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Pressure and Strain Rate Dependence of Dynamic Recovery in NaCl

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Abstract

Crystals of NaCl have been deformed at room temperature in compression at pressures up to 10 kb at two different strain rates having a ratio of 24. At both strain rates, τ_{III} (the stress for the onset of stage III work hardening) decreases with pressure up to a pressure of about 5 kb, whereupon, within experimental error, no further change is observed. The decrease in τ_{III} (up to 5 kb) is more rapid at high strain rate ($d \ln \tau_{III}/dP \simeq -0{\cdot}34$/kb) than at low strain rate ($d \ln \tau_{III}/dP \simeq -0{\cdot}25$/kb) so the strain-rate sensitivity of τ_{III}, $(\partial \ln \tau_{III}/\partial \ln \dot{\epsilon})_{T,P}$, is decreased by about an order of magnitude between 1 atm and 5 kb.

Stage III work hardening in NaCl is believed to be controlled by the thermally activated, stress-assisted, cross-slipping of screw dislocations. The decrease of τ_{III} with pressure may be qualitatively associated with an increase in the stacking-fault energy γ, which is dependent on pressure through a strong dilatation of the lattice in the vicinity of the fault. From cross-slipping theory the dependence of the strain-rate sensitivity of τ_{III} on pressure may be calculated. The small increase predicted is, however, in clear disagreement with the present results.

§ 1. Introduction

The work hardening behaviour of NaCl has been examined in considerable detail by Hesse (1965) and Davidge and Pratt (1964). In particular, Hesse, has examined the dependence of the stress for stage III work hardening, τ_{III}, on strain rate and temperature and established a relation of the form

$$\ln(\tau_{III}/\tau_0) = (kT/A)\ln(\dot{\epsilon}/\dot{\epsilon}_0), \qquad (1)$$

where $\dot{\epsilon}$ is the strain rate, T the absolute temperature, k Boltzmann's constant, τ_0 and $\dot{\epsilon}_0$ are constants independent of T and $\dot{\epsilon}$ and A is a function of the stacking-fault energy, γ . Equation(1) derives from the cross-slip theory of Seeger, Berner and Wolf (1959) (SBW) and of Haasen (1958), leading Hesse to suggest that one may associate dynamic recovery in NaCl with the onset of thermally activated, stress-assisted cross slip of screw dislocations. Experimental support of the cross slip mechanism for stage III followed when the appearance of cross slip traces at the onset of stage III was observed in the electron microscope (by replication) by Matucha and Haasen (1967) and Matucha (1968). More recently Davis and Gordon (1969 a) and Aladag, Davis and Gordon (1970) have found that τ_{III} in NaCl is strongly dependent on pressure. According to a qualitative application of the SBW cross slip theory, this is associated with an enhancement of the cross slip process at high pressure (Davis and Gordon 1969 a, b) through an increase of the stacking-fault energy, γ, with pressure. This latter proposal follows from the work of Fontaine (1968) who predicted that stacking faults in the alkali halides should cause a strong local dilatation of the lattice ($\epsilon_0 = \delta d_{110}/d_{110} \simeq 0{\cdot}3$) and thus γ should be sensitive to pressure. Subsequently Haasen, Davis, Aladag and Gordon (1970) found that using the SBW theory, and incorporating the pressure dependence of γ according to Fontaine and Haasen (1969), one can predict a value of $(d \ln \tau_{III}/dP)_{1\,atm}$ which is in fair agreement with previous experimental data. This suggests that the influence of pressure on stage III in NaCl may be described in some detail by the SBW theory. It is of interest, therefore, to pursue this subject further by examining the decrease of τ_{III} with pressure as a function of strain rate so that comparison may be made with predictions of the theory. This may be done conveniently by comparing τ–ϵ curves for two significantly different strain rates.

