Influence of impurities in polygonization of fcc metals of low stacking fault energy

Several studies performed on metal polygonization [1–3] indicate that impurities lock the dislocations and prevent their motion during a thermal treatment of previously deformed crystals. Nevertheless the study made on polygonization of Cu showed contradictory results. Young [4, 5] and Wei *et al.* [6] observed polygonization in OFHC Cu, but not in 99.999% Cu, and pointed out that polygonization in Cu is very slow and occurs upon prolonged annealing at temperatures near the melting point. Seeger and Schoeck [7, 8] suggested that, in metals of low stacking fault energy like Cu, Ag and Au, the dislocations would be extended into widely separated partials, and thus climbing would be difficult. The fact that polygonization has been observed [4, 5] in OFHC Cu, but not in 99.999% Cu, was explained by considering that impurities tend to allow the partial dislocations to recombine. More recently, French workers [9–13], showed that pure Cu polygonizes more easily than impure.

In order to clarify the effect of impurities in climbing of the extended dislocations in fcc metals, and to find the effect of the stacking fault energy, the present study was carried out in Cu and Ag crystals.

The samples were single crystals grown from OFHC 99.98% Cu, 99.999% Cu, 99.98% Ag and 99.999% Ag. The surfaces in study were parallel to the (1 1 1) plane and the axis of the specimen on such plane was approximately on the [1 $\bar{1}$ 0] direction, so that only one gliding system predominates during the deformation of the crystals. The single crystals were first annealed at high temperature to lower the initial number of dislocations, then deformed by bending with the [1 1 1] axis parallel to the bending axis. The deformations were small enough (1 to 1.3%) in order to prevent possible recrystallization of the metals during the successive thermal annealings. To follow the evolution of the polygonization substructure, the deformed crystals were isothermally annealed under vacuum (10^{-5} mm of Hg) at two different temperatures, and at accumulative periods of 5 h up to a total of 200 h for each temperature. Details of deformations and annealings are shown in Table I. After every annealing, the surfaces were electropolished conveniently and etched. Livingston's [14–16] modified dislocation etchant was used to follow the motion of dislocations on the Cu surfaces. Levinstein and Robinson's [17] chemical etching and the modified one [18, 19] were used for the Ag crystals. A Reichert MeF Microscope was employed to observe the characteristic etch pit structures. The advance of polygonization was estimated with an error of ±10% after running over the whole crystal surfaces.

TABLE I Orientation, deformation and thermal annealings of the copper and the silver single crystals

Purity (%)	Deviation from the (1 1 1) plane	Deformation (%)	Annealing temperature (° C)
Cu			
OFHC 99.98	3°30′	1.1	700
OFHC 99.98	3°	1.3	1000
99.999	3°	1.1	700
99.999	3°30′	1.3	1000
Ag			
99.98	3°	1.2	700
99.98	2°	1.0	900
99.999	3°	1.1	700
99.999	2°	1.0	900

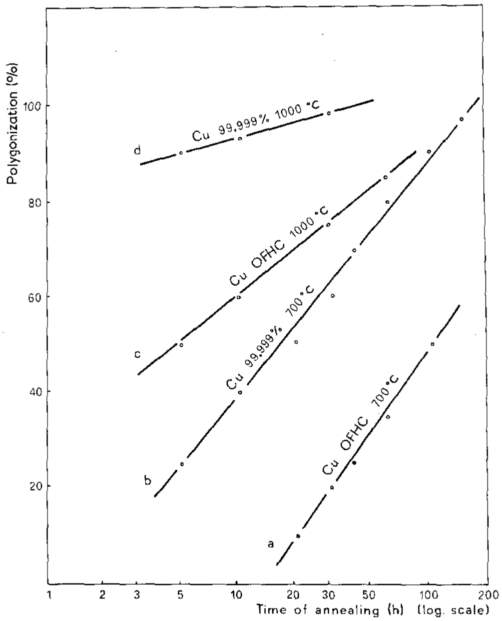

Figure 1 Advance of polygonization versus time of annealing in OFHC Cu and in 99.999% Cu, at 700 and 1000° C.

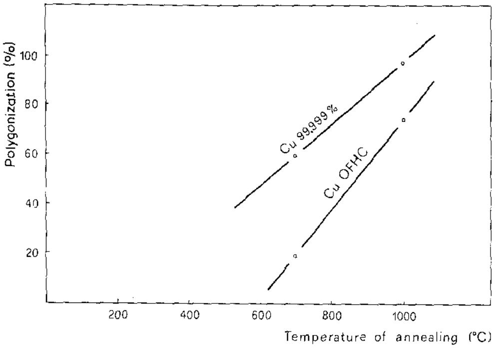

Figure 2 Advance of polygonization versus temperature of annealing in OFHC Cu and in 99.999% Cu.

