Plastic deformation of NaCl single crystal with superposition of ultrasonic oscillatory stress

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A variation of deformation current as well as a reduction in static flow stress has been observed when an ultrasonic oscillatory stress was superimposed during the plastic deformation of NaCl single crystals. It is found that the activation volume can be estimated by two methods using the deformation current: one is from the stress amplitude and the other from the stress decrement at the removal of the oscillation. Good agreement has been obtained between experiment and theory by fitting these activation volumes to that obtained by the strain rate change test method. The dislocation velocity is found to increase with the aid of the oscillatory stress during the stress relaxation period.

1. Introduction

It is known that the static flow stress decreases when ultrasonic oscillatory stress is superimposed during plastic deformation of metals. This phenomenon was found by Blaha and Langenecker [1] when an oscillation of 800 kHz was superimposed during the deformation of Zn single crystals. Therefore it has been called the Blaha effect and has been widely applied for industrial purposes, e.g. wire drawing or rolling [2, 3]. In spite of a general tendency to consider that the reduction in flow stress is due to a temperature rise of the material during deformation [4], to an abrupt increase in mobile dislocation density [5], or to a reduction in internal stress [6, 7], the Blaha effect has been interpreted on the basis of stress superimposition [1–14].

In this paper, we have observed the Blaha effect as well as the deformation current of NaCl single crystals at the reduction in flow stress. We attempt to interpret the following experimental results by the above mentioned superimposition theory.

2. Experimental procedure

Two kinds of NaCl single crystals, one pure and the other doped with 0.1 mol% $CaCl_2$, were grown by the Kyropoulos method in air. The ingots were cleaved into 4 mm x 7 mm x 14 mm pieces and annealed at 740° C for 24 h in N_2 gas followed by cooling to room temperature at the rate of 40° C h^{-1}. The initial dislocation density of the annealed specimens, counted after they were etched in acetone and dried, was about 3×10^4 cm^{-2} and distributed randomly. A resonator with a frequeny of 42 kHz was connected to the testing machine, a Shimadzu DSS-500. The specimens were compressed with a strain rate of 2.38×10^{-5} sec^{-1} and the oscillatory stress was applied in the same direction as the compression. The size of the specimen was not in resonance with the applied frequency and the length of the specimen was about ten times less than the wavelength of the applied frequency. Therefore, the oscillatory stress was homogeneous throughout the specimen.

The wider pair of the surfaces of a specimen, which were parallel to the compression axis, were turned into electrodes by painting with silver paste in order to measure the electric current during deformation. The outline is shown in Fig. 1. The electrodes were connected to an electrometer (Takeda-Riken TR-8651), the response time of which is less than 0.5 sec. The effect of the response time can be neglected in this test, because the time of variation was much longer than the

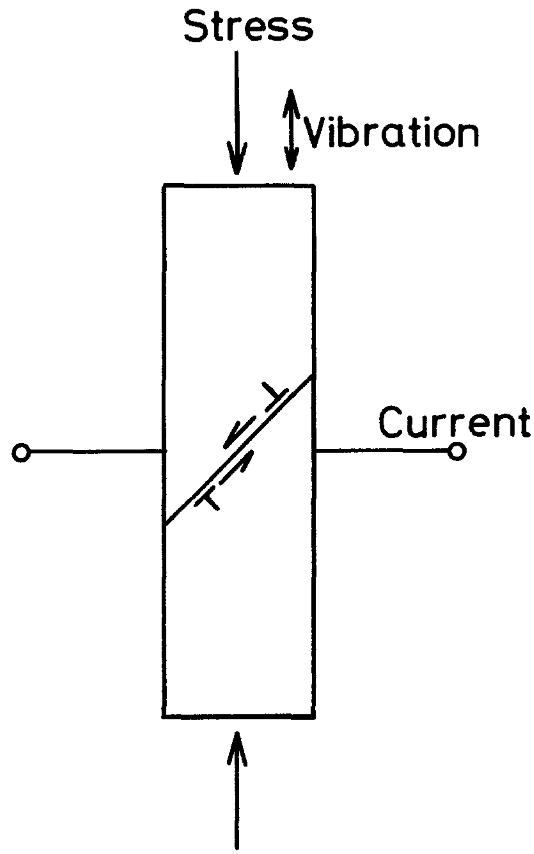


Figure 1 Specimens under the compressive stress combined by the oscillatory stress.

