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Dislocation resistivity in Cu: dependence of the deviations from Matthiessen's rule on temperature, dislocation density and impurity content

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Abstract. Measurements of dislocation resistivity and the deviations from Matthiessen's rule (DMRs) are reported for polycrystalline Cu samples with various dislocation densities, impurities and phonons. Since this DMR can reach the same order of magnitude as the dislocation resistivity itself, its quantification is essential in using the electrical resistivity for dislocation density measurement. In the framework of the classical two-group model, a formula for the DMR is derived for the three independent scatterers. The fits by this formula are satisfactory provided that isotropic phonon scattering is chosen for samples with high dislocation densities. While samples with a high purity (residual resistivity ratio (RRR) ≥ 300) show dislocation scattering anisotropies $A_{dis} \simeq 0.1$ irrespective of dislocation density, the more impure material (purity 99.99%) (RRR = 130) exhibits a significant decrease in A_{dis} at low dislocation densities, indicating some contribution of enhanced strain fields in the dislocation structure. This leads to an enhanced DMR contribution in this material and makes the determination of the dislocation density N by the electrical resistivity method less reliable. Nevertheless, in the whole purity range considered, the electrical resistivity yields at least the same measuring resolution as TEM of about $\Delta N \simeq 1 \times 10^9 \text{ cm}^{-2}$, but a better performance for high dislocation densities up to the order of 10^{14} cm^{-2} .

1. Introduction

Deviations from Matthiessen's rule (DMRs) have been thoroughly investigated for a long time, which is well reflected in the reviews of, for example Bass [1] and Cimberle *et al* [2]. However, the overwhelming part of their papers was concerned with point defects with respect to temperature, i.e. the phonon density, and not so much with other crystal defects such as dislocations. This means a real lack of knowledge because it has turned out in the last few decades that many other crystal defects such as large- and small-angle grain boundaries and point defect clusters basically consist of certain arrays of dislocations [4, 5]. Moreover, efforts have been made in the last few years to use the electrical resistivity as a measuring tool to determine the dislocation density [3] and, consequently, the total grain boundary area [6]. Therefore the first tasks were to check the pre-conditions for this, one being the question of the scattering contribution of the dislocation core or strain field [3]. Furthermore DMRs (which are expected to arise when different scatterers with different scattering anisotropies coexist [7]) impede proper determination of the dislocation density using the electrical resistivity if they cannot be accounted for in the quantitatively correct way.

So it is the aim of the present paper to investigate thoroughly the DMRs in the presence of dislocations, as a function of their density, impurity content and temperature. This also

appeared to be a good idea since research in the last few years has revealed the specific dislocation resistivity to be largely independent of dislocation density and arrangement because of the dominance of *scattering by the core* [3, 8, 9] or at least by *near-core regions* [10]. Concerning the quantification of the DMR itself, the conditions under which the concept of the classical *two-group model* (TGM) can be used are also of interest. This question was also treated in a recent paper [11] which discusses the possibility of simultaneous description of the DMR and the low-field Hall effect by the TGM.

2. Experimental details

Samples were prepared from high-purity copper with different impurity contents as shown in table 1, (for simplification the different copper types will be denoted by the letters A, B, M and Z as indicated).

Table 1. Impurity contents of the different types of copper used.

Element	Impurity content (ppm) for the following types of Cu			
	MBOFN Cu (purity, 99.99%)	OFE 101 Cu (purity, 99.99%)	Cu (purity, 99.99+%)	Cu (purity, 99.9999%)
	Material Z	Material M	Material A	Material B
Ag	9	14	7	
Zn	1	0.8		
Pb	8	5		<0.05
Fe	3.2	4	2	
Sb	1.8	1.4		<0.10
S	22	15		<0.10
As	1.5	1		
Ni	3	3		
P	5	<1		
Ca			<1	
Mg				<0.05
Mn				0.40
Co				<0.05
Au				<0.05
Si				0.2
Sn				<0.05

Pieces of materials B, M and Z were cut by spark erosion and rolled to thicknesses between 250 and 100 μm . The rolling was performed in a sandwich with copper plates so that any contamination by iron atoms was avoided. Material A was delivered as a foil of 250 μm thickness which could be used directly as the sample material. The copper foils were then annealed in a high vacuum (annealing time t , annealing temperature T and total pressure p) to give different residual resistivities ρ_i at 4.2 K as shown in table 2.

Material A was additionally annealed at a partial oxygen pressure $p^* = 4 \times 10^{-5}$ mbar [12] in order to oxidize the Fe impurities [13], lowering the residual resistivity by a factor of almost 10.

The foils were deformed by tension up to a true strain $\varepsilon = 0.15$ and by rolling to $0.15 \leq \varepsilon \leq 1.04$. The rolling was again performed with copper plates to avoid direct contact with the rolls. The samples were cut by spark erosion to ensure well defined leads for current and potential difference.

Table 2. Annealing conditions.

Cu type	t (h)	T (°C)	p (mbar)	$\rho_i(4.2 \text{ K})$ (nΩ cm)
A	16	950	2×10^{-7}	5.51
	10	950	2×10^{-4}	0.598
B	16	500	2×10^{-7}	1.182
M	16	600	1×10^{-6}	5.681
Z	16	600	1×10^{-6}	12.546

The measuring current was 0.5 A; the voltage was measured with a resolution of ± 1 nV. The electrical resistivities of all deformed samples were measured at 4.2 K, 77 K and room temperature in Dewar baths of liquid helium, liquid nitrogen and alcohol. The temperature of the alcohol was measured with a Beckmann thermometer with an accuracy ΔT of ± 0.01 °C. Temperature-dependent measurements between 4.2 and 150 K were performed in an evaporation cryostat with controlled helium flow [14]. The measurement temperature was kept constant to ± 0.01 K for $T > 40$ K and to ± 0.005 K for $T < 40$ K. The temperature was measured by means of a carbon glass resistor which gave highly reproducible results.