§ 2. Experimental

The mechanical testing device (minitester) employed has been described in detail by Gordon and Mike (1967) and Davis and Gordon (1968). The drive components of the system consist of an 1800 r.p.m. syncronous motor, a planetary gear system, and 0·3175 cm pitch drive screw. By adjusting the gear ratio one can achieve different compression rates. Here we have employed gear trains of 60 000 : 1 and 2500 : 1 reduction for compression rates of 0·00952 cm/min and 0·229 cm/min respectively, i.e. a strain-rate ratio of 24. The minitester is placed inside a pressure vessel and pressure generated by a 200 000 p.s.i. capacity Harwood system; pressure is monitored by a manganin cell. Samples are tested in pentane, with a trace of oil for lubrication of moving parts. The use of a low viscosity fluid such as pentane is essential to minimize the viscous drag on the motor at high pressure. It is found that no decrease in motor speed obtains to pressures of about 8·5 kb; at 10 kb a decrease of up to 20% is possible. For the strain-rate ratio employed here a 20% change from nominal speed is negligible.

A large batch of annealed and cleaved samples was obtained from the Harshaw Chemical Co., with nominal dimensions of $1{\cdot}27 \times 0{\cdot}635 \times 0{\cdot}635$ cm. The sample ends are lapped flat and parallel in a vee-block and the sample is tested in compression at room temperature. The compression platens are lubricated with oil or a PTFE sheet. The top platen has a hemispherical head which bears on the load cell and is free to rotate about the bearing point. This facilitates alignment of the system and approximates the condition of laterally free platens. Specimens deform primarily on one family of parallel $\{110\}\ \langle\bar{1}10\rangle$ slip systems. For the specimen shape employed here one finds the nominal shear strain is approximately four times the compressive strain. Thus, for the compression rates noted above the shear strain rates are $\dot{\epsilon} = 5 \times 10^{-4}$/sec and $\dot{\epsilon} = 1{\cdot}2 \times 10^{-2}$/sec. At a compression of about 20% (0·254 cm) the sample ends remain parallel to within about 0·018 cm. Some difficulty is encountered with specimens which barrel excessively by slipping on two sets of orthogonal slip planes or which distort by slipping on oblique {110} slip planes. This is easily noted by the appearance of the crystal after deformation; τ–ϵ curves in these cases show excessive work hardening rates and are omitted from these results.

In the work of Aladag *et al.* (1970) (ADG) on NaCl a variation of $d\tau_{III}/dP$ (at $\dot{\epsilon} \simeq 5 \times 10^{-4}$/sec) for different sets of crystals was noted. It is necessary, therefore, to establish the pressure behaviour of the τ–ϵ curves at the low strain rate for the present material in order to be certain of a valid comparison with new data at the high strain rate. We note also that here τ_{III} is defined, following Hesse (1965), as the stress at which the τ–ϵ curve deviates by 1% (in τ) from the linear extrapolation of stage II. ADG employed the technique discussed by Haasen (1958) where the intersection of straight lines drawn through stages II and III is denoted as τ_{III}. The latter technique appears less certain and is abandoned here.