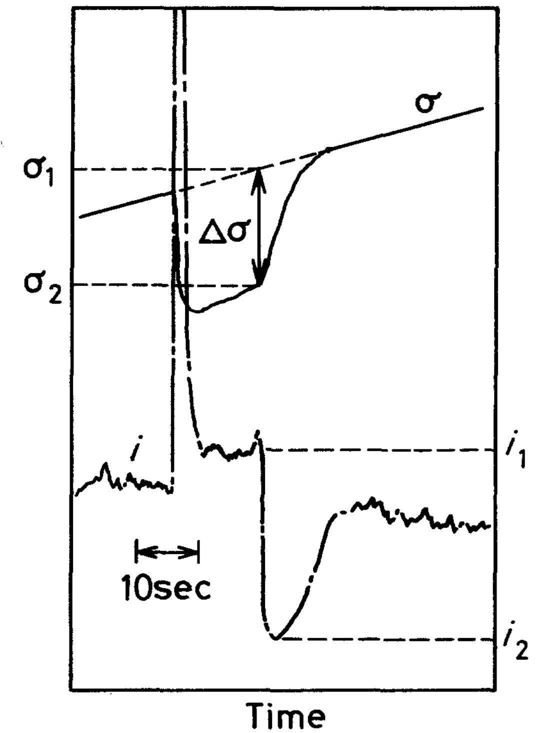


Figure 2 Variation of the stress decrement $\Delta\sigma$ and the deformation current i under the superimposition of ultrasonic oscillatory stress.

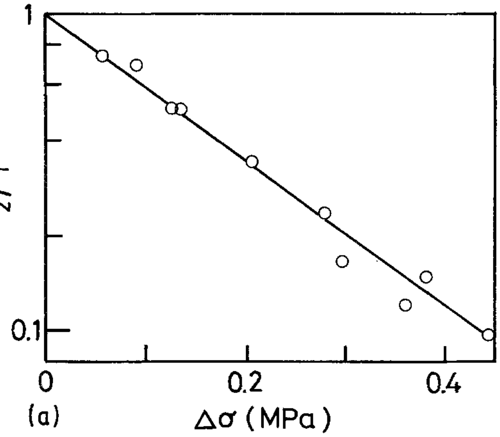
response time. It should be mentioned that such electric current carried by charged dislocations is usually measured by the introduction of asymmetry into the deformation system [15]. But, even in a symmetric system shown in Fig. 1, a little asymmetry should exist. And, if we are not concerned with the sign, the current carried by dislocations will be detected, as has been observed in the stress relaxation experiment reported by Ohring *et al.* [16]. As for the magnitude of the current, it changes somewhat depending on the conditions of the experiments, but the linear relation between the logarithm of the ratio of the current immediately before and after the oscillator is turned off, $\ln (i_2/i_1)$, and the stress decrement, $\Delta\sigma$, always holds. (See below.)

