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On the martensitic transformation temperature and its stress dependence in Cu–Zn and Cu–Zn–Al single crystals

R Romero†‡ and M Ahlers†

† Centro Atómico Bariloche, 8400 SC de Bariloche, Rio Negro, Argentina

‡ Universidad Nacional del Centro de la Provincia de Buenos Aires, Pinto 399, 7000 Tandil, Argentina

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Abstract. The critical stress to induce martensite in Cu–Zn and Cu–Zn–Al single crystals has been determined down to 80 K. It has been found that the stress increases linearly with increasing temperature. From this result it is deduced that the higher vibrational entropy of the β -phase compared with the martensite is due solely to the soft transverse $[\xi\xi 0]$ phonon modes, the other modes remaining largely unaffected by the transformation. This result is consistent with the predictions of Zener on the stability of β -phases in the noble-metal alloys.

The linearity in the temperature dependence of the critical stress has been used to obtain transformation temperatures by a linear extrapolation to zero stress. The results for alloys with Zn concentrations between 40 and 43 at.% Zn can be expressed as $M_s(\text{K}) = 2686 - 64C_{\text{Zn}}$. This dependence is different from what has been reported in the literature before.

Finally, the influences of previous plastic deformation in the β -phase and of a stress-induced transformation of the martensite to the FCT phase are considered.

1. Introduction

The entropy difference between the high-temperature β -phase and the martensite with a 9R or 18R structure is due to a change in the vibrational properties of the lattices, since the configurational part does not change during the diffusionless martensitic transformation. The vibrational properties are often described in terms of a Debye temperature θ_D which is around room temperature for β -phase Cu–Zn (Veal and Rayne 1962, Gillette and Granada 1984). If the β -phase and the martensite differ in their Debye temperatures, this difference can be determined from the measured entropy difference; it has been found to be $\theta_D(\text{M}) - \theta_D(\beta) = 14 \pm 1 \text{ K}$ (Ahlers 1986). However, from the Debye model, it is expected that at temperatures below θ_D the entropy difference becomes temperature dependent, and this influence should manifest itself also in the temperature dependence of the critical stress τ_M which is necessary to induce martensite at temperatures T above the temperature M_s for spontaneous martensite. It implies also that the composition dependence of M_s below θ_D is in part due to a vibrational contribution, an aspect which is important for the evaluation of the relative stability of the martensitic phases. For this reason the temperature dependence of τ_M has been determined down to liquid-air temperature. The results, which are reported in this

paper, show that the Debye model is not adequate for describing the entropy difference between β and the 9R or 18R martensite in Cu–Zn and Cu–Zn–Al.

The analysis of the temperature dependence of τ_M involves also the question of the extent to which modifications can occur, by plastic (micro)flow due to dislocation movement in the β -phase matrix or in the martensite plates if the stress is sufficiently high.

It has been shown that martensitic 9R Cu–Zn single crystals start to deform at a critical stress which is strongly composition dependent, leading to face-centred martensite (Ahlers 1986), and there is evidence that above this stress the temperature dependence of the critical stress $\tau_M(T)$ changes (Delaey *et al* 1979). In addition, the transformation stress can approach the critical stress for plastic flow in the β -phase, and the microplasticity or macroplasticity of the β -phase interacts with the martensite formation (Romero and Ahlers 1989). Whether these changes are also present in the single crystals has now been analysed and is reported in this paper.

Knowing the temperature dependence of the critical stress to induce martensite, we can then infer M_s for spontaneous martensite, once the critical stress at a given temperature has been determined. Using published data for τ_M at liquid-air temperature (Romero and Ahlers 1988), in addition to the measured data, M_s has been redetermined for Cu–Zn with between 40 and 43 at. % Zn. It will be seen that our results differ from those of other researchers (Pops and Massalski 1964, Pops and Ridley 1970).

2. Experimental procedures

High-purity (all 99.999%) Cu, Zn and Al were melted in sealed quartz tubes under an Ar atmosphere in a resistance furnace. The melt was vigorously shaken in order to obtain a homogeneous solution. Subsequently, it was quenched into water at room temperature. Pieces (of mass about 20 g) were then cut from the ingot and were re-encapsulated in quartz tubes to grow the single crystals by the Bridgman method.

