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### Influence of Hydrostatic Pressure of the Flow Stress in Polycrystalline NaCl

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#### ABSTRACT

By etch-pitting NaCl polycrystals which had been subjected to a hydrostatic pressure of 8 or 10 kbar it was established that dislocations are generated during the pressurization treatment. The effect of these freshly nucleated dislocations on the stress-strain relationship at atmospheric pressure is established and the relevance of these observations to the interpretations of experiments carried out under pressure is discussed.

Aladag, Davis and Gordon (1970) have recently reported that the yield stress of polycrystalline sodium chloride is reduced by about 20% if the specimen is tested under a hydrostatic pressure of 10 kbar. Their material was 'seasoned' at 10 kbar and then some specimens were compressed at atmospheric pressure whilst others were tested under pressure; results on unpressurized material were not reported. Aladag *et al.* attributed the difference in behaviour to increased ease of cross-slip at high pressure and did not consider the possibility of irreversible effects of the pressurization on the mechanical properties of sodium chloride. As solids with the cubic structure have isotropic linear compressibilities, local shear stresses should not be expected in the presence of grain boundaries on the application of hydrostatic pressure. It has, however, been demonstrated (Evans, Redfern and Wronski 1970) that dislocations are generated in the region of a tilt boundary in a bicrystal of NaCl subjected to a hydrostatic pressure of 10 kbar. It was accordingly decided to extend this investigation to NaCl polycrystals.

The specimens, of approximately square cross section ~10 mm high, were sectioned from polycrystalline AnalaR grade NaCl. Details of the experimental techniques have been previously reported (Evans *et al.* 1970). The total cation and anion impurity concentration was (20 p.p.m.), which precludes the presence of precipitates and pre-precipitates. The grain sizes were not uniform, the average grain diameters being ~1 mm. Pressurizations were carried out at 8 and 10 kbar and compression testing on a modified Hounsfield tensometer at strain rates of  $\sim 2 \times 10^{-4} \text{ sec}^{-1}$  at atmospheric temperature and pressure. Etch-pitting experiments

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revealed that dislocations were generated, sometimes in well-defined  $\langle 110 \rangle$  slip bands, near high angle boundaries whilst the specimens were subjected to the high hydrostatic pressure. Figure 1 illustrates a detail from a polycrystalline sample, where a  $\{100\}$  face has been etched before and after pressurization at 8 kbar. The edge dislocation etch-pit trace, AB, should be noted in particular; this motion arises, we suggest, from shear stresses developed from the grain boundary, XY.

Fig. 1

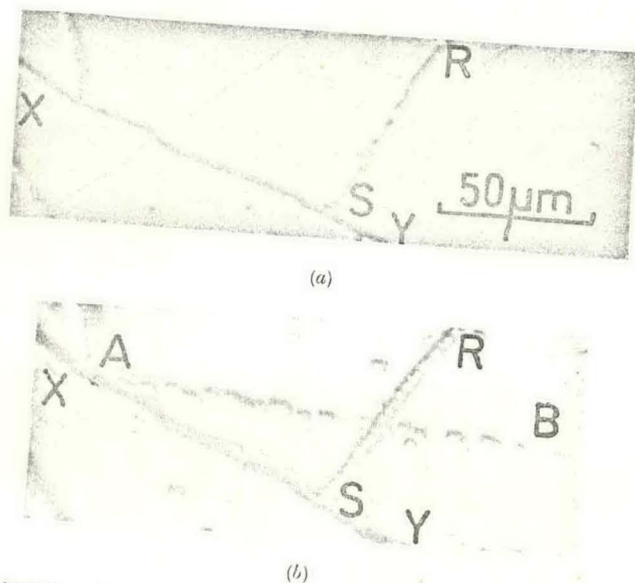


Fig. 1. A  $\{100\}$  face of a NaCl polycrystal (a) before and (b) after pressurization at 8 kbar. In (b) note the line of screw dislocation etch pits AB, in  $\langle 100 \rangle$ , associated with the grain boundary XSY following the pressurization and crossing the sub-boundary RS.

