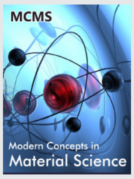

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Research article

Study on Dislocation-Divalent Cations Interaction in Potassium Chloride Crystals by Strain-Rate Cycling Tests Combined with Ultrasonic Oscillation

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Abstract

The strain-rate cycling tests between 2.2×10^{-5} and 1.1×10^{-4} s^{-1} were carried out in association with the ultrasonic oscillation (20 kHz) for $KCl:Zn^{2+}$ (0.1 mol% in melt) single crystals at 102 to 299 K. As a result, the $\Delta\tau$ vs. λ curves at a given strain also have stair-like shape below 200 K, as described those for other crystals so far. $\Delta\tau$ is the stress drop by the application or removal of ultrasonic oscillation during plastic deformation. λ is the strain-rate sensitivity of flow stress, which is derived from the strain-rate cycling in the middle of ultrasonic oscillation with constant stress amplitude. Observation of $\Delta\tau$ vs. λ gives us the information on dislocation motion on the slip plane containing Zn^{2+}-cation vacancy dipoles and forest dislocations mainly, during plastic deformation.

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

Introduction

A large number of investigations on strength of materials have been made with alkali halide crystals [1-5]. This is because the crystals are readily available and transparency. Furthermore, they have simple crystal structure such as rock salt type, compared with some metals. Therefore, alkali halide contained additives is an excellent material for an investigation of mechanical properties.

When alkali halide crystals are doped with divalent cations, the cations induce positive ion vacancies to conserve the electrical neutrality. Then, they are expected to be paired with the vacancies (e.g., [6]). Figure 1 shows a schematic diagram of vicinity around divalent cations in a monovalent ionic crystal. The pairs are termed Impurity-Vacancy dipoles, which are abbreviated to I-V dipoles. Asymmetrical distortions are produced around the I-V dipoles. Those interact strongly with mobile dislocations in the crystal [7] and cause rapid hardening. The hardening is distinguished from gradual hardening due to the defects of cubic symmetry. With regard to the gradual hardening, it has been described for sodium halide crystals doped with monovalent cations ($NaCl:Li^{+}$ and $NaBr:Li^{+}$) in the previous papers [8-10]. Here, the rapid hardening is studied for $KCl:Zn^{2+}$ crystals by the original method: the strain-rate cycling tests combined with ultrasonic oscillation.

Experimental Procedure

An ingot of KCl doped with Zn^{2+} (0.1 mol% in melt) was grown by the Kyropoulos method in air. As a specimen, some KCl:Zn^{2+} single crystals used in this study were made by cleaving the ingot to the size 5×5×15 mm^3. The specimens were prepared by the heat treatment below. KCl:Zn^{2+} single crystals were annealed at 973 K for 24 h and were gradually cooled to room temperature at 40 Kh^{-1} for the purpose of reducing dislocation density as much as possible. And further, the crystals were kept at 673 K for 30 min, following by quenching to room temperature to disperse the isolated I-V dipoles into them immediately before the following tests.

The strain-rate cycling tests between $\dot{\varepsilon}_1$ (2.2×10^{-5} s^{-1}) and $\dot{\varepsilon}_2$ (1.1×10^{-4} s^{-1}) were conducted in association with ultrasonic oscillation (20 kHz), keeping the stress amplitude (τ_v) constant during plastic compression at 102 to 299 K. The main testing machine (Instron 5565) connected with resonator is shown in Figure 2(a) and a close-up photograph of deforming specimen is presented in Figure 2(b). Application or removal of ultrasonic oscillation causes the stress drop $(\Delta\tau)$ during the deformation tests. On the basis of stress increment $(\Delta\tau')$ due to strain-rate cycling, the strain-rate sensitivity (λ) of flow stress was derived in this study. This was already described in the previous paper [11].

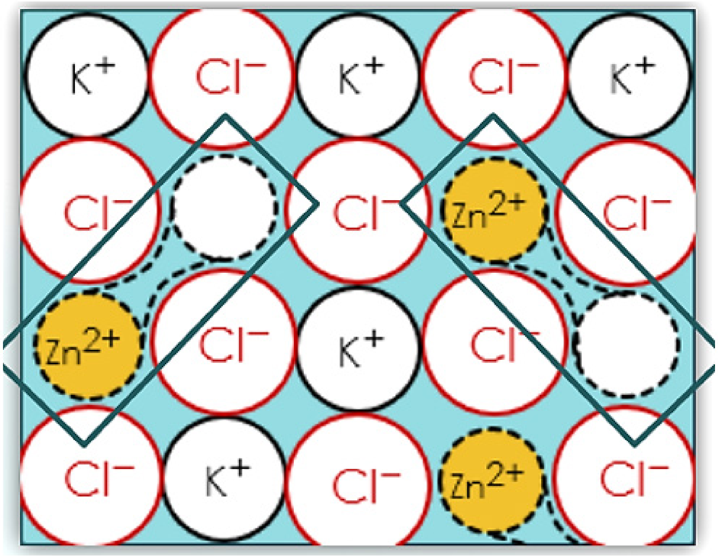

Figure 1: A section through a (111) plane of the KCl:Zn^{2+} single crystal. Tetragonal lattice distortions are formed around Zn^{2+} ion-cation vacancy dipoles in the crystal.