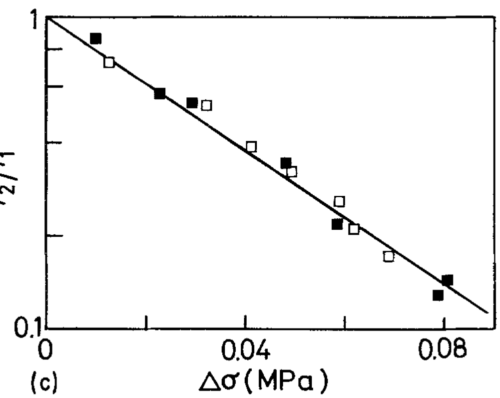
The vibrational strain amplitude was measured through the output voltage from the bridge containing a semiconductor strain gauge stuck to the specimens. All the experiments were carried out at room temperature. The temperature rise of the specimen due to the superimposition of ultrasonic oscillation was so small, less than 1° C, that the influence of temperature could be neglected.

3. Results and discussion

3.1. Relation between stress decrement and deformation current

Fig. 2 shows the stress decrement, $\Delta\sigma$, and the variation in the deformation current, i, due to the charged dislocations when oscillating stress was superimposed during the plastic deformation of a NaCl single crystal. The $\Delta\sigma$ values can be arbitrarily chosen within 10% of the flow stress magnitude, σ. It can be seen that the stress decreases rapidly and the current increases the moment the oscillator was turned on, and the stress increases to an initial stationary level and the current dips down when the oscillator was turned off. These phenomena suggest that the plastic strain rate increases rapidly by the superimposition of ultrasonic oscillatory stress and decreases by removal of the oscillation. Assuming that the dislocation density is kept constant throughout the whole process, the velocity of dislocation motion changes directly by the superimposition





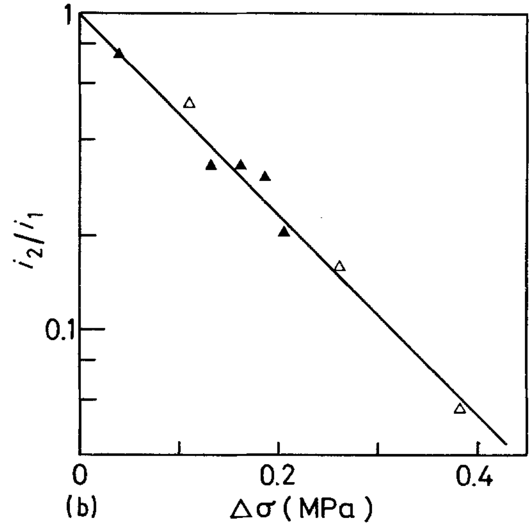


Figure 3 Relation between the i_2/i_1 and the $\Delta\sigma$ for pure NaCl single crystals. The logarithm of the i_2/i_1 is linear against $\Delta\sigma$. (a) At the flow stress of 6 MPa and the frequency of 42 kHz. (b) At the flow stress of 4 MPa and the frequency of 42 kHz (△), and 22 kHz (▲). (c) Vibration with the frequencies of 200 kHz (□) and 30 kHz (■) applied in the direction perpendicular to the compression.

of the ultrasonic stress, and it returns to the original stationary value when it is taken off. Here, we define the currents immediately before and after the oscillator being turned off as i_1 and i_2, respectively, as shown in Fig. 2. It should be mentioned that a similar result to Fig. 2 was obtained by using the switching-on process, but the experimental error was higher.

The relation between $\Delta\sigma$ and the ratio of i_2 to i_1 for the flow stress of 6 MPa at the frequency of 42 kHz is shown in Fig. 3a. The data were obtained without unloading and successively for the same specimen. It is shown that the logarithm of i_2/i_1 is linear to $\Delta\sigma$, though the $\Delta\sigma$ values have been chosen arbitrarily within 10% of the σ-values as has been mentioned. The slopes of the straight lines decrease with increasing strain. The single straight line shown in Fig. 3b shows that the plots of i_2/i_1 against $\Delta\sigma$ are the same for both 22 kHz and 42 kHz at a stress of 4 MPa, when tests were carried out for the same specimen in a narrow deformation range. Fig. 3c shows the special case when the oscillatory stress was superimposed perpendicular to the compression axis of the crystal. It is seen from the figure that the logarithms of the i_2/i_1 against $\Delta\sigma$ are the same for both frequencies of 200 and 30 kHz. Thus, it is understood that the ratio i_2/i_1 does not depend on the frequency superimposed, nor on the direction of vibrations, but depends on the $\Delta\sigma$ and the flow stress.