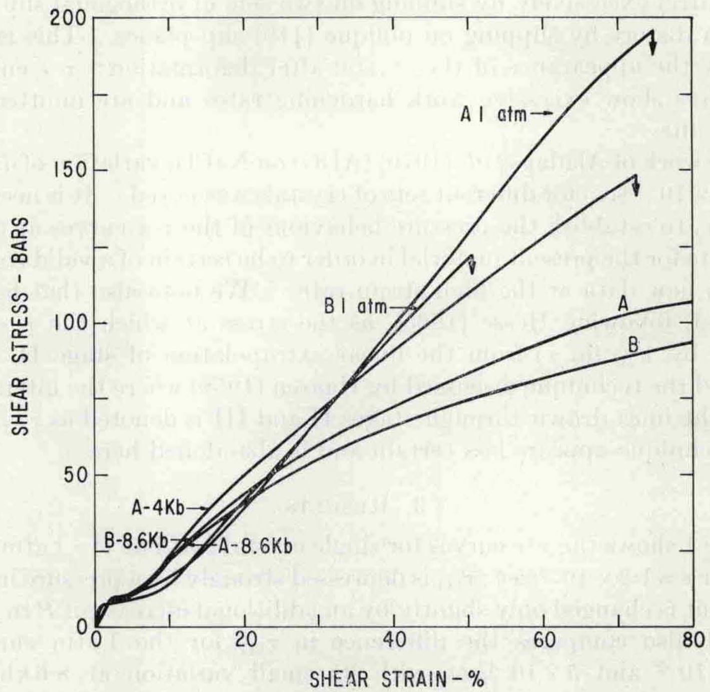
§ 3. Results

Figure 1 shows the τ–ϵ curves for single crystal NaCl at $P = 1$ atm, 4 and 8·6 kb for $\dot{\epsilon} = 1{\cdot}2 \times 10^{-2}$/sec; τ_{III} is depressed strongly by a pressure increase of 4 kb but is changed only slightly by an additional increase of P to 8·6 kb. Figure 1 also compares the difference in τ_{III} for the 1 atm curves at $\dot{\epsilon} = 1{\cdot}2 \times 10^{-2}$ and 5×10^{-4}/sec with its small variation at 8·6 kb. For these curves $\tau_s = \tau_c/2$ and $\epsilon_s = 4\epsilon_c$, where s and c refer to shear and compression.

Considering the data in more detail, it is found that τ_I, the extrapolated critical resolved shear stress, is independent of P and $\dot{\epsilon}$ within experimental scatter; at $\dot{\epsilon} = 5 \times 10^{-4}$/sec, $\tau_I = 8{\cdot}0 \pm 1{\cdot}2$ bars and at $\dot{\epsilon} = 1{\cdot}2 \times 10^{-2}$/sec, $\tau_I = 8{\cdot}7 \pm 1{\cdot}4$ bars for the range 0–10 kb, where the limits represent the standard deviation. (In order to obtain an accurate estimate of the pressure dependence of τ in stage I it is necessary to perform pressure cycling tests (Davis and Gordon 1968).) At the low strain rate, the stress for initiation of stage II, τ_{II}, is roughly independent of pressure ($\tau_{II} = 12{\cdot}4 \pm 1{\cdot}7$ bars); at $\dot{\epsilon} = 1{\cdot}2 \times 10^{-2}$/sec, τ_{II} averages $13 \pm 1{\cdot}8$ bars at 2 kb and above but appears to be slightly greater at 1 atm ($16{\cdot}6 \pm 2{\cdot}1$ bars) where the transition from stage I to stage II is very gradual.

The scatter in the data for θ_I and θ_{II}, the slopes of stages I and II, respectively, is quite considerable, but in each case trends are apparent which may be characterized by a least squares, straight line fit. For $\dot{\epsilon} = 5 \times 10^{-4}$/sec $\theta_I = 1{\cdot}36P + 71$ (19) and $\theta_{II} = -10P + 306$ (32), while at $\dot{\epsilon} = 1{\cdot}2 \times 10^{-2}$/sec, $\theta_I = 3{\cdot}2P + 76$ (20) and $\theta_{II} = -9{\cdot}4P + 316$ (54), where θ is given in bars/unit shear strain when P is in kilobars; numbers in parentheses are the square root of the error mean square for each equation and indicate the magnitude of the scatter of the data points about the fitted straight line. At high pressure, then, θ_I increases slightly, while θ_{II} decreases by about $\frac{1}{3}$ between 0 and 10 kb.

Fig. 1



$\tau-\epsilon$ curves for 1 atm, 4 and 8·6 kb for $\dot{\epsilon}=1{\cdot}2\times10^{-2}$ sec^{-1} (marked A) and 1 atm and 8·6 kb for $\dot{\epsilon}=5\times10^{-4}$ sec^{-1} (marked B); position of horizontal arrows indicates τ_{III}. Curves at 1 atm and 4 kb terminated by fracture, those at 8·68 kb did not. The strain to fracture at 1 atm scatters over a considerable range independent of $\dot{\epsilon}$. Curves at 8·6 kb are typical also of those run at 6·9 and 10 kb.