The shape factors G of all samples were determined by electrical measurements after a second annealing treatment at 500 °C from

$$G = \frac{R(293.2 \text{ K}) - R(4.2 \text{ K})}{\rho_p(293.2 \text{ K})} \quad (1)$$

assuming Matthiessen's rule for these samples at 293 K. The absolute error in G was lower than 1%. The value for the phonon resistivity $\rho_p(293.2 \text{ K})$ was taken from the literature [15] to be 1679 nΩ cm.

The measured dislocation resistivity (rd) of the samples is given as

$$rd(T) = \rho_{p,i,d} - \rho_{p,i,d=0} \quad (2)$$

which is the difference between the specific resistivity of the deformed sample, with phonons (subscript p), impurities (subscript i) and dislocations (subscript d) as scatterers, and the specific resistivity of the undeformed 'reference' sample (subscript $d \equiv 0$) with the conditions before deformation. The resistivity of the reference sample was in good agreement (± 0.1 nΩ cm) with the values of the deformed samples after the second annealing procedure.

The quantity rd contains not only the true dislocation resistivity ρ_d but also some DMRs due to the different scattering mechanisms in the deformed and in the undeformed condition, respectively:

$$\rho = \rho_p + \rho_i + \rho_d + \delta_{pid} \quad \rho_{Ref} = \rho_p + \rho_i + \delta_{pi} \quad (3)$$

where ρ and ρ_{Ref} are the resistivities of the deformed sample and reference sample, respectively, and δ is the DMR. It then follows that

$$rd = \rho - \rho_{Ref} = \rho_d + \delta_{pid} - \delta_{pi}. \quad (4)$$

In all cases, $\delta_{pid} \gg \delta_{pi}$ is valid and therefore rd reflects mainly the first DMR contribution. At 4.2 K (where ρ_p was set to zero), $rd(4.2 \text{ K})$ contains not only ρ_d but also the DMR caused by the common presence of impurities and dislocations δ_{id} , which therefore must be known well enough to find the true dislocation resistivity ρ_d .

3. Formulation of the two-group model for three independent scatterers

The TGM divides the Fermi surface into two characteristic regions of necks and bellies (with non-interacting 'groups' of electrons), each of them having a typical contribution σ_n and σ_b to the total electrical conductivity σ :

$$\sigma = \sigma_n + \sigma_b \quad (5a)$$

and therefore

$$\frac{1}{\rho} = \frac{1}{\rho^n} + \frac{1}{\rho^b}. \quad (5b)$$

In each group the sum of partial resistivities caused by the different scattering mechanisms j gives the total resistivity of the group:

$$\rho^n = \sum_j \rho_j^n \quad (6a)$$

and

$$\rho^b = \sum_j \rho_j^b. \quad (6b)$$

From (5b), one obtains

$$\rho = \frac{\rho^n \rho^b}{\rho^n + \rho^b} \quad (7)$$

and

$$\rho_j = \frac{\rho_j^n \rho_j^b}{\rho_j^n + \rho_j^b}. \quad (8)$$

From (6) and (7),

$$\rho = \left(\sum_j \rho_j^n \right) \left(\sum_j \rho_j^b \right) / \left(\left(\sum_j \rho_j^n \right) + \left(\sum_j \rho_j^b \right) \right). \quad (9)$$

The DMR δ is given by

$$\delta = \rho - \sum_j \rho_j \quad (10)$$

and can be written as (by inserting (8) and (9) into (10))

$$\delta = \left(\sum_j \rho_j^n \right) \left(\sum_j \rho_j^b \right) / \left(\left(\sum_j \rho_j^n \right) + \left(\sum_j \rho_j^b \right) \right) - \sum_j \frac{\rho_j^n \rho_j^b}{\rho_j^n + \rho_j^b} \quad (11)$$

which results for three scattering mechanisms $j = 1, 2, 3$ in

$$\delta = \frac{(\rho_1^n + \rho_1^b)(\rho_2^b \rho_3^n - \rho_2^n \rho_3^b)^2 + (\rho_2^n + \rho_2^b)(\rho_3^b \rho_1^n - \rho_3^n \rho_1^b)^2 + (\rho_3^n + \rho_3^b)(\rho_1^b \rho_2^n - \rho_1^n \rho_2^b)^2}{(\rho_3^n + \rho_3^b + \rho_2^n + \rho_2^b + \rho_1^n + \rho_1^b)(\rho_3^n + \rho_3^b)(\rho_2^n + \rho_2^b)(\rho_1^n + \rho_1^b)}$$

$$= \sum_{j=1}^3 (\rho_{j+2}^n + \rho_{j+2}^b)(\rho_j^b \rho_{j+1}^n - \rho_j^n \rho_{j+1}^b)^2 / \left(\prod_{j=1}^3 (\rho_j^n + \rho_j^b) \right) \left(\sum_{j=1}^3 \rho_j^b + \rho_j^n \right) \quad (12)$$

with $\rho_4 \equiv \rho_1$, $\rho_5 \equiv \rho_2$.