The relation between the i_2/i_1 and $\Delta\sigma$ is given by

$$i_2/i_1 = \exp(-\gamma\Delta\sigma), \qquad (1)$$

where γ is a constant which depends on the flow stress. It is known that the mobile dislocations carry electric charges, and the current due to these charges reveals plenty of information on the motion of dislocations [15].

Ohring *et al.* [16] have shown that the current density, i, is expressed by the formula

$$i = Afc\rho v \qquad (2)$$

where A is the density of charges per unit length of dislocation, f the net fraction of dislocations moving in the direction of observed current, c the magnitude of the average charge for the carrier, ρ the mobile dislocation density, and v the average velocity of dislocations. When a dislocation surmounts an obstacle with the aid of external shear stress and thermal fluctuations, the velocity of the dislocation is given by

$$v = s\nu \exp\left(-\frac{H_0 - \tau V}{kT}\right), \tag{3}$$

where s is the mean distance that dislocation moves between obstacles, ν the attempt frequency, H_0 the Helmholtz energy, V the activation volume, and kT has its usual meaning [17]. The dislocation velocity under the superimposition of the oscillatory stress with the amplitude τ_v is given by

$$V(t) = s\nu \exp\left[-\frac{H_0 - (\tau + q\tau_v \sin \omega t)V}{kT}\right] \mathrm{d}t \tag{4}$$

where t is time, q the effective function of oscillatory stress, and ω the angular frequency. The average dislocation velocity corresponding to the measured strain rate is obtained by taking an average of the dislocation velocity over a cycle,

$$\langle v_1 \rangle = \frac{\omega}{2\pi} \times \int_0^{2\pi/\omega} s\nu \exp\left[-\frac{H_0 - (\tau + q\tau_v \sin \omega t)V}{kT}\right] \mathrm{d}t \tag{5}$$

Assuming that the dislocation velocity immediately after the removal of the oscillatory stress, v_2, is given by

$$v_2 = s\nu \exp\left(-\frac{H_0 - \tau V}{kT}\right), \tag{6}$$

and using Equations 2, 5 and 6, we obtain

$$\frac{i_2}{i_1} = \frac{v_2}{\langle v_1 \rangle} = \left[\frac{\omega}{2\pi}\int_0^{2\pi/\omega} \exp\left(\frac{Vq\tau_v \sin \omega t}{kT}\right)\mathrm{d}t\right]^{-1} = \left[I_0\left(\frac{q\tau_v V}{kT}\right)\right]^{-1}, \tag{7}$$

where I_0 is the Bessel function of zeroth degree.

On the other hand, according to the stress superimposition mechanism [12, 13],

$$\frac{K\Delta\tau V}{kT} = P \ln I_0\left(\frac{q\tau_v V}{kT}\right), \tag{8}$$

where K, $0 \leqslant K \leqslant 1$, is the factor indicating the fraction of stress decrement to the effective stress. (For instance, the case $K = 0$ indicates that the stress decrement is due only to the decrement of the internal stress and not to the decrement of the effective stress. The situation is vice versa for $K = 1$.) P is the factor indicating the change in the mobile dislocation density and $\Delta\tau = \Delta\sigma/2$. Then, combining Equations 7 and 8, we obtain

$$\frac{i_2}{i_1} = \exp\left[\left(-\frac{K}{P}\right)\left(\frac{\Delta\tau V}{kT}\right)\right] \tag{9}$$

Equation 9 is found to correspond to Equation 1, and there is a relation between the constants, i.e.

$$\gamma = \frac{KV}{2PkT} \tag{10}$$

Thus, if we measure $\Delta\sigma$, τ_v, i_1, and i_2, we can obtain the activation volume by the two methods, corresponding to Equation 7 and Equation 9. Assuming $K/P = 1$, the activation volume can be obtained by inserting the slopes of the straight lines found in Figs. 3a, b and c into Equation 9. The results are 4.4×10^{-20}, 6.0×10^{-20}, and 2.0×10^{-19} cm^3, respectively. These values are not always equal to that obtained by the strain rate change test. We will see the details of the problem in the next section.

