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Interaction between a dislocation and impurities in KCl doped with Li^+ or Na^+

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A strain-rate cycling test during the Blaha effect measurement was carried out in stage I, II and III of stress-strain curve at 80–300 K. On the basis of the relation curve of strain-rate sensitivity and stress decrement due to oscillation, the influence of strain on the interaction between a dislocation and impurities was investigated for KCl : Li^+ (0.5 mol% in the melt) and KCl : Na^+ (0.5 mol% in the melt). It has been so far reported that the curve seems to reflect the influence of ultrasonic oscillation on the dislocation motion on the slip plane containing many weak obstacles such as impurities and a few strong ones such as forest dislocations. As a result, it seemed that the Na^+ ions in KCl : Na^+ contribute to dislocation multiplication in the three stages, whereas the Li^+ ions in KCl : Li^+ did not contribute to it. Furthermore, the effective stress due to the impurities decreased with increasing strain at almost all the temperatures for KCl : Na^+. Accordingly, the critical temperature, at which the effective stress is zero, was larger in stage I, compared with that in stage II for KCl : Na^+ from the dependence of effective stress on the temperature.

1. Introduction

It has been reported that a strain-rate cycling test during the Blaha effect measurement can separate the contributions arising from the interaction between a dislocation and impurities and between the dislocations themselves during plastic deformation [1, 2]. Recently, the information on the interaction between a mobile dislocation and impurities has been obtained from the relation between strain-rate sensitivity and stress decrement due to oscillation for KCl doped with divalent impurities [3–5] or monovalent ones [6, 7]. The relation between strain-rate sensitivity and stress decrement seems to reflect the influence of ultrasonic oscillation on the dislocation motion on the slip plane containing many weak obstacles such as impurities and a few strong ones such as forest dislocations [3–6].

In this study, the strain-rate cycling test during the Blaha effect measurement is carried out for KCl doped with monovalent impurities in stage I, II and III of stress-strain curve. The purpose of this paper is to investigate the influence of shear strain on the interaction between a dislocation and impurities. The study is discussed on the basis of the variation of the relation curve of strain-rate sensitivity and stress decrement with shear strain. The effective stress due to impurities and the critical temperature, at which the effective stress is zero, are examined in the different plastic deformation region.

2. Experimental procedure

Three kinds of single crystals, KCl doped with Li^+ (0.5 mol% in the melt), KCl doped with Na^+ (0.5 mol% in the melt) and KCl, were deformed by compression along the $\langle 100 \rangle$ axis and the ultrasonic oscillatory stress was applied by a resonator in the same direction as the compression. The stability of temperature during the test was kept within 2 K. The size of the specimens was $5 \times 5 \times 15$ mm^3 and the resonant frequency was 20 kHz.

The stress drop due to superposition of oscillatory stress during plastic deformation is $\triangle\tau$, which increases with the stress amplitude at a given temperature and shear strain [3]. The stress change due to the strain-rate cycling is $\triangle\tau'$, when the strain-rate cycling is carried out keeping the stress amplitude constant. $\triangle\tau'/\triangle \ln \dot{\varepsilon}$ is obtained as the strain-rate sensitivity. The strain-rate cycling tests made between the crosshead speads of 20 and 100 μm min^{-1} were carried out at temperatures from 80–300 K. The details of the strain-rate cycling test during the Blaha effect measurement are described in the previous papers [3, 7].

3. Results

3.1. Relation between the strain-rate sensitivity and the stress decrement

Fig. 1 shows the relation between strain-rate sensitivity and stress decrement during the Blaha effect measurement at various shear strains for KCl : Na^+ at 180 K. The strain-rate sensitivity varies with stress decrement at a given shear strain and the relation curve of strain-rate sensitivity and stress decrement shifts upward with increasing shear strain. The same phenomenon as Fig. 1 is also observed for KCl : Li^+. We denote the first bending point at low stress decrement by τ_{p1} and the second

