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Tensile properties of irradiated Cu single crystals and their temperature dependence

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Abstract

Single crystal copper was irradiated with 590 MeV protons to a dose of 0.01 dpa at room temperature. Irradiated and unirradiated tensile samples were deformed and relaxation tests were performed at temperatures between 77 and 293 K. The tests show a strong temperature dependence of the flow stress of irradiated samples as compared to the unirradiated case. Deformed microstructures in the unirradiated and irradiated samples were investigated by transmission electron microscopy. The plastic deformation mechanism of the irradiated samples is dislocation slip, while in the unirradiated sample at large strains and at 77 K, twinning is observed. In the irradiated case, strain localization in the form of defect-free channel takes place, over the temperature range from 77 to 293 K. Deformation processes are analyzed through the determination of the activation volumes and energies of the deformation mechanisms, as deduced from relaxation tests. The activation energy in unirradiated Cu has an approximate value of 1.6 eV. In the irradiated samples it is suggested that more than one deformation process is operative, in the temperature range from 77 to 293 K.

1. Introduction

Metallic single crystals are useful candidates to study dislocation processes associated with mechanical properties in irradiated metals because of the absence of grain boundary effects. They have been used to investigate defects due to irradiation and the subsequent hardening in pure single crystals of Cu [1–8], Pd [9], Ni [10].

In the present work, we try to identify the origin of the deformation processes of irradiated Cu single crystal, by investigating the temperature dependences of dislocation mechanisms at the various deformation stages. Results are presented on the activation energies deduced from the activation volume measurements for unirradiated and irradiated single crystal Cu as a function of temperature. The possible associated deformation mechanisms are discussed in relation with the microstructure observed in transmission electron microscopy (TEM).

2. Experimental procedure

Single crystal rods of pure 99.999% copper of 20 mm in diameter were provided by Goodfellow Cambridge Ltd. The tensile specimens of 2.5 mm gauge width and 5.5 mm gauge length were cut from the plates by EDM. The tensile orientation is close to ⟨0 1 1⟩, where little or no easy glide is expected to be present. After mechanical polishing of the surfaces, the final thickness was about 300 μm, and was further reduced to about 250 μm after removing the deformed surface layer by electro-chemical polishing. All specimens were annealed in a vacuum of about 10^{-5} mbar at 1273 K for 2 h followed by a slow cooling to room temperature (293 K).

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Tensile specimens were irradiated in the PIREX facility [11] installed in the 590 MeV proton beam of the accelerator located at the Paul Scherrer Institut in Switzerland, to doses 10^{-2} dpa. The typical dose rate is 5×10^{-7} dpa s^{-1}. The irradiation temperature is around 60–70 °C, which represents $T_{irr} = 0.25T_M$, where T_M is the absolute melting temperature of Cu.

The tensile tests were carried out at 293 K in air and at low temperatures in alcohol mixed with liquid nitrogen or in pure liquid nitrogen. All tests were performed at a cross-head speed of 0.3 μm s^{-1}, corresponding to a shear strain rate of about 5×10^{-1} s^{-1}.

Stress relaxation tests have been performed to obtain the activation volumes, which were used to calculate the thermal activation energy [7,12,13]. The same tensile machine was used, and the method of successive relaxation tests with a constant relaxation time was applied. At low temperatures the duration of each stress relaxation lasts 30 s so that the temperature for each relaxation is be constant. The effective activation volume was corrected by using the technique of successive relaxation tests following the first stress relaxation, the later indicating only the apparent activation volume [14].

TEM specimens of the deformed unirradiated specimen and of the irradiated and deformed ones were prepared. The specimens were first mechanically polished and then jet-electro-polished at 293 K and 9 V in a solution of 25% HPO_3, 25% methanol, and 25% water. TEM examinations were performed in a JEOL 2010 microscope operated at 200 kV.

3. Results

3.1. The tensile properties and their temperature dependence

Tensile tests on unirradiated and irradiated Cu single crystal were carried out at 77, 173, 233, and 293 K. The shear stress–shear strain curves for the unirradiated Cu are shown in Fig. 1(a). The yield shear stress (measured at 0.2% shear strain) increases monotonically with decreasing temperature from 14 MPa at 293 K to 27 MPa at 77 K. Samples irradiated at a dose of 0.01 dpa were deformed at the same temperatures as the unirradiated ones. The yield shear stress of the irradiated Cu is more strongly dependent on the test temperature than that of the unirradiated one, as it increases with decreasing temperature from 37 MPa at 293 K to 83 MPa at 77 K.

Contrary to the case of the unirradiated material (Fig. 2(a)), the work hardening rate ($\theta = d\tau/d\gamma$) of the irradiated Cu is less dependent on testing temperature, as displayed in Fig. 2(b).