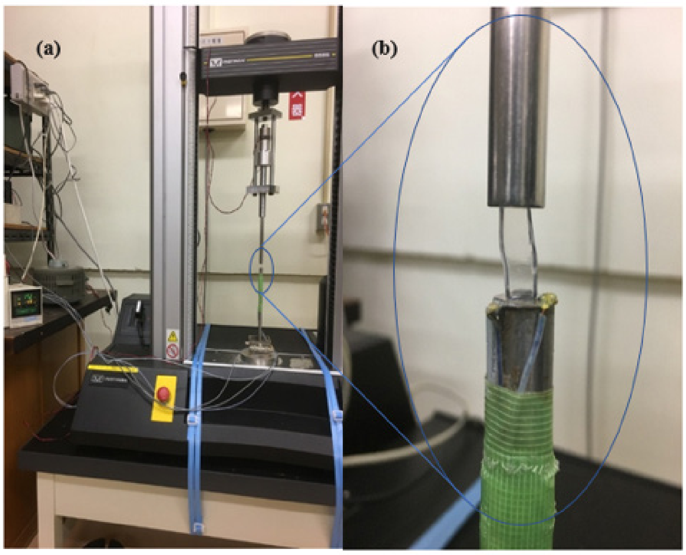

Figure 2: Experimental apparatus: (a) main testing machine connected with resonator and (b) scaled-up representation of the sample compressed by deforming of the strain-rate cycling tests combined with ultrasonic oscillation at room temperature (299 K).

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Results and Discussion

The strain hardening curve is shown in Figure 3 for the specimen at 128 K. As the characteristic behavior of rock salt structural single crystal, three-stage strain hardening is observed in the figure, although the stress versus strain curve is bumpy because of the strain-rate cycling in addition to the ultrasonic oscillation. An enlarged relative curve of Figure 3 below 5.1 % is shown in Figure 4. And further scaled up (a) and (b) in Figure 4 are presented in (a’) and (b’) in the figure, respectively. The (a’) corresponds to the case of strain-rate cycling without oscillation (i.e., τ_v =0), while (b’) that in the middle of ultrasonic oscillatory stress (τ_v =10 (arb. units)). The stress drop $\Delta\tau_1$ occurs by the application of ultrasonic oscillation during the plastic deformation. And also, the removal of it yields almost the same stress rise $\Delta\tau_2$ as $\Delta\tau_1$ in Figure 4(b’). The strain-rate cycling test keeping the stress amplitude τ_v constant makes the stress change $(\Delta\tau')$ on the stress versus strain in the figure. The strain-rate sensitivity λ, which is inversely proportional to the average length of dislocation segments (12), of flow stress is obtained by using $\Delta\tau'$. That is to say, λ value was estimated from $\Delta\tau'/\Delta\ln\dot{\varepsilon}$ (=$\Delta\tau'$/1.609).

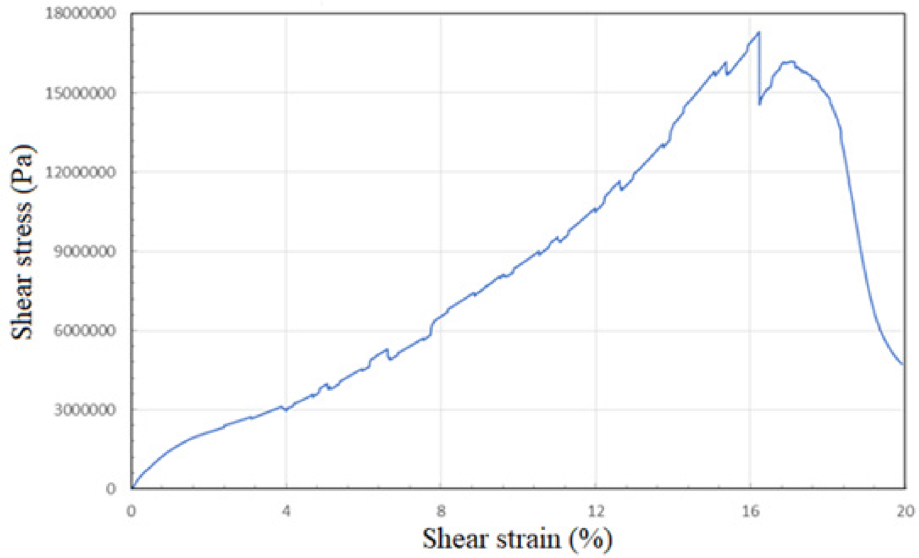

Figure 3: Stress – strain curve by the strain-rate cycling tests combined with ultrasonic oscillation during plastic deformation of KCl:Zn^{2+} (0.1 mol% in melt) at 128 K.