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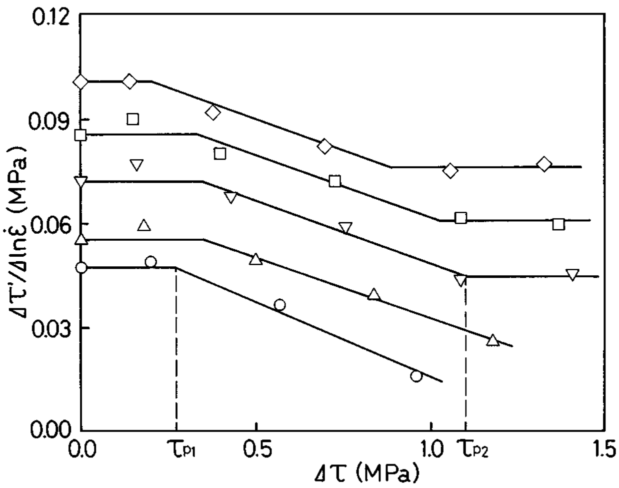

Figure 1 Relationship between the strain-rate sensitivity and the stress decrement for KCl : Na^{+} (0.5 mol% in the melt) at 180 K. ε: (○) 6%, (△) 12%, (▽) 17%, (□) 20%, (◊) 23%.

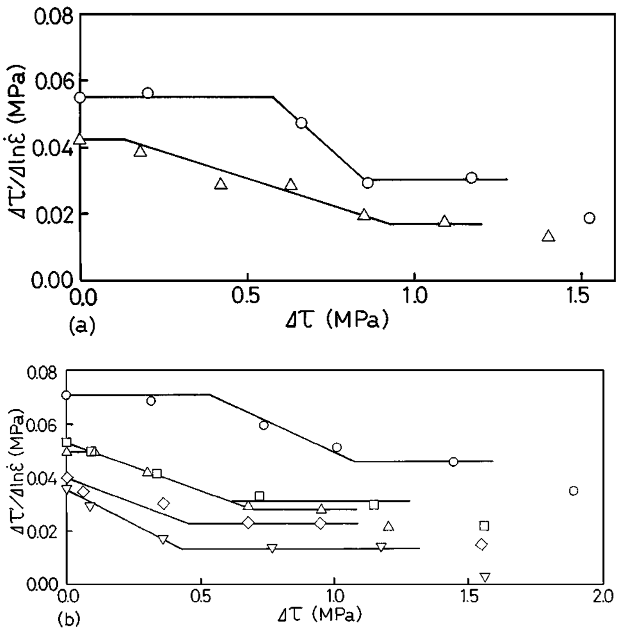

Figure 3 Relationship between the strain-rate sensitivity and the stress decrement for (a) KCl : Li^{+} (0.5 mol% in the melt) at various conditions: (○) 160 K and $\varepsilon = 17\%$, (△) 192 K and $\varepsilon = 15\%$ and (b) KCl : Na^{+} (0.5 mol% in the melt) at various conditions: (○) 147 K and $\varepsilon = 14\%$, (△) 251 K and $\varepsilon = 19\%$, (▽) 279 K and $\varepsilon = 20\%$, (□) 279 K and $\varepsilon = 26\%$, (◊) 300 K and $\varepsilon = 28\%$.

one at high stress decrement by τ_{p2}. τ_{p1} is considered to represent the effective stress due to only one type of the impurities which lie on the dislocation with the largest separation when the dislocation moves forward with the help of oscillation [3]. τ_{p2} is reported to be the stress decrement at which the ultrasonic oscillatory stress helps the dislocation break away from almost all weak obstacles [2].

A third bending point is found on the relation curves of strain-rate sensitivity and stress decrement for KCl : Li^{+} at shear strains of 33, 35 and 38% as shown in Fig. 2. We denote the third bending point by τ_{p3}. However, τ_{p3} does not appear on the curves at shear strains of 25 and 30%, because high stress amplitude could not be applied to the specimen during the compression test. The lengths of second plateau regions on the curves at shear strains of 33, 35 and 38% are 0.64, 0.69 and 0.75 MPa, respectively. The second plateau region where the obstacles to the dislocation motion are considered to be only strong ones such as forest dislocations [3] increases with increasing shear strain. The appearance of existence of τ_{p3} is also observed on the relation curves of strain-rate sensitivity and stress decrement at 160 and 192 K for KCl : Li^{+} in Fig. 3a and at 147, 251, 279 and 300 K for KCl : Na^{+} in Fig. 3b.

