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Research Article

Study on Dislocation-Dopants Interaction in KCl: Zn^{2+} Single Crystals by Strain-Rate Cycling Tests Combined with Ultrasonic Oscillation

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Abstract

Combination method of strain-rate cycling tests with the ultrasonic oscillation was conducted for KCl:Zn^{2+} (0.1 mol.% in melt) crystals at the temperature of 102 to 299 K and for KCl crystal at 128 K. Relative curve between $\Delta\tau$ and λ has stair-like shape at a given strain for the impure KCl crystal. That is to say, $\Delta\tau$ values (τ_{p1}) at first bending point are observed on the curve. As for the nominal pure KCl crystal, τ_{p1} does not appear on the $\Delta\tau$ vs. λ curve at the low temperature. Therefore, τ_{p1} depends on the dopant ions (i.e., Zn^{2+}) in the KCl crystal. The τ_{p1} tends to be gradually depressed with increasing temperature in similar as the variation of yield stress with it. The critical temperature (*Tc*), at which τ_{p1} becomes zero, is near 217 K. Above the *Tc*, it is considered that the dopant Zn^{2+} ions no longer act as obstacles for dislocation motion in the impure crystal during plastic deformation.

Keywords: Mobile dislocation; Yield stress; Effective stress; Strain-rate cycling test; Ultrasonic oscillation

Introduction

Study on dislocation-point defects interaction has been carried out on the basis of $\Delta\tau$ versus λ below room temperature so far (e.g., [1,2]). $\Delta\tau$ is the stress change due to the application or removal of ultrasonic oscillation. λ is the strain-rate sensitivity of flow stress and is given by the following Equation (1)

$$\lambda = \frac{kT}{bLd} = \frac{\Delta\tau'}{\Delta \ln \dot{\varepsilon}} = \frac{\Delta\tau'}{1.609} \quad (1)$$

where *b* is the magnitude of Burgers vector, *L* is the average length of dislocation segments, *d* is the activation distance, and *kT* has its usual meaning. That is to say, the λ value is inversely proportional to the *L* [3] and was derived here from the stress charge ($\Delta\tau'$) due to the strain-rate cycling between $\dot{\varepsilon}_1$ (2.2×10^{-5} s^{-1}) and $\dot{\varepsilon}_2$ (1.1×10^{-4} s^{-1}) in the middle of ultrasonic oscillation with constant stress amplitude. The $\Delta\tau$ versus λ curve, which is obtained from the values of $\Delta\tau$ versus λ at the same strain, reflects the effect of ultrasonic oscillation on the free flight motion of dislocation on the slip plane containing forest dislocations and point defects such as dopant ions during the plastic deformation of ionic crystal (e.g., [1]). The information on the dislocation motion has been given by the original combination method of strain-rate cycling tests with the ultrasonic oscillation for the many kinds of materials, for example, the sodium halide crystals doped with monovalent cations (NaBr:Li^+ [4-6] and NaCl:Li^+ [6]) or irradiated by X-ray at room temperature [7]. Motion of dislocation on the slip plane contained obstacles such as Zn^{2+}-cation vacancy dipoles, which cause rapid hardening, is further investigated here by the original method, following on from the previous paper [8].

Experimental Procedure

Nominal pure KCl and KCl single crystals doped with Zn^{2+} (0.1 mol.% in melt) were cleaved to the size 5×5×15 mm^3 and were subjected to the following heat treatments. Annealing at 973 K for 24 h, these crystals were gradually cooled to room temperature to remove as much internal strain as possible. Furthermore, the impure crystals only were quenched after keeping at 673 K for 30 min for the purpose of dispersing the Zn^{2+}-cationic vacancy dipoles into them immediately before the original combination tests described in the introduction section.

