity limiting curve, which is obtained from the resistivity formula $\rho^{-1} = (e^2/3)\Lambda_F v_F N(E_F)$ under the constraints that the mean free path of the conduction electrons is replaced by the average atomic distance of 4 Å and that the Fermi velocity v_F is reduced to $\frac{1}{8}$ that of the free electron value of 10^8 cm s^{-1}. It was noted that the types (*d*) and (*e*) appear only in the

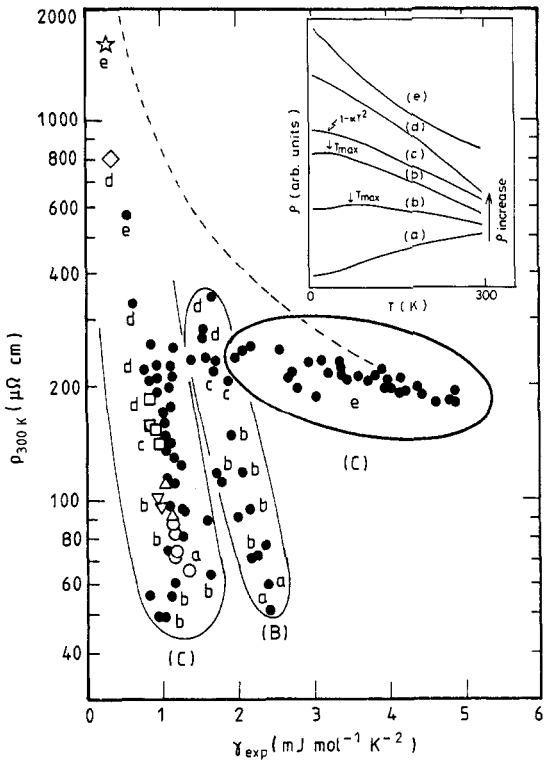

Figure 6. Interrelationship between the measured value of $\rho_{300\,K}$ and the electronic specific heat coefficient γ_{exp} for sp-electron quasicrystals and non-magnetic amorphous alloys. The data for the amorphous alloys are shown with full circles without differentiating each alloy system (for more detail see Mizutani *et al* (1990a)). The symbols for quasicrystals are as follows: (▽) Mg–Zn–Al (Matsuda *et al* 1989), (○) Mg–Al–Cu, (△) Mg–Al–Ag, (□) Mg–Zn–Ga (Mizutani *et al* 1990b), (◇) Al–Li–Cu (Kimura *et al* 1989) and (☆) Al–Cu–Ru (present data). A broken curve is drawn as a guide for a possible high-resistivity limiting curve, beyond which no data would appear (see text for more detail). The whole set of data is divided into three families encircled by (A), (B) and (C), depending on the degree of d-states at the Fermi level: (A) sp-electron systems, (B) systems in which d-states coexist with sp-states and (C) d-electron systems. The ρ–T types are marked for representative data points. The ρ–T curves of types (*a*)–(*e*) are schematically shown in the inset.

vicinity of this limiting curve in non-magnetic amorphous alloys (Mizutani 1988a, b, Mizutani *et al* 1990a). The data for the quasicrystals are apparently not exceptional to this rule: the data for the Al–Li–Cu (type (*d*)) and the Al–Cu–Ru (type (*e*)) *do* fall near this limiting curve.

Kimura *et al* (1989) analysed the ρ–T data of type (*d*) by plotting conductivity, rather than the resistivity, against temperature. They found that the conductivity increases in proportion to the square-root of temperature below about 20 K and almost linearly above 20 K and attributed this to effects associated with the weak localisation of conduction electrons. The same conclusion has been drawn in high-resistivity amorphous alloys of types (*d*) and (*e*) (Mizutani *et al* 1990a, Mizutani 1988a, b). It may be worthwhile mentioning that both types (*d*) and (*e*) exhibit the same features when analysed in terms of conductivity and, hence, the scattering mechanism involved would be essentially the

