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Interaction between a dislocation and impurities in KCl single crystals

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The strain-rate cycling test during the Blaha effect at 77–220 K has been carried out for three kinds of single crystals, KCl, KCl doped with Sr^{2+}, and KCl doped with various impurities. The temperature and impurity dependence of the relation between strain-rate sensitivity and stress decrement, as well as the effective stress, τ_{p1}, due to only one type of impurity lying on the dislocation with the largest separation, has been investigated. From the temperature dependence of τ_{p1}, the force–distance profile between a dislocation and impurities was obtained. The critical temperature, T_c, for KCl: Sr^{2+} was found to be about 227 K.

1. Introduction

Interaction between a dislocation and a solute atom has been so far investigated by yield stress [1–5], internal friction [6–11], or direct observation [12–14]. Recently, Ohgaku and Takeuchi have reported that the strain-rate cycling test during the Blaha effect measurement can separate the effective stress due to a weak obstacle such as an impurity, from that due to dislocation cutting [15, 16]. The Blaha effect is the phenomenon where static flow stress decreases when an ultrasonic oscillatory stress is superimposed. Ohgaku and Takeuchi reported that strain-rate sensitivity varies with the stress decrement at a given strain, and the relation between the strain-rate sensitivity and the stress decrement provides information on the interaction between a dislocation and an impurity. However, the report concerns only room temperature.

We have measured the strain-rate sensitivity and the stress decrement due to oscillation for three kinds of KCl single crystals at lower temperature. The purpose of this paper was to investigate the temperature and impurity dependence of the relation between the strain-rate sensitivity and the stress decrement. Furthermore, we discuss the temperature dependence of the effective stress due to only one type of weak obstacle.

2. Experimental procedure

Three kinds of single crystal used in this work, KCl, KCl doped with Sr^{2+}(0.035, 0.050, 0.065 mol % in the melt), and KCl doped with various impurities sodium, calcium, manganese, nickel, strontium, silver, caesium, barium, thallium and lead (0.050 mol %, respectively, in the melt), were grown from the melt of a superfine reagent of powders by the Kyropoulos method in air. The specimens, which were obtained from the ingots by cleaving to the size $5 \times 5 \times 15$ mm^3, were annealed at 973 K for 24 h in order to reduce dislocation density as far as possible, followed by cooling to room temperature at the rate of 40 K h^{-1}. Furthermore, the specimens were held at 673 K for 30 min, followed by quenching to room temperature immediately before the test, in order to disperse the impurities into them.

A schematic illustration of the apparatus is shown in Fig. 1. A resonator composed of a vibrator and a horn with the resonant frequency of 20 kHz was attached to the testing machine, Shimadzu DSS-500. The specimens were deformed by compression along the ⟨1 0 0⟩ axis and the ultrasonic oscillatory stress was applied by the resonator in the same direction as the compression. The amplitude of the oscillatory stress was observed by a piezoelectric transducer.

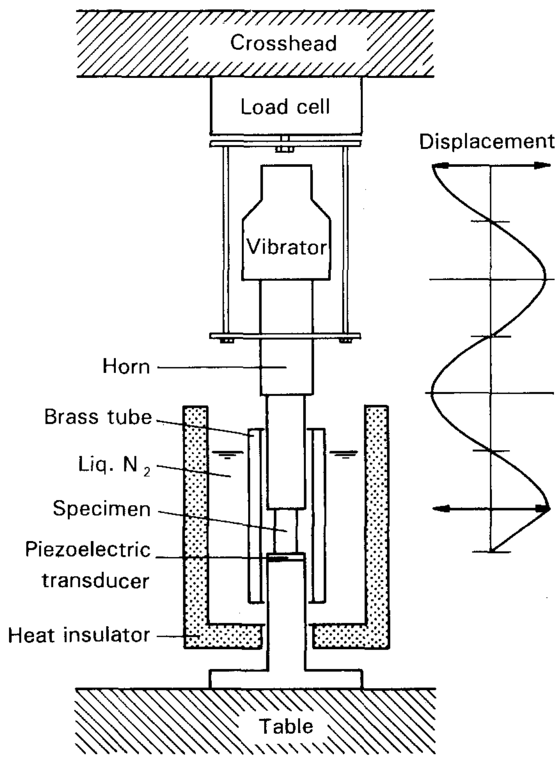

Figure 1 Schematic illustration of the apparatus.