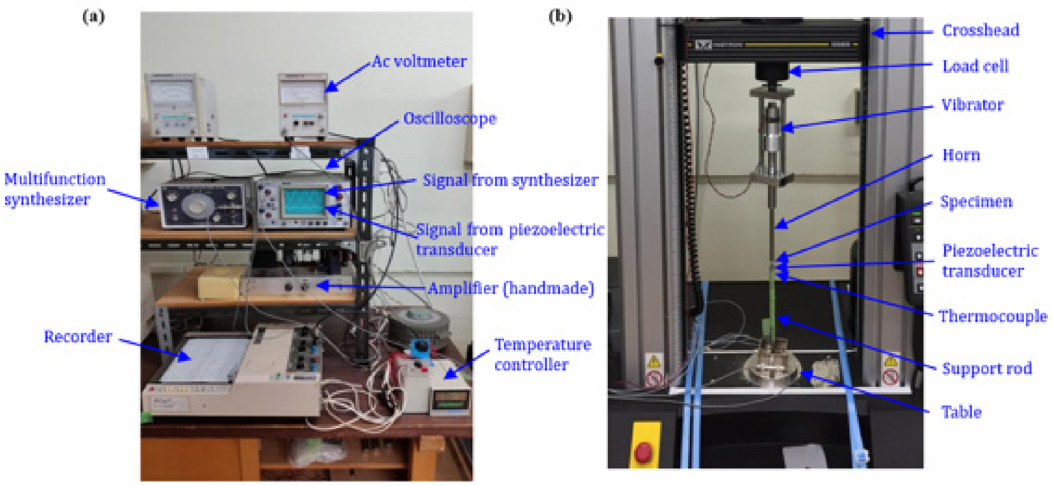

Figure 1: Experimental apparatus: (a) ultrasonic generator and temperature controller, etc.; (b) compressive testing machine connected with resonator.

Figure 1(a) and (b) show the photographs of experimental apparatus. Strain-rate cycling tests between the strain rates of 2.2×10^{-5} and 1.1×10^{-4} s^{-1} were conducted in association with ultrasonic oscillation (20 kHz), using this test machine (Instron 5565) connected with resonator and keeping the stress amplitude constant during plastic compression at 102 to 299 K. This experimental method was detailed in the paper [1].

Results and Discussion

In Figure 2, the $\Delta\tau$ versus λ is shown for KCl:Zn^{2+} crystals at various temperatures and has stair-like shape. Two bending points of τ_{p1} (at low stress decrement $\Delta\tau$) and τ_{p2} (at high $\Delta\tau$) are observed on the variation of λ with $\Delta\tau$ at 134, 180, and 188 K in the figure. And further, these two stress values seem to be smaller at higher temperature, although the second bending point (i.e., τ_{p2}) does not appear on the relationship line at 117 and 124 K since the ultrasonic oscillation with larger stress amplitude could not be applied to the crystals during the tests.

With regard to the nominal pure KCl single crystal at the low temperature of 128 K, the $\Delta\tau$ versus λ curves are shown in Figure 3. Blue, red, and gray solid lines represent the relationships at the strains of 10, 11, and 12 %. In low $\Delta\tau$, the plateau place does not appear and λ all slopes downwards with rising $\Delta\tau$. No the first bending points (i.e., τ_{p1}) appear on the relationship lines for the KCl crystal and second bending ones (i.e., τ_{p2}) only can be seen in the figure. Similar phenomenon as Figure 3 is also seen at other low temperatures. Therefore, the τ_{p1}, observed for the impure KCl crystals in Figure 2, is considered to represent the effective stress due to the dopant ions Zn^{2+} in the crystal. And also, the relationship line between $\Delta\tau$ and λ rises upwards as strain increases, although there is some scatter in the plot data as shown in Figure 3. This is caused by the strain-rate sensitivity due to the dislocation cuttings, because the dislocation cuttings increase in the process of plastic deformation [9]. This leads to the increase in λ with strain.