With anisotropy parameters A_j [7, 16] for each scattering mechanism j given by

$$A_j = \left(\frac{\tau_n}{\tau_b} \right)_j \quad (13)$$

the DMR δ for three independent scatterers can be written as

$$\delta = \sum_{j=1}^3 \rho_j \rho_{j+1} A_{j+2} b (A_j - A_{j+1})^2 / \sum_{j=1}^3 \rho_j (1 + b A_j)^2 A_{j+1} A_{j+2} \quad (14)$$

with $A_4 \equiv A_1$, $A_5 \equiv A_2$. b is the band-structure term [16]:

$$b = \left(\int_n v dS \right) / \left(\int_b v dS \right) \quad (15)$$

where v is the Fermi velocity.

4. Results

4.1. Measurements

Typical results of the DMR measurements in Cu polycrystals with different impurity contents are shown in figure 1. For characterization of DMR we used the expression

$$D \equiv \frac{rd(T)}{rd(4.2 \text{ K})} - 1. \quad (16)$$

From equation (4) and the above comments, D follows as

$$D = \frac{\rho_d + \delta_{pid} - \delta_{pi}}{\rho_d + \delta_{id}} - 1. \quad (17)$$

Since $\delta_{pi} \ll \delta_{pid}$ and $\delta_{id} \ll \rho_d$ we arrive at

$$D \simeq \frac{\rho_d + \delta_{pid}}{\rho_d} - 1 \quad (18)$$

which in fact reflects the temperature dependence of the DMR $\equiv \delta_{pid}$; D equals zero if no DMR occurs. While at low strains D depends on the impurity content of material (figures 1(a) and 1(b)), this dependence becomes markedly smaller with increasing strain (figure 1(c)). Nevertheless, the temperature variation $D(T)$ remains considerable; rd differs by a factor of about 2 between the lowest and the highest temperatures measured. This is also reflected when comparing the DMRs at selected temperatures, in the full range of dislocation densities in figure 2. The values of $rd(4.2 \text{ K})$ plotted on the x axis are divided by $\rho_i(4.2 \text{ K})$ to give an impression of the contribution of dislocations in relation to the impurities. The $\rho_i(4.2 \text{ K})$ -values were obtained from the RRR and shape factor G of annealed samples (the material-specific values of $\rho_i(4.2 \text{ K})$ are given in table 2). Material Z with the largest impurity content is also included in figure 2. For all materials a tendency of $D(T)$ to decrease with increasing dislocation density can be recognized.