3.2. Dependence of τ_{p1} and τ_{p2} on the shear strain

The relation between strain-rate sensitivity and stress decrement at various shear strains is shown in Fig. 4 for KCl : Na^{+} at 169 K. Both the τ_{p1} and the τ_{p2} almost decrease with increasing shear strain. The same phenomenon as Fig. 4 also appears on the relation curves of strain-rate sensitivity and stress decrement for KCl : I^{-} (0.5 mol% in the melt) at 205 K [6] and KCl : Sr^{2+} (0.05 mol% in the melt) at 200 K [3]. The dependence of τ_{p1} and τ_{p2} on shear strain at various temperatures is shown in Fig. 5a for KCl : Li^{+} and Fig. 5b for KCl : Na^{+}. The τ_{p1} and the τ_{p2} decrease with increasing shear strain at almost all the temperatures for KCl :Na^{+}. However, it can not be discerned for KCl : Li^{+}.

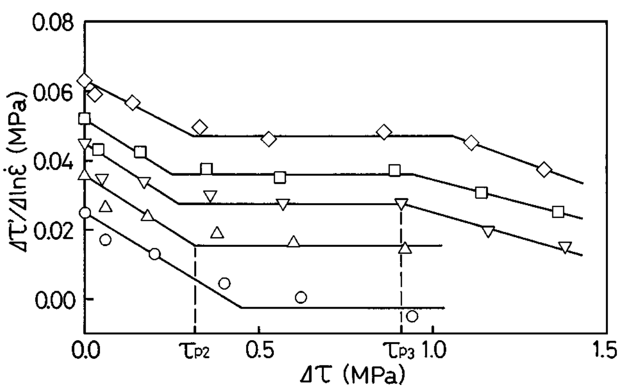

Figure 2 Relationship between the strain-rate sensitivity and the stress decrement for KCl : Li^{+} (0.5 mol% in the melt) at 249 K. ε: (○) 25%, (△) 30%, (▽) 33%, (□) 35%, (◊) 38%.

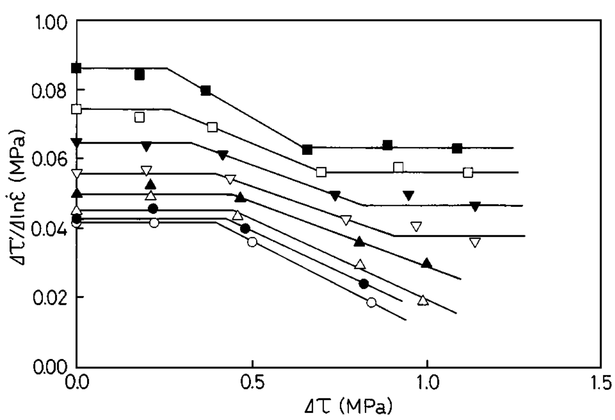

Figure 4 Relationship between the strain-rate sensitivity and the stress decrement for KCl : Na^{+} (0.5 mol% in the melt) at 169 K. ε: (○) 6%, (●) 8%, (△) 10%, (▲) 12%, (▽) 14%, (▼) 16%, (□) 18%, (■) 20%.