Typical results of the activation volume measurements as a function of stress are shown in Fig. 3. While in the unirradiated Cu the activation volume decreases rapidly with strain at all testing temperatures, it remains almost constant in the irradiated case. For the latter, values between 100–400 b^3 and 60–150 b^3 are found at 173 and 77 K, respectively.

3.2. The deformation microstructure

The deformation microstructure of unirradiated specimens was shown in Fig. 4, at a different temperature of 293 K (Fig. 4(a)) and 77 K (Fig. 4(b)) observed under TEM. The microstructure observed in the samples deformed at 233 and 173 K shows a similar elongated dislocation cell structure as that of Fig. 4(a), characteristic of stage III. At 77 K, twinning lamellae are observed (Fig. 4(b)), as indicated by the diffraction pattern attached at the right bottom in Fig. 4(b).

Fig. 4(c)–(h) show the deformation microstructure of Cu irradiated to 0.01 dpa at the different temperatures. TEM samples were cut from the region close to the shoulders of the specimens of the gauge length, where the strain is lower than in the gauge length (Fig. 4(c), (e)

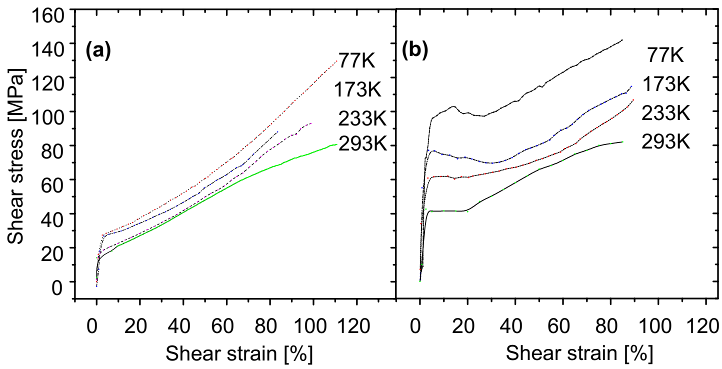

Fig. 1. Shear stress–shear strain curves as a function of temperature for (a) unirradiated single crystal Cu, and (b) Cu irradiated to a dose of 0.01 dpa at RT.

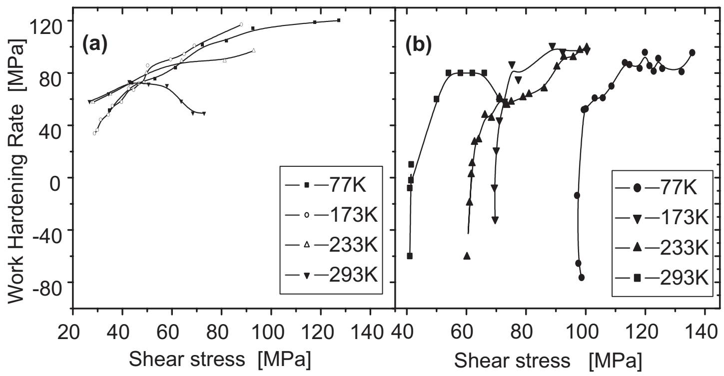

Fig. 2. Work hardening as a function of shear stress of (a) unirradiated Cu and (b) Cu irradiated to 0.01 dpa, both tested at 293, 233, 173, and 77 K (continues lines eye guidelines only).

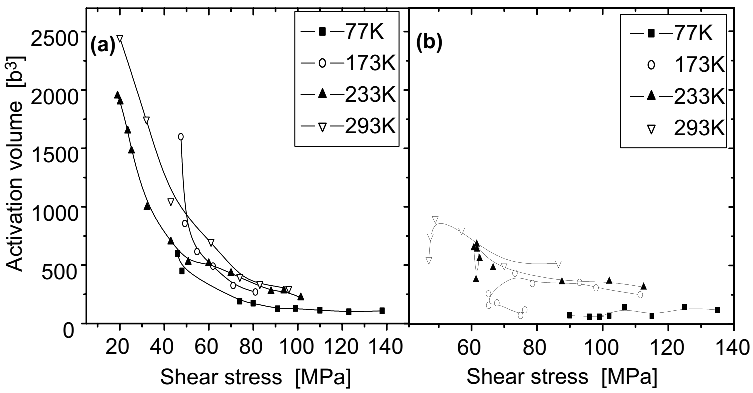

Fig. 3. Effective activation volume–shear stress response as a function of temperature in (a) unirradiated Cu and (b) Cu irradiated to 0.01 dpa at RT (continues lines eye guidelines only).