Figures 2, 3 and 4 indicate the dependence on P and $\dot{\epsilon}$ of τ_{III}, or the stress for initiation of stage III, $(\epsilon_{\mathrm{III}}-\epsilon_{\mathrm{II}})$, or the range of stage II, and ϵ_{II}, or the range of stage I, respectively. In each case it is noted that the stress or strain variable decreases with P, the decrease being more rapid at the higher strain rate. A least squares analysis of the data between 1 atm and 4 kb of figs. 2 and 3 yields

$$(\partial \ln \tau_{\mathrm{III}}/\partial P)\simeq -0{\cdot}34/\mathrm{kb}$$

and

$$(\partial \ln (\epsilon_{\mathrm{III}}-\epsilon_{\mathrm{II}})/\partial P)\simeq -0{\cdot}31/\mathrm{kb}$$

for

$$\dot{\epsilon}=1{\cdot}2\times10^{-2}/\mathrm{sec}$$

and

$$(\partial \ln \tau_{\mathrm{III}}/\partial P)\simeq -0{\cdot}25/\mathrm{kb}$$

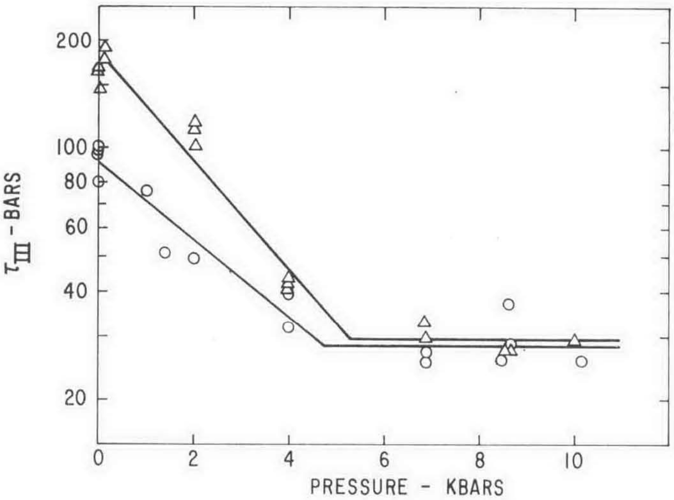
and

$$(\partial \ln (\epsilon_{\mathrm{III}}-\epsilon_{\mathrm{II}})/\partial P)\simeq -0{\cdot}26/\mathrm{kb}$$

for

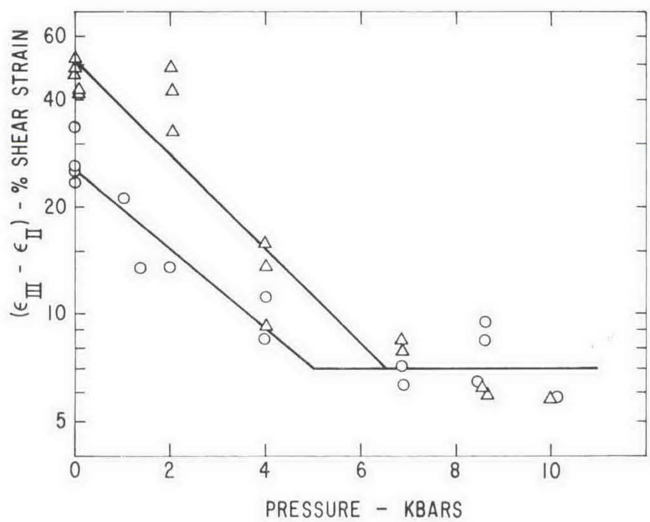
$$\dot{\epsilon}=5\times10^{-4}/\mathrm{sec}.$$

Fig. 2



Dependence of the stress for initiation of stage III, τ_{III}, on pressure; circles refer to $\dot{\epsilon}=5\times10^{-4}\,\mathrm{sec}^{-1}$ and triangles to $\dot{\epsilon}=1{\cdot}2\times10^{-2}\,\mathrm{sec}^{-1}$. The straight lines between 1 atm and 4 kb are fitted by least squares; the slopes are given in the text.