In order to grow the binary Cu–Zn single crystals, care was taken to ensure that the liquid remained only slightly above the melting point. In this way, it was possible to grow pore-free single crystals. Since the β -Cu–Zn alloys studied here decompose at a rather high temperature, the encapsulated sample was kept in contact with a piece of steel at 1100 K, while being taken out of the furnace, and then was rapidly quenched into water at room temperature by breaking the quartz tube. The Zn loss during the single-crystal growth and the quenching procedure was found to be negligible, and so no corrections to the original compositions were necessary. Attempts were made to anneal the quenched single crystals afterwards by heating them to a temperature within the range of the β -phase stability. This procedure had to be abandoned, since it generally resulted in the formation of polycrystals, and in an abnormally large scatter in transformation temperatures. For this reason the quenched Cu–Zn single crystals were not heat treated further but were kept not less than 10 days at room temperature prior to their transformation; this time was considered sufficient for any quenched-in defects to be annealed out. The Cu–Zn–Al alloys which were used had a β -phase stability range which extended to lower temperatures, and therefore the samples could be air cooled.

The Cu–Zn and Cu–Zn–Al single crystals were then spark machined to cylindrical tensile samples of about 3 mm diameter and 10 mm length, with thicker ends of 6 mm diameter and 5 mm length, in order to fit them into the grips of the deformation machine.

The damaged surface layer was eliminated by an electropolish in a solution of 13% HNO_3 in CH_3OH , and the sample surface was then studied by optical microscopy.

The samples were deformed in tension in an Instron 1123 deformation machine. A cryostat was designed to keep the sample temperature constant in the temperature range from 80 K to room temperature. The sample temperature was measured by soldering chromel-alumel thermocouples to both ends of the sample. The difference between both sample ends was always less than 1 K.

3. Experimental results

In figure 1 the critical resolved transformation stress is shown as a function of temperature for four Cu-Zn single crystals with various Zn contents: 40 at.% Zn (sample 1),

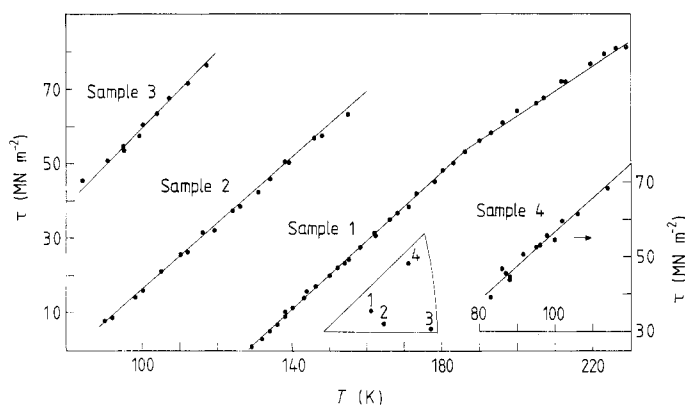


Figure 1. The critical resolved transformation stress as a function of temperature for Cu-40 at.% Zn (sample 1), Cu-40.5 at.% Zn (sample 2), Cu-41 at.% Zn (sample 3) and Cu-41.5 at.% Zn (sample 4). The orientations of the tensile axis are given in the unit triangle with the numbers indicating the samples.

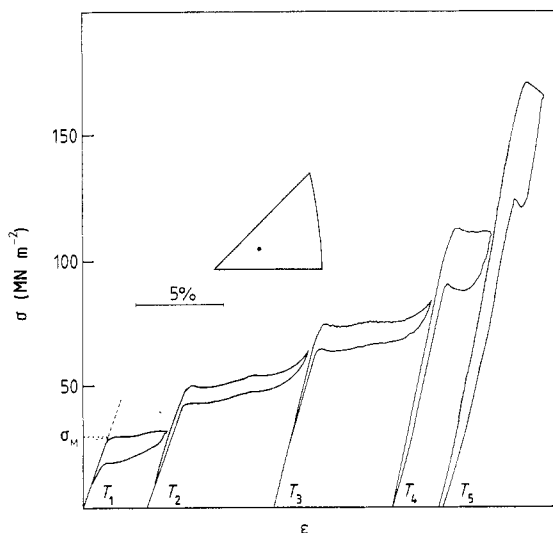


Figure 2. Stress-strain curves for loading and unloading of sample 1 (figure 1) at consecutively higher temperatures $T_1 = 146$ K, $T_2 = 154$ K, $T_3 = 166$ K, $T_4 = 186$ K and $T_5 = 227$ K.