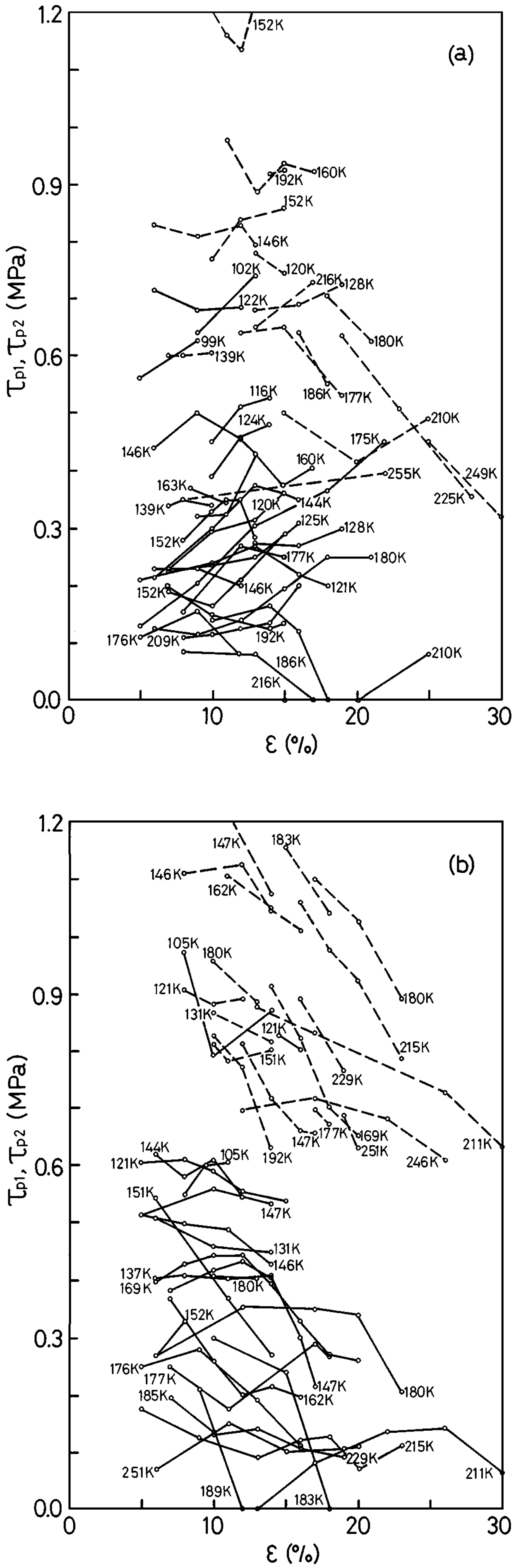

Figure 5 Dependence of (o——o) τ_{p1} and (o– – –o) τ_{p2} on the shear strain at various temperatures for (a) KCl : Li^+ (0.5 mol% in the melt) and (b) KCl : Na^+ (0.5 mol% in the melt).

4. Discussion

4.1. Strain-rate sensitivity at second plateau region on the relation curve of strain-rate sensitivity and stress decrement

The phenomenon that the relation curve of strain-rate sensitivity and stress decrement shifts upward with increasing shear strain in Fig. 1 may be caused by the part of the strain-rate sensitivity due to dislocation cutting process as reported for KCl : Sr^{2+} [3]. Because, when the waiting time for the thermal activation equals at two kinds of obstacles such as impurities and forest dislocations, the reciprocal of the activation volume, v, is given by [8, 9]

$$1/v = 1/v_p + 1/v_s \tag{1}$$

where the suffixes of p and s are due to impurities and dislocation cutting process, respectively. The strain-rate sensitivity at second plateau region on the relation curve of strain-rate sensitivity and stress decrement is considered to be due to dislocation cutting process [3].

Three-stage strain hardening has been clearly established as the characteristic behaviour of rock salt structure single crystals deforming predominantly by single glide [10]. The three-stage strain hardening is also obtained for KCl [11, 12]. In this study, it is observed at above 124 K for KCl : Li^+ and at above 105 K for KCl : Na^+. The strain hardening curves are shown in Fig. 6a for KCl : Li^+ at 160 K, b for KCl : Na^+ at 169 K and c for KCl at 142 K. We investigate the strain-rate sensitivity at second plateau region on the relation curve of strain-rate sensitivity and stress decrement at the following two shear strains. One is the boundary between stage I and II, namely, A in Fig. 6. The other is that between stage II and III, namely, B in Fig. 6. The former strain-rate sensitivity is hereafter termed $(\triangle\tau'/\triangle \ln\dot{\varepsilon})_{\text{I–II}}$ and the latter one $(\triangle\tau'/\triangle \ln\dot{\varepsilon})_{\text{II–III}}$. The result for the strain-rate sensitivity is shown in Fig. 7a

