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An *in situ* transmission electron microscope study of dislocation glide in Cu–Ge alloys

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Properties of Cu–2 and –6 at% Ge were investigated at several temperatures by *in situ* deformation experiments in an electron microscope. In particular the change of homogeneous to heterogeneous glide with alloy concentration and deformation temperature was studied. The friction stress opposing dislocation glide was estimated from analysis of groups of defects and numerical calculations and the results are connected with models based on solute atom/dislocation interactions.

1. Introduction

The mode of deformation of alloys varies with the concentration of solute atoms and the temperature. In some cases, the deformation is homogeneous, i.e. it occurs by the motion of many dislocations, each gliding in its own slip plane. In other cases, the whole deformation is localized in bands consisting of a few planes, where a large number of coplanar dislocations glide in a collective movement.

In earlier investigations the dynamic movement of dislocation groups in differently concentrated copper–germanium alloys was studied by deformation tests and high-speed cinematography [1, 2]. The investigation of these materials at the scale of single dislocations by *in situ* deformation in a high-voltage microscope and calculations of dislocation interaction forces is an attempt to pin-point the mechanisms which control the drastically different glide processes in low and high concentrated alloys. Characteristic concentrations, 2 and 6 at % Ge were chosen. These alloys present homogeneous and heterogeneous plastic deformation, respectively. The variations of these phenomena during hardening and at high temperature will be considered.

2. Experimental details

2.1. Alloys

The alloys used were copper–2 and –6 at % germanium. The bulk material consisted of cylindrical single crystals the axis of which is oriented at 48° from the chosen glide direction and 45° from chosen (primary) glide plane.

From earlier deformation tests [1], the concentration and temperature dependence of the critical resolved shear stress (CRSS) was obtained (Fig. 1). For electron microscope observation, the crystals were cut into slices by an acid string saw. They were then cloth polished with an electrolyte down to 80 μm and finally jet polished for TEM observations around the centre. During all the preparation the orientation of the specimen was carefully controlled by Laue diagrams. For observation of the deformation structure, a convenient orientation was to chose the glide direction parallel to the specimen and glide plane at 45°. The tensile axis was deduced from this choice and the specimen cut. However, in order to activate a given glide system, the orientation of *in situ* specimens is open to discussion because the Schmid law is not always observed in thin samples [3]. In fact, in a material exhibiting heterogeneous deformation, the multiplication of numerous dislocations in the same glide plane induces the formation of a crack if the glide direction is not parallel to the specimen plane. The specimens can then be destroyed at the beginning of *in situ* deformation.

2.2. Deformation procedure

The specimen was glued on the special tensile holders which were made in the Laboratoire d'Optique Electronique du C.N.R.S. in Toulouse [4, 5]. In most cases, observations and recording were performed simultaneously with deformation. However, in the case of heterogeneous deformation, the sudden vigorous movement of dislocations shook the specimen and observations were no longer possible. Deformation increments were then applied to the specimen and observations were made as the load was released. The usual precautions were used to protect the specimen from radiation damage: low beam, short observation time, reasonable tension for electron acceleration (800 kV).

3. Observations and results

3.1. Easy glide at room temperature

3.1.1. Cu–2% Ge

Fig. 2 shows a characteristic aspect of the deformation structure in this alloy under load. Dislocations are arranged rather homogeneously in the whole specimen.

