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Interaction between a dislocation and an impurity in KCl: Mg^{2+} single crystals

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The interaction between a dislocation and the impurity in KCl: Mg^{2+} (0.035 mol% in the melt) was investigated at 77–178 K with respect to the two models: one is the Fleischer's model and the other the Fleischer's model taking account of the Friedel relation. The latter is termed the F-F. The dependence of strain-rate sensitivity due to the impurities on temperature for the specimen was appropriate to the Fleischer's model than the F-F. Furthermore, the activation enthalpy, ΔH, for the Fleischer's model appeared to be nearly proportional to the temperature in comparison with the F-F. The Friedel relation between effective stress and average length of the dislocation segments is exact for most weak obstacles to dislocation motion. However, above-mentioned results mean that the Friedel relation is not suitable for the interaction between a dislocation and the impurity in the specimen. Then, the value of $\Delta H(T_c)$ at the Fleischer's model was found to be 0.61 eV. $\Delta H(T_c)$ corresponds to the activation enthalpy for overcoming of the strain field around the impurity by a dislocation at 0 K. In addition, the Gibbs free energy, ΔG_0, concerning the dislocation motion was determined to be between 0.42 and 0.48 eV on the basis of the following equation

$$\partial \ln \dot{\varepsilon}/\partial \tau = \{\Delta G_0/(kT\tau_{p0})\}\left\{1-(T/T_c)^{1/2}\right\}^{-1}(T/T_c)^{1/2} + \partial \ln \dot{\varepsilon}_0/\partial \tau$$

where k is the Boltzmann's constant, T the temperature, T_c the critical temperature at which the effective stress due to the impurities is zero, τ_{p0} the effective shear stress without thermal activation, and $\dot{\varepsilon}_0$ the frequency factor.

1. Introduction

On the basis of the relative curves of strain–rate sensitivity and stress decrement due to oscillation, we have investigated the interaction between a dislocation and an impurity for KCl doped with divalent impurities [1–3]. The curve seemed to reflect the influence of ultrasonic oscillation on the dislocation motion on the slip plane containing many impurities and a few forest dislocations [1, 4]. When alkali halide crystals are doped with divalent impurities, the impurities are expected to be paired with positive ion vacancies. The pairs are termed I–V dipoles. Then, tetragonal lattice distortions are considered to be produced around the I–V dipoles [5]. The asymmetrical distortions which cause solution hardening interact strongly with mobile dislocations. Fleischer and Hibbard [6] and Johnston *et al.* [7] named the solution hardening "rapid hardening." Furthermore, Fleischer has discussed the solution hardening of LiF: Mg^{2+} (80 p.p.m.), whereas the Friedel relation [8] was not taken into his model [5]. It is well known that the Friedel relation between the effective stress and the average length of dislocation segments is exact for most weak obstacles to dislocation motion at low solute concentration. We investigate whether the Friedel relation is valid for the interaction between a dislocation and an impurity in KCl:Mg^{2+}, which is expected to cause the rapid hardening. This is examined from various methods. In addition, the activation enthalpy and the Gibbs free energy for overcoming the impurity by a dislocation are obtained in this paper.

2. Experimental procedure

The single crystals, which are KCl doped with Mg^{2+} (0.035 mol% in the melt), were deformed by compression at 77 to 178 K and strain-rate cycling tests were carried out during superposition of oscillation. The size of the specimens was about $5 \times 5 \times 15$ mm^3. Then, the stress drop due to superposition of oscillatory stress is $\Delta\tau$. The stress change due to the strain-rate cycling is $\Delta\tau'$ and $\Delta\tau'/\Delta \ln \dot{\varepsilon}$ was used as a measure of the strain-rate sensitivity. In this paper, the strain-rate sensitivity is abbreviated to SRS. The details of the compression test and the preparation for the specimens were described in the previous papers [1, 3].