40.5 at.% Zn (sample 2), 41.0 at.% Zn (sample 3) and 41.5 at.% Zn (sample 4). The orientations of the crystals are given in the unit stereographic triangle. Some stress-strain curves for sample 1 are plotted in figure 2. Since, on unloading, the original β -phase is restored, the martensite could be induced repeatedly at different temperatures T without any additional intermediate treatments. In order to calculate the resolved stress, it is assumed that the most favourable martensite variant is induced—a reasonable assumption (Arneodo and Ahlers 1974). From figure 1 it can be seen that the stress is a linear function of temperature T down to the lowest measured T . Only sample 1 shows a deviation at a higher temperature $T > 186$ K. Figure 2 indicates that below 186 K the hysteresis of sample 1 is small and changes little with temperature, whereas at 186 K it starts to increase rapidly.

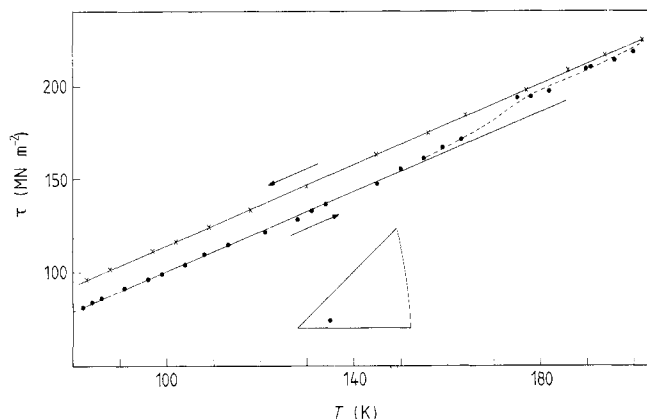


Figure 3. The critical resolved transformation stress as a function of temperature for a Cu-28.09 at.% Zn-9.95 at.% Al single crystal deformed in tension. Transformation cycles started at 82 K, were repeated at successively higher temperatures up to 202 K (●) and then were performed at decreasing temperatures (×) (see arrows).

In order to determine whether the linear temperature dependence of the critical resolved transformation stress is a general phenomenon, a Cu-28.09 at.% Zn-9.95 at.% Al single crystal was also tested and also used to analyse the influence of prior plastic deformation in the β -phase on the temperature dependence of the stress. The results are shown in figure 3. The transformation cycles were started at 80 K and then repeated at successively higher temperatures for this crystal. It can be seen that the stress does indeed depend linearly on temperature for this ternary alloy up to a temperature of 155 K. Between 155 and 170 K a slight increase compared with the linearly extrapolated curve can be seen. However, no permanent deformation is retained on unloading, according to the measured stress-strain curves. If this change in slope is real (which is difficult to decide because of its smallness and the errors involved in determining it from the load-elongation curve), then it must be related to microflow. From 175 K upwards, plastic flow in the β -phase precedes the martensitic transformation, its amount depending on the temperature and the previous deformation, thus leading to the shift in τ observed in figure 3. On reaching 202 K, the temperature was again decreased and the transformation cycles measured. The crystal remained in the plastically deformed state that it had reached at 202 K. It can be seen from figure 3 that the critical stress decreases linearly with decreasing temperature in the whole temperature range, with the same slope as for the undeformed crystal but displaced to lower temperatures. (For the calculation of the resolved stress the change in the crystal orientation due to the previous plastic deformation has been taken into account.)