Such a generation and motion of dislocations betoken a significant shear stress operating on the  $\{110\}$  planes in the sample interior. This stress must inevitably be in excess of 5–10 bar, which is the range of experimentally observed, critically resolved shear stresses in single crystals of similar purity.

The authors consider that these features of the pressurization treatment of NaCl samples have some significance in the discussion of the results of Aladag *et al.* (1970) on polycrystalline NaCl. We suggest that their

'seasoning' treatment at 10 kbar, given to all polycrystalline samples, may have introduced and activated dislocation sources, on the evidence from our present work. Such a dislocation activity will have an influence on any subsequent deformation which the crystal is required to undergo, in particular if free dislocations are present in all grains. It is to be expected that the flow stress of an unpressurized polycrystalline sample would be higher than of samples tested either subsequent to pressurization or at pressure, because of the absence of activated dislocation sources. Difference in behaviour in the last two conditions (reported by Aladag *et al.*) can be accounted for by considering that plastic flow in samples tested at 10 kbar is supported not only by the applied stress but also the inherent shear stresses due to the hydrostatic pressure near the grain boundary dislocation sources. Although the samples tested at atmospheric pressure contain mobile dislocations introduced during 'seasoning', the plastic flow is in this case supported solely by the applied shear stress resolved onto the active slip planes. The 20% reduction in yield stress,  $\sigma_Y$ , observed by these authors, may represent the contribution from the additional pressurization shear stresses.

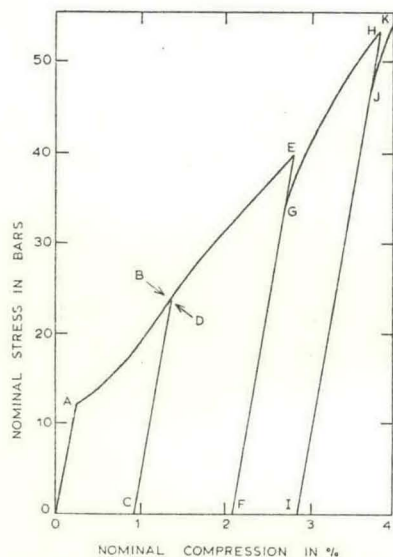
In our mechanical properties investigations the yield stress in precipitate-free single crystals was found to be insensitive to pressurization, which is in line with the lack of dislocation generation in single crystals subjected to hydrostatic pressure (Evans *et al.* 1970). Yield stresses of unpressurized and pressurized polycrystals were also determined, but the scatter in the values of the yield stress of unpressurized samples (11–16 bar) precludes for the time being a quantitative evaluation of the effects of pressurization on as-annealed specimens. The confining pressure, however, was reported by Aladag *et al.* (1970) to influence the entire stress-strain relationship of a polycrystal; we suggest, *inter alia*, through the generation of dislocations. A quantitative test of this hypothesis, therefore, is the pressurization and compression of pre-compressed polycrystals. If fresh dislocations are generated by the high pressure treatment, the stress to re-initiate macroscopic plastic flow should be smaller than the stress reached in the test on an unpressurized sample.

These experiments were carried out and, for 10 kbar pressurizations, reductions in the flow stress of  $\sim 4$  bar (20–40%  $\sigma_Y$ ) were observed (e.g. fig. 2). By carrying out unload-reload experiments it was established that a real effect of pressurization was being studied. Pressurization of strained pure monocrystalline specimens did not result in a discernible effect on the flow stress.

An estimate of the number of new dislocations contributing to plastic flow in a pressurized polycrystal can be made using a modification of the model Johnston and Gilman (1959) proposed for the deformation of LiF monocrystals. Hahn (1962) and Cottrell (1963) extended the theory to the deformation of polycrystals of body-centred cubic transition metals and Mellor and Wronski (1970 a) recently used the same semiquantitative approach to interpret pressurization effects in polycrystalline chromium.



Fig. 2



The initial part (OAB) of the nominal stress-compression curve of a NaCl polycrystal tested at room temperature at a rate of  $\sim 1.8 \times 10^{-4} \text{ sec}^{-1}$ . The specimen was then unloaded (BC), reloaded (CDE), unloaded (EF), pressurized at 10 kbar, reloaded (FGH), unloaded (HI), repressurized at 10 kbar and reloaded (IJK). All the compressions were carried out at atmospheric pressure.