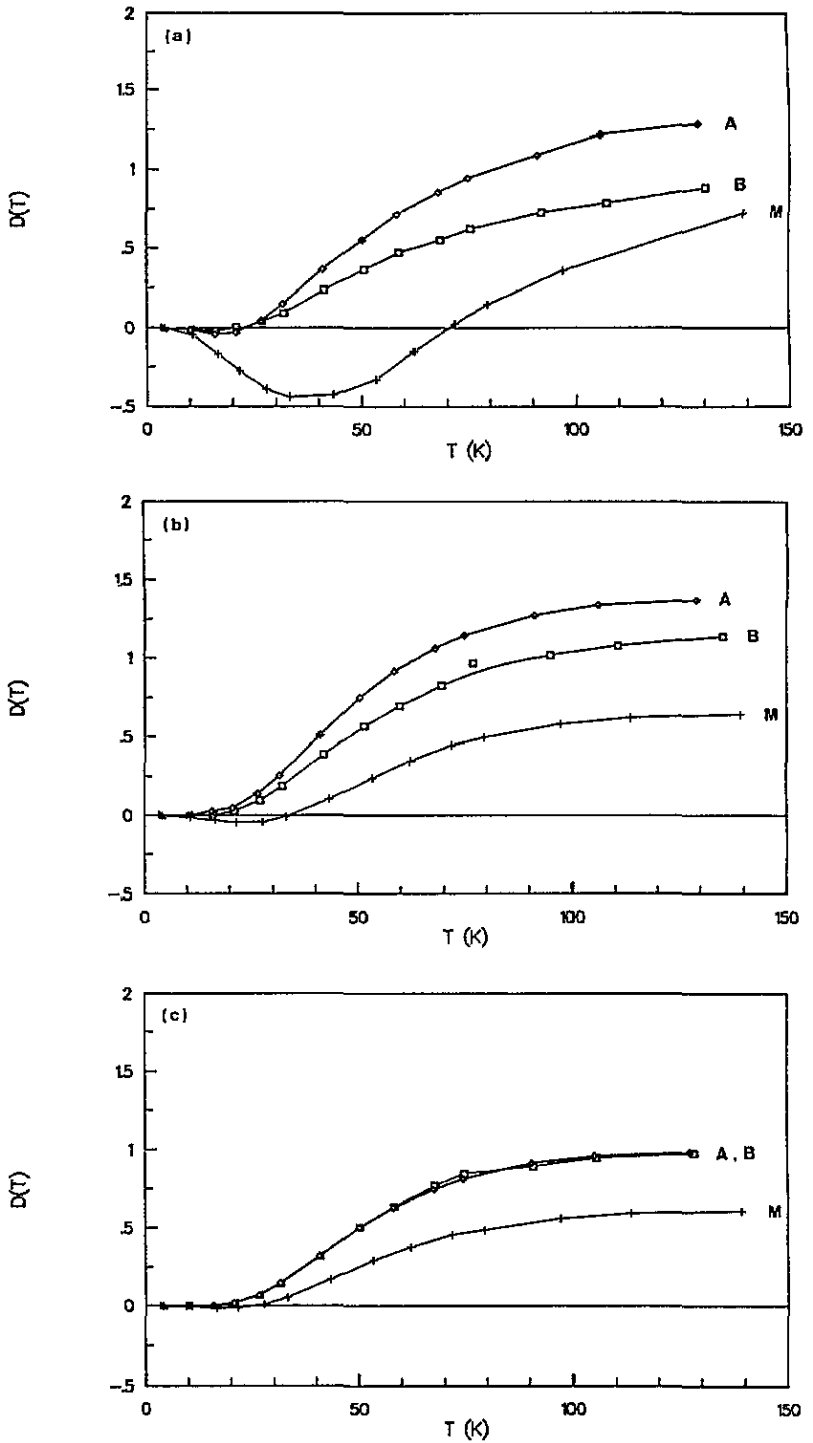


Figure 1. Typical variations in measured normalized dislocation resistivity $D \equiv rd(T)/rd(4.2 \text{ K}) - 1$, as a function of measuring temperature T , after different true strains ϵ , for different materials A (\diamond), B (\square) and M ($+$) (for details, see section 2): (a) $\epsilon = 0.05$; (b) $\epsilon = 0.17$; (c) $\epsilon = 0.5$. The lines are drawn as a guide to the eye.

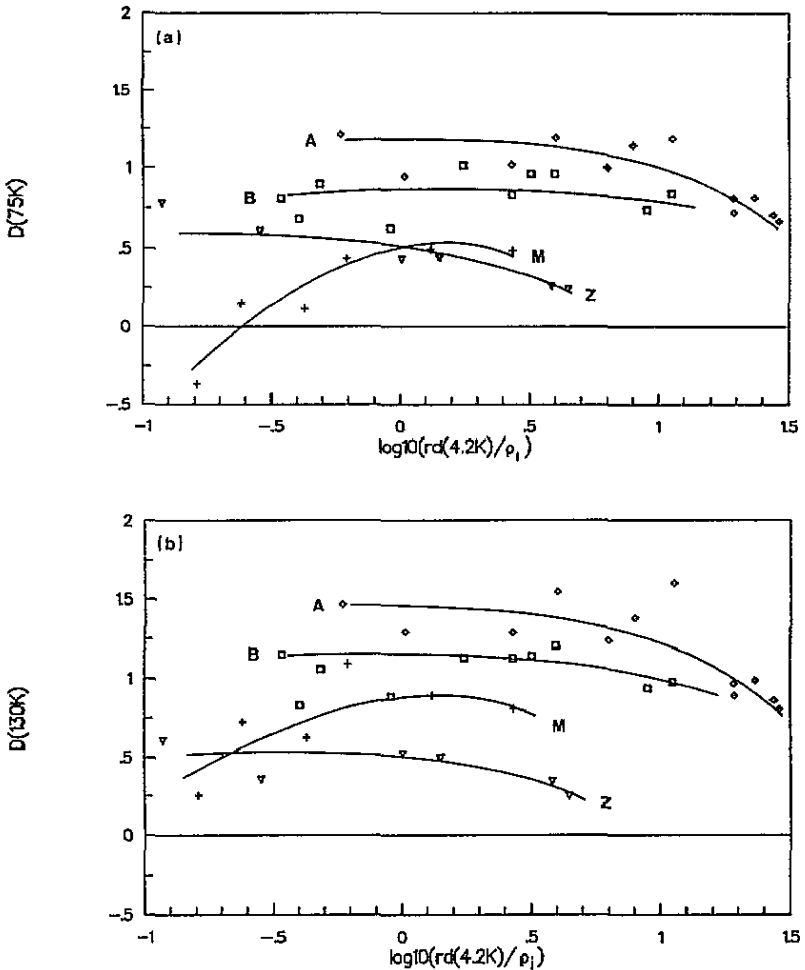


Figure 2. Comparison of experimental values $D(T)$ of different materials A (\diamond), B (\square), M ($+$) and Z (∇) as a function of residual dislocation resistivity $rd(4.2\text{ K})$ after different deformations ε : (a) $T = 75\text{ K}$; (b) $T = 130\text{ K}$.

4.2. Fitting calculations according to the two-group model

We tried to achieve an optimum fit to the experimental $D(T)$ -values by means of equation (14) for the calculation of δ_{pid} . This was done for fixed values of the band-structure term b and the scattering anisotropy A_{im} of impurities, with the dislocation scattering anisotropy A_{dis} as the only free fitted parameter and result of interest. As for the phonon anisotropy involved, it turned out that beyond a certain strain ε_c a good fit of experimental data required us to use *isotropic phonons for all temperatures* (figure 3(b)). For samples with $\varepsilon < \varepsilon_c$, good fits of $D(T)$ were achieved using some *quasi-isotropic phonon characteristics* to model the scattering processes by phonons more isotropic than usual (figure 3(a)). In the quasi-isotropic phonon model, A_{ph} was taken as the arithmetic mean between $A_{\text{ph}} = 1$ (isotropic) and A_{ph} as given by Barnard [17]. For the limit of temperature T approaching 0 K we then found $A_{\text{ph}} \approx 0.6$. Only in some special cases (in the limit of low dislocation densities and additionally $rd/\rho_i \ll 1$) was it necessary to use the *usual phonon anisotropy*

dependence as given by Barnard [17] to obtain a good fit. In figure 4, the three phonon anisotropy characteristics used are demonstrated graphically.