The hysteresis between the stress to induce the martensite on loading and that on unloading at small transformation strains increased to a $\Delta\tau$ -value of between 3.1 and 4.3 MN m⁻² for the deformed crystal at temperatures between 83 and 109 K, compared with a $\Delta\tau$ -value of between 1.1 and 2.2 MN m⁻² for the undeformed crystal. The hysteresis increase is smaller than the shift of the $\tau(T)$ curve by deformation, which implies that plastic deformation in the β -phase stabilises it with respect to the martensite (see Romero and Ahlers 1989).

The slope $d\tau/dT$ has been measured for several Cu-Zn single crystals, among them the samples used for figure 1. The results are plotted in figure 4. No systematic composition dependence is found, nor could an influence of the crystal orientation (figure 1) be established. The average value obtained from figure 4 is

$$d\tau/dT = 0.89 \pm 0.07 \text{ MN m}^{-2} \text{ K}^{-1}.$$

This value agrees within the experimental scatter with that published by Arneodo and Ahlers (1974) of $0.96 \pm 0.04 \text{ MN m}^{-2} \text{ K}^{-1}$ for Cu-Zn from 39.2 to 40.4 at.% Zn. The slope for the ternary alloy shown in figure 3 is slightly higher; it is $d\tau/dT = 1.08 \text{ MN m}^{-2} \text{ K}^{-1}$, similar to data reported by Saburi *et al* (1982) and Sato *et al* (1981).

The observation that down to 80 K the critical stress to induce martensite depends linearly on temperature makes it possible to extrapolate measured transformation stresses to zero stress in order to obtain an extrapolated M_s temperature. Above 80 K this M_s should agree with the measured value for spontaneously formed martensite, but for M_s below 80 K this is not necessarily the case. In figure 5 are plotted in addition to the measured M_s for Cu-40 at.% Zn, the M_s -values which were obtained by extrapolating the $\tau(T)$ dependence to zero stress (indicated by crosses in figure 5). Since $d\tau/dT$ is independent (at least within experimental scatter) of composition, from the measured

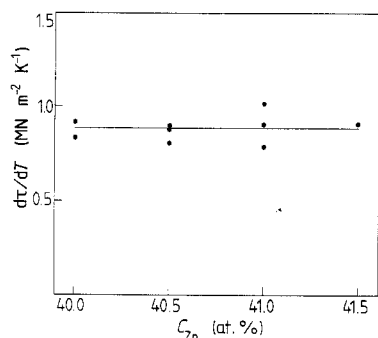


Figure 4. The temperature dependence of the critical resolved transformation stress $d\tau/dT$ as a function of Zn concentration for Cu-Zn single crystals of different compositions.

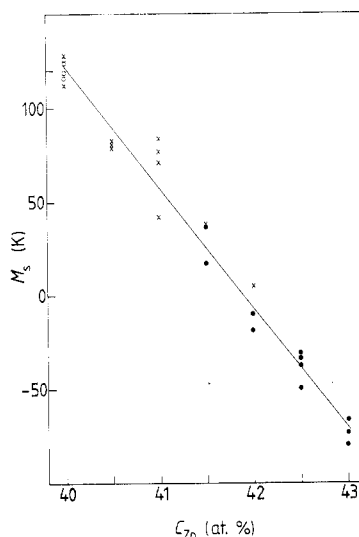


Figure 5. Measured and extrapolated transformation temperatures for Cu-Zn alloys: ○, M_s obtained on cooling without stress (for Cu-40 at.% Zn only); ×, M_s obtained from an extrapolation of the $\tau(T)$ dependence; ●, M_s obtained from stress measurements at 80 K.

stresses to induce martensite at 80 K (Romero and Ahlers 1988) an extrapolated M_s has also been obtained by using a $d\tau/dT = 0.89 \text{ MN m}^{-2} \text{ K}^{-1}$ (full circles in figure 5). All these data can be reasonably well presented by the relationship

$$M_s \text{ (K)} = 2686 - 64C_{Zn}$$

where C_{Zn} is expressed in atomic per cent and the slope dM_s/dC_{Zn} is measured with an uncertainty of $64 \pm 2 \text{ K at. \%}^{-1}$.

