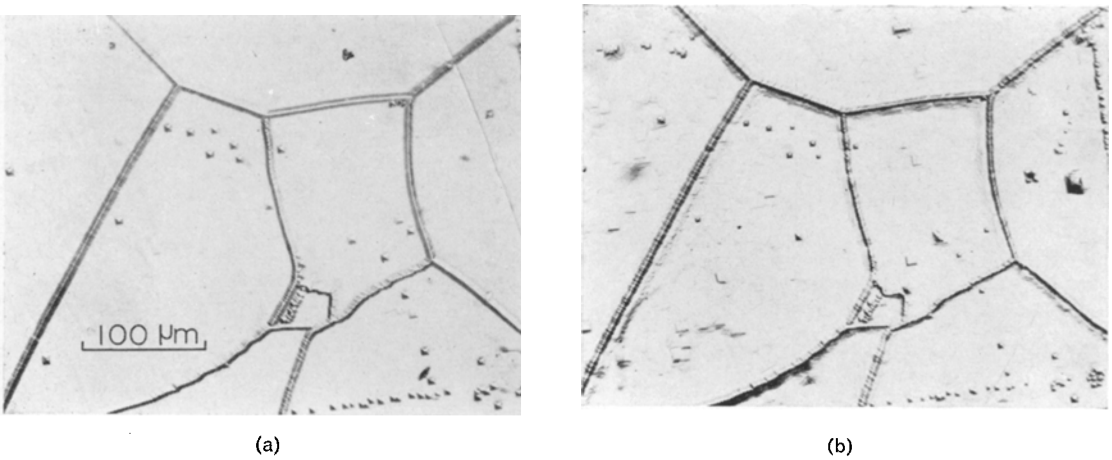
Letters

The Generation of Dislocations in Bicrystals of Sodium Chloride by Hydrostatic Pressure

It is now generally recognised that the application of a hydrostatic pressure of the order of 10 kbars can result in the generation of dislocations in polycrystals of cubic metals, e.g. iron-carbon alloys [1] and chromium [2]. As solids possessing the cubic structure have isotropic linear compressibilities, the presence of grain-boundaries, *to the first approximation*, should not result in local shear stresses [3]. Bullen and Wain [2] and Das and Radcliffe [4] have accordingly suggested that elastic discontinuities, e.g. particles or voids, are responsible for the generation of dislocations under hydrostatic pressure. The mechanical properties of Si–Fe are affected by pressurisation similarly as those of steel and chromium; Worthington [5], however, did not observe nucleation of dislocations at precipitate particles, but suggested that the pressurisation treatment resulted in an increase in activity of grain-boundary sources (during subsequent straining). In another study of pressurised chromium Mellor and Wronski [6] report that arrays of dislocations were seen which appeared to have been generated by precipitate particles *and* by grain-boundaries. For the hexagonal metals, e.g. cadmium and zinc possessing high degrees of anisotropy in the linear compressibility [3], no deformation has been observed in monocrystalline specimens pressurised at 26 kbars, but polycrystals exhibited grain-boundary migration, slip, multiple slip and twinning.

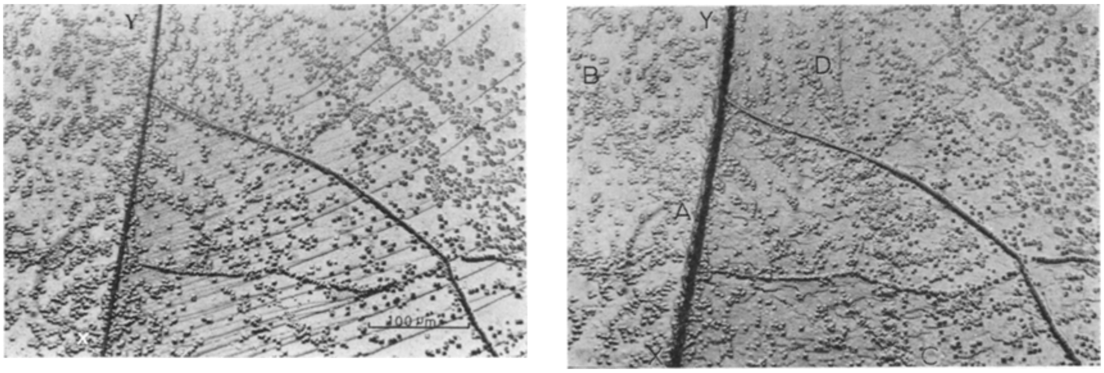
Similar considerations should apply to non-metallic crystalline solids, which, however, appear not to have been investigated. We have initiated accordingly a programme of study of pressurisation effects in ionic solids, initially in the cubic sodium chloride. The purpose of this communication is to report the effect of a 10 kbar pressurisation on the dislocation substructure of mono- and bi-crystalline specimens.