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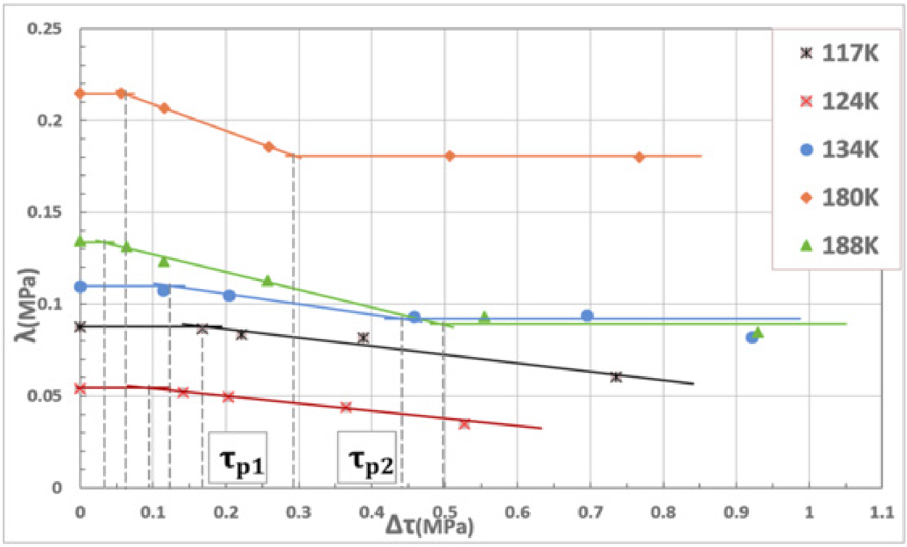

Figure 2: Stress decrement ($\Delta\tau$) versus the strain-rate sensitivity (λ) of flow stress for KCl:Zn^{2+} (0.1mol.% in melt) single crystals at various temperatures. τ_{p1} and τ_{p2} are $\Delta\tau$ values at first and second bending points on the stair-like relationships.

Figure 4 shows the variation of τ_{p1} with temperature for KCl:Zn^{2+} crystals. τ_{p1} value tends to decrease with increasing temperature and becomes zero above around 217 K. Dislocations overcome the obstacles (i.e., Zn^{2+}-cation vacancy dipoles) on the slip plane with the help of thermal activation. At higher temperature, it will be easier to overcome the obstacles by mobile dislocations.

Figure 5 (a) shows the compressive stress versus strain curve in the early deformation region for the KCl: Zn^{2+} crystal at 124 K. Yield stress τ_y has been given by the stress at the intersection of the tangent to the plastic deformation region and the straight line extrapolated from the elastic deformation one in the stress-strain curve [10], as denoted in the figure. Here, the value of τ_y is about 1.5 MPa for the crystal at 124 K. Temperature dependence of τ_y is shown for the crystals below room temperature in Figure 5 (b). Solid curve in the figure was determined as to be fitted to the data plots using the least-squares method. τ_y decreases with increasing temperature and seems to approach constant stress above 200 K.

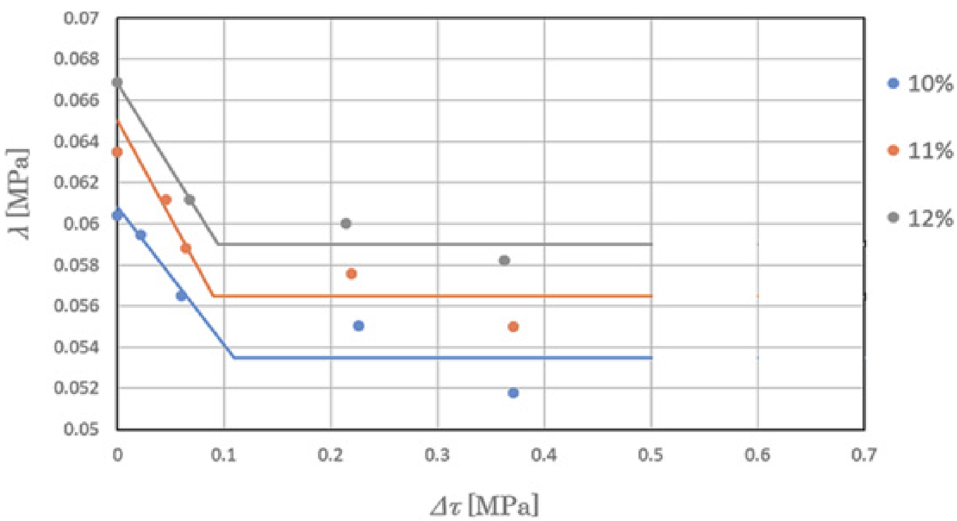

Figure 3: Stress decrement ($\Delta\tau$) versus the strain-rate sensitivity (λ)of flow stress for nominal pure KCl single crystal at 128 K and strains of 10 to 12%.