3.2. Dependence of stress decrement on stress amplitude

The dependences of the activation volume on the static flow stress obtained by means of the strain rate change test, Equation 10, and Equation 7 for pure NaCl single crystals are shown in Fig. 4, assuming K/P and q are unity. Since these activation volumes should be equal to one another, log–log plottings of the data between the activation volumes and the stress were arranged on a straight line by sliding the lines obtained by Equations 10 and 7 parallel to the ordinate until they corresponded to that obtained by the strain rate change test. The result is shown in Fig. 5, from which we obtain $K/P = 0.37$, and $q = 0.74$. Assuming that K is equal to unity, i.e. that $\Delta\sigma$ corresponds only to the decrement of the effective stress, we obtain $P = 2.7$. This means that the mobile dislocation density under the superimposition of the oscillatory stress is 2.7 times larger than that without the oscillation. On the contrary, assuming that P is equal to unity, i.e. that the mobile dislocation density under the superimposition of the oscillatory stress is equal to that without the oscillation, we obtain $K = 0.37$. This indicates that the effective stress decrement is equal to the fraction of $0.37\,\Delta\sigma$ and the other fraction of $\Delta\sigma$ is the internal stress decrement.

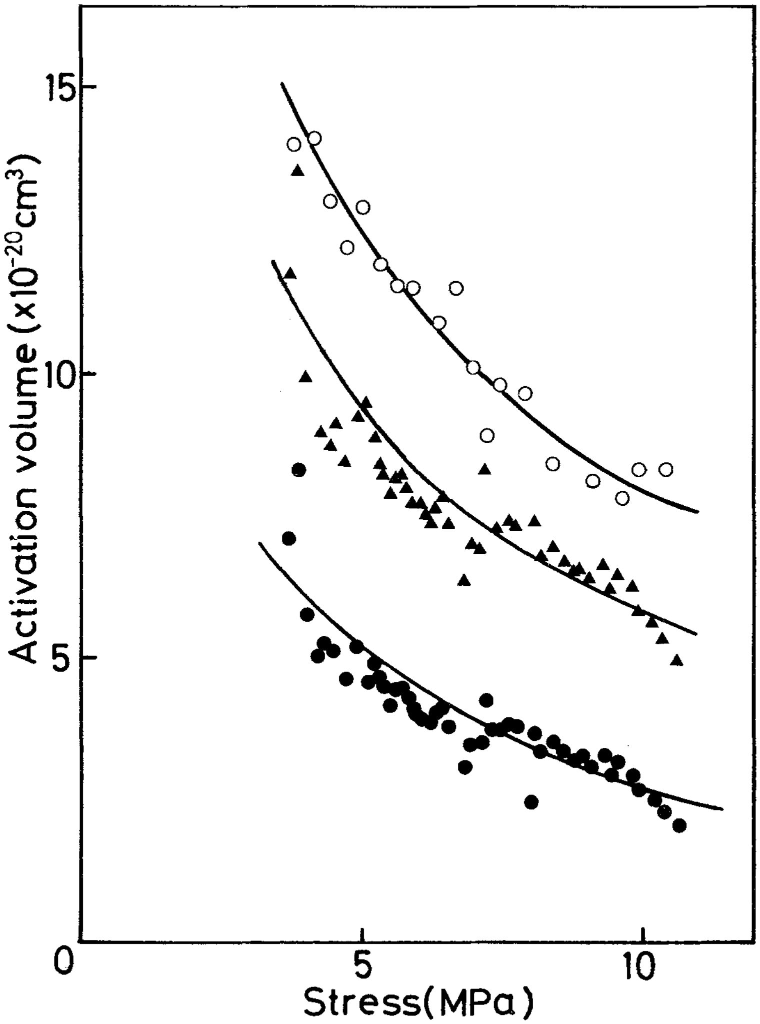


Figure 4 Activation volumes calculated by the three methods, the strain rate change test (○), Equation 9 (●), and Equation 7 (▲), respectively, assuming that $K/P = 1$.

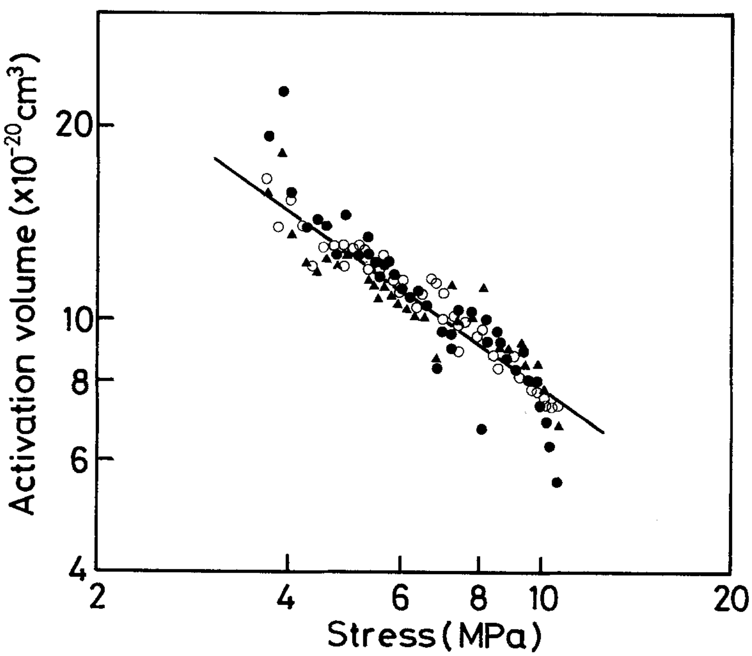


Figure 5 Superimposition of the three log V against log σ plots. These superimpositions give K/P and q, since all the three activation volumes should be the same.

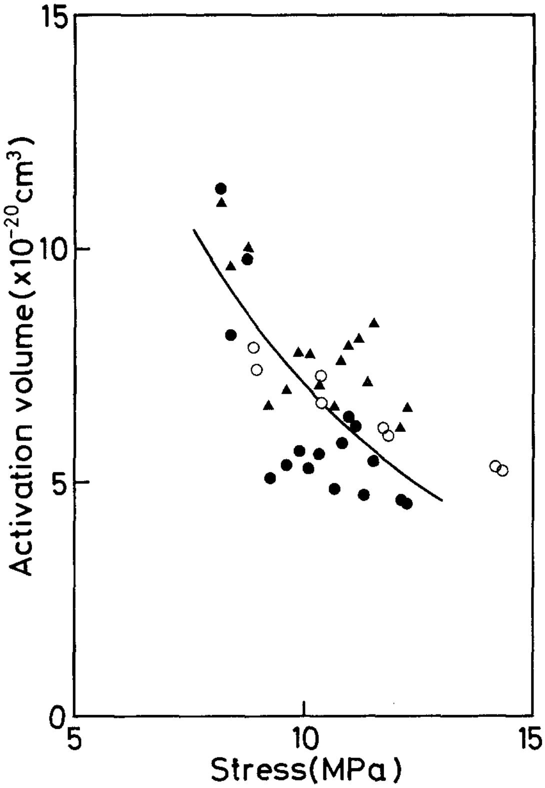


Figure 6 Activation volume for the NaCl: Ca (0.1 mol%) crystal. The symbols have the same meaning as in Fig. 5. The values are nearly equal to one another.