All these workers incorporate the empirical formula for the mean dislocation velocity,

$$v = \left( \frac{\sigma}{\sigma_0} \right)^n \quad \dots \quad (1)$$

into the relation for plastic strain rate,

$$\dot{\epsilon} = \Phi b \rho_Y v_Y = \Phi b \rho_Y \left( \frac{\sigma_Y}{\sigma_0} \right)^n, \quad \dots \quad (2)$$

where  $\sigma$  is the tensile stress,  $\Phi$  an orientation factor (0.5),  $b$  the Burgers vector,  $\sigma_0$  and  $n$  constants and  $\rho_Y$ ,  $v_Y$  and  $\sigma_Y$  the values of, respectively, the mean dislocation density,  $\rho$ ,  $v$  and  $\sigma$  when macroscopic plastic flow commences. For the conditions:  $b \approx 4 \times 10^{-8} \text{ cm}$ ,  $\sigma_Y \approx 13 \text{ bar}$  ( $\sigma(A)$  of fig. 2),  $\dot{\epsilon} \approx 1.8 \times 10^{-4} \text{ sec}^{-1}$  and (Gutmanas, Nadgornyi and Stepanov 1963)  $\sigma_0 \approx 20 \text{ bar}$ ,  $n \approx 8$ ,  $\rho_Y$  and  $v_Y$  evaluate to  $\sim 3 \times 10^5 \text{ cm}^{-2}$  and  $\sim 3 \times 10^{-2} \text{ cm sec}^{-1}$ , respectively.

As macroscopic work hardening takes place at approximately constant strain rate (temperature and pressure), the product of the mobile dislocation density and their mean velocity must remain constant (Johnston and Gilman 1960, Mellor and Wronski 1970b). It has been observed by Johnston and Gilman (1960) in work hardened LiF crystals that the extra stress beyond  $\sigma_Y$  is just sufficient to maintain the same dislocation velocity as in the unstrained material. This behaviour has been assumed to be general (Hahn 1962, Cottrell 1963) and it follows therefore that during work hardening the mobile dislocation density and mean velocity remain approximately constant, and equal to  $\rho_Y$  and  $v_Y$ , respectively. The estimated value of  $\rho$  at E (as at B or H) of fig. 2 is therefore  $3 \times 10^5 \text{ cm}^{-2}$  (much lower than the total—mobile and immobile—dislocation density).

If fresh dislocations are generated during the pressurization treatment they may influence further deformation process both by moving to cause plastic flow and by acting as obstacles to gliding dislocations. Most of the dislocations present contribute to the latter process, but, in a work-hardened crystal, only a relatively small fraction (the free) take part in the former. It is therefore suggested that in a polycrystalline sample, where the grain boundaries act as specific obstacles to slip propagation, freshly nucleated dislocations, in the vicinities of these boundaries, would contribute principally to the former process—i.e. making slip easier. It will be assumed that the work-hardened structure opposing the movement of free dislocations is unaffected. Following pressurization the stress to reinitiate plastic flow (G of fig. 2) was reduced by  $\Delta\sigma \approx 4 \text{ bar}$ ;  $\sigma_Y - \Delta\sigma$  was therefore substituted for  $\sigma_Y$  into relation (2) and the new mobile dislocation density evaluated as  $\sim 5 \times 10^6 \text{ cm}^{-2}$ .

This result implies that probably  $> 10^7 \text{ cm}^{-2}$  dislocations are produced by pressurizing polycrystals of NaCl at 10 kbar (not all are expected to be mobile when a sample is reloaded), a figure an order of magnitude larger than our experiments with bicrystals (Evans *et al.* 1970) suggest. Bearing in mind the semi-quantitative nature of this analysis (Hahn 1962, Cottrell 1963, Mellor and Wronski 1970a) and that the dislocation velocity parameters,  $\sigma_0$  and  $n$ , for these boules of sodium chloride have not been determined, we suggest the agreement between the experimental results and the analysis is encouraging. The etch-pit densities in the strained polycrystals were too large for quantitative (metallographic) determination of the number of the pressurization-induced dislocations.

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