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Because the wavelength, which is 225 mm, is 15 times as long as the length of the specimen, the strain of the specimen is considered to be homogeneous.

The strain-rate cycling test during the Blaha effect measurement is illustrated in Fig. 2. Superposition of oscillatory stress, τ_v, during plastic deformation causes a stress drop, $\Delta\tau$. Keeping the stress amplitude, τ_v, constant, strain-rate cycling between the strain rates of $\dot{\varepsilon}_1$ and $\dot{\varepsilon}_2$ is carried out. Then, the stress change due to the strain-rate cycling is $\Delta\tau'$. The strain-rate cycling tests made between the crosshead speeds of 20 and 100 μm min^{-1} were performed at temperatures from 77–220 K. The strain-rate sensitivity, $\Delta\tau'/\Delta \ln \dot{\varepsilon}$, is given by $\Delta\tau'/1.609\ \Delta\tau'$.

Fig. 3 shows the variation of the strain-rate sensitivity and the stress decrement with the shear strain for KCl: Sr^{2+}(0.050 mol %) at 200 K. The relation between the strain-rate sensitivity and the stress decrement at a given strain, which is shown in Fig. 4, is obtained from Fig. 3.

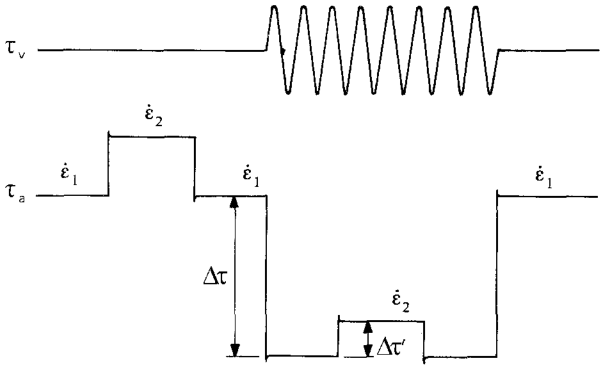

Figure 2 Variation of applied shear stress, τ_a, when the strain-rate cycling between the strain rate, $\dot{\varepsilon}_1$ and $\dot{\varepsilon}_2$, is carried out under superposition of ultrasonic oscillatory shear stress, τ_v.

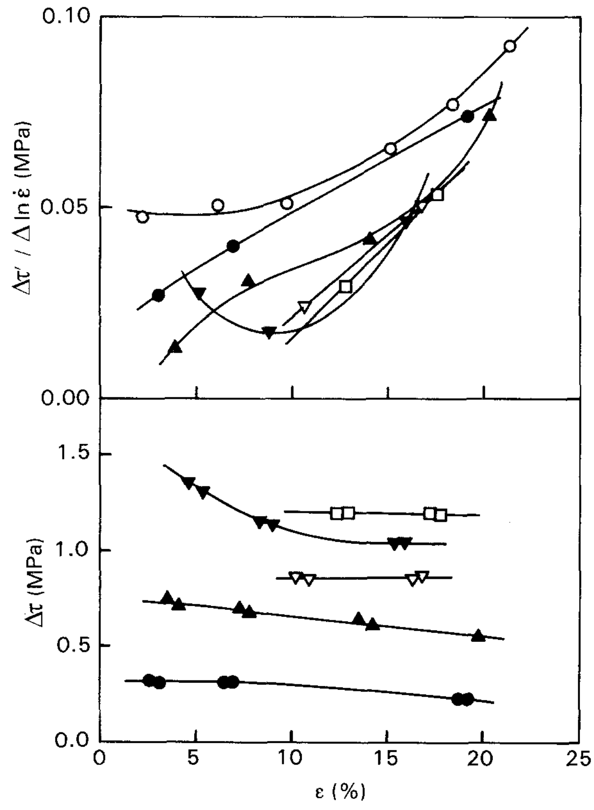

Figure 3 Variation of the strain-rate sensitivity, $\Delta\tau'/\Delta \ln\dot{\varepsilon}$, and the stress decrement, $\Delta\tau$, with shear strain for KCl: Sr^{2+} (0.050 mol %) at 200 K. τ_v (arb. units): (○) 0, (●) 10, (▲) 25, (▽) 35, (▼) 45, (□) 50.