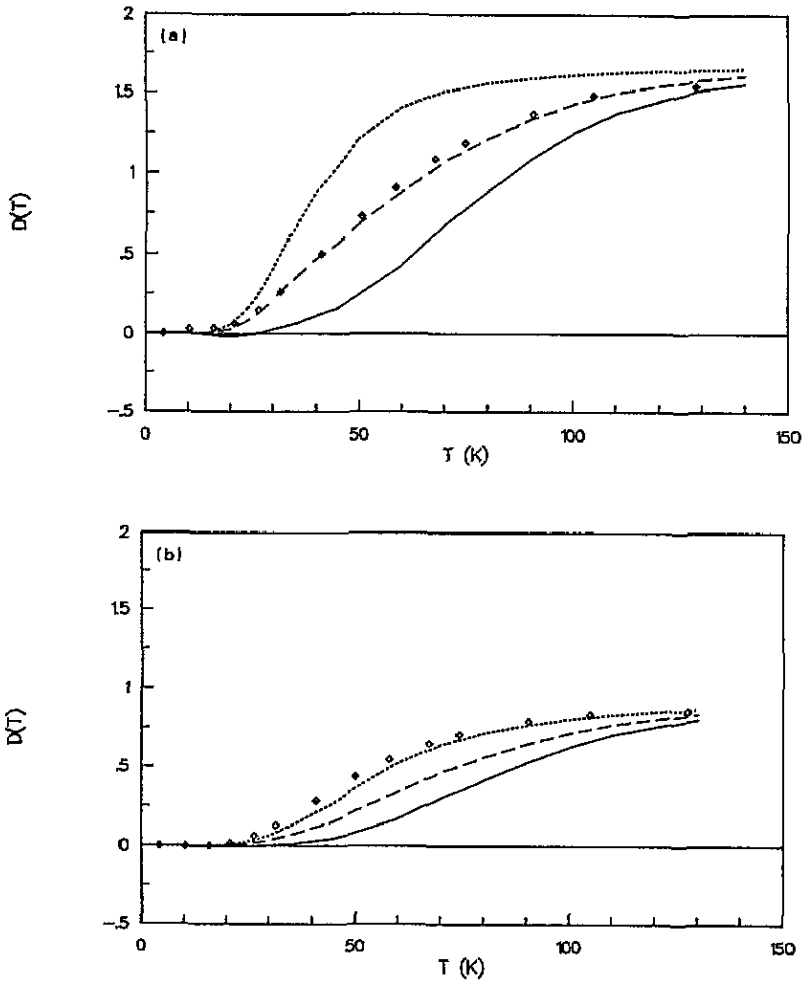


Figure 3. Fits of $D(T)$ curves with the TGM by different characteristics of phonon scattering anisotropy (material A; $\epsilon_c = 0.35$) for an applied strain ϵ of (a) $0.13 < \epsilon_c$ and (b) $0.82 > \epsilon_c$: , isotropic phonons; ----, quasi-isotropic phonons; —, anisotropic phonons (see also figure 4); \diamond , experimental values.

Figure 5 presents the resulting fitted values of dislocation scattering anisotropy A_{dis} for all materials investigated, each for two fixed values of impurity scattering anisotropy (for comments see discussion).

5. Discussion

Before analysing the calculated values for dislocation scattering anisotropy A_{dis} as a function of dislocation density, content and scattering anisotropy of impurities, we have to discuss

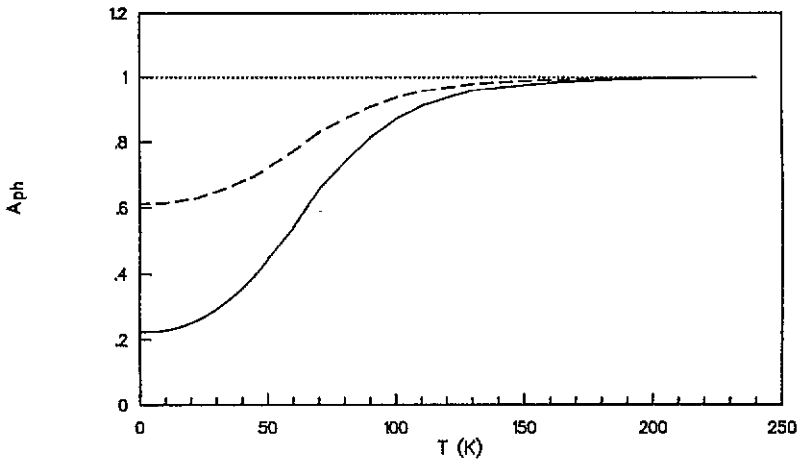


Figure 4. Differences in temperature characteristics of electron-phonon scattering anisotropy A_{ph} . ·····, isotropic phonons; ----, quasi-isotropic phonons; —, anisotropic phonons. (After Barnard [17].)

the increasing temperature dependence of phonon anisotropy for decreasing dislocation density. In our opinion the main reason for this is that the TGM neglects the contribution of *small-angle scattering processes* to the phonon scattering anisotropy. Obviously, these are strongly sensitive to the number of additional defects; this means that the isotropic character of scattering increases as the number of additional defects increases. An analogous example for alloying atoms as defects in various concentrations has been given by Gröger [19] in Hall-effect studies of several Cu-Be alloys. The idea that both the number of dislocations and the number of impurity atoms are important for the phonon anisotropy behaviour is confirmed by the following. Although ε_c is different in the different materials according to their impurity contents, we find that $rd(4.2 \text{ K}, \varepsilon_c) + \rho_i$ is about 11–13 nΩ cm. For example, for material A, $\varepsilon_c \simeq 0.35$ (dislocation density $N \simeq 1.35 \times 10^{15} \text{ m}^{-2}$) and, for material M, $\varepsilon_c \simeq 0.17$ ($N \simeq 5 \times 10^{14} \text{ m}^{-2}$).