3. Discussion for the applicability of the Friedel relation to the interaction between a dislocation and the impurity

3.1. Dependence of the SRS due to the impurities on the temperature

The force-distance relation, which represents the interaction between a dislocation and an impurity, is investigated with respect to the two models. One is the Fleischer's model [5] and the other the Fleischer's model taking account of the Friedel relation. The latter is hereafter termed the F-F. The SRS due to the impurities for the Fleischer's model is obtained as

$$\partial\tau/\partial \ln\dot{\varepsilon} = (\tau_{p0}/T_c)\{(T_c/T)^{1/2} - 1\}T/\alpha \qquad (1)$$

where T_c is the critical temperature T, at which τ_{p1} is zero, τ_{p0} is the effective shear stress due to the impurities without thermal activation and α is a constant. τ_{p0} is the value of τ_{p1} at 0 K. τ_{p1} is considered to represent the effective stress due to only one type of the impurities which lie on the dislocation when the dislocation moves forward with the help of oscillation [1, 4]. That for the F-F is expressed by

$$\partial\tau/\partial \ln\dot{\varepsilon} = \{3\tau_{p0}T/(2T_c)\}(T_c/T)^{1/2}\{1 - (T/T_c)^{1/2}\}^2/\alpha \qquad (2)$$

The process of leading Equations 1 and 2 was described in the paper [9]. The values of τ_{p0} and T_c for Equation 1 can be derived from

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

and those for Equation 2 from

$$(\tau_{p1}/\tau_{p0})^{1/3} = 1 - (T/T_c)^{1/2} \qquad (4)$$

The relation between τ_{p1} and temperature is considered to reveal the force-distance relation between a dislocation and an impurity [1, 4]. Therefore, the interaction between a dislocation and the impurity for $KCl{:}Mg^{2+}$ is approximated by the F-F in Fig. 1a and by the Fleischer's model in Fig. 1b. The values of T_c and τ_{p0}, which are obtained from Fig. 1a and b, are given in Table I. The two values for the Fleischer's model are small in comparison with those for the F-F. The separation of various models between a dislocation and an impurity has been carried out through the variation of the thermal component of the yield stress with temperature [11]. However, the difference in the linear relationship of effective stress and temperature for the two models cannot be observed within the temperature as shown in Fig. 1a and b. Ono [12] also concluded as follows. It was impossible to select one of several models on the basis of an experimentally obtained relationship of stress and temperature.

Fig. 2 shows the dependence of SRS due to the impurities on temperature for the specimen. The relative curve of SRS due to the impurities and temperature for the Fleischer's model and that for the F-F are represented as a solid and a dashed lines, respectively. The two curves are derived from the calculations of Equations 1 and 2. The SRS due to the impurities for the F-F is obviously larger than that for the Fleischer's model below about 100 K as shown in Fig. 2. The open circles correspond to $(\Delta\tau'/\Delta\ln\dot{\varepsilon})_p$ for the specimen, which are considered to be the SRS due to impurities [1, 3, 4]. The $(\Delta\tau'/\Delta\ln\dot{\varepsilon})_p$ is represented by the difference between SRS at the first plateau region and at the second one on the relative curve of SRS and stress decrement. The solid line in Fig. 3 shows the relative curve of SRS and stress decrement for $KCl{:}Mg^{2+}$ at 113 K.

TABLE I Values of τ_{p0} and T_c for the specimen at the two models

	τ_{p0} (MPa)	T_c (K)
Fleischer's model	5.64	191 [3]
F-F	17.91 [10]	199 [10]

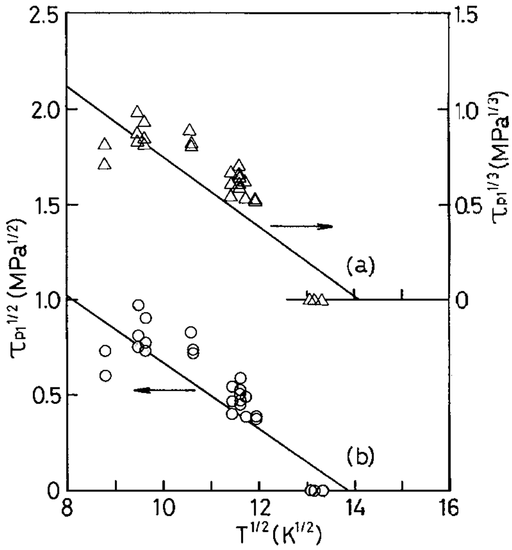

Figure 1 Linear plots of the effective shear stress and the temperature for $KCl{:}Mg^{2+}$ (0.035 mol% in the melt) at the two models: (a) the F-F and (b) the Fleischer's model.