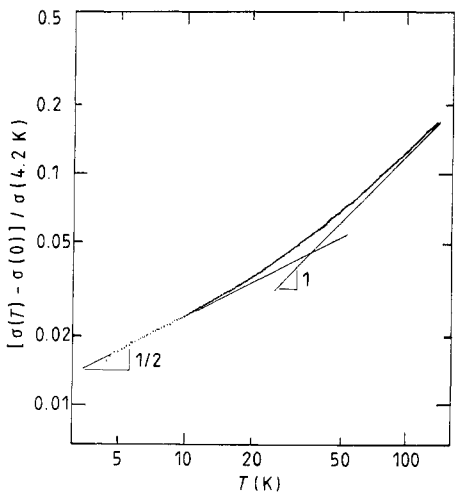

Figure 7. Log$[(\sigma(T) - \sigma(0))/\sigma(4.2\,\mathrm{K})]$ against log T for the annealed $Al_{68}Cu_{17}Ru_{15}$ quasicrystal. The data shown in figure 4 has been converted into this form. The slopes of the lines are $\frac{1}{2}$ and 1.

same. The ρ–T data shown in figure 4 is converted to the conductivity versus temperature and reproduced in figure 7. It is clear that the square-root and linear temperature dependences hold valid below and above approximately 20 K, respectively. We, therefore, ascertain that the scattering mechanism in the Al–Cu–Ru quasicrystal is dominated by the weak localisation effects.

Finally, we discuss the effect of an enhancement in the quasi-crystallinity on the resistivity of a quasicrystal. As discussed in the preceding section, the heat-treatment of the ribbon sample at 850 °C for 24 h definitely reduced the FWHM value of the diffraction lines and caused a substantial increase in resistivity. A similar phenomenon has been observed in the Mg–Zn–Ga quasicrystal, where a sharpening in the x-ray diffraction profile upon annealing results in a definite increase in resistivity (Mizutani *et al* 1990b). They are inclined to believe that the reduction in the phason strains and the possible grain growth are most likely responsible for the reduction in the FWHM of the x-ray diffraction lines and that an enhancement in the quasi-crystallinity contributes to an increase, rather than a decrease, in the resistivity. This is contrary to what one might naturally expect for crystalline metals.

Within the framework of the generalised Faber–Ziman theory, which is constructed on the basis of the Boltzmann transport equation with the second-order perturbation theory, the electrical resistivity would be decreased if the total area under the structure factor is reduced, at least, in the integration range 0–$2k_F$. An increase in resistivity within this model might be still logically possible, provided that the sharpening of the diffraction lines proceeds so as to satisfy the $2k_F = K_P$ condition without reducing the total area under the structure factor below $2k_F$. However, it must be borne in mind that the theory based on the Boltzmann transport equation breaks down when the mean free path of conduction electrons becomes comparable to an average atomic distance and that this condition is indeed satisfied in the present quasi-crystalline sample, as discussed above.

An alternative explanation may be found in the recent theory put forward by Fresard *et al* (1990a, b). They applied the effective medium approximation (EMA) to a disordered system and showed that a locally existing coherent atomic structure in the liquid or amorphous solids produces a structure-induced minimum in the electron density of states as a result of multiple-scatterings. They also predicted that an enhancement in

resistivity would occur when the Fermi level coincides with a structure-induced minimum and also that a negative TCR would occur due to the thermal broadening of the Fermi distribution function. We consider their model to be applicable to the electron transport of sp-electron quasicrystals, where the coherent multiple-scattering or the Bragg scattering is expected to occur more predominantly without being capable of forming the Bloch states. The experimental findings that the resistivity for quasicrystals always becomes higher than for the corresponding amorphous state may provide some clue to the presence of a mechanism in which the local coherent scattering enhances the resistivity. Studies along these lines are now under consideration.

Acknowledgments

We are grateful to Professor M Itoh, Shimane University, and Dr H Tsunetsugu, The Institute for Solid State Physics, The University of Tokyo, for valuable discussions in connection with the local coherent multiple scattering expected in the quasiperiodic lattice.

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