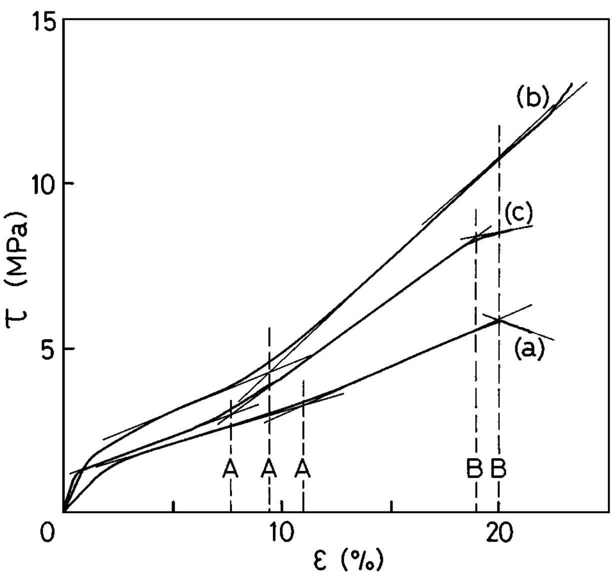

Figure 6 Shear stress-shear strain curves for (a) KCl : Li^+ (0.5 mol% in the melt) at 160 K, (b) KCl : Na^+ (0.5 mol% in the melt) at 169 K and (c) KCl at 142 K.

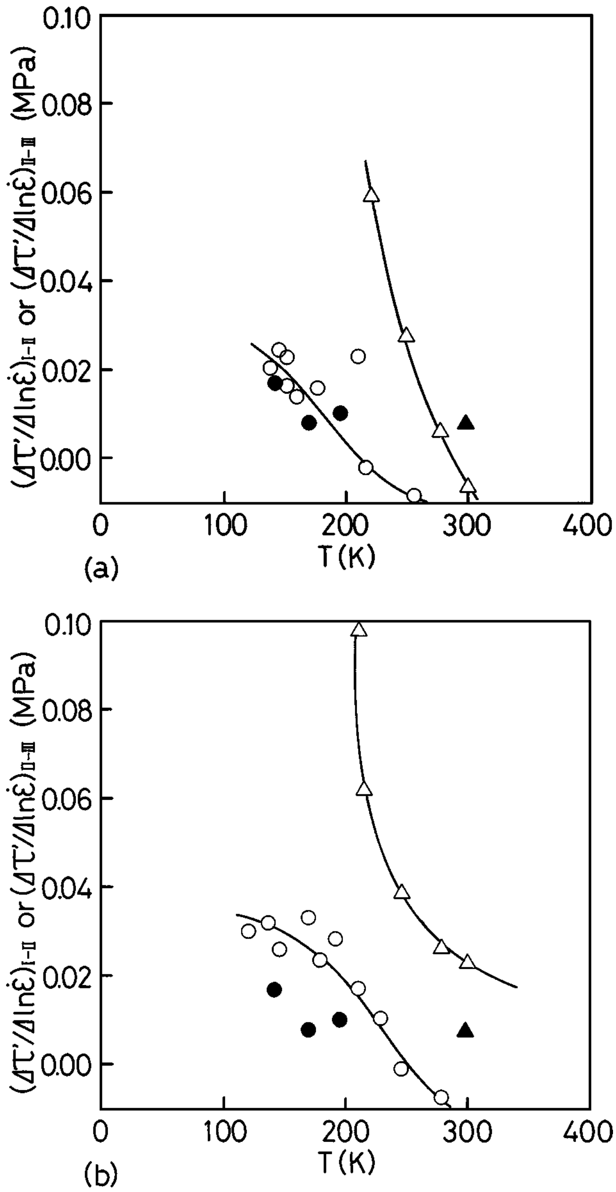

Figure 7 Dependence of $(\triangle\tau'/\triangle\ln\dot{\varepsilon})_{\mathrm{I-II}}$ and $(\triangle\tau'/\triangle\ln\dot{\varepsilon})_{\mathrm{II-III}}$ on the temperature for (a) KCl : Li^+ (0.5 mol% in the melt) and KCl and (b) KCl : Na^+ (0.5 mol% in the melt) and KCl. $(\triangle\tau'/\triangle\ln\dot{\varepsilon})_{\mathrm{I-II}}$: (○) for KCl : Li^+ and KCl : Na^+ and (●) for KCl. $(\triangle\tau'/\triangle\ln\dot{\varepsilon})_{\mathrm{II-III}}$: (△) for KCl : Li^+ and KCl : Na^+ and (▲) for KCl.