The experiments were performed on specimens cleaved in {100} orientation from several single crystals and one bicrystal grown from Analar NaCl by the Czochralski method. The grain-boundary misorientation was 8°. The specimen dimensions were approximately 10 × 2 × 2 mm. The specimens were annealed at 650° C to remove internal stresses produced by cleavage and then cooled to room temperature over a period of 35 h. All specimens were then polished and etched and several areas of their surfaces photographed. The polishing solution was a mixture of 1 part methyl alcohol with 1 part ethyl alcohol and the etchant was 1 part methyl alcohol with 2 parts glacial acetic acid. The



(a) (b)

Figure 1 (a) An area of a {100} face of a NaCl single crystal in a slowly-cooled condition after etching. Note the presence of sub-grain-boundaries. (b) The same area photographed after a 5 minute pressurisation at 10 kbars, polishing (to remove 15μm off the surface) and etching. Note the precise correlation between the etch-pits and their position in comparison with (a).



(a) (b)

Figure 2 (a) An area about a tilt aboundary XY on the {100} faces of a NaCl bicrystal in a slowly-cooled condition after etching. (b) The same area photographed after a 5 minute pressurisation at 10 kbars, polishing (to remove 15μm off the surface) and etching. Note the dislocation distributions AB and CD and that the mean etch pit density has increased in comparison with (a).

specimens were then subjected to a pressure of 10 kbars for 5 min in a piston-cylinder apparatus in a mixture of normal and iso-pentane. Following pressurisation the specimens were cleaned, dried, repolished and etched.

All the observations on monocrystalline specimens, including those of areas adjoining sub-boundaries (misorientations of the order of 10 min) failed to reveal the presence of fresh dislocations. An example is presented in fig. 1 which shows an area of the {100} face before and after pressurisation at 10 kbar. The observations with the bicrystal specimens revealed that existing dislocations had moved and fresh dislocations had been generated during the pressurisation cycle. The number of dislocations in areas adjoining the grain-boundary was observed in some instances to increase and in others to decrease. The mean dislocation density, ρ, however, in all larger areas examined before and after pressurisation either was approximately the same or increased, indicating that in the entire volume of the bicrystalline specimen it had increased. Fig. 2 shows an area where ρ has increased from 2.5×10^6 cm^{-2} (a) to 3.4×10^6 cm^{-2} (b) due to the pressurisation at 10 kbar. It should be noted that fresh dislocations are present, particularly along AB and CD. To ascertain whether this was purely a surface phenomenon the specimen was repolished and etched several times after fig. 2b was taken. It was established that the pronounced distribution of etch-pits CD ($\sim$ 200 μm in length) persisted to a depth of $\sim$ 100 μm. The features of the area of fig. 2 after 100 μm had been removed resembled, in fact, fairly closely, those of the unpressurised surface region. It is of course impossible to know whether dislocation activity at that depth had taken place as there the unpressurised state could not be determined. (The features of the opposite face of the specimen were entirely different.)

The bicrystal from which the specimens were prepared contained <20 ppm of cation and anion impurity. No precipitates are thought to be present in crystals of this purity and none were detected. In the absence of even small inelastic discontinuities, other than the grain-boundary, the generation of fresh dislocations during pressurisation is tentatively attributed to the presence of the grain-boundary.