For low dislocation densities and $rd(4.2 \text{ K})/\rho_i \ll 1$ the strong temperature dependence of phonon scattering anisotropy as given by Barnard was found only for the impure materials M and Z whereas the quasi-isotropic characteristics appear for the pure materials A and B. This change in the temperature dependence of A_{ph} below 80 K with dislocation density and impurity content makes it clear that the classical TGM is rather overstrained in giving a full characterization of electron-phonon scattering processes because of the relaxation time approximation involved.

Considering now the calculated values A_{dis} for different dislocation densities in materials with different impurity contents (figure 5), the general behaviour is a slow decrease in A_{dis} with decreasing dislocation resistivity. Below a certain dislocation resistivity ($rd(4.2 \text{ K}) \leq (2-3)\rho_i$), however, the value of A_{dis} depends sensitively on the value A_{im} (impurity scattering anisotropy) chosen, i.e. it either strongly decreases (for $A_{im} = 1$; isotropic) or increases (for $A_{im} < 1$; anisotropic) in approaching the smallest dislocation resistivities measured (figure 5). Although DHVA measurements yield relaxation times which are not weighted correctly for the case of electrical resistivity, it can be seen from such results on dilute alloys [18] that, in general, A_{im} does not equal 1. Thus it is preferable to measure the anisotropies A_{im}^* by low-field Hall-effect (LFHE) experiments on undeformed

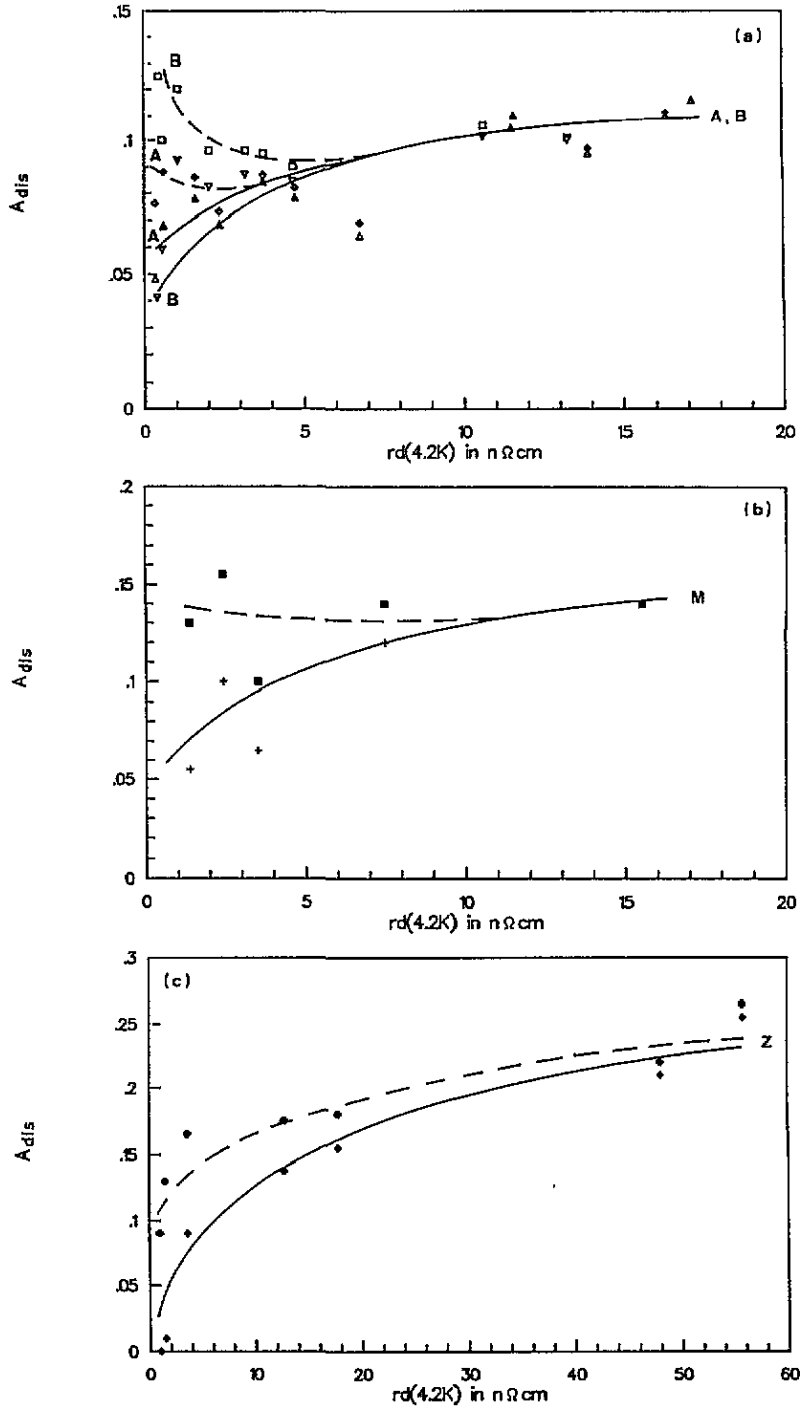


Figure 5. Anisotropies A_{dis} of electron-dislocation scattering, from fitting calculations with $b = 0.219$; calculations were done with $A_{im} = 1$ (—) as well as the experimental value A_{im}^* from the Hall effect [11] (---). The lines are drawn as guide to the eye: (a) material A (\diamond , for $A_{im}^* = 0.256$; Δ , for $A_{im} = 1$) and material B (\square , for $A_{im}^* = 0.161$; ∇ , for $A_{im} = 1$); (b) material M (\boxplus , for $A_{im}^* = 0.224$; $+$, for $A_{im} = 1$); (c) Material Z ($\#$, for $A_{im}^* = 0.313$; ϕ , for $A_{im} = 1$).