3. Results

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

The relation between the strain-rate sensitivity and the stress decrement obtained by the method mentioned above is shown in Figs 4 and 5 for KCl: Sr^{2+}(0.050 mol %). Fig. 4 corresponds to the case of one specimen at several strains. Fig. 5 concerns several temperatures. As can be seen from Figs 4 and 5, there are two bending points on each curve, and there are two plateau regions: the first plateau region ranges below the first bending point, τ_{p1}, at low stress decrement and the second one extends from the second bending point, τ_{p2}, at high stress decrement. The second plateau region is considered to correspond to the plateau region reported by Ohgaku and Takeuchi [16]. The strain-rate sensitivity decreases with the stress decrement between the two bending points. Furthermore, Fig. 4 shows the influence of the shear strain on the relation between the strain-rate sensitivity and the stress decrement. The curve shifts upwards with increasing shear strain. This phenomenon is

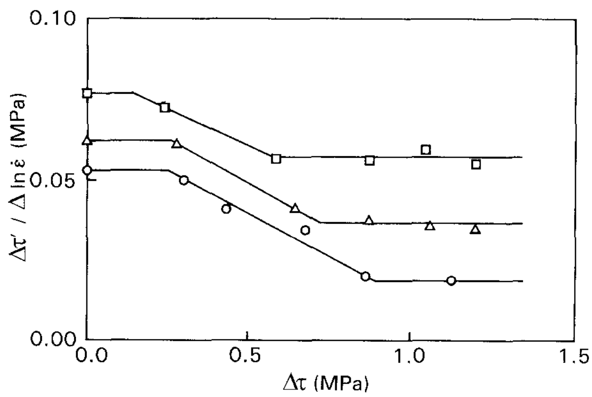

Figure 4 Relation between the strain-rate sensitivity and the stress decrement for KCl: Sr^{2+} (0.050 mol %) at 200 K. ε: (○) 10%, (△) 14%, (□) 18%.

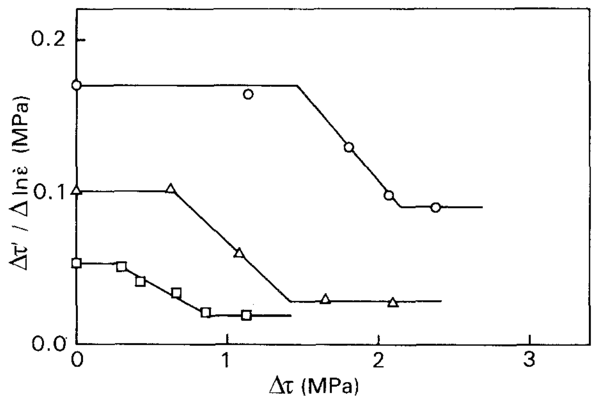

Figure 5 Relation between the strain-rate sensitivity and the stress decrement for KCl: Sr^{2+} (0.050 mol %) at various temperatures: (○) 103 K, ε = 9% (△) 133 K, ε = 8% (□) 200 K, ε = 10%.

caused by that part of the strain-rate sensitivity which depends on dislocation cuttings. Because the dislocation cuttings increase with increasing strain, the strain-rate sensitivity increases [16]. Fig. 5 shows the influence of temperature on the relation between the strain-rate sensitivity and the stress decrement. As the temperature is lower, τ_{p1} is larger.

Fig. 6 shows the relation between the strain-rate sensitivity and the stress decrement for nominally pure KCl. In contrast to KCl: Sr^{2+}, there is only one bending point on each curve which is considered to correspond to τ_{p2}. The bending point shifts in the direction of higher stress decrement with decreasing temperature.

It is shown in Fig. 7 that no bending point appears for KCl containing various impurities. The strain-rate sensitivity only decreases with increasing stress decrement.