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References

1. S. V. RADCLIFFE and H. WARLIMONT, *Phys. Stat. Sol.* 7 (1964) K67.
2. A. BALL, F. P. BULLEN, and H. L. WAIN, *Mat. Sci. Eng.* 3 (1969) 283.
3. T. E. DAVIDSON, J. C. UY, and A. P. LEE, *Trans. AIME* 233 (1965) 820.
4. G. DAS and S. V. RADCLIFFE, *Phil. Mag.* **20** (1969) 589.

5. P. J. WORTHINGTON, *ibid* **19** (1969) 663.
6. H. G. MELLOR and A. S. WRONSKI, *Acta Met.* in the press.

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Influence of Cold-work on Subsequent Precipitation in an Al–20 wt% Ag Alloy

Precipitation in a supersaturated alloy is known to be altered considerably by a plastic deformation after solution-annealing and before ageing. This effect has been investigated particularly in several aluminium alloys by means of transmission electron microscopy (TEM) in the last few years (e.g. [1-5]). In the case of Al–Ag alloys however, no TEM-studies of precipitation after cold-work (especially heavy cold-work up to 99% thickness reduction) have been done to the authors' knowledge. Recently, Krause and Laird [6] examined an Al–15 wt % Ag alloy cold-rolled 50% and subsequently aged 1 day at 160° within the framework of an investigation of fatigue properties of aluminium alloys.

It is the purpose of this paper to present some results of an Al–20 wt % Ag alloy (prepared from 99.99% Al) in the following conditions: solution annealed 1 day 540° C (quenched in water of 15° C), then cold-worked by rolling with 10, 25, 50, 80, 90 or 99% thickness reduction and aged at 200, 250 or 300° C between 1 min and 1 day. Thin foils transparent for 100 kV electrons were prepared by the standard window technique using an electrolyte of 20% $HClO_4$ + 80% C_2H_5OH at 4° C and 17 V.

Without deformation, the decomposition follows the usual, well-known sequence [7] of spherical GP-zones, plate-like metastable γ' precipitates on $\{111\}$-matrix planes and the equilibrium phase γ, grown usually in a lamellar, discontinuous manner starting from grain-boundaries (fig. 1). However, according to Laird and Aaronson [8], γ may also grow continuously by a direct transition of γ' plates into the γ crystal structure by means of misfit dislocations. In this case, which was also observed in the present study in the case of undeformed specimens, the γ' and γ plates may not be distinguished easily.

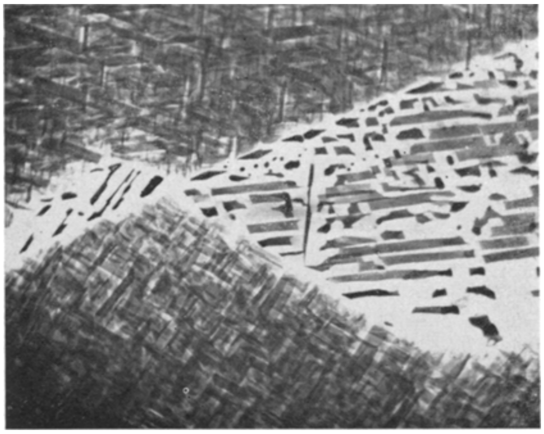

Figure 1 Al–20% Ag, 10 h 300° C. γ' in the grain interior, γ discontinuously grown from grain-boundaries (undeformed specimen) (× 2600).

After deformation in the solution-annealed state, the dislocation structure observed was similar to that described earlier [2] in the case of Al–4 wt % Cu, i.e., it consists of short, irregularly shaped dislocation lines and loops more or less randomly distributed after low or medium degrees of deformation (up to 50%). After higher deformation there develops a kind of cell structure, with "thick walls" (see fig. 1 in [1]). The cell diameter was 1 to 2 μm and the wall thickness about 0.5 μm.

The results of precipitation after deformation may be described as follows. After 10% cold-rolling, γ' formation was observed to be accelerated drastically on account of suppression of the GP-zones, in accordance with Murakami and Kawano [9] (mechanical, electrical and X-ray studies). This is due to the easier nucleation of γ' on dislocations introduced by the deformation. The γ' plates are much more numerous and much smaller than without de-