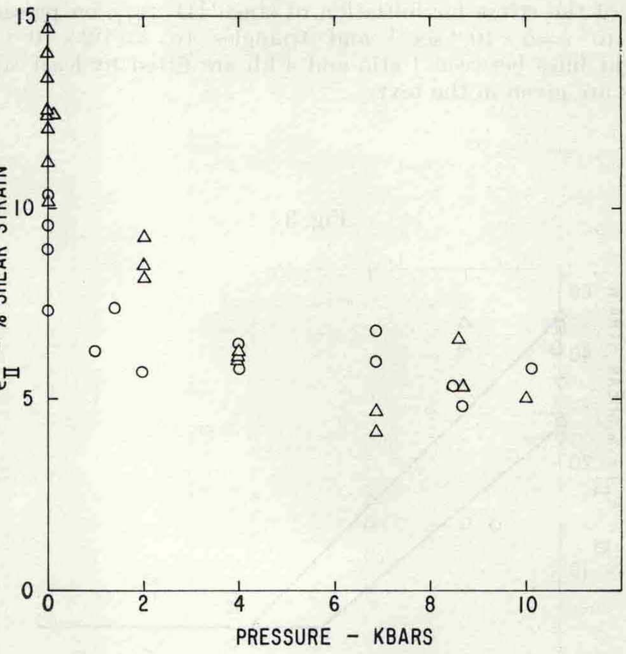
Fig. 3



Dependence of the range of stage II, $(\dot{\epsilon}_{\mathrm{III}}-\epsilon_{\mathrm{III}})$, on pressure, where symbols are as for fig. 2. The sloping lines are fitted by least squares; above 6 kb a simple means of all the data points is plotted.

In figs. 2 and 3 it also is apparent that the effect on P of the initiation of stage III saturates in the vicinity of 5 kb for both strain rates. According to our qualitative association of decreasing τ_{III} with increasing γ, noted in the introduction, it would follow that either the increase of γ saturates or the stacking-fault width becomes so narrow it is no longer sensitive to increasing γ. In fig. 2, which is of most interest, the horizontal lines represent the mean values (from 5 to 10 kb) of τ_{III} at each strain rate. It is also readily apparent from fig. 2 that the strain rate sensitivity of τ_{III} decreases greatly with increase in pressure. Extracting points from the solid lines shown one finds that $(\partial \ln \tau_{III}/\partial \ln \dot{\epsilon}) = 0{\cdot}22$ at 1 atm (in good agreement with the 1 atm data of Hesse) then decreases to 0·099 at 4 kb and finally becomes roughly constant at 0·01 between 5 and 10 kb. The last value is probably only reliable within a factor of 2 or 3. A far greater number of samples would have to be tested to fix it more accurately. It is clear, however, that a strong decrease of $(\partial \ln \tau_{III}/\partial \ln \dot{\epsilon})$ with pressure is well established.

Fig. 4



Dependence of the range of stage I, ϵ_{II}, on pressure; circles are for $\dot{\epsilon} = 5 \times 10^{-4}$ sec^{-1} and triangles for $\dot{\epsilon} = 1{\cdot}2 \times 10^{-2}$ sec^{-1}.

On comparison of the present τ–ϵ curves with those reported by Aladag *et al.* reasonable agreement is found. The stronger dependence of τ_{III} and $(\epsilon_{III} - \epsilon_{II})$ on P noted here is, in considerable measure, due to the 1%

offset method of computing τ_{III} adopted. Using the straight line intersection technique leads to larger values of τ_{III}, especially at high pressure where the rate of work hardening in stage III is reduced.

§ 4. Discussion

Except for the moderate decrease of θ_{II}, the influence of pressure on stages I and II of deformation observed is consistent with previous work (Davis and Gordon 1968, 1969 a) and thus will not be considered further. Based on the increase in elastic constants with pressure θ_{II} would be expected to increase slightly; apparently some other parameter, such as the slip distance, is mildly P sensitive.

Turning to stage III the parameter A in the cross slip equation (eqn. (1)), as derived by Wolf (1960) for the slip geometry of f.c.c. metals, is given by

$$A=(0\cdot352\,Gb^3)/\{(1+n/900)(1+180\gamma/Gb)\}, \quad . \quad . \quad . \quad . \quad (2)$$

where G is the shear modulus, b the Burgers vector and n the number of dislocations in a pile-up. As discussed by Thornton, Mitchell and Hirsch (1962) eqn. (2) is strictly valid only for $Gb^3/A \simeq 4$ to 7; for NaCl $Gb^3/A \simeq 47$. Thus numerically and due to differing slip geometry eqn. (2) is not appropriate for NaCl. However, to compute parameters of interest here one may simplify the expression for A to

$$A=G^2b^4/\beta\gamma, \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (3)$$

where β is an unspecified parameter (Haasen 1965). Using Fontaine's (1968) calculation for γ (195 ergs/cm^2) and Hesse's data for A to calculate β one finds $(\partial \ln \tau_{III}/\partial P)_{1\,\mathrm{atm}} \simeq -0\cdot02\,\mathrm{kb}^{-1}$ for $\dot\epsilon \sim 10^{-4}$/sec (Haasen *et al.* 1970). On comparison of this result with the present data ($\partial \ln \tau_{III}/\partial P \simeq -0\cdot25$/kb, fig. 2) the relatively poor agreement is apparent.