The experimental results for Cu are represented in Figs. 1 and 2. It was observed that by annealing OFHC Cu at 700° C, as well as at 1000° C, polygonization in the full extension of the specimen surfaces was not reached. In the case of high purity Cu, nearly 100% polygonization was observed during the annealings at both temperatures in study.

In the study with the Ag crystals, polygonization could not be detected in 99.98% Ag at the temperatures and times of the present study. In 99.999% Ag, a slow motion of dislocations after the first 5 h of annealing at 700° C was observed. Approximately 10% of the crystal surface was polygonized after 90 h of annealing at 700° C, and the advance was slow with further annealings. Nevertheless, by annealing 99.999% crystals at 900° C a clear and an advanced polygonization was observed.

The experimental results led to the following observations: (a) A negative effect of impurities in polygonization of Cu and Ag. (b) The climb of dislocations in Cu require neither very high temperatures nor long time of annealing. Because of the percentage of polygonization observed at 700° C, it is obvious that some polygonization may occur at lower temperatures. This is also demonstrated if the plots of Fig. 2 are extrapolated to 0% polygonization, revealing low starting temperatures of polygonization in both Cu purities. (c) The necessity of high annealing temperatures for polygonization to occur in Ag crystals.

Comparing the results obtained for Ag with those for Cu, one arrives at the following conclusions:

(1) Impurities affect climbing of dislocations more in silver crystals than in copper. No polygonization was observed in Ag 99.98% even at the highest temperature of the present study (95% of the absolute melting point of Ag). On the other hand, 99.98% Cu probably polygonizes at less than 700° C (72% of the absolute melting point of Cu).

(2) 99.999% Ag polygonizes slower than 99.999% Cu. Advanced polygonization of high purity Cu was observed after only 10 h of annealing at 700° C, but Ag required 90 h of annealing to show some polygonization at 700° C.

The conclusions indicate that the different behaviour of the metals may be attributed to the difference in their stacking fault energy. Table II shows the values of stacking fault energy of Cu and Ag obtained by different investigators. The values in Ag are consistently low, in spite of the different techniques used. The dislocations must be extended into partials, more widely separated in this metal than in Cu, and thus their climb is difficult, as postulated by Seeger [7, 8].

TABLE II Values of stacking fault energy in Cu and in Ag obtained by different workers

	Cu (erg cm^{-2})	Ag (erg cm^{-2})
Berner (1960) [20]	170	30
Bolling *et al.* (1962) [21]	163	20
Thornton, Mitchell and Hirsch (1962) [22]	60	20
Loretto *et al.* (1964) [23]		21
Gallagher (1964) [24]		20 ± 7
Gallagher and Washburn (1966) [25]		22
Dillamore and Smallman (1965) [26]	85 ± 30	21 ± 7
Ruff and Ives (1967) [27]		23
Cockayne *et al.* (1971) [28]	41 ± 9	16.3 ± 1.7
Clarebrough (1974) [29]		14.17 ± 2.13
Englert, Tompa and Bullough (1970) [30]	70	
Stobb and Sworn (1971) [31]	41	

These conclusions lead to two hypotheses about the mechanism of polygonization of the metals in study:

(1) The stacking fault energy in Cu is not very low, thus the partials are not too separated. This assumption would be possible, bearing in mind that the values of stacking fault energy in Cu obtained by different investigators do not coincide (Table II) and in some cases are high: 170 and 163 erg cm^{-2} [20, 21].

(2) The stacking fault energy in Cu is relatively low. Thus the probable situation is that the extended dislocations can climb directly as shown elsewhere [32], or that the tenor of impurities in 99.999% Cu is enough to saturate the dislocations and to allow the recombination of the partials and their further climbing.

As the stacking fault energy in Ag is low, this second mechanism is also considered for Ag. After a certain limit, the impurities would start to make difficult the climbing of dislocations, as observed in both metals.

On the other hand, it is suggested that the contradictory results obtained by certain workers [4–6] in relation to polygonization in 99.999% Cu, are due principally to the techniques used to detect the climbing of dislocations. It is important to emphasize the peculiarity presented by Cu related to the anisotropic behaviour of the crystal planes [33, 34]. Thus the etchant appears to be selective of the crystallographic orientation of the crystal [24].

Acknowledgements

The authors thank the Consejo Nacional de Investigaciones Científicas y Técnicas of Argentina and the Centro Argentino de Información del Cobre for financial support.

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Received 29 October 1976
and accepted 8 Febraury 1977

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