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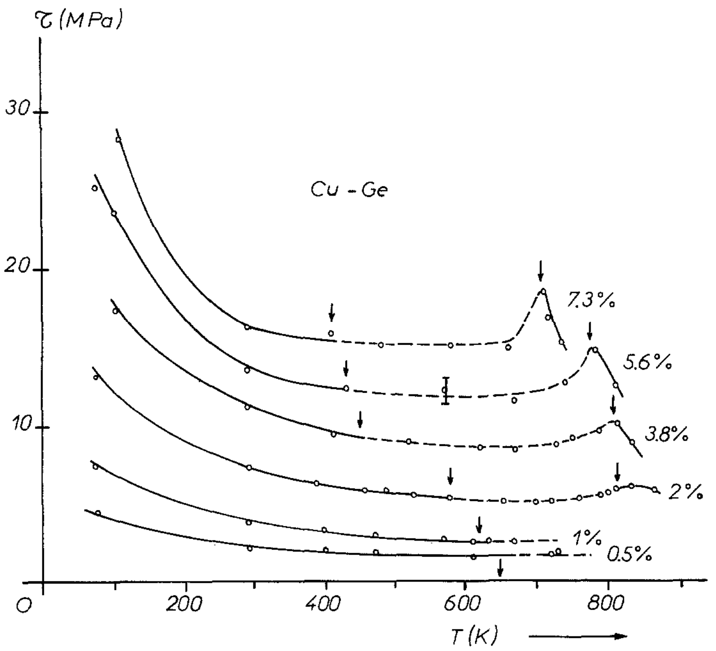


Figure 1 Temperature and concentration dependence of the CRSS in the easy glide stage [1, 2]. The PLC effect has been observed in the temperature range between the arrows (dashed curve line).

Dislocation movement can be observed on the screen of the microscope: they jump over distances of 0.1 to 0.5 μm, and waiting times last for several seconds.

3.1.2. Cu–6% Ge

The deformation mode is quite different from the preceding one (Fig. 3). There are a few active glide planes grouped together in bands, and separated by undeformed zones. Numerous dislocations glide in each plane with a velocity too high to be observed or measured by *in situ* methods ($v > 5\,\mu\text{m}\,\text{sec}^{-1}$).

3.2. Deformation of Cu–6% Ge in easy glide at various temperatures

3.2.1. 110 K observations

At 110 K image sharpness is better than at room temperature. This is due to lower thermal vibrations, i.e. diffracting planes are better defined. The critical resolved shear stress (CRSS) is 24 MPa. Dislocation movement is similar to that at room temperature: planar glide with very high local velocity. Fig. 4a shows an example of the microstructure under stress. In the bands, dislocations are very closely spaced: there are about 80 dislocations in this group. Several comments may be made. (1) Dislocations are rather straight. Thus, there is no possibility of determining the direction of their motion (according to their curvature) before they stop. This also means that surface friction forces are not too high as compared with the volume friction forces; (2) slip traces cut across the group; (3) foreign dislocations are trapped in the group. Either they were attracted, or they were pulled after an intersection.

3.2.2. 500 K observations

At 500 K the CRSS is 13 MPa. Fig. 5 shows that groups of dislocations still exist, but with larger spacings between dislocations than at lower temperature. This means that the stress on the leading dislocations is lower. In this temperature range the Portevin–Le Chatelier effect appears, if the deformation rate in these experiments is not too different from that used for the experiments in Fig. 1.

3.2.3. High-temperature observations (700 and 820 K)

These experiments were performed near the range of

Figure 2 Cu–2 at % Ge. Room temperature.

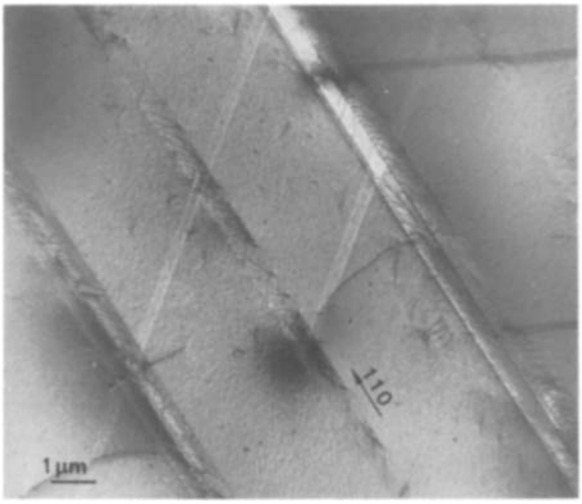


Figure 3 Cu–6 at % Ge. Room temperature.