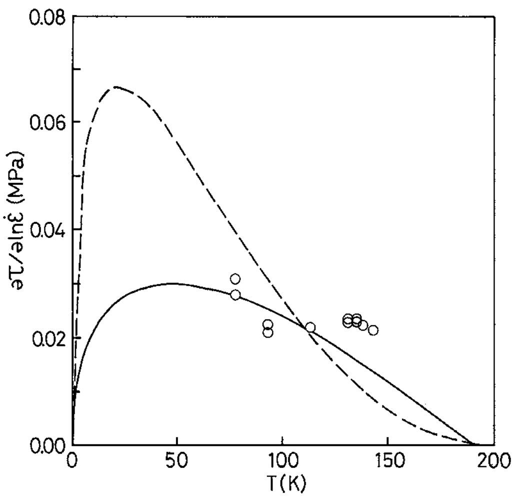

Figure 2 Relationship between the strain-rate sensitivity due to the impurities and temperature for $KCl{:}Mg^{2+}$ (0.035 mol% in the melt). (——) corresponds to the dependence of the strain-rate sensitivity on temperature for the Fleischer's model and (– – – –) that for the F-F. (O): $(\Delta\tau'/\Delta\ln\dot{\varepsilon})_p$ for the specimen.

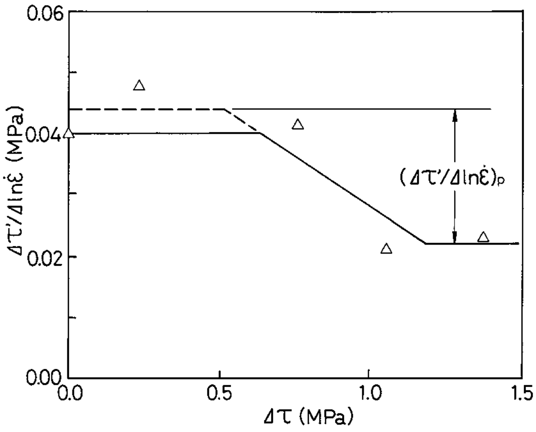

Figure 3 Relationship between the strain-rate sensitivity and the stress decrement for KCl:Mg^{2+} (0.035 mol% in the melt) at 113 K and $\varepsilon = 9\%$.

The value of $\Delta\tau'/\Delta \ln \dot{\varepsilon}$ at which $\Delta\tau$ is 0.23 MPa is obviously larger than that at which $\Delta\tau$ is 0 MPa as can be seen from open triangles in Fig. 3. Therefore, the SRS at the first plateau region is assumed to be in the middle of those at which $\Delta\tau$ is 0 and 0.23 MPa. That is, the $(\Delta\tau'/\Delta \ln \dot{\varepsilon})_p$ in Fig. 3 corresponds to the SRS due to the impurities for the specimen at 113 K. Fig. 4 shows the curve for KCl:Mg^{2+} at 143 K. It is the curve at the smallest temperature at which τ_{p1} does not appear. Then, the $(\Delta\tau'/\Delta \ln \dot{\varepsilon})_p$ is assumed to be the difference between the SRS at which the curve intersects the ordinate and at plateau region, since τ_{p1} first becomes zero at 143 K within the accuracy. Therefore, the $(\Delta\tau'/\Delta \ln \dot{\varepsilon})_p$ at this temperature would be 0.0215 MPa from Fig. 4.

As can be seen from Fig. 2, the open circles are approximated to the solid line rather than the dashed one within the present temperatures. That is, the Fleischer's model seems to be more suitable than the F-F. This means that the Friedel relation is not appropriate to the interaction between a dislocation and the impurity in the specimen. If the $(\Delta\tau'/\Delta \ln \dot{\varepsilon})_p$ can be obtained below about 100 K, it will be more distinctly determined whether the Friedel relation is appropriate for the specimen.

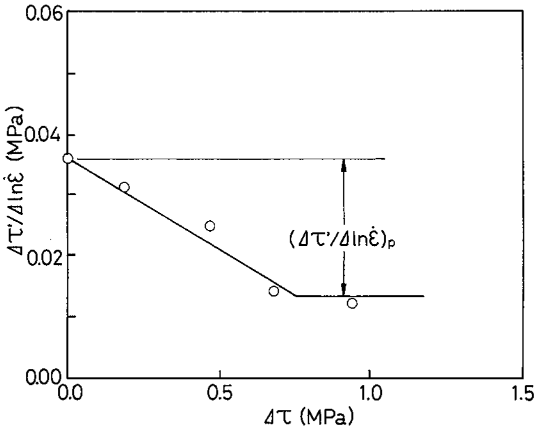

Figure 4 Relationship between the strain-rate sensitivity and the stress decrement for KCl:Mg^{2+} (0.035 mol% in the melt) at 143 K and $\varepsilon = 10\%$.

3.2. Relation between the effective shear stress and the activation volume for the interaction between a dislocation and the impurities

The measurement of activation volume and its variation with strain (flow stress) have been investigated in order to identify the rate-controlling mechanism to dislocation motion [13–19]. The consideration of the activation volume as dependent on the effective stress is useful in examining the force-distance relation between a dislocation and an impurity [20]. At a given concentration of impurity activation volume varies with effective stress according to the shape of the force-distance curve [21]. The activation volume, v^*, is given by

$$v^* = kT(\partial \ln \dot{\varepsilon}/\partial\tau)_T \tag{5}$$

where k is the Boltzmann's constant. The v^* for the Fleischer's model is expressed by substitution of Equation 1 into Equation 5 as follows

$$v^* = \alpha k(T_c/\tau_{p0})\left\{(T_c/T)^{1/2} - 1\right\}^{-1} \tag{6}$$

Similarly from Equations 2 and 5, that for the F-F is expressed by

$$v^* = \{2\alpha k T_c/(3\tau_{p0})\}\,(T/T_c)^{1/2}\left\{1 - (T/T_c)^{1/2}\right\}^{-2} \tag{7}$$

The results of calculations for v^* at the two models are shown in Fig. 5 for the specimen. The τ_{p1} for the Fleischer's model of ordinate in the figure is obtained from Equation 3 by the following equation

$$\tau_{p1} = \tau_{p0}\left\{1 - (T/T_c)^{1/2}\right\}^2 \tag{8}$$

and that for the F-F from Equation 4 by

$$\tau_{p1} = \tau_{p0}\left\{1 - (T/T_c)^{1/2}\right\}^3 \tag{9}$$

A solid and a dashed lines represent the relative curve of v^* and τ_{p1} for the Fleischer's model and that for the F-F, respectively. The open circles in Fig. 5 correspond

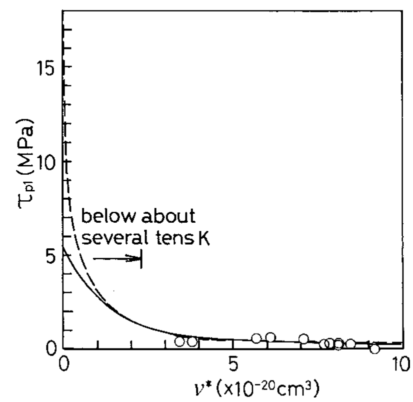

Figure 5 Relationship between the effective shear stress and the activation volume for the interaction between a dislocation and the impurities in KCl:Mg^{2+} (0.035 mol% in the melt). (——) corresponds to the dependence of the activation volume on the effective shear stress for the Fleischer's model and (– – – – –) that for the F-F. (O): $kT(\Delta \ln \dot{\varepsilon}/\Delta\tau')_p$ for the specimen.

to the dependence of v^* calculated from $(\Delta\tau'/\Delta \ln \dot{\varepsilon})_p$ on τ_{p1} for the specimen. That is, the v^* is given by

$$v^* = kT(\Delta \ln \dot{\varepsilon}/\Delta\tau')_p \quad (10)$$

No difference between the two curves can be almost observed above the v^* of about 1×10^{-20} cm^3. Below 1×10^{-20} cm^3, however, τ_{p1} for the F-F increases more markedly with decreasing v^* (temperature) than that for the Fleischer's model. As can be seen from Fig. 5, it is impossible to determine which curve is appropriate for the open circles within the present temperatures. If the $(\Delta\tau'/\Delta \ln \dot{\varepsilon})_p$ can be obtained at still lower temperature, it may be distinguished whether the Friedel relation is appropriate for the specimen from the figure. However, the Peierls mechanism can be adapted to the deformation of alkali halide crystals, such as LiF, NaCl, NaBr and KCl, at the low temperature [22–24]. It has been reported for KCl-4 mol% KBr single crystals that the mobile dislocations are impeded by the Peierls barriers in addition to the solute ions below about several tens K [17]. Therefore, there is difficulty in obtaining τ_{p1} and $(\Delta\tau'/\Delta \ln \dot{\varepsilon})_p$ from the relative curve of SRS and stress decrement for the specimen at the low temperature region at which the Peierls barriers additionally control the dislocation motion. As mentioned above, the reason is that the curve seems to reflect the dislocation motion on the slip plane containing two kinds of obstacles: impurities and forest dislocations. On the basis of v^* calculated from $(\Delta\tau'/\Delta \ln \dot{\varepsilon})_p$, namely, from Equation 10, it is difficult to determine the applicability of the Friedel relation for the specimen.

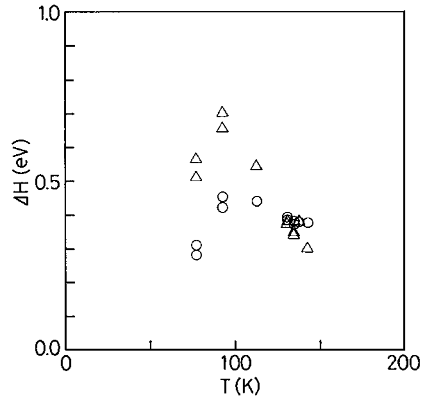

Figure 6 Relationship between the temperature and the activation enthalpy for the interaction between a dislocation and the impurity in KCl:Mg^{2+} at the two models: (○) the Fleischer's model and (Δ) the F-F.

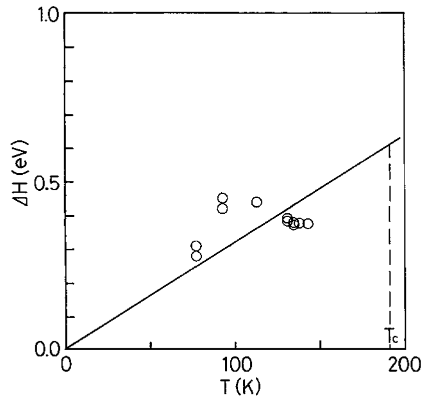

Figure 7 Proportional relationship between the temperature and the activation enthalpy for the interaction between a dislocation and the impurity in KCl:Mg^{2+} at the Fleischer's model.

3.3. Relation between temperature and the activation enthalpy for the interaction between a dislocation and the impurity

When a dislocation overcomes the impurity with the aid of thermal fluctuation, activation enthalpy, ΔH, for the Fleischer's model is given by [2, 3]

$$\Delta H = -kT^2(\Delta\, ln\, \dot{\varepsilon}/\Delta\tau')_p \left\{1 - (T/T_c)^{-1/2}\right\}\tau_{p0}/T_c \quad (11)$$

On the other hand, ΔH for the F-F is calculated from [9]

$$\Delta H = -kT^2(\Delta \ln \dot{\varepsilon}/\Delta\tau')_p \{-3\tau_{p0}/(2T_c)\} \times (T_c/T)^{1/2}\left\{1 - (T/T_c)^{1/2}\right\}^2 \quad (12)$$

Assuming that the changes in entropy are neglected, ΔH is in proportion to the temperature [25, 26]. That is, ΔH is expressed by

$$\Delta H = \alpha kT \quad (13)$$

We investigate the proportional relationships of temperature and the activation enthalpy at the two models for the specimen. The results of Equations 11 and 12 are represented by open circles and triangles in Fig. 6. The open circles appear to be nearly proportional to the temperature, compared with the open triangles. The $\Delta H(T)$ for the Fleischer's model seems to be satisfied with Equation 13 within the temperature range. Then, we can find that the Fleischer's model is suitable for the specimen rather than the F-F from Fig. 6. Therefore, the Friedel relation seems not to be appropriate to the interaction between a dislocation and the impurity in the specimen.

Fig. 7 shows the proportional relation between temperature and the activation enthalpy at the Fleischer's model for the specimen. The value of $\Delta H(T_c)$ taken from Fig. 7 is 0.61 eV. It corresponds to the activation enthalpy for overcoming of the strain field around the I–V dipole by a dislocation at 0 K. The value of $\Delta H(T_c)$ at the F-F is 0.73 eV.

4. Gibbs free energy for overcoming of tetragonal lattice distortions by a dislocation

When the force-distance relation between a dislocation and an impurity can be approximated by the Fleischer's model, the Gibbs free energy for overcoming of the impurity by the dislocation, ΔG, was expressed by [5]

$$\Delta G = \Delta G_0 \left\{1 - (\tau/\tau_0)^{1/2}\right\}^2, (\Delta G_0 = \tau_0\, Lb^2) \quad (14)$$

where τ_0 is the effective shear stress τ at the temperature of 0 K, L is the average length of dislocation segments and b is the magnitude of the Burgers vector. From an Arrhenius equation for the thermally activated deformation rate, $\dot{\varepsilon}$, the Gibbs free energy of activation is also expressed as

$$\Delta G = \alpha kT, (\alpha = \ln(\dot{\varepsilon}_0/\dot{\varepsilon})) \tag{15}$$

where $\dot{\varepsilon}_0$ is a frequency factor. Differentiating the combining Equations 14 and 15 with respect to the shear stress gives

$$\partial \ln \dot{\varepsilon}/\partial \tau = \{\Delta G_0/(kT\tau_0)\}(\tau_0/\tau)^{1/2}\{1-(\tau/\tau_0)^{1/2}\} + \partial \ln \dot{\varepsilon}_0/\partial \tau \tag{16}$$

Substituting Equation 3 in Equation 16 gives

$$\partial \ln \dot{\varepsilon}/\partial \tau = \{\Delta G_0/(kT\tau_{p0})\}\{1-(T/T_c)^{1/2}\}^{-1} \times (T/T_c)^{1/2} + \partial \ln \dot{\varepsilon}_0/\partial \tau \tag{17}$$

where τ_0 is replaced by τ_{p0}. The result of calculations of Equation 17 is shown by the open circles in Fig. 8. The $\partial \ln \dot{\varepsilon}/\partial \tau$ in Equation 17 corresponds to the $(\Delta \ln \dot{\varepsilon}/\Delta \tau')_p$ for the specimen in stage II of stress-strain curve. Only from the data denoted by open circles, it is difficult to obtain the ΔG_0 for the specimen on the basis of the slope of a line. We examine the value of $(\Delta \ln \dot{\varepsilon}/\Delta \tau')_p$ at which the line intersects the ordinate in Fig. 8. It corresponds to the $\partial \ln \dot{\varepsilon}_0/\partial \tau$ in Equation 17. The $\dot{\varepsilon}_0$ is usually expressed by [27, 28]

$$\dot{\varepsilon}_0 = \rho b^2 \nu_D (L_0/L)^2 \tag{18}$$

where ρ is the density of mobile dislocations, ν_D is the Debye frequency and L_0 is the average spacing of impurities on the slip plane. Differentiating the natural logarithmic equation of Equation 18 with respect to the effective shear stress gives

$$\partial \ln \dot{\varepsilon}_0/\partial \tau = (\partial \ln \rho/\partial \tau) - 2(\partial \ln L/\partial \tau) \tag{19}$$

The $\partial \ln L/\partial \tau$ is zero in the Fleischer's model. Therefore, the $\partial \ln \dot{\varepsilon}_0/\partial \tau$ in Equation 17 for the Fleischer's model can be expressed from Equation 19 by

$$\partial \ln \dot{\varepsilon}_0/\partial \tau = \Delta \ln \dot{\varepsilon}_0/\Delta \tau' = \Delta \ln \rho/\Delta \tau' \tag{20}$$

The $\Delta\rho/\Delta\tau'$ is previously obtained for KCl doped with Ca^{2+} (0.035 and 0.065 mol% in the melt), Sr^{2+} (0.035, 0.050 and 0.065 mol% in the melt) or Ba^{2+} (0.050 and 0.065 mol% in the melt) in stage II [29]. However, no difference of $\Delta\rho/\Delta\tau'$ among the three kinds of specimens can be discriminated because of scattering $(\Delta \ln \dot{\varepsilon}/\Delta \tau')_p$. The results of $\Delta \ln \rho/\Delta \tau'$ are tabulated in Table II. The value of $\Delta \ln \rho/\Delta \tau'$ ranges from 8.94 to 13.06 MPa^{-1} and may not be influenced by the size and the concentration of divalent impurity. Assuming that the magnitude of the $\Delta \ln \rho/\Delta \tau'$ for KCl:Mg^{2+} is around those for the KCl doped with Ca^{2+}, Sr^{2+} or Ba^{2+}, the $\partial \ln \dot{\varepsilon}_0/\partial \tau$ in Equation 17 is represented by solid circles in Fig. 8. Then, the ΔG_0, which is obtained from the slope of dashed lines, seems to be between 0.42 and 0.48 eV for the specimen.

When KCl single crystals are doped with Mg^{2+}, tetragonal lattice distortions are expected to be produced around the I-V dipoles. The tetragonality, $\Delta\varepsilon$, is calculated from the following equation [10]:

$$\Delta\varepsilon = 3.81\Delta G_0/(b^3\mu) \tag{21}$$

where μ is the shear modulus for [110] direction at 0 K and is assumed to be 1.01×10^{10} Pa [30]. The value of $\Delta\varepsilon$ for KCl:Mg^{2+} would be within the range of 0.29 to 0.33.

The values of ΔG_0 and $\Delta\varepsilon$ at the F-F are also given in Table III. The ΔG_0 is calculated on the basis of the equation [10]:

$$\partial \ln \dot{\varepsilon}/\partial \tau = \{2\Delta G_0/(3kT\tau_{p0})\}\{1-(T/T_c)^{1/2}\}^{-2} \times (T/T_c)^{1/2} + \partial \ln \dot{\varepsilon}_0/\partial \tau \tag{22}$$

The two values may be slightly small in contrast to those at the Fleischer's model from Table III.

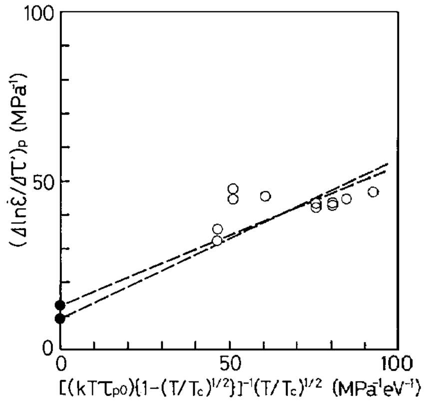

Figure 8 Linear plots of Equation 17 for KCl:Mg^{2+} (0.035 mol% in the melt). (○): $(\Delta \ln \dot{\varepsilon}/\Delta \tau')_p$. (●): $\partial \ln \dot{\varepsilon}_0/\partial \tau$.

TABLE II Values of c and $\Delta \ln \rho/\Delta \tau'$ at 0 K

Specimen	(mol% in the melt)	c (p.p.m.)[a]	$\Delta \ln \rho/\Delta \tau'$ (MPa^{-1})
KCl:Ca^{2+}	(0.035)	43.1	13.06
	(0.065)	43.5	13.05
KCl:Sr^{2+}	(0.035)	55.2	8.94
	(0.050)	98.3	8.97
	(0.065)	121.8	8.98
KCl:Ba^{2+}	(0.050)	9.2	12.38
	(0.065)	28.3	12.41

[a]The concentration of divalent impurities is obtained by dielectric loss measurement.

TABLE III Values of ΔG_0 and $\Delta\varepsilon$ for the specimen at the two models

	ΔG_0 (eV)	$\Delta\varepsilon$
Fleischer's model	0.42–0.48	0.29–0.33
F-F	0.40–0.46	0.27–0.31

5. Conclusions

1. The relation between $(\Delta\tau'/\Delta \ln \dot{\varepsilon})_p$ and temperature for the specimen is approximated to the dependence of SRS due to the impurities on temperature at the Fleischer's model rather than the F-F in Fig. 2. Furthermore, the ΔH calculated from Equation 11 for the Fleischer's model seems to be nearly proportional to the temperature in comparison with the F-F. These results show that the Fleischer's model is more suitable than the F-F. Therefore, it is considered that the Friedel relation is not appropriate to the interaction between a dislocation and the impurity in the specimen, though it is exact for most weak obstacles to dislocation motion.

2. Assuming that the changes in entropy are neglected, the value of $\Delta H(T_c)$ at the Fleischer's model is found to be 0.61 eV for the specimen. It is obtained from the proportional relation of $\Delta H(T)$ as shown in Fig. 7.

3. Although the ΔG_0 for the specimen cannot be obtained only from the result of calculations of Equation 17, we attempt to estimate the ΔG_0 through examining the value of the $\partial \ln \dot{\varepsilon}_0/\partial\tau$. The $\partial \ln \dot{\varepsilon}_0/\partial\tau$ for the Fleischer's model corresponds to $\Delta \ln \rho/\Delta\tau'$. As a result, the ΔG_0 is considered to be between 0.42 and 0.48 eV from the slope of dashed lines in Fig. 8. In addition, the $\Delta\varepsilon$ for the specimen would be within the range of 0.29 to 0.33 on the basis of the value of ΔG_0.

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