4. Discussion

The most surprising result of the present experiments is the linear temperature dependence of the critical stress to induce martensite down to liquid-air temperature, which would not be expected if the Debye model for the description of the vibrational entropy holds. Before this aspect is discussed, it is necessary to show that the linearity is not the artificial superposition of a non-linear term compatible with a Debye model and contributions from plasticity in the martensite or in the β -phase.

In a single crystal of 9R martensite, a 3R face-centred structure is induced at a critical stress τ_B . This stress has been measured (Arneodo and Ahlers 1974). For samples 1–4 in figure 1 this stress is reached at τ_B -values of 57 MN m^{-2} , 88 MN m^{-2} , 1366 MN m^{-2} and 193 MN m^{-2} , respectively. For sample 1, one indeed finds the change in slope at this value whereas, for the other samples, τ_B is higher, and the linearity extends to higher stresses. It is concluded that the transformation from 9R to 3R does not modify the linear $\tau_M(T)$ dependence, provided that τ_M remains below τ_B . Measurements of $d\tau/dT$ above this critical stress should not be used, however, to determine ΔS . This range can be recognised easily from the increased hysteresis in the stress–strain curves (see figure 2). It is unlikely that the change in slope is due to a modification in the Clausius–Clapeyron equation, because the transformation mode changes, as has been shown in a detailed study by Sato *et al* (1981) of Cu–Zn–Al.

It is known that the presence of dislocations due to plastic deformation in the β -phase increases the critical stress to induce martensite (Romero and Ahlers 1988b). Below the yield stress, microflow can occur, in Cu–Zn it has been observed to set in at 0.6–0.7 of the critical shear stress for plastic deformation (Ito and Nakayama 1986). The deviation from linearity on loading in figure 3 below the yield stress for macroscopic plastic deformation probably is due to microyield.

This microyield may lead to a curvature of the $\tau(T)$ relationship and, if averaged, may give a higher $d\tau/dT$ than justified. In order to avoid this error, it is more convenient to increase the stresses during decreasing the temperature since, as seen in figure 3, the relationship remains linear, and the slope is not modified. This result is not surprising since dislocations are not expected to change the vibrational modes in a significant way, at least not for the low dislocation densities which have been introduced in the present conditions.

The linearity of the temperature dependence of τ_M therefore is real, and as will be shown more important conclusions on the entropy change during the transformation can be drawn. This result is incompatible with the assumption that both phases can be described by a Debye model with slightly different Debye temperatures, since this implies strong deviations from linearity above 80 K (Ahlers 1986, Romero and Ahlers 1988). In figure 5 is drawn the expected line (full line A), using the high-temperature measurements to adjust the difference in Debye temperatures. It can be seen that the

discrepancy with the measured values lies far outside the experimental scatter. In fact, a similar failure had also been noted by Abbé *et al* (1984) who found that the specific heat experiments below 40 K did not support a Debye model for β -phase Cu-Zn-Al alloys. By adding an Einstein term with a temperature of 78 K, they could adjust their results well. They used the Einstein approximation to describe the transverse low-energy vibration mode, corresponding to the $\{110\}\langle 110\rangle$ shear wave, which they had analysed experimentally by neutron spectroscopy.

Our results can be explained if the entropy difference between both phases, β and martensite, is due to low-energy vibration modes in the β -phase which are fully excited at 80 K, leaving unaltered during the phase transformation all other modes, which are described by the Debye spectrum during the phase transformation.

For a quantitative description, let us assume that the specific heat is the sum of an Einstein (indicated by a subscript E) and a Debye (indicated by a subscript D) term in both phases:

$$C_p = (1 - p)f_D(T, \theta_D) + pf_E(T, \theta_E)$$

where $p < 1$ is the fraction described by the Einstein term. According to the Debye model

$$f_D(T, \theta_D) = 3R\left(\frac{T}{\theta_D}\right)^3 \int_0^{\theta/T} x^4 \exp x (\exp x - 1)^{-2} dx$$

and, correspondingly for the Einstein contribution,

$$f_E(T, \theta_E) = 3R(\theta_E/T)^2 \exp(\theta_E/T)[\exp(\theta_E/T) - 1]^{-2}$$