samples with minimum dislocation density [11]. These values were used for the fitting calculations, yielding the graphs of A_{dis} in figure 5. The anisotropy parameters A_{im}^* of impurity scattering are surprisingly small and very different from unity. As already argued in [11] for polycrystalline Cu samples in comparison with single-crystal samples, these values are probably dominated by the scattering (anisotropy) of grain boundaries which are thought to consist of certain arrays of dislocations and therefore have similar scattering anisotropies. After the measured values A_{im}^* are included in the calculations, they now exhibit some increase in A_{dis} when proceeding to lowest dislocation resistivities, except for material Z with the highest impurity content. Thus a general feature of the results for A_{dis} in the low dislocation density range is roughly constant or slightly increasing characteristics on decreasing the dislocation resistivity, with an absolute value of about $A_{\text{dis}} = 0.1$ † for the 'pure' materials A and B (having impurity resistivities of less than $1.5 \text{ n}\Omega \text{ cm}$) and only a slightly higher value of $A_{\text{dis}} \simeq 0.14$ in material M ($\rho_i = 5.68 \text{ n}\Omega \text{ cm}$).

In great contrast with that, material Z with an impurity resistivity of $12 \text{ n}\Omega \text{ cm}$ shows quite a different behaviour of A_{dis} as a function of (low) dislocation resistivity. Regardless of the choice of A_{im} , all values A_{dis} decrease markedly with decreasing dislocation content. Considering the impurity contents and impurity types contained in this material (see section 2), we suggest the following explanation. Besides the high impurity content itself, material Z contains phosphorus which is known to be an effective constituent in strengthening Cu. In contrast with the (fewer) constituents in materials A, B and M, the phosphorus atoms act as strong barriers to the moving dislocations in deformation, favouring the formation of stress-(strain)-field-intensive dislocation arrays. If we recall the findings of Barnard *et al* [21] from a combined resistivity-TEM investigation of deformed Ag, arrays of pinned dislocations with their considerable strain fields are suspected to provide particularly high scattering anisotropies A_{dis} of the order of 0.01, in contrast with low-energy dislocation arrays exhibiting, as mentioned, an A_{dis} -value of about 0.1.

From equation (14) it is obvious that the resulting DMRs become larger with increasing difference between the anisotropy parameters $A_{1,2,3}$ involved and/or with increasing resistivities $\rho_{1,2,3}$. From this point it is quite clear that, at temperatures $T > 100 \text{ K}$, a higher number of impurities only leads to enhanced DMRs if the scattering anisotropy A_{im} is highly different from the anisotropy of the combined dislocation-phonon system.

At 4.2 K , when no phonons are present, DMRs occur only as a result of impurities and dislocations. Larger DMRs are expected in impure materials particularly with dislocation pinning obstacles (e.g. material Z) and on proceeding to lower dislocation densities. This yields larger corrections if one wants to measure dislocation densities exactly using the electrical resistivity. These corrections are considerable (a factor of about 2 in the region $\varepsilon \leq 0.2$ for the pure materials A and B and a factor of up to 10 for material Z) where A_{im} is about 1, which means that the values of A_{im} and A_{dis} are highly different. However, as the values of A_{im} measured by the LFHE are closer to A_{dis} than to unity, the correction of the measured quantity $rd(4.2 \text{ K})$ to the 'true' dislocation resistivity ρ_d decreases rather drastically.

If we take the measured values of A_{im}^* as the anisotropy parameters for impurity scattering, the correction in dislocation density N does not exceed a few per cent. An estimate of the maximum correction for the sample materials used can be obtained from the values of $A_{\text{im}} \simeq 0.6$ resulting from LFHE measurements on copper single crystals [11],

† The band-structure term b slightly influences the absolute value of A_{dis} but has no effect on the general tendencies presented here. Choosing $b = 0.219$ minimizes the difference between the averages of resistivity and Hall effect [11, 20], and the corresponding neck angle of 18.7° is in good accordance with the usual models of the copper Fermi surface. Therefore, this b -value accounts for the most reliable A_{dis} -values calculated here.

which corresponds to the limit of dominant impurity scattering in polycrystals. In this case of vanishing grain boundary influences the corrections in the region $\varepsilon < 0.2$ are a factor of about 1.7 for the most impure material Z and smaller than 1.25 for all the others. For $\varepsilon > 0.2$ the values of $rd(4.2\text{ K})$ and ρ_d differ by less than 10%. In all cases (as shown in figure 6) the difference between measured and corrected dislocation densities is largest with the most impure material. It is clear that the correction to ρ_d at temperatures $T > 4.2\text{ K}$, e.g. at $T = 77\text{ K}$ with measurements in liquid nitrogen, is much more complicated because of the uncertainty in A_{ph} . Without any correction the accuracy of determination of dislocation densities at $T = 77\text{ K}$ cannot be in general higher than a factor of 3–5 even in very pure materials.