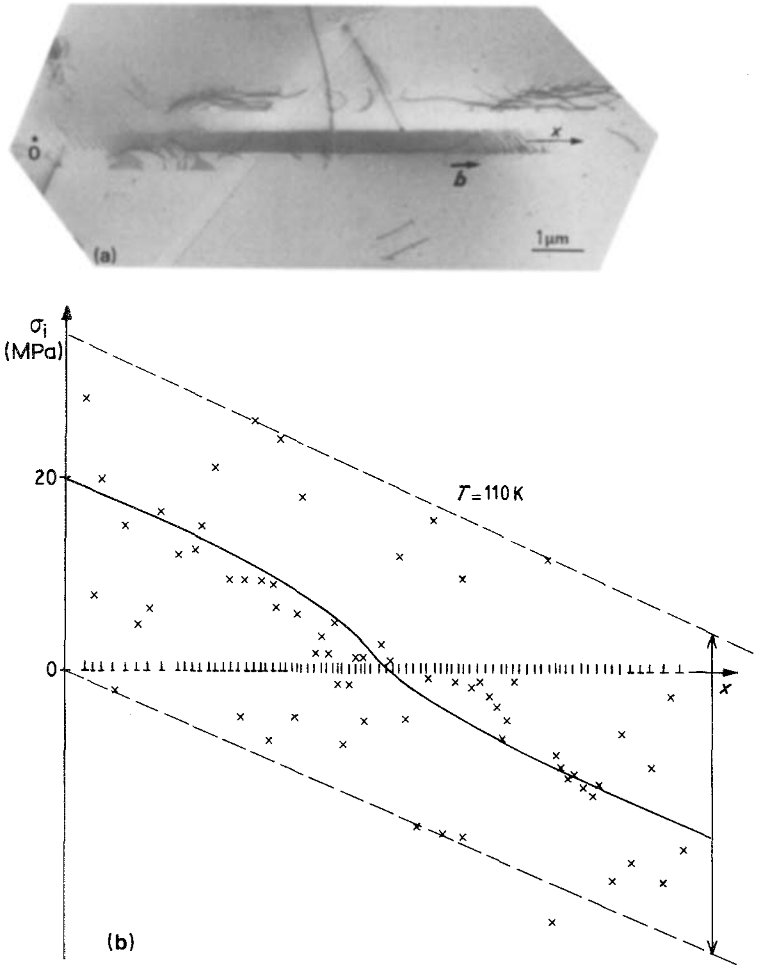


Figure 4 (a) Cu–6 at% Ge. T = 110 K. (b) Group analysis. Interaction stress along the group.

the peak on the CRSS curve of Fig. 1 (15 MPa). As temperature rises, fewer and fewer plane groups of dislocations are observed and isolated dislocations appear which can move freely. Glide traces are numerous (Fig. 6) and image recording of dislocation movement is possible. Isolated dislocations glide two or three times faster than dislocations in groups. The dislocation velocity is 2 to 5 µm $\sec^{-1}$.

3.3. Cu–6 at% Ge: deformation in the hardening stage

To obtain some information about the hardening stage of Cu–6% Ge solid solution, some microtensile specimens were oriented for double glide. Predeformed specimens were also used. Fig. 7 shows the crossing of two glide systems A and C. C is active and several dislocations have passed through. On the other