Fig. 6 shows the activation volumes for NaCl: Ca (0.1 mol%) obtained by the three methods similar to Fig. 4. These values are nearly equal to one another at the same flow stress. It seems that K/P and q are equal to unity. That is, the stress decrement corresponds only to the effective stress decrement and no change in the mobile dislocation density occurs. This indicates that the oscillating stress acts directly on the dislocation motion.

The calculated dependence of $\Delta\sigma$ on the oscillatory stress amplitude and on the activation volume by using Equation 8 is shown in Fig. 7 by a solid line. In this figure, the measured data for pure NaCl and NaCl: Ca single crystals are also plotted. The plots for pure NaCl are in good agreement with the calculated curve, but those for NaCl: Ca are not in good agreement. This suggests that K/P and/or q depends on the deformation of the NaCl: Ca single crystals.

3.3. Change in deformation current due to superimposition of oscillatory stress during stress relaxation

It is plausible that the strain rate increases under the superimposition of the oscillatory stress. The effect can be observed during the stress relaxation process. Fig. 8 shows that the deformation current increases by the superimposition of the ultrasonic

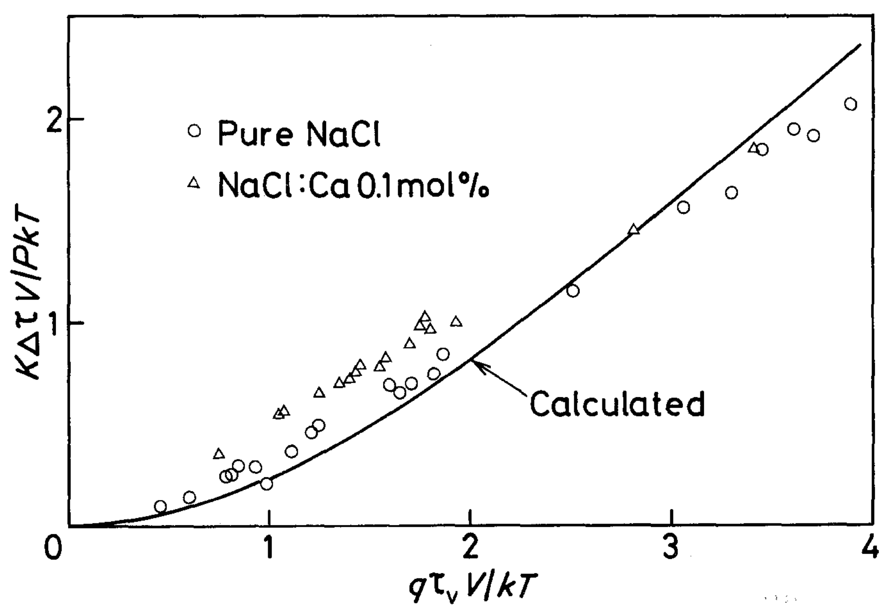


Figure 7 Comparison of experiments and theory. The plots for pure NaCl single crystal (○) is in good agreement with the calculated curve (solid line). The plots for NaCl:Ca (△) deviate a little from the calculated curve.