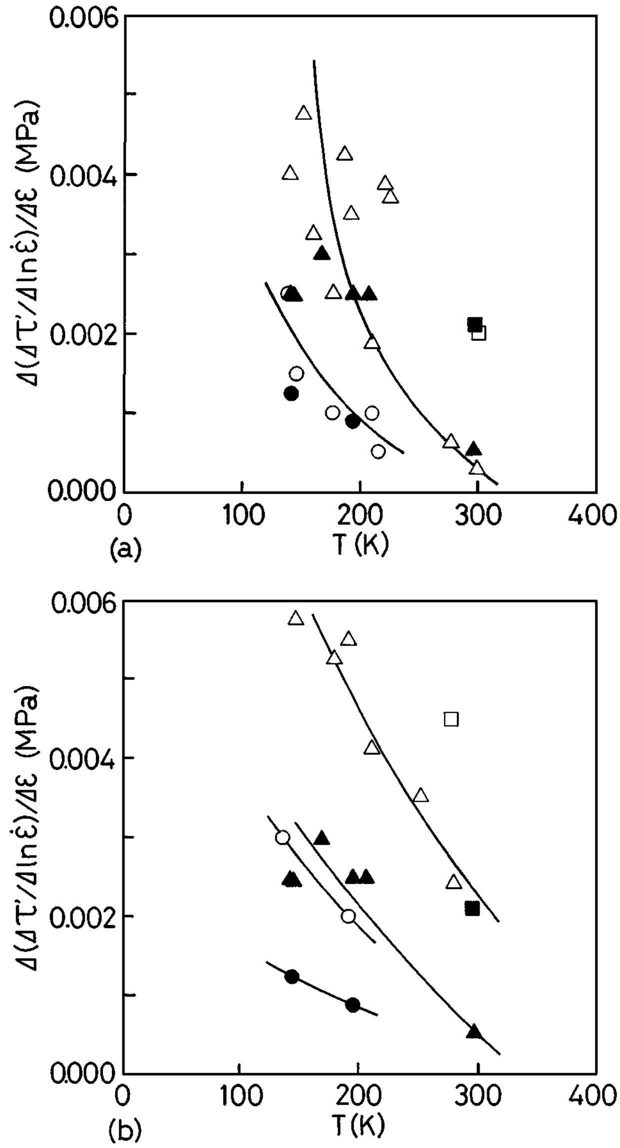

Figure 8 Dependence of $\triangle(\triangle\tau'/\triangle\ln\dot{\varepsilon})/\triangle\varepsilon$ on the temperature for (a) KCl : Li^+ (0.5 mol% in the melt) and KCl and (b) KCl : Na^+ (0.5 mol% in the melt) and KCl in the different plastic deformation region: (○) for KCl : Li^+ and KCl : Na^+ and (●) for KCl in stage I; (△) for KCl : Li^+ and KCl : Na^+ and (▲) for KCl in stage II; (□) for KCl : Li^+ and KCl : Na^+ and (■) for KCl in stage III.