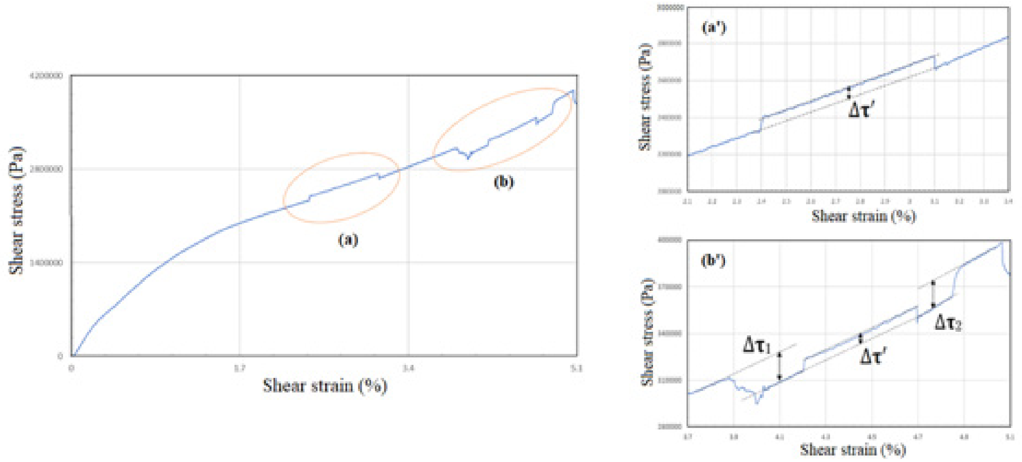

Figure 4: Stress – strain curve below 5.1 % by the strain-rate cycling tests between $\dot{\varepsilon}_1$ (2.2×10^{-5} s^{-1}) and $\dot{\varepsilon}_2$ (1.1×10^{-4} s^{-1}) in association with ultrasonic oscillation during plastic deformation of KCl:Zn^{2+} (0.1 mol% in melt) at 128 K. The curves (a’) and (b’) are scaled-up representations of corresponding the curves (a) τ_v =0 (arb. units) and (b) τ_v =10 (arb. units) in Figure 4.

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Figure 5(a, b) shows the strain dependence of stress decrement $\Delta\tau$ due to the oscillation and strain-rate sensitivity λ of flow stress at various stress amplitude τ_v for KCl:Zn^{2+} (0.1 mol% in melt) at 124 K. The values of $\Delta\tau$ (i.e., $\Delta\tau_1$ and $\Delta\tau_2$) are almost constant independently of strain and become larger and larger with the increase of τ_v (i.e., 0, 10, 20, 40, 60 (arb. units)). On the other hand, λ values tend to increase gradually with strain at all τ_v and decrease with τ_v at a given strain for the specimen. The variation of λ with the stress amplitude at a given strain is small at low or high τ_v, as can be seen in Figure 5(b). Here, the $\Delta\tau$ and λ were noted at the constant strain of 5 to 10 %, where solid lines are drawn at each strain in the figure. The values of $\Delta\tau$ and λ at the same strain are plotted in Figure 6. The solid lines in the figure are to guide a reader's eye. As described the $\Delta\tau$ vs. λ curves for sodium halide crystals contained monovalent cations (Li$^+$ ions) or irradiated by the X-ray at room temperature so far [8-10,13], those for KCl:Zn^{2+} crystal at 124 K also have stair-like shape: the first plateau place ranges until first bending point (see τ_{p1} in Figure 6) and second one extends from the second bending point (see τ_{p2} in the figure). λ decreases with $\Delta\tau$ between the two bending points. This is observed below 200 K.