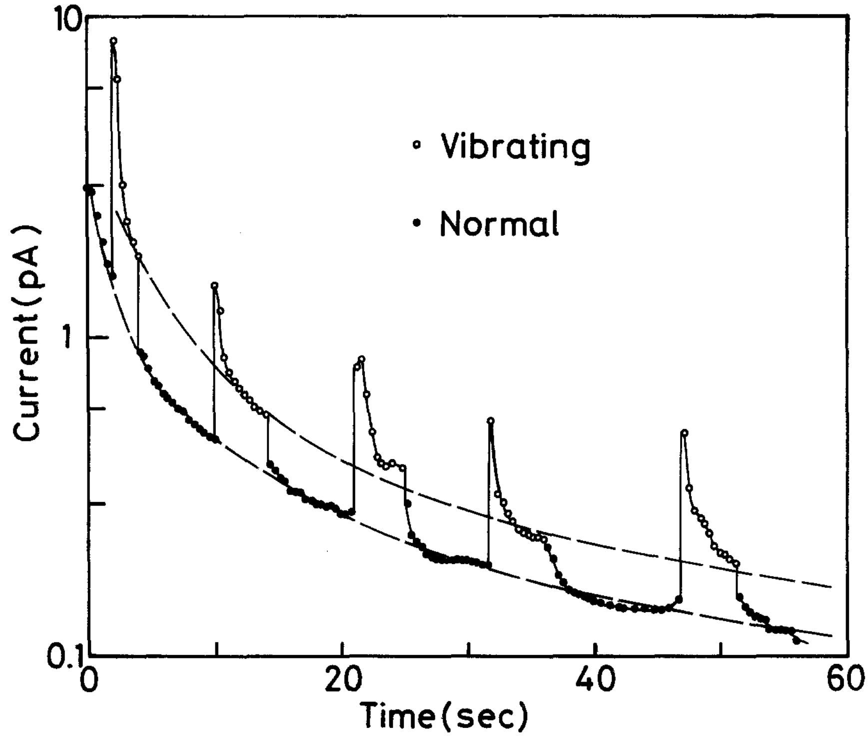


Figure 8 Variation of the deformation current due to the superimposition of oscillatory stress during the stress relaxation. The strain rate is shown to be larger when the oscillation is superimposed than not superimposed. The dashed lines indicate the variation of current with and without the superimposition, respectively.

oscillatory stress with the stress amplitude of 0.24 MPa. The current varies rapidly at the application and the removal of the oscillation. However, it is seen from the figure that there are components of the current which decrease continuously even at the moments of application and removal of the oscillation. This is shown by the two dashed lines. These two lines can be superimposed by translating one of the curves along the ordinate except for the initial 10 sec of the relaxation. This superimposition and the stress amplitude give an activation volume of 2.2×10^{-20} cm^3 by means of Equation 7 assuming $q = 1$. This value for the activation volume is less than that measured immediately before the start of the stress relaxation in the same manner, 4.4×10^{-20} cm^3, and that from the strain rate change test, 5.2×10^{-20} cm^3. Because it is expected that the activation volumes should have the same value independent of the methods, the above differences in the values should have been caused by the difference in qs, which are really not unity. Comparing those activation volumes obtained by using Equation 7 to that obtained by the strain rate change test, we get $q = 0.42$ for the stress relaxation and $q = 0.85$ for the static deformation, because the strain rate change test is independent of q. The ratio, i_2/i_1, in the early part of the stress relaxation is less than that in the later part, as shown in Fig. 8. It is considered that the value obtained from the data at the early part of the relaxation, $q = 0.66$, is closer to the value obtained from the static deformation ($q = 0.85$) rather than at the later part ($q = 0.42$). This suggests that the mechanism of the stress relaxation is different at the early and later parts, though further study of the problem is necessary.

4. Conclusion

The stress decrement and the variation of deformation current caused by the intermittent superimposition of ultrasonic oscillatory stress were measured during the deformation of pure NaCl and NaCl : Ca (0.1 mol %) single crystals. It is found that the activation volume can be estimated by two different methods concerning the deformation current, one using the stress amplitude and the other the stress decrement at the removal of the oscillation. A good agreement between the measurement and the stress superimposition theory applied for pure NaCl single crystals was obtained by fitting the activation volumes estimated by the above two methods to that obtained by the strain rate change test.

The variation of the deformation current is observed during the stress relaxation when the oscillation was superimposed. This indicates that the strain rate under the superimposition of ultrasonic oscillation is larger than that without the superimposition.

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