for KCl : Li^+ and KCl and b for KCl : Na^+ and KCl. The open symbols correspond to that for KCl : Li^+ and KCl : Na^+ and the solid ones that for KCl. The curves in Fig. 7a and b are to guide the reader's eye. Comparing the $(\triangle\tau'/\triangle\ln\dot{\varepsilon})_{\mathrm{I-II}}$ and the $(\triangle\tau'/\triangle\ln\dot{\varepsilon})_{\mathrm{II-III}}$ for KCl : Li^+ and KCl : Na^+ with those for KCl, great difference is not seen between KCl : Li^+ and KCl, however those for KCl : Na^+ are obviously large as against those for KCl. That is, the Li^+ ions in KCl : Li^+ have no influence on both $(\triangle\tau'/\triangle\ln\dot{\varepsilon})_{\mathrm{I-II}}$ and $(\triangle\tau'/\triangle\ln\dot{\varepsilon})_{\mathrm{II-III}}$. However, the Na^+ ions in KCl : Na^+ seem to make those large. Therefore, the forest dislocation density seems to increase by the Na^+ ions in KCl : Na^+ within the temperature. But the Li^+ ions in KCl : Li^+ do not influence it. Furthermore, the variation of strain-rate sensitivity at the second plateau region on the relation curve of strain-rate sensitivity and stress decrement with shear strain, $\triangle(\triangle\tau'/\triangle\ln\dot{\varepsilon})/\triangle\varepsilon$, is investigated for the three kinds of specimens in Fig. 8a and b. The relations of temperature and $\triangle(\triangle\tau'/\triangle\ln\dot{\varepsilon})/\triangle\varepsilon$ in stage I, II and III are represented by a circle, a triangle and a square, respectively. The $\triangle(\triangle\tau'/\triangle\ln\dot{\varepsilon})/\triangle\varepsilon$ in stage II is obviously larger than that in stage I at a given temperature. This phenomenon may suggest that there is only a slight increase in forest dislocation density with the shear strain in stage I but there is a rapid increase with that in stage II. The $\triangle(\triangle\tau'/\triangle\ln\dot{\varepsilon})/\triangle\varepsilon$ in stage III is larger than that in stage II at a given temperature. The $\triangle(\triangle\tau'/\triangle\ln\dot{\varepsilon})/\triangle\varepsilon$ in stage I and II increases with decreasing temperature for the three kinds of specimens. Both $(\triangle\tau'/\triangle\ln\dot{\varepsilon})_{\mathrm{I-II}}$ and $(\triangle\tau'/\triangle\ln\dot{\varepsilon})_{\mathrm{II-III}}$ also increase with decreasing temperature for KCl : Li^+ and KCl : Na^+. Thus, when the specimens are deformed by the compression test in stage I and II within the temperature, it will be predicted that the forest dislocation density at a given shear strain increases with decreasing temperature. When Ge single crystals were deformed by tensile test at a constant strain rate of $1.1 \times 10^{-4}\ \mathrm{s}^{-1}$ at 873–993 K, the dislocation density on the surface of specimens increases with decreasing temperature in the same deformation region of stage I and II [13]. The experimental result for Ge single crystals agrees with the above-mentioned prediction. According to Fig. 8a and b, $\triangle(\triangle\tau'/\triangle \ln\dot{\varepsilon})/\triangle\varepsilon$ depends greatly on the temperature in stage II in contrast to that in stage I for the three kinds of specimens. We can also observe that the value of $\triangle(\triangle\tau'/\triangle \ln\dot{\varepsilon})/\triangle\varepsilon$ for KCl : Li^+ is almost the same as that for KCl but the value of $\triangle(\triangle\tau'/\triangle \ln\dot{\varepsilon})/\triangle\varepsilon$ for KCl : Na^+ is remarkably large as compared with that for KCl in stage I, II and III. Consequently, it seems that the Na^+ ions in KCl : Na^+ contribute to dislocation multiplication in the three stages within the temperature, whereas the Li^+ ions in KCl : Li^+ do not contribute to it.

4.2. Critical temperature

In the previous paper [7], the force-distance relation between a dislocation and the impurity was investigated for KCl : Li^+ and KCl : Na^+. As a result, the square force-distance relation seemed to be the most suitable of the three [14], which are a square one, a parabolic one and a triangular one, taking account of the Friedel relation [15] for KCl : Na^+. Then, the relation formula of τ_{p1} and temperature, which reveals the force-distance relation between a dislocation and an impurity [3, 6], is given by [7]

$$(\tau_{p1}/\tau_{p0})^{2/3} = 1 - (T/T_c) \tag{2}$$

where τ_{p0} is the effective stress due to the impurities without thermal activation and T_c is the critical temperature at which τ_{p1} is zero. The values of τ_{p0} and T_c can be obtained through the research on the linear plots of Equation 2. τ_{p0} is determined by extrapolating the linear relationship of τ_{p1} and temperature to 0 K. Since the τ_{p1} for KCl : Na^+ decreases with increasing shear strain at almost all the temperatures in Fig. 5b, we investigate the values of T_c and τ_{p0} for KCl : Na^+ on the basis of the relation between τ_{p1} and temperature in the different plastic deformation region. The τ_{p1} in stage I is got from the relation curve of strain-rate sensitivity and stress decrement at the smallest shear strain in stage I and the τ_{p1} in stage II from the curve at the largest shear strain in stage II. The results for T_c and τ_{p0} are given in Table I. The T_c in stage I is larger than that in stage II and the τ_{p0} in stage I is slightly larger than that in stage II.