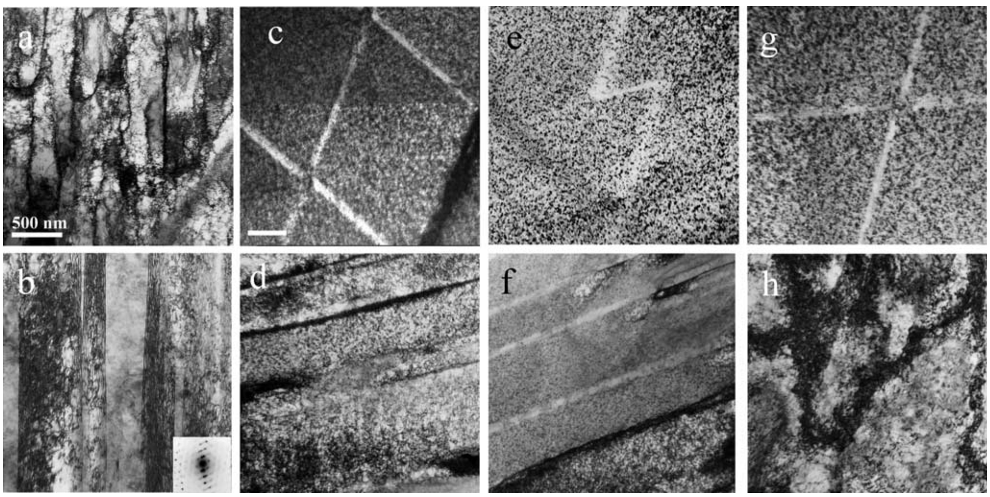

Fig. 4. TEM micrographs of deformation microstructure in unirradiated Cu specimens: (a) dislocation cells at RT and (b) twinning lamellae at 77 K. From (c) to (h), top and bottom row of images relate to regions close to the shoulder and regions of the gauge length of tensile specimens, respectively. The first column (c, d), second column (e, f), and third column (g,h) relate to the deformation structure at 233, 173, and 77 K, respectively. Figures (a) and (b) have the same scale, and (c)–(g) have another but equal scale.

and (g)), and the regions near the fracture of the specimens (Fig. 4(d), (f) and (h)) where the strains exceeded 100%. The first, second, and third columns of images correspond to samples deformed at 233, 173, and 77 K, respectively. The TEM observation performed after a deformation at 293 K shows a dislocation cell structure, as that shown in Fig. 4(a). The highly strained gauge section of the irradiated material at 233 K also shows dislocation cells but no channels or dislocation braids or bands (Fig. 4(d)). At 177 K, a mixed structure of defect-free channels together with a dislocation cell structure comparable to that found in stage III is present (Fig. 4(f)). Defect-free channels formed earlier in the test are apparently being destroyed by the dense formation of dislocation cells, but a high density of defect clusters subsists within the cells in the region between the channels. The microstructure in the shoulder region of the same irradiated specimen (Fig. 4(e) and (g)) consists, at all temperatures, of defect-free channels and undeformed structure containing the original high density of irradiation induced defects.

The deformation takes place exclusively by dislocation slip in irradiated Cu, but in the case of unirradiated Cu deformed at 77 K at a strain of ~100% twinning has been observed.

4. Discussion

4.1. Plastic deformation of the Cu single crystal

In fcc metals such as Ni and Cu, the twin nucleus and hence the stress needed to propagate twins relates to the stacking fault energy (SFE). Carter and Holmes [15] and Hirth and Lothe [16] reported a value of 125 $\mathrm{mJ\,m^{-2}}$ for Ni, which is more than two times that of Cu (45 $\mathrm{mJ\,m^{-2}}$). Tensile tests show that in pure Ni there is no twinning even at 4.2 K, while pure Cu shows twinning in the same conditions [17].

In Cu the calculated twinning stress derived from Zerilli–Amstrong equation [18] by Meyers et al. [19] with a strain rate of 5×10^{-5} $\mathrm{s^{-1}}$ is around 110 MPa at temperatures between 77 and 293 K. The TEM observation shown in Fig. 4 clearly indicates that only after heavy deformation at 77 K deformation twinning takes place. Additional conditions must exist other than the above criteria, as no twinning is observed in the unirradiated Cu at other testing temperatures (where the stress overcomes the 110 MPa limit) or in the irradiated material, where the whole tensile curve at 77 K is above this stress limit. In this last case, the operative deformation mode is dislocation channelling, which is expected to actually relax the local stress and thus may impede twinning.

In the unirradiated material, only the tensile curve obtained at 293 K shows an initial increase of the work hardening to values between 40 and 70 MPa, leading to almost constant stage at ≈70 MPa (stage II). For tests at lower temperatures, the work hardening increases continuously, with little or no temperature dependence. In contrast, the irradiated Cu shows at all testing temperatures an initial increase followed by work hardening at a level of approximately 80 MPa.