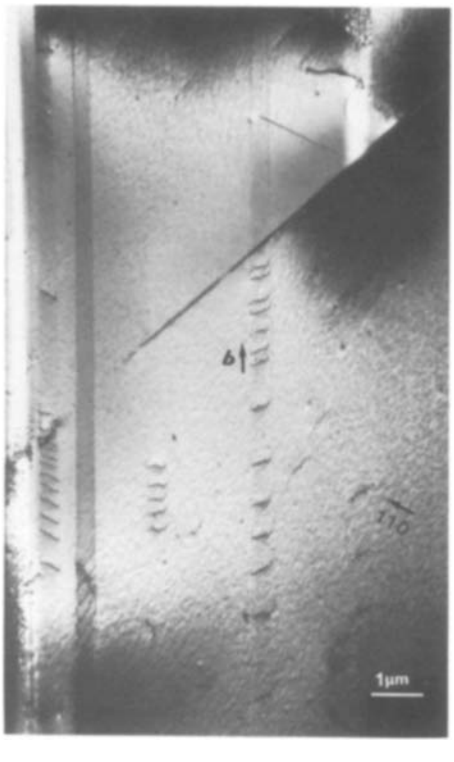


Figure 5 Cu–6 at% Ge. T = 500 K.

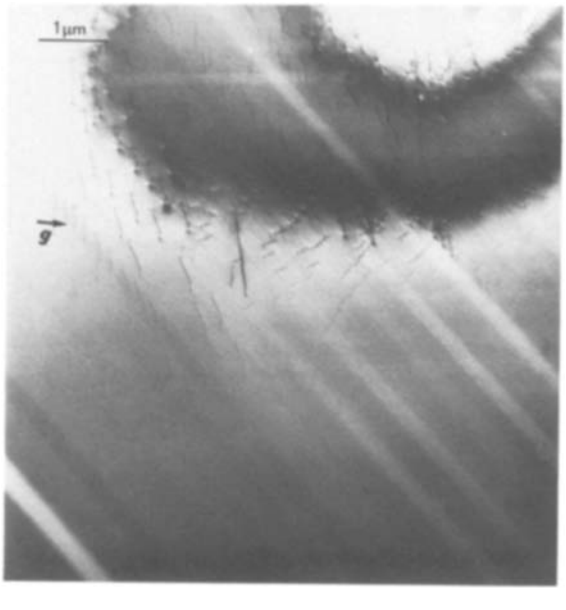
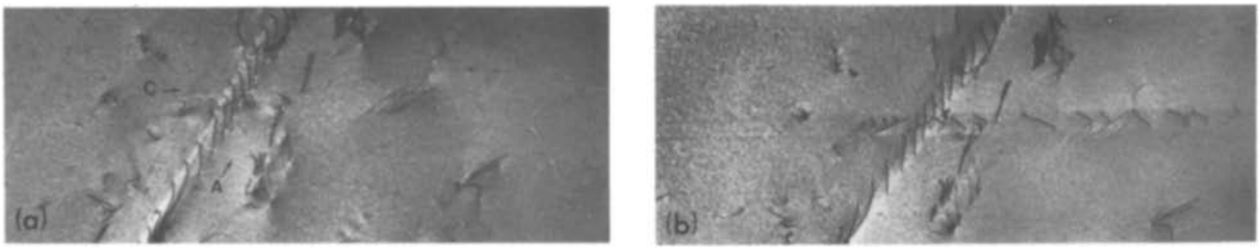


Figure 6 Cu–6 at% Ge. T = 700 K.



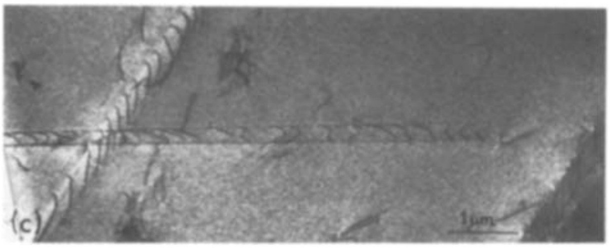


Figure 7 Cu–6 at % Ge. Hardening stage. (a) $t = 0$, (b) $t = 5$ min, (c) $t = 10$ min.

hand, often strong barriers are formed which cannot be crossed by a pile up of one hundred dislocations.