TABLE I Values of T_c and τ_{p0} in the different plastic deformation region for KCl : Na^+ (0.5 mol% in the melt)

Plastic deformation region	T_c (K)	τ_{p0} (MPa)
Stage I	269	1.36
Stage II	260	1.34

5. Conclusion

1. Comparing the $(\triangle\tau'/\triangle \ln\dot{\varepsilon})_{I-II}$ and the $(\triangle\tau'/\triangle \ln\dot{\varepsilon})_{II-III}$ for KCl : Li^+ with those for KCl, great difference is not seen. The value of $\triangle(\triangle\tau'/\triangle \ln\dot{\varepsilon})/\triangle\varepsilon$ for KCl : Li^+ is also almost the same as that for KCl. However, both the $(\triangle\tau'/\triangle \ln\dot{\varepsilon})_{I-II}$ and the $(\triangle\tau'/\triangle \ln\dot{\varepsilon})_{II-III}$ for KCl : Na^+ are obviously large as against those for KCl within the temperature. The value of $\triangle(\triangle\tau'/\triangle \ln\dot{\varepsilon})/\triangle\varepsilon$ for KCl : Na^+ is also larger than that for KCl in stage I, II and III. Therefore, it seems that the Na^+ ions in KCl : Na^+ make the forest dislocation density increased and contribute to dislocation multiplication in the three stages within the temperature. But the Li^+ ions in KCl : Li^+ do not influence the forest dislocation density.

2. The phenomenon that both τ_{p1} and τ_{p2} decrease with increasing shear strain is observed at almost all the temperatures for KCl : Na^+. However, it can not be discerned for KCl : Li^+. As a result, the T_c in stage I is larger than that in stage II and the τ_{p0} in stage I is slightly larger than that in stage II for KCl : Na^+.

References

1. T. OHGAKU and N. TAKEUCHI, *Phys. Status Solidi (a)* **111** (1989) 165.
2. *Idem., ibid.* **118** (1990) 153.
3. Y. KOHZUKI, T. OHGAKU and N. TAKEUCHI, *J. Mater. Sci.* **28** (1993) 3612.
4. *Idem., ibid.* **28** (1993) 6329.
5. *Idem., ibid.* **30** (1995) 101.
6. T. OHGAKU and N. TAKEUCHI, *Phys. Status Solidi (a)* **134** (1992) 397.
7. Y. KOHZUKI, *J. Mater. Sci.* **33** (1998) 5613.
8. U. F. KOCKS, *Trans. JIM Suppl.* **9** (1968) 1.
9. U. F. KOCKS, A. S. ARGON and M. F. ASHBY, "Progress in Materials Science, Vol. 19," edited by Bruce Chalmers, J. W. Christian and T. B. Massalski (Pergamon Press, New York, 1975) p. 160.
10. M. T. SPRACKLING, "The Plastic Deformation of Simple Ionic Crystals," edited by A. M. Alper, J. L. Margrave and A. S. Nowick (Academic Press, London, 1976) p. 203.
11. T. H. ALDEN, *Trans. Met. Soc. AIME* **230** (1964) 649.
12. L. A. DAVIS and R. B. GORDON, *J. Appl. Phys.* **40** (1969) 4507.
13. K. KOJIMA and K. SUMINO, *Cryst. Latt. Defects* **2** (1971) 147.
14. A. J. E. FOREMAN and M. J. MAKIN, *Philos. Mag.* **14** (1966) 911.
15. J. FRIEDEL, "Dislocations" (Pergamon Press, Oxford, 1964) p. 224.

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