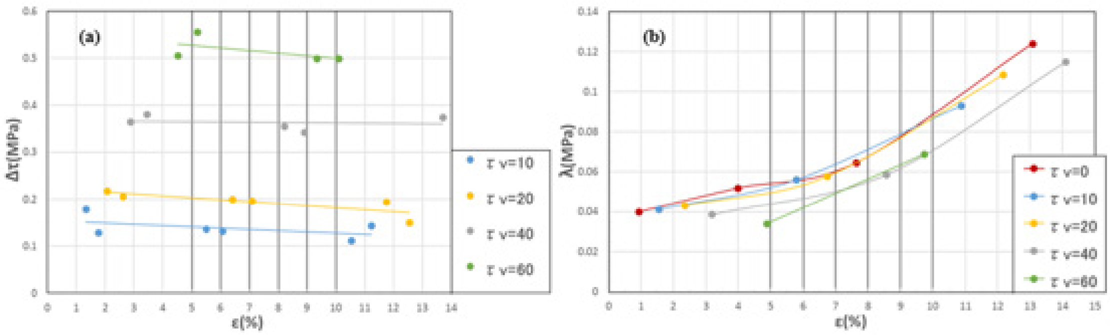

Figure 5: Variation of (a) stress decrement $(\Delta\tau)$ and (b) the strain-rate sensitivity $\left(\lambda\left(=\Delta\tau'/\Delta\ln\dot{\varepsilon}\right)\right)$ of flow stress with shear strain (ε) and various stress amplitude (τ_v (arb. units)) for KCl:Zn^{2+} (0.1mol% in melt) at 124 K.

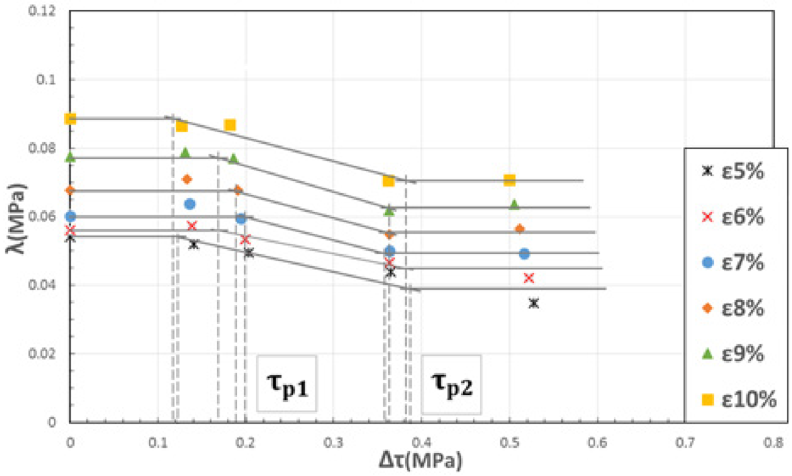

Figure 6: Stress decrement $(\Delta\tau)$ vs. λ for KCl:Zn^{2+} (0.1 mol% in melt) at various strains and 124 K. These plots data in the relative curves are based on the values of $\Delta\tau$ and λ at each stress amplitude τ_v and constant strain within 5 to 10 % in Figure 5.

The aspect of $\Delta\tau$ versus λ curve reflects the movement of dislocation on the slip plane containing the weak obstacles such as Zn^{2+}-cation vacancy dipoles and the strong ones of forest dislocations mainly, during plastic deformation of KCl:Zn^{2+} crystal. In the first plateau place, mobile dislocations are pinned by all the weak obstacles. By oscillation with high stress amplitude, the dislocation begins to break away from them at the effective stress τ_{p1} due to the dopants. This leads to the appearance of decrease in λ between two bending points on the relative curve of $\Delta\tau$ and λ, since λ is inversely proportional to the average length of dislocation segments. Applying oscillation with even higher stress amplitude (i.e., the effective stress more than τ_{p2} in Figure 6), Zn^{2+}-cation vacancy dipoles no longer act as the impedimenta for mobile dislocations in the second plateau place. Strong obstacles such as forest dislocations etc. act as that there.

Figure 6 concerns the influence of the shear strain on the $\Delta\tau$ versus λ curve for KCl:Zn^{2+} (0.1 mol% in melt) crystal. Namely, the curve shifts upward in the process of plastic deformation by the compression. This result is attributed to the phenomenon that the λ due to dislocation cuttings increases with increasing strain, because forest dislocations multiply with it. But the effective stress τ_{p1} is not almost influenced by the multiplication of them, as can be seen in Figure 6.

Conclusion

By the strain-rate cycling in the middle of ultrasonic oscillation with constant stress amplitude, the stair-like relative curves of $\Delta\tau$ versus λ are also observed for KCl:Zn^{2+} (0.1 mol% in melt) single crystals below 200 K, which are similar to the relative curve for the sodium halide crystals contained monovalent cations (Li^+ ions) or irradiated by the X-ray at room temperature. The $\Delta\tau$ versus λ at a given strain reflects the effect of ultrasonic oscillation on free flight motion of dislocation on the slip plane containing mainly two kinds of obstacles (one is the weak obstacles such as Zn^{2+}-cation vacancy dipoles and the other the strong ones of forest dislocations) during plastic deformation of KCl:Zn^{2+} crystal. As the plastic deformation proceeds by the compression, the curve shifts upward with strain.

Acknowledgement

None.

Conflict of Interest

No conflict of interest.

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