Combining eqns. (1) and (3) one may derive an expression for the strain-rate sensitivity of τ_{III} given by

$$(\partial \ln \tau_{III}/\partial \ln \dot\epsilon)=\beta\gamma kT/G^2b^4. \quad . \quad . \quad . \quad . \quad . \quad . \quad (4)$$

Then

$$\partial(\partial \ln \tau_{III}/\partial \ln \dot\epsilon)/\partial P = (\beta\gamma kT/G^2b^4)\{\partial \ln \gamma/\partial P - 2\partial \ln G/\partial P - 4\partial \ln b/\partial P\}, \quad (5)$$

assuming β independent of P. Setting the strain-rate sensitivity equal to Z one has for the relative change of Z with P,

$$(\partial \ln Z/\partial P)_{1\,\mathrm{atm}}=\{\partial \ln \gamma/\partial P - 2\partial \ln G/\partial P - 4\partial \ln b/\partial P\}_{1\,\mathrm{atm}}. \quad (6)$$

Inserting appropriate values for the derivatives: $\partial \ln \gamma/\partial P \sim 0\cdot028\,\mathrm{kb}^{-1}$ (Fontaine and Haasen 1969), $\partial \ln K_s/\partial P \simeq 0\cdot0147\,\mathrm{kb}^{-1}$ (inserting the more appropriate screw dislocation stress field elastic constant, K_s, for G) and $\partial \ln b/\partial P \sim -0{\cdot}0014\,\mathrm{kb}^{-1}$ (Davis and Gordon 1968) one finds

$$(\partial \ln Z/\partial P)_{1\,\mathrm{atm}} \sim +0{\cdot}005\,\mathrm{kb}^{-1};$$

the value computed from fig. 2, between 1 atm and 4 kb, is

$$-\ln(0{\cdot}22/0{\cdot}099)/4 \simeq -0{\cdot}2\,\mathrm{kb}^{-1},$$

i.e. the SBW theory predicts a small increase of strain-rate sensitivity of τ_{III} with pressure, while experimentally a very strong decrease is observed. This considerable discrepancy cannot be eliminated by any simple manipulation, e.g. if β is allowed to change with P in eqn. (5) to account for the observed large decrease of Z, then this simultaneously leads to the inadmissable requirement that τ_{III} must increase with P.

It is apparent, therefore, that a straightforward application of the SBW theory to the present data is not possible. As discussed by Aladag *et al.*, the only apparent alternative explanation for the reduction of τ_{III} with pressure requires that τ_{100}, the stress for motion of dislocations on the (100) plane, must decrease with pressure. This is theoretically unattractive because it requires a negative activation volume. In fact, it is now experimentally established (Davis and Gordon 1970, unpublished data) that pressure has no significant effect on the flow stress or work hardening of NaCl crystals oriented for (100) slip (compressed parallel to ⟨111⟩). Hence, our qualitative association of the decrease in τ_{III} with the enhanced recombination of dilated stacking faults apparently remains reasonable.