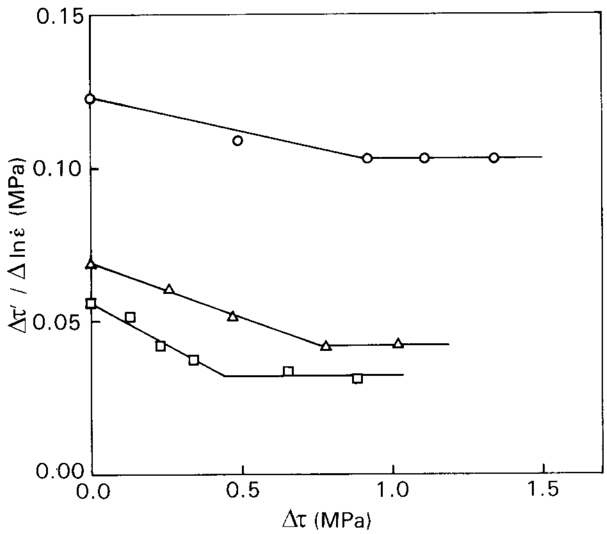

Figure 6 Relation between the strain-rate sensitivity and the stress decrement for KCl at various temperatures: (○) 77 K, (△) 94 K, (□) 168 K.

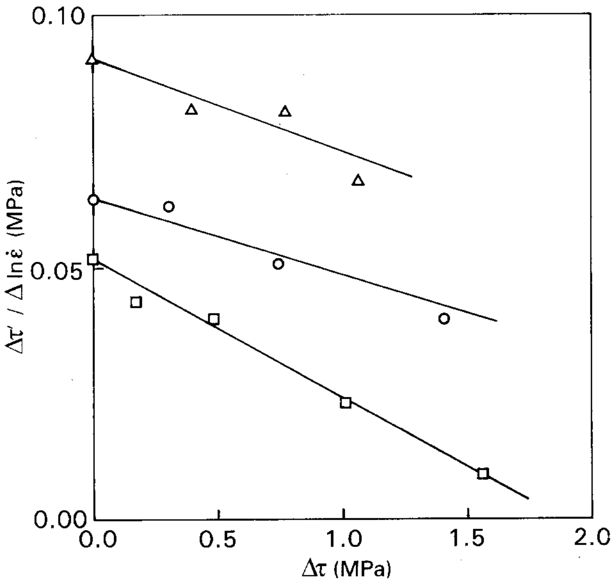

Figure 7 Relation between the strain-rate sensitivity and the stress decrement for KCl doped with various impurities at various temperatures: (○) 151 K, (△) 159, (□) 195 K.

3.2 Dependence of τ_{p1} and τ_{p2} on the yield stress for KCl: Sr^{2+}

It is clear from Figs 4–7 that the curve of the strain-rate sensitivity and the stress decrement has two bending points and two plateau regions only for KCl: Sr^{2+}, which is considered to have only one type of impurity. Thus, τ_{p1} and τ_{p2} may depend on impurity concentration. Fig. 8 shows the dependence of τ_{p1} and τ_{p2} on the yield stress for KCl: Sr^{2+} at 150 K. The plots correspond to the case when the Sr^{2+} concentration is 0.035, 0.050, and 0.065 mol % from the bottom. It can be seen from this figure that both τ_{p1} and τ_{p2} are approximately proportional to the yield stress. This means that τ_{p1} and τ_{p2} increase, depending on the impurity concentration, because the yield stress generally increases with increasing impurity concentration [1–5].

4. Discussion

4.1. Relation between the strain-rate sensitivity and the stress decrement for KCl: Sr^{2+}

The above experimental facts that the curve of the strain-rate sensitivity and stress decrement for only KCl: Sr^{2+} has two bending points and two plateau regions, and both τ_{p1} and τ_{p2} depend on Sr^{2+} concentration, suggest that the phenomena shown in Figs 4 and 5 are attributable to the interaction between a dislocation and only one type of obstacle. The reason for this is discussed below.

The strain-rate sensitivity relates to the activation volume, i.e. the average length of dislocation segment. In addition, it is reported that the length of the dislocation segment increases and the strain-rate sensitivity decreases when the ultrasonic oscillatory stress is applied at room temperature during plastic deformation and that the plateau region is due to the dislocation cuttings when oscillations cause the dislocation to break away from all weak obstacles [16].