(R is the gas constant). The temperature dependence of the critical stress is given by the Clausius-Clapeyron equation

$$d\tau/dT = \Delta S/\gamma V$$

where

$$\Delta S = S_\beta - S_M = \int_0^T \frac{C_p(\beta) - C_p(M)}{T} dT$$

is the entropy difference per mole; γ is the transformation strain ($\gamma = 0.17$, independent of composition and temperature; if a dependence exists, it is too small to change the results significantly). V is the molar volume ($V = 7.68 \text{ cm}^3 \text{ mol}^{-1}$). After integration, we obtain

$$\tau = \tau_0 + (3RTp/\gamma V) \ln\left[\frac{1 - \exp[-\theta_E(M)/T]}{1 - \exp[-\theta_E(\beta)/T]}\right].$$

For high temperatures this simplifies to

$$\tau = \tau_0 + (3RT/\gamma V)p \ln[\theta_E(M)/\theta_E(\beta)].$$

The same relationship holds also for the Debye model ($p = 1$, and θ_E replaced by θ_D). From $d\tau/dT$ a value for $p \ln[\theta_E(M)/\theta_E(\beta)]$ of $(4.7 \pm 0.4) \times 10^{-2}$ is obtained.

Additional arguments are now required to deduce each of the three parameters in the product $p \ln[\theta_E(M)/\theta_E(\beta)]$. $\theta_E(\beta)$ can be estimated from the phonon branch for the $[\xi\xi\xi 0]$ transverse mode measured by Guenin *et al* (1982). For Cu-Zn-Al alloys of two widely differing compositions, they find close to the martensitic transformation a frequency ν_E of 1.2×10^{12} Hz near the Brillouin zone boundary. The identification of

this value with the Einstein frequency leads to an Einstein temperature $\theta_E(\beta)$ of 60 K. The fraction p of modes which can be considered as belonging to this group is more difficult to estimate. If we take all states whose wavevector does not differ by more than 10° from the $[\xi\xi\xi 0]$ direction in the reciprocal lattice and take into account that there are 12 equivalent $[\xi\xi\xi 0]$ directions in the reciprocal lattice, and three polarisation branches, we can estimate p to be 0.03 (it does not seem to be very realistic to describe the transverse vibration modes near $[\xi\xi\xi 0]$ by an Einstein term but, since we are only interested in the behaviour at $T > \theta_E$, this description is thought to be reasonable). The third quantity, $\theta_E(M)$, is then $\theta_E(M) = 287$ K, of the expected order of magnitude for the Debye temperature of the close-packed phase. This indicates that the modes in the martensite, which belong to the soft branch of the β -phase, should be better described by a Debye term. For this reason, a refinement has been made, describing the martensite solely by the Debye model, with a Debye temperature $\theta_D(M)$ of 265 K, equal to that of the β -phase. Using $\theta_E(\beta) = 60$ K, a fraction $p = 0.04$ is deduced, close to that estimated above.

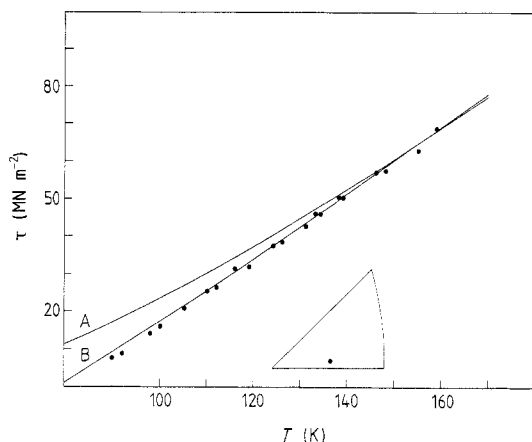


Figure 6. Critical resolved transformation stress as a function of temperature for Cu-40.5 at.% Zn (sample 2 of figure 1): ●, experimental data; curve A, predictions from the Debye model; curve B, predictions if an Einstein term for the β -phase is included.