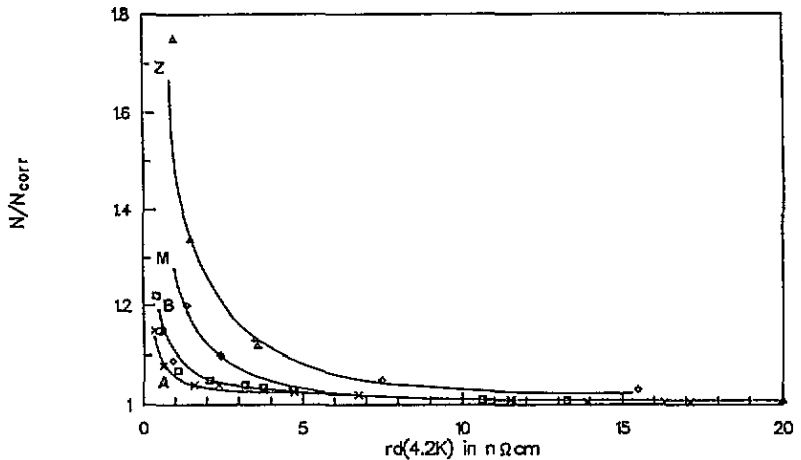


Figure 6. Factor N/N_{corr} to correct the dislocation density because of contributions from DMRS (calculated for $A_{im} = 0.6$): symbols are as figure 2.

A final test of the reliability of resistivity measurement as a method for dislocation density can be achieved by comparing the results with those of TEM investigations [22, 23]. At $\varepsilon = 0.1$ and $\varepsilon = 0.05$ we find that $N = (2-2.6) \times 10^{10}\text{ cm}^{-2}$ and $N = (1-1.3) \times 10^{10}\text{ cm}^{-2}$, respectively, whereas TEM yields $N = 3 \times 10^{10}\text{ cm}^{-2}$ and $N = 1.3 \times 10^{10}\text{ cm}^{-2}$. So we can conclude that resistivity leads, to a high degree, to the same results on dislocation density as the analyses by TEM do. Also, the measurement errors are of the same order of magnitude: $\Delta N \simeq 5 \times 10^8\text{ cm}^{-2}$. However, it is well known that dislocation density measurements by TEM are restricted to N not larger than some 10^{10} cm^{-2} which is clearly not the case with resistivity measurements. Because of numerous findings that the strain field of dislocation (arrays) plays only a negligible role in electron-dislocation scattering compared with the dislocation core [3, 8, 9], the resistivity method in principle can be applied with no serious limit up to the highest dislocation densities, except when the distance between dislocations becomes smaller than $2b$ (b is the Burgers vector). This leads to an overlap of dislocation cores and thus to an apparent decrease in specific dislocation resistivity in spite of an actual increase in the dislocation density [6]. Such small dislocation distances can of course only be reached locally, i.e. at small- and large-angle grain boundaries. Here, Nakamichi [6] indeed measured a decrease in specific dislocation resistivity with a high-resolution SQUID method. Thus, the above dislocation distance of $2b$ can be regarded as some upper limit where the resistivity method correctly operates;

it corresponds to a dislocation density of about $4 \times 10^{14} \text{ cm}^{-2}$, which means that this method exceeds the TEM technique by at least four orders of magnitude. Moreover, besides the advantages of a comparably simple experimental method, it yields an average global dislocation density for a sample volume which is in most cases much more representative for comparisons with other physical properties (strength, neutron diffraction, stored energy, etc) than the microvolumes studied by TEM. The TEM method also sometimes suffers from influences of the preparation method and is mostly restricted to room-temperature observations.

6. Conclusions

(1) In the framework of the TGM and an exact equation for three independent scatterers, various DMRS in the presence of dislocations can be calculated, i.e. for different dislocation densities, impurity contents and temperatures.

(2) Satisfactory fits of DMRS caused by dislocations require isotropic phonon scattering at high dislocation and/or impurity contents, and increasing anisotropy of phonon scattering when these defect densities are smaller. This specific behaviour is attributed to the variable contribution of small-angle scattering processes to phonon scattering anisotropy which is not taken into account in the classical TGM.

(3) In high-purity Cu and in internally oxidized Cu, the anisotropy A_{dis} of electron-dislocation scattering is roughly independent of both dislocation and impurity contents, giving about $A_{\text{dis}} \simeq 0.1$. In Cu with a typical purity of 99.99% and special constituents which favour the formation of high-internal-strain-field arrays of dislocations, A_{dis} decreases drastically in the low-density range by about one order of magnitude, reaching $A_{\text{dis}} \simeq 0.01$.

(4) In the case of special types of strain field formation due to some sort of impurities, the DMR increases strongly as well as the corresponding correction which is necessary to obtain the true value of dislocation density. Therefore, using the resistivity for dislocation density measurement, the resolvable dislocation density is about twice that in the pure material.

(5) Compared with usual TEM techniques, the resistivity method allows at least the same accuracy at low dislocation densities, provided that the impurity content does not exceed 99.99%. However, proceeding to high dislocation densities, the resistivity method markedly exceeds the limits of TEM (a few 10^{10} cm^{-2}) and even reaches 10^{14} cm^{-2} where the overlap of dislocation cores occurs. Furthermore, the resistivity method is a relatively simple method and offers all advantages of a non-local defect density measurement.

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