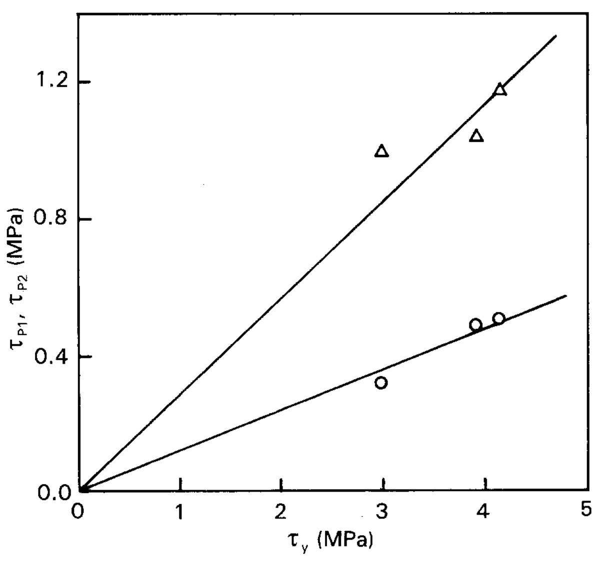

Figure 8 Dependence of τ_{p1} and τ_{p2} on the yield stress, τ_y, for KCl: Sr^{2+} at 150 K. (○, △) Concentration of Sr^{2+} = 0.035, 0.050, 0.065 mol % from the left.

Therefore, the first plateau region, as well as the second one, indicates that the average length of the dislocation segment remains constant there. Thus, application of oscillations with low stress amplitude cannot influence the average length of the dislocation segment at low temperature, but it can do so at high temperature, such as room temperature. Therefore, the plateau region appears at low stress decrement in Figs 4 and 5. Now we imagine a dislocation pinned by many weak obstacles and bowing by applied stress between a few strong obstacles during stationary plastic deformation. When the stress amplitude increases, the dislocation begins to break away from weak obstacles by oscillation between the strong ones and the average length of the dislocation begins to increase. The strain-rate sensitivity starts to decrease at the stress decrement of τ_{p1}. This τ_{p1} should depend on temperature, and on type and density of the obstacle. Consequently, the phenomena shown in Figs 4–7 reflect the influence of ultrasonic oscillation on the dislocation motion on the slip plane containing many weak obstacles and a few strong ones, during plastic deformation. Furthermore, τ_{p1} is considered to represent the effective stress due to the weak obstacles which lie on the dislocation with the largest separation, because the dislocation begins to break away from these weak obstacles with the help of oscillation. The stress decrement at which the ultrasonic oscillatory stress helps the dislocation break away from all weak obstacles is τ_{p2}, as reported by Ohgaku and Takeuchi [16]. At a stress decrement more than τ_{p2} the obstacles to the dislocation motion are only strong ones such as dislocation cuttings.

The curve of the strain-rate sensitivity and the stress decrement for the specimen containing many types of obstacle should be obtained by superimposition of various curves for each obstacle. Therefore, the bending points and plateaus should not be clear. The curve shown in Fig. 7 corresponds to this case. KCl crystal may contain a small amount of various impurities, although none were added. As a result, the first bending point does not appear. The second bending point, however, appears because the amount of impurities is small.

4.2. Dependence of τ_{p1} and τ_{p2} on the temperature for KCl: Sr^{2+}

If τ_{p1} is the effective stress due to the weak obstacles with the largest separation, it should depend on temperature and impurity concentration. Fig. 9a–c show the dependence of τ_{p1} and τ_{p2} on temperature for KCl: Sr^{2+} (0.065, 0.050 and 0.035 mol %, respectively). It is clear from the figures that τ_{p1} and τ_{p2} tend to increase with decreasing temperature for three specimens. Both τ_{p1} and τ_{p2} increase with increasing concentration of Sr^{2+} at a given temperature. The critical temperature, T_c, at which the curves intersect the abscissa and τ_{p1} is zero, may be determined from these figures. Then, T_c appears to be constant independently of the Sr^{2+} concentration. It is clear from these phenomena that τ_{p1} corresponds to the effective stress due to the weak obstacles which lie on the dislocation with the largest