It is of interest, then, to consider the source of the discrepancy between theory and experiment further. Kocks, Chen, Rigney and Schaefer (1966) have indicated the difficulties encountered in establishing accurate τ_{III} values. In consideration of this Mecking and Lücke (1969) have proposed a method of analysis which uses the whole τ–ϵ curve rather than just τ_{III} to characterize dynamic recovery. In the present case, however, the change of strain rate sensitivity of τ_{III} with pressure is much too large to be attributed to any uncertainty in analysis. Turning to the theory, if eqns. (4) to (6) are taken as fundamentally correct and we expect correspondence between theory and experiment, it appears necessary to insert for τ_{III} in eqn. (1) some effective stress τ_{III}^*, rather than the applied stress. Gupta and Li (1970) have shown that the effective stress τ^* is a small portion of the applied stress in the work hardening of NaCl; if we may assume that τ_{III}^* is similarly small relative to τ_{III} it follows that a large change of τ_{III} would be required to produce a small change of τ_{III}^*. Similarly $(\partial \ln \tau_{\mathrm{III}}^*/\partial \ln \dot{\epsilon})$ could be relatively unchanged by pressure even though $(\partial \ln \tau_{\mathrm{III}}/\partial \ln \dot{\epsilon})$ decreases sharply. If τ_{III}^* should replace τ_{III} it follows that the calculation of Haasen *et al.* for $(\partial \ln \tau_{\mathrm{III}}/\partial P)_{1\,\mathrm{atm}}$, which requires the data of Hesse for τ_{III}, applied, versus T and $\dot{\epsilon}$, is questionable. Equation (6), however, does not require Hesse's data and thus could be a valid prediction of the relative change of $(\partial \ln \tau_{\mathrm{III}}^*/\partial \ln \dot{\epsilon})$ with pressure.

Alternatively, some other modification of the SBW theory may be required. For example, Peissker (1965) has suggested that the rate 'constant' $\dot{\epsilon}_0$ in eqn. (1) may be stress dependent, i.e. the density of potential cross slip sites $=f(\tau)$. Following Peissker's approach, $\dot{\epsilon}_0 \propto \tau_{III}{}^m$ and $2 \geqslant m \geqslant 0$. Equation (4) then becomes

$$Z = (\beta\gamma kT/G^2b^4)(1 + m\beta\gamma kT/G^2b^4)^{-1}. \quad . \quad . \quad . \quad . \quad . \quad . \quad (7)$$

If we denote the right-hand side of eqn. (6) as S and assume m is independent of pressure, from eqn. (7) we have

$$(\partial \ln Z/\partial P) = S - mZS. \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (8)$$

With $m=2$, $Z_{1\,\text{atm}} = 0{\cdot}22$, and $S = 0{\cdot}005$/kb (eqn. (6)), eqn. (8) yields $(\partial \ln Z/\partial P)_{1\,\text{atm}} \simeq +0{\cdot}003$/kb, i.e. the result of eqn. (6) is modified only slightly. If, in eqn. (8), $m \simeq 200$ we find $(\partial \ln Z/\partial P)_{1\,\text{atm}} \simeq -0{\cdot}2\,\text{kb}^{-1}$, in agreement with our experimental results. Such a large value of m is physically unreasonable, however, because it simultaneously requires that the parameter A in eqn. (1) be negative. In addition to this serious complication we can show that $(\partial \ln \tau_{III}/\partial P)$ is not a function of m. Hence even on introducing $\dot{\epsilon}_0 \propto \tau_{III}{}^m$ we cannot thereby rationalize the rapid decrease of τ_{III} with pressure. It is uncertain how else the SBW theory might be modified to explain the present data.

The difficulties encountered here in the application of the SBW theory to pressure effects in NaCl have a parallel in the case of some f.c.c. alloys. Gallagher (1968) and Gallagher and Liu (1969) have examined the change of γ on addition of Zn to Ag as determined from extended node measurements and as deduced from the SBW theory. In the latter case values of γ are calculated from the strain rate sensitivity of τ_{III}; in the above we have simply followed the inverse procedure of calculating $(\partial \ln \tau_{III}/\partial \ln \dot{\epsilon})$ versus P given a predicted change of γ with pressure. For Ag–Zn, extended node measurements indicate a smooth decrease of γ with increasing electron to atom (e/a) ratio; the τ_{III} method indicates an increase in γ (by a factor of 2) up to $e/a \simeq 1{\cdot}02$ followed by a decrease at larger e/a. That is, while γ is actually decreasing, the strain rate sensitivity of τ_{III} (and, according to eqn. (4), γ) is increasing. Gallagher and Liu conclude that the SBW theory does not yield a satisfactory quantitative explanation of dynamic recovery in these f.c.c. alloys; the present results indicate that the same is also true for NaCl.

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