Structure of Cross-Slip Bands and Brittle-to-Ductile Transition in Ionic Crystals^{*1}

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The causal relation between cross-slip structure and brittle-to-ductile transition has been examined using NaCl single crystals with the [100] axis. At around room temperature, the crystals exhibit cleavage fracture after a few percent elongation, although their yield stress is lower than 2 MPa. When the temperature is raised, brittle-to-ductile transition takes place at around 400 and 500 K with the respective strain rates of 5.5×10^{-6} and $5.5 \times 10^{-5} \text{ s}^{-1}$. The transition temperatures correspond well to those for the abundant operation of cross slip. The cross-slip lines are not parallel to a specific crystallographic plane, but are widely distributed in angles of $10^\circ \sim 40^\circ$ from the slip line of the primary plane (101) on the (001) surface. Even in high magnification images observed using an UHV-AFM, cross-slip lines appear to be wavy, indicating the cross slip on indefinite planes. Dislocations emitted from a crack tip can extend sideways along a moving crack front by the cross slip on indefinite planes, and reduce local stress intensities to impede the crack advance by crack tip shielding due to dislocations.

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1. Introduction

Dislocations in alkali halide crystals with NaCl structure are mobile on {110} slip planes and operate actively to cause very low yield stress at room temperature.¹⁻⁴⁾ Such dislocation activity should suppress brittle fracture. However, at room temperature, the ionic crystals exhibit cleavage fracture and the dislocation activity does not cause the increase of fracture toughness K_{IC} .²⁻⁵⁾ When temperature is raised, brittle-to-ductile transition takes place to increase the K_{IC} at above 400 K.⁴⁾ Such brittle-ductile behavior has been suggested to relate to the limited number of independent slip systems.^{6,7)} However, no detailed mechanism was treated in connection with the shielding/anti-shielding of a crack tip by dislocations.

In the present study, in order to understand the transition mechanism, the change of slip band structure, in particular of cross-slip structure, was examined using NaCl crystals deformed at temperatures below and beyond the transition temperature. Detailed cross-slip structure was observed by means of an ultra-high vacuum atomic force microscopy (UHV-AFM). The mechanism of crack tip shielding by dislocations to cause the transition will be discussed in connection with the change of slip band structure.

2. Experimental Procedure

Single crystals of NaCl (99.95% purity) were used. Rectangular crystals (3 mm × 3 mm × 50 mm) oriented to a (100) axis were cleaved out from a mother crystal. In order to prepare tensile specimens, two brass balls with a through hole were fixed to both ends of each crystals by molten rock salt (see Fig. 1) and then the crystals were subjected to thermal cyclic annealing for 33 ks at temperatures between 933 to 1033 K to decrease dislocation density. Tensile tests were car-

Fig. 1 Size and orientation of NaCl specimens prepared for tensile tests. (a) As cleaved from a mother crystal, (b) Brass balls are fixed to both ends of a crystal.

ried out between room temperature and 623 K at strain rates of 5.5×10^{-5} and $5.5 \times 10^{-6} \text{ s}^{-1}$ using an Instron-type machine (Shimazu Autograph AG-50KNE) equipped with an infrared light furnace. Observation of slip lines was made on the {001} side surface of specimens using an optical microscope (Nikon Optiphot) and an UHV-AFM (Omicron). After tensile tests, fracture surface was observed in order to determine whether the fracture mode was brittle or ductile.

3. Experimental Results

3.1 Stress-strain curves

Figures 2 and 3 show the stress strain curves of NaCl crystals deformed at various temperatures at strain rates of 5.5×10^{-6} and $5.5 \times 10^{-5} \text{ s}^{-1}$, respectively. As is seen from the figures, the strain to failure is markedly increased at 423 K for $5.5 \times 10^{-6} \text{ s}^{-1}$ and at 523 K for $5.5 \times 10^{-5} \text{ s}^{-1}$. Below those temperatures, specimens were fractured by cleavage to exhibit small elongation, while, above the temperatures, failure by necking was observed, followed by decrease in the strain to failure with further increasing temperature. In the high temperature range, stress-strain curves often exhibit irregular feature, which seems to be caused by the interaction

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Fig. 2 Stress-strain curves of NaCl crystals deformed at various temperatures with a strain rate of $5.5 \times 10^{-6} \text{ s}^{-1}$.

Fig. 3 Stress-strain curves of NaCl crystals deformed at various temperatures with a strain rate of $5.5 \times 10^{-5} \text{ s}^{-1}$.

of dislocations with point defects produced through the plastic deformation (some kind of Portevin-Le Chatelier effect).

Temperature dependence of strain to failure is shown in Fig. 4, where our data of apparent fracture toughness K_{IC}^* for NaCl crystals are also plotted against temperature.⁴⁾ In tensile tests, sharp transition from brittle to ductile behavior appears at around 400 and 500 K for strain rates of 5.5×10^{-6} and $5.5 \times 10^{-5} \text{ s}^{-1}$, respectively. It is noteworthy that the tenfold increase of strain rates raises the transition temperature by 100 K. Furthermore, the sharp transition takes place in tensile tests in contrast with that of fracture toughness tests where K_{IC}^* values gradually increase with increasing temperature.

3.2 Slip band observations

Figure 5 shows optical micrographs of slip lines observed on NaCl crystals deformed at a strain rate of $5.5 \times 10^{-5} \text{ s}^{-1}$ at 423, 473 and 523 K. Slip lines are mostly straight and parallel to the primary plane at 423 and 473 K below the transition temperature. At 523 K, *i.e.*, above the transition temperature, slip lines exhibit zigzag feature, indicating the active operation of cross slip. Similar results were also obtained at $5.5 \times 10^{-6} \text{ s}^{-1}$ where slip lines became zigzag at 423 K and above. Those results suggest that cross slip may play an important role in ductile-to-brittle transition. Using optical micrographs for the (001) surface of specimens deformed at above the transition temperature, we measured the angle between the cross and primary slip lines. Frequencies of the angle are shown for specimens deformed at 523 K at a strain rate of $5.5 \times 10^{-5} \text{ s}^{-1}$ in Fig. 6. The angles of cross-slip lines to the primary slip line are widely distributed in the range of $10^\circ \sim 40^\circ$. This suggests that cross slip does not operate

Fig. 4 Strain to failure at strain rates of 5.5×10^{-6} and $5.5 \times 10^{-5} \text{ s}^{-1}$ for NaCl crystals tested at various temperatures. Here fracture toughness data of K_{IC}^* are also plotted.⁴⁾

Fig. 5 Optical micrographs of slip lines in NaCl crystals deformed with a strain rate of $5.5 \times 10^{-5} \text{ s}^{-1}$ at 423 K (the top photograph), 473 K (the middle) and 523 K (the bottom).

toward a specific crystallographic plane and that it does on the indefinite planes including the slip direction. In the case where the primary system is defined as $(10\bar{1})[101]$, cross slip may operate toward the (010) plane,⁷⁾ whose slip line is per-

Fig. 6 Observed frequency plotted against the angle of observed cross slip line to the primary slip line of