The deformation of specimens predeformed up to the end of easy glide ($\varepsilon \sim 10\%$) shows that the previously formed microstructure is stabilized by either barriers and dislocation junctions, or in numerous multipoles. The deformation continues by activation of new planar groups in new slip planes which destroy the initial microstructure. After a sufficient amount of deformation, light glide traces appear, characteristic of the movement of individual dislocations (Fig. 8).

3.4. Friction stress

The observation of numerous groups which are immobile under stress suggests that a strong friction force exists in the material which prevents the dislocations from moving despite the applied or interaction stresses [6]. Estimation of the friction stress was performed by analysing immobile groups, just after they have stopped at the end of a relaxation period.

Figure 8 Cu–6 at % Ge predeformed ($\varepsilon = 10\%$, end of easy glide), further deformed *in situ* at room temperature.

3.4.1. Analysis of dislocation groups

A dislocation is immobile in a group if the resultant stress on it is equal to zero: $\sigma_e + \sigma_i + \sigma_f = 0$, where σ_e is the effective stress applied to the group, σ_i the resultant stress produced by all the other dislocations of the group, and σ_f the friction stress. Calculation of σ_i from the positions of the observed dislocations and the assumption that σ_e equals CRSS would enable one to obtain σ_f at each point where there is a stopped dislocation. However, the distances between dislocations in the resting group may be different from those of the moving one. Then, the friction stress calculated by the above equation may be different from that felt by the moving dislocation. Thus $|\sigma_e + \sigma_i| \leqslant \sigma_f$ where σ_f is here a limiting value which must be surpassed by the local stress to move the dislocation. In a chosen group (Fig. 4a), far from foreign dislocations, σ_i values are computed using a programme of numerical calculation of interaction forces in an anisotropic medium [7]. They are plotted against position x of each dislocation (Fig. 4b).

In fact, a region exists where each dislocation can stay, where the resultant stress is lower than the friction stress. This corresponds to an uncertainty Δx in the position of each dislocation. Thus each dislocation may take a position on any point of a thin rectangle of Δx in width, $2\sigma_f$ in height, and the average position of which is given by the equation $\sigma_i + \sigma_e = 0$. This provides a scattering of points on the graph. However, there are other possible reasons for this scattering, such as errors in measurement of the dislocation position on the micrographs, curvature of the dislocations, and approximations in the calculations where segments are supposed in an infinite medium.

A method of polynomial interpolation [7] permits the smoothing of the position of the segments against their number. The interaction stress along the smoothed group is shown by the full line curve (Fig. 4b). There are two ways for making use of these curves:

(a) The calculated positions of the dislocations show the shape of a free group under the effective stress alone. The full line curve then shows the distribution of $\sigma_i (= -\sigma_e)$ along the group. The friction, which balances the stress equation on each