The predicted $\tau(T)$ curve is also shown in figure 6. Indeed the temperature dependence of the stress remains linear down to 80 K, at least within the experimental uncertainty. A small temperature dependence could be expected if $\theta_E(\beta)$ remains constant. However, it has been shown that C' increases with increasing temperature (Guenin *et al* 1977) and, since the frequency at the Brillouin zone boundary is proportional to C' , a slight increase in $\theta_E(\beta)$ with increasing temperature is expected which leads to a decrease in $d\tau/dT$ and almost compensates for the calculated increase.

From the present discussion, we thus conclude that the higher vibrational entropy of the β -phase compared with the close-packed martensites is solely due to the soft transverse $[\xi\xi\xi 0]$ vibration modes, in this way confirming experimentally the hypothesis presented by Zener (1948) on the stability of the BCC phases in the β -brasses.

The most important quantity which enters into the entropy difference is the phonon frequency ν_E at the Brillouin zone boundary which is proportional to C' , since the dispersion curve can be well approximated by a sine dependence on the wavevector k . The observation by Guenin *et al* (1982) that ν_E is nearly the same for two very different Cu-Zn-Al concentrations (Cu-19.3 at.% Zn-13 at.% Al and Cu-7.2 at.% Zn-23.1 at.% Al) is consistent with the small composition dependence of ΔS deduced from the measured $d\tau/dT$ (Ahlers 1986). By measuring the elastic constant C' , and the Debye

temperature for martensite or for β' which, according to our discussion, are the same, it should be possible to obtain the entropy difference between the close-packed and the BCC structures for other alloys.

The observation of a linear temperature dependence of the critical stress to induce martensite makes it possible to deduce M_s for spontaneous martensite, if a stress at a temperature above liquid-air temperature is available. In an earlier publication (Romero and Ahlers 1988) these values have been reported for Cu–Zn at liquid-air temperature. They have been used for the determination of M_s , together with those obtained by a direct measurement during cooling, provided that M_s is above 80 K, or by extrapolating the critical stress for single variant formation, measured as a function of temperature. All these data give a consistent result for the composition dependence of M_s and show a linear decrease in M_s with increasing Zn content. The results differ from those obtained by Pops and Massalski (1964) in polycrystals for what they called 'burst' martensite but are in better agreement for their 'thermo-elastic' martensite, which according to Saburi and Wayman (1977) and Sato and Takezawa (1968) is due to quenched-in stresses, and is absent when the stresses are eliminated. The reason for the difference is not known but it is possible that the reheating of the quenched polycrystals has an effect, as suggested by our own results. A similar discrepancy with the results of Pops and Massalski (1964) has also been noted by Mwamba and Delaey (1982). It is worth mentioning that the present empirical relationship fits considerably better the c/a dependence of M_s , which has been deduced for ternary Cu–Zn–Ga, Cu–Zn–Al and Cu–Zn–Si alloys (Ahlers 1974). Here c/a is the tetragonality of a basic face-centred structure from which 9R is derived formally by introducing stacking faults on each third plane. The question of whether for all Cu-based alloys the same c/a dependence of M_s exists remains still open.

In another paper (Ahlers 1986) the enthalpy difference between β and martensite had been deduced, assuming that the Debye model is valid and that therefore the composition dependence of M_s had to be corrected. This correction was applied, using the data of Pops and Massalski. It turns out, however, that the corrected curve ($M_s^0 = 2700 - 65.09C_{Zn}$) is almost identical with the relationship deduced here and that therefore all conclusions on the relative phase stability discussed there (Ahlers 1986) remain valid.

5. Conclusions

(i) The high-temperature stability of the BCC β -phase in Cu–Zn and Cu–Zn–Al is due solely to the soft transverse lattice [$\xi\xi 0$] vibrational modes, related to the C' elastic constants.

(ii) The temperature dependence of the critical stress to induce martensite above M_s is modified by the formation of a face-centred martensite phase only if its stress is exceeded.

(iii) The entropy difference between β and martensite is not affected by the presence of dislocations.

(iv) By extrapolating the temperature dependence of the critical stress to induce martensite to zero stress, an extrapolated M_s temperature is deduced, which is a linear function of composition between Cu–40 at.% Zn and Cu–43 at.% Zn and is given as (where C_{Zn} is in atomic per cent)

$$M_s \text{ (K)} = 2686 - 64C_{Zn}.$$

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