4.2. The temperature dependence of the flow stress

In the presence of both an athermal (τ_μ) and thermal component (τ^*) of the stress, the total flow stress can be written as

$$\tau = \tau_\mu + \tau^*. \tag{1}$$

If the flow stress is controlled by a single deformation mechanism, the deformation rate is given by the following equation:

$$\dot{\gamma}_\mathrm{p} = \dot{\gamma}_0 \exp\left(\frac{-\Delta G(\tau^*)}{kT}\right). \tag{2}$$

The activation energy ΔG of this mechanism can be calculated [12] according to

$$\Delta G = \delta G + \int_{\tau_\mathrm{m}}^{\tau} V\,\mathrm{d}\tau, \tag{3}$$

where τ_m is the largest measured yield stress and ΔG represents the free energy between 0 and 77 K, which is not measured. V is the activation volume. From Eq. (2), for a fixed strain rate, ΔG depends linearly on temperature:

$$\Delta G = \alpha kT \tag{4}$$

and δG can be determined by requiring that $\Delta G = 0$ at 0 K. Fig. 5(a) shows the correlations between resolved shear stress, temperature, and activation energy for unirradiated samples

$$\int_{\tau_\mathrm{m}}^{\tau} V\,\mathrm{d}\tau \quad \text{and} \quad \Delta G = \delta G + \int_{\tau_\mathrm{m}}^{\tau} V\,\mathrm{d}\tau.$$

In this case $\int_{\tau_\mathrm{m}}^{\tau} V\,\mathrm{d}\tau$, and thus ΔG is indeed linear with temperature. The derivation of the values of δG and ΔG is straightforward.

In the unirradiated case an activation energy, δG of approximately 1.6 eV can be extrapolated from Fig. 5(a).

The yield shear stress of the irradiated samples shows a strong temperature dependence from 77 to 293 K which suggests that a different deformation mechanism is operative, related to the irradiation-induced defects and which, according to Fig. 1(b), still controls the overall flow stress. This result is comparable to that of Blewitt et al. [1], who found a similar temperature

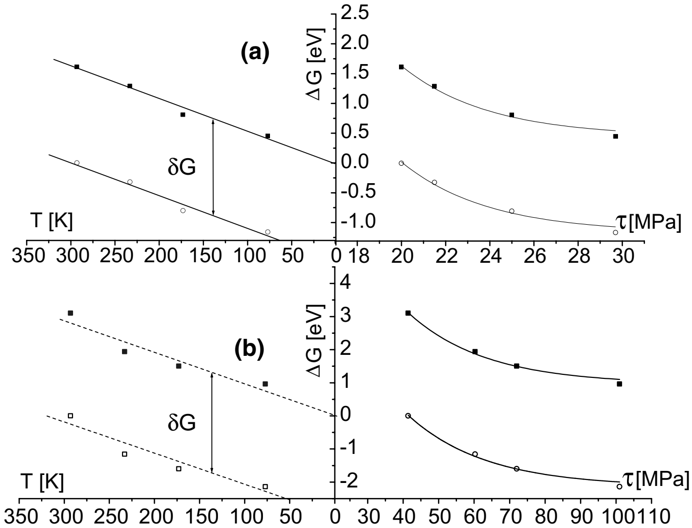

Fig. 5. Stress and temperature dependence of the activation energy for (a) unirradiated Cu and (b) Cu irradiated to 0.01 dpa. The open symbols are $\int_{\tau_m}^{\tau} V\,d\tau$, and the closed symbols are ΔG.

dependence of the yield stress of irradiated Cu single crystals at doses higher than $\sim 10^{-3}$ dpa.

The argument can be extended into the ΔG, τ, T behaviour shown in Fig. 5(b). In opposition to the unirradiated case, the ΔG vs T does not show a lineal fit, so no single value can be extrapolated to 0 K. When in the irradiated case values at all temperatures are used, the extrapolated value is $\delta G = 3.1$ eV (Fig. 5(b)), which is probably too large to be considered.

One possible reason for this result is that more than one deformation mechanism is operative in this regime and then Eq. (2) is not anymore valid.

5. Conclusions

1. The yield shear stress of the irradiated single crystal Cu has a strong dependence on the test temperature in the range from 77 to 293 K.
2. The initial plastic deformation of Cu irradiated to 0.01 dpa shows a typical strain localization in defect-free channels in the temperature range from 77 to 293 K. A dislocation cell structure is formed at high strains in all cases. At 77 K, in unirradiated specimens, twinning lamellae are observed at large strains.
3. The calculation of thermal activation parameters shows a temperature and stress dependence of the activation energy in unirradiated samples. Above 293 K, the yield shear stress is athermal. The results in the irradiated Cu suggest that more than one deformation process is operative.

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