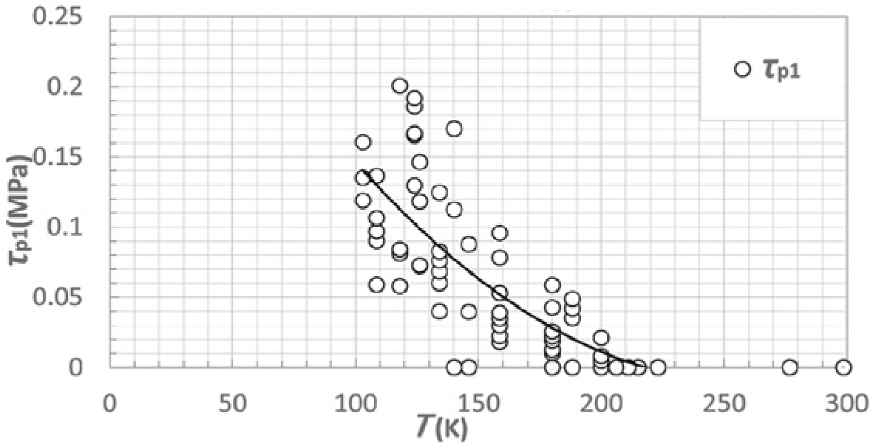

Figure 4: Temperature dependence of τ_{p1} for KCl: Zn^{2+} (0.1mol.% in melt) single crystals.

Figure 6 is concerned with the dependence of τ_{p1}, τ_{p2}, and τ_y on temperature for the KCl:Zn^{2+} crystals. The open circles and triangles represent τ_{p1} and τ_{p2}, respectively, and the solid circles the yield stress τ_y The curves for three kinds of stress are to guide the reader's eye by the least-squares method. As mentioned in the previous paper [8], it has been reported that Zn^{2+}-cationic vacancy dipoles do not act as the obstacles for mobile dislocation by the application of the stress more than τ_{p2}, where strong obstacles such as the forest dislocations behave as obstacles. τ_{p1} and τ_{p2} become gradually large with decreasing temperature as the variation of τ_y within the temperature. The difference between τ_{p1} and τ_{p2} might not be seen and is almost constant at a given temperature in the figure. This may suggest the almost unchanged distribution of the separation between the obstacles (dopant ions Zn^{2+}) on the mobile dislocation independently of temperature.

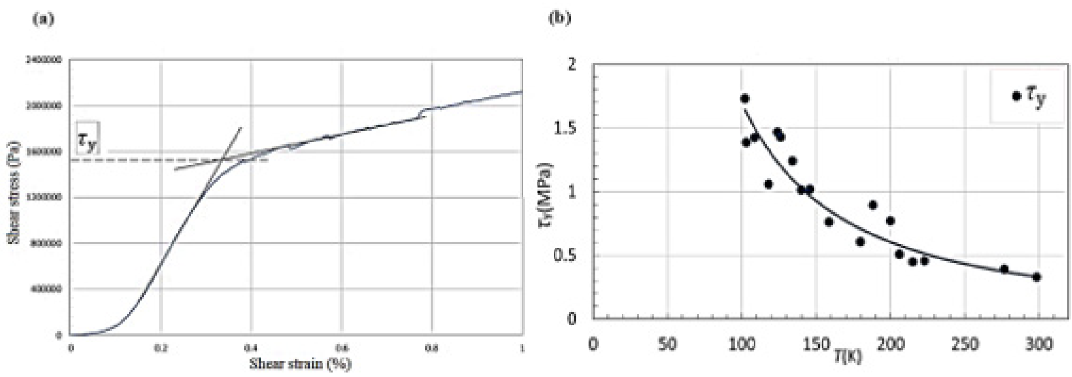

Figure 5: The yield stress (τ_y) of KCl:Zn^{2+} (0.1mol.% in melt) single crystal. (a) Stress – strain curve in the early stage of deformation at 124 K and (b) the variation of yield stress with temperature for the crystals.