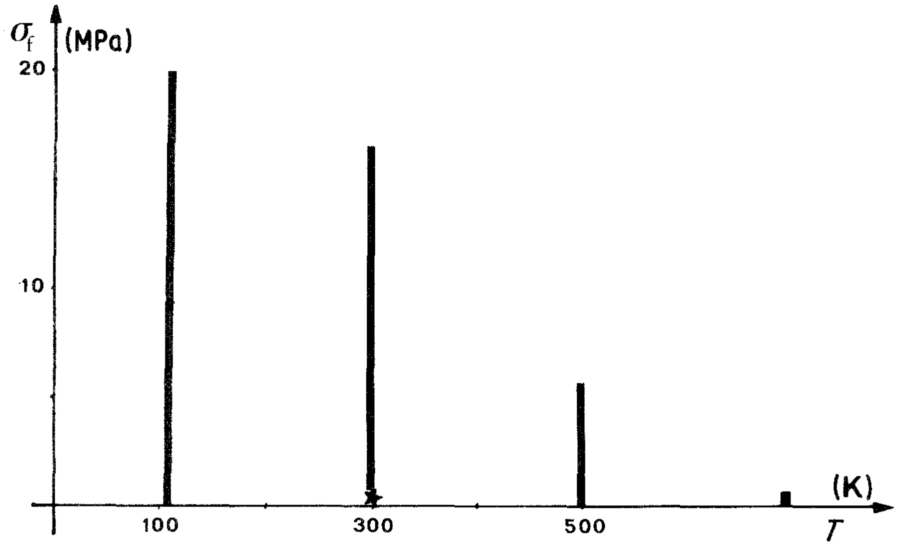


Figure 9 Cu–6 at % Ge. Temperature dependence of the friction stress. (*) Room temperature value for Cu–2 at % Ge.

dislocation, is given by the maximum difference between the calculated value of true $\sigma_i(x)$ and the smoothed curve.

(b) One can suppose that the scattering is due to measurements or calculations. Then, all dislocations are immobile under a friction stress given by the smoothed curve. Assuming σ_e is uniform and high enough for the local stress $(\sigma_i + \sigma_e)$ to be positive, variations of σ_i are linked to the variations of σ_f.
In any case, similar results are found, for the interesting value of σ_f for Cu–6% Ge.

3.4.2. Results

Friction forces have also been estimated by considering the distance from the surface over which the image force overcomes the friction, thus bending the dislocation away from its bulk shape. The results confirm the previous values of σ_f. Fig. 9 presents the variations of σ_f with temperature for the Cu–6% Ge alloy. At low temperatures, σ_f has the same magnitude as the CRSS. It decreases as the temperature increases and becomes quite small for high temperatures.

4. Discussion

4.1. Homogeneous/heterogeneous deformation

The observations show that the deformation becomes heterogeneous when the germanium concentration is increased. At room temperature, there may be several reasons for this phenomenon:

(a) Difficulty for cross-slip. From pure copper to 10 at % germanium (which is the limit of F C C structure of Cu–Ge alloys), the stacking fault energy decreases to nearly zero. For Cu–2 at % Ge, the coefficient $\gamma/\mu\boldsymbol{b}$, where $\boldsymbol{b}$ = Burgers' vector, is near that for pure copper. For 6%, it is three times smaller. Then, dislocations in Cu–2% Ge have the same probability for cross-slip as in pure copper. For 6% Ge, however, cross-slip is much more difficult. Dislocations emitted by a source are constrained to glide in their original slip plane.

(b) Obstacles. We can suppose that the material contains obstacles to dislocation glide, the strength of which increases with concentration. They are supposed to consist of groups of solute atoms [1, 2, 8], perhaps with preferential short-range ordering. In the Cu–6% Ge alloy the isolated dislocations would not be able to cross the obstacles. Only the pile-ups of dislocations, with a stress concentration at their head higher than the applied stress, are able to overcome them. The pile-ups are formed and stay together because of the gradual destruction of the (short-range ordered) obstacles by the cutting dislocations.

(c) Sources. At low concentration, sources are numerous (low friction), and produce a few dislocations under a low applied stress. At higher concentration, only those sources situated in regions where the effective stress is higher than the friction stress are able to operate, suddenly emitting many dislocations again due to the destruction of obstacles. The scarcity of sources is a reason for heterogeneous deformation.

In fact none of these reasons is predominant. They are simultaneous and complementary. We will discuss below the role of solute atoms on the dislocation glide.

4.2. Deformation at various temperatures; dislocation/solute atoms interaction

The strain field around a dislocation affects the distribution of the solute atom in its surroundings. A germanium atom is attracted by the dilated zones. As the dislocation glides, this "cloud" moves with it by diffusion, if the temperature is high enough. Several authors (e.g. [9, 10]) have calculated the behaviour of a moving dislocation in this case.

4.2.1 Low temperatures

At low temperatures friction forces are high in the material (up to 20 MPa). This value is near the CRSS (Fig. 1). Grown-in dislocations are locked by their solute atmosphere and need some overstress or local stress concentration to form initial sources for mobile dislocations. The stress against velocity curve ($\sigma(v)$) is shown in Fig. 10. Application of a stress σ_f is necessary to move mobile dislocations. Only in the first moments after escaping from its atmosphere and during action of local overstress, can the first few dislocations glide with high velocity v_0; under stationary conditions

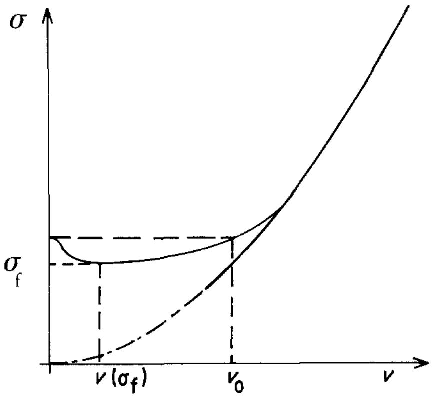


Figure 10 Stress necessary for a dislocation to move at low temperature. (–·–·–·–) Theoretical curve for a thermally activated movement.

(corresponding to macroscopic CRSS measurements) the smaller value $v(\sigma_f)$ will be reached. Accordingly, at low tempeatures, the CRSS is well explained [1, 2] assuming a thermally activated movement of dislocations across obstacles consisting of groups of solute atoms.

4.2.2. Intermediate temperatures

These are the domain of the Portevin–Le Chatelier effect (cf. Fig. 1). Numerous authors have studied this phenomenon. For instance, according to Penning [11] the instability of the deformation curve can be explained by a negative slope of the stress against strain-rate curve $\sigma(\dot{\varepsilon})$. Recently, Kubin and Estrin [12] have purposed a mathematical model which provides a link between the microscopic and the macroscopic aspects in such a case. Yoshinaga and Morozumi [10] calculated the distribution of the solute atmosphere in the case of a model system of carbon interstitials in vanadium confirming the general shape of the force against glide velocity curve $F(v)$ used in [11, 12], with three domains where the behaviour of the solute atoms is different (Fig. 11): (a) $0 < v < v_{\mathrm{CH}}$. The solute atoms follow the dislocation, but the force exerted on it increases with velocity. The extent of this domain is negligible at room temperature but increases with increasing temperature and F_{m} corresponds to the friction force. (b) $v_{\mathrm{CH}} < v < v_{\mathrm{CL}}$. The dislocation escapes from its solute atmosphere, and the force decreases as v increases. Thus, it is an instability zone. (c) $v > v_{\mathrm{CL}}$. The velocity is very high and the solute atoms seem immobile for the dislocation. Various phonon dragging processes cause the parabolic increase of $F(v)$ for high v values.

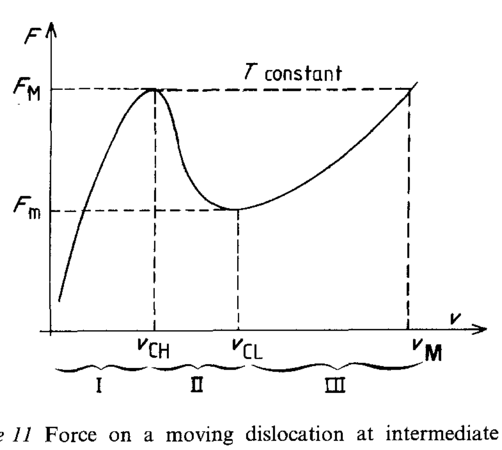


Figure 11 Force on a moving dislocation at intermediate temperatures. Theoretical curve.

The dislocations escape from their solute atmosphere and accelerate to v_{M} when the applied stress is high enough to overcome the friction. Then, if a too rapid deformation corresponds to v_{M}, F decreases to F_{m} and the dislocation stops. So, F has to increase again to F_{M} for the deformation to continue. The applied force is oscillating.

This simple model needs to be extended by accounting for the collective dislocation motion [13] in our case of heterogeneous glide. This may explain that the extreme values of this domain of temperature (Fig. 1) which correspond to the positive slope part of the CRSS curves are lower than that which can be calculated by the formulae of Yoshinaga or Cottrell [9, 10] formulae.

4.2.3. High temperatures

The friction force is very low. Many sources can operate easily and single dislocations are mobile. The deformation is homogeneous. However, it is the domain where the solute atoms are more and more mobile. Now the solute atmosphere around the dislocation will be established and be redistributed even during its motion: the interaction force increases. The dislocation velocity decreases or a higher effective stress is necessary to maintain it at the same level. At still higher temperatures, diffusion becomes so rapid that the formation of an atmosphere around the moving dislocation will be less favourable. This may involve the observed peak in the CRSS curve (Fig. 1).

4.2.4. The friction stress

The dislocation/solute atom interaction can involve two kinds of friction stresses: (a) the dynamic friction which is encountered by the moving dislocations and controls the dislocation velocity; (b) the static friction caused by the rearrangement of solute atoms around the dislocations stopped for a long time, by the Suzuki effect for instance. It controls the unpinning of the defects. The rate of atomic rearrangements around the dislocations is very important: it is not very fast for the Cu–2% Ge alloy. On the other hand, it is strong and rapid for the 6% Ge, especially as a plane dislocation group is an effective trap for solute atoms [14].

Our measurements give the static friction. In the 2% Ge alloy, the dynamic friction is near to the local stress since the dislocations glide slowly in a jerky movement over limited distances. The high velocity of the dislocations in the 6% Ge alloy shows that the dynamic friction is quite faint compared to the static one which controls their start.

4.3. Hardening stage of Cu–6 at % Ge alloy

Li [14] has shown that "horizontal" dislocation groups in the same glide plane produce a long-range stress. Therefore, they exert a strong force on the surrounding defects and can attract them to create lower energy networks as observed (multipoles, Lomer–Cottrell barriers). This phenomenon produces early hardening and stops the activity of the first glide system. This assumption is confirmed by Peissker [15] who measured the extent of the easy glide stage length against concentration in copper–germanium alloys. There is a maximum in this stage for a germanium concentration between 2 and 3% which corresponds to the appearance of heterogeneous deformation. For higher concentrations the easy glide stage begins to shorten. In spite of high local strain on a few active slip planes, the average external strain decreases with increasing heterogeneity of slip. The primary glide system cannot run for a long time but, due to high local stresses, it creates in the material a network of barriers and very strong junctions, providing quick hardening. Higher stresses are needed for further deformation. Then the applied stress overcomes the friction value and many single dislocations can glide. The deformation is homogeneous again.

5. Conclusion

The measurements and observations presented in this work have been analysed by consideration of the interaction between dislocations and solute atoms. Although the physical nature of the friction stress on dislocations still is not well defined, it is possible to give simple explanations for the behaviour of Cu–Ge alloys during deformation.

The dislocations created are rather homogeneously distributed in the material when the effective stress they support is higher than or equal to the static friction. This is the case for 2% Ge at room temperature, and for 6% Ge at high temperature and in stage 2 at room temperature. On the other hand, Cu–6% Ge shows heterogeneous glide and discontinuous motion of dislocations owing to strong locking of resting dislocations, local overstress to activate sources, formation of dislocation groups due to obstacle destruction, and small dynamic friction compared to static friction.

The dislocation velocity is controlled by the temperature. At low and room temperature, in the domain of jerky dislocation movement, the average velocity is determined by the waiting times at obstacles. High temperature is the domain of solute atmosphere dragging. The dislocations move continuously with low velocity.

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