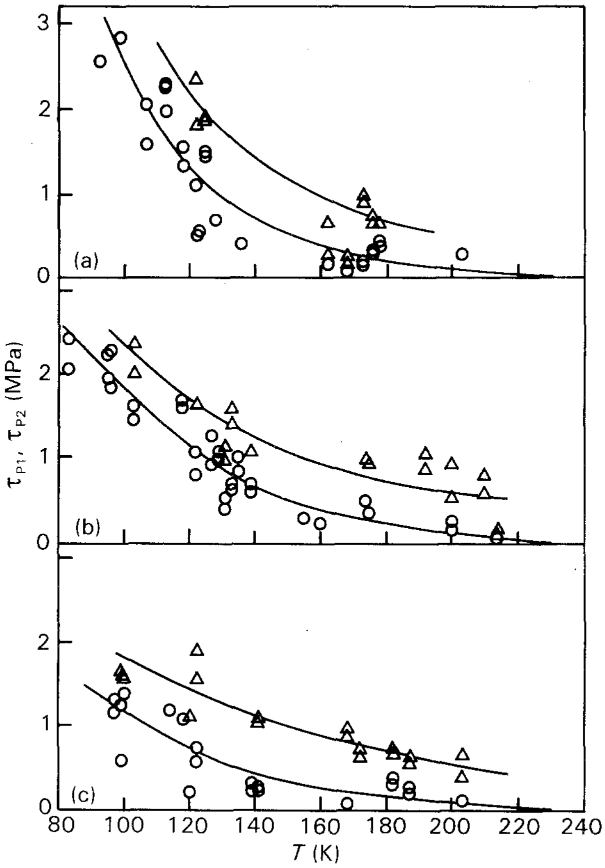

Figure 9 Dependence of τ_{p1} and τ_{p2} on temperature for KCl: Sr^{2+}. The Sr^{2+} concentration is (a) 0.065, (b) 0.050, and (c) 0.035 mol %.

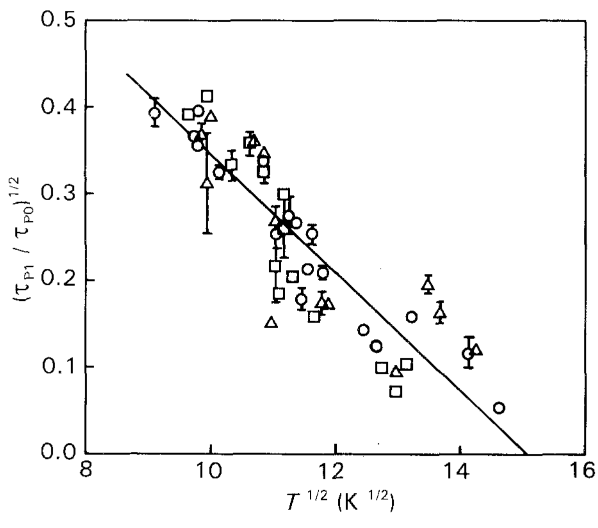

Figure 10 Linear plots of $(\tau_{p1}/\tau_{p0})^{1/2}$ and $T^{1/2}$ for KCl: Sr^{2+}: (△) 0.035 mol %, (○) 0.050 mol %, (□) 0.065 mol %.

separation when the dislocation moves forward. Observation of τ_{p1}, therefore, provides information on the interaction between a dislocation and weak obstacles. That is, the temperature dependence of τ_{p1} reveals the force–distance profile which expresses the interaction between a dislocation and weak obstacles.

The tetragonal distortion resulting from the introduction of the divalent cations into alkali halides is generally formed in the lattice, and it is well known that the Fleischer's model, which is the most successful one for the dramatic hardening of divalent cations, is suitable for the interaction between a dislocation and

TABLE I Values of τ_{p0}

KCl: Sr^{2+} (mol %)	τ_{p0}(MPa)
0.035	9.01
0.050	14.52
0.065	16.71

obstacles [17]. Then, the relation between the effective stress and the temperature can be approximated as

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

where τ_{p0} is the effective stress due to the weak obstacles without thermal activation. Fig. 10 shows that the relation between τ_{p1} and temperature for KCl: Sr^{2+} agrees with the above equation.

We can also determine the critical temperature. The value of the critical temperature, T_c, is about 227 K for KCl: Sr^{2+}, and τ_{p0}, which is obtained by extraporating the curve to 0 K, increases with Sr^{2+} concentration. The values of τ_{p0} are given in Table I.

5. Conclusion

When the strain-rate cycling test during the Blaha effect measurement is carried out, the dependence of the strain-rate sensitivity on the stress decrement provides information on the interaction between a dislocation and weak obstacles. The plots of the strain-rate sensitivity and stress decrement for KCl: Sr^{2+} have two bending points and two plateau regions, as shown in Figs 4 and 5. The first bending point, τ_{p1}, corresponds to the effective stress due to weak obstacles which have the largest separation on the mobile dislocation. The temperature dependence of τ_{p1} reveals the force–distance profile between a dislocation and obstacles, and T_c is 227 K for KCl: Sr^{2+}.

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