The critical temperature T_c at which the intersection with the abscissa of τ_{p1} versus temperature curve in Figure 4 and τ_{p1} becomes zero is 217 K, as mentioned above. There, Zn^{2+}-cationic vacancy dipoles no longer act as impedimenta to movement dislocation in the KCl:Zn^{2+} crystal. That is to say, the dislocations move forward, overcoming the dopants only with the help of thermal activation above the temperature of *Tc* (i.e., 217 K) during the compressive deformation of the crystal. Above near the *Tc*, τ_y also seems to approach a constant value due to internal stress, as can be seen in Figure 6.

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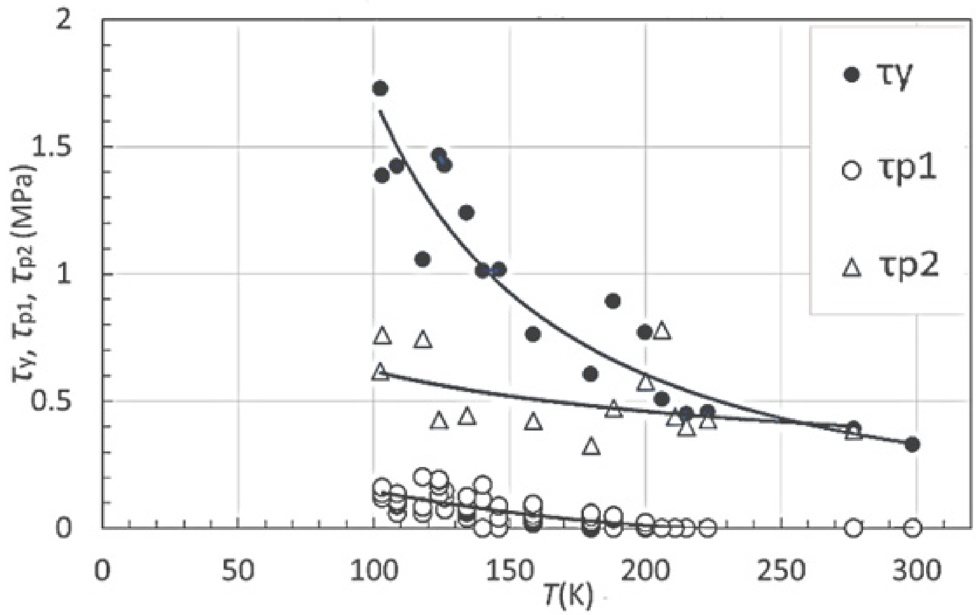

Figure 6: Temperature dependence of τ_{p1}, τ_{p2} and τ_y for KCl:Zn^{2+} (0.1mol.% in melt) single crystals. These τ_{p1} and τ_{p2} data are based on the $\Delta\tau$ versus λ at each temperature. τ_{p1} and τ_y plots correspond to the data in figures 4 and 5(b).

Conclusion

The information on motion of dislocations on the slip plane contained obstacles such as Zn^{2+}-cationic vacancy dipoles was obtained by the original method of strain-rate cycling tests combined with the ultrasonic oscillation for KCl:Zn^{2+} single crystals.

The first bending points τ_{p1} are observed at low stress decrement $\Delta\tau$ on the stair-like $\Delta\tau$ versus λ relationships for KCl:Zn^{2+} crystals at 117 to 188 K, however τ_{p1} does not appear for the nominal pure KCl crystal at the low temperature. This means that the stress τ_{p1} is due to the dopant ions Zn^{2+} in the crystal. τ_{p1} value gradually decreases with increasing temperature as the variation of yield stress and becomes zero at 217 K. Above the critical temperature of 217 K, Zn^{2+}-cationic vacancy dipoles no longer behave as obstacles for mobile dislocations and dislocations break-away from them only with the help of thermal activation during the plastic compression of the KCl:Zn^{2+} single crystal.

Acknowledgement

None.

Conflict of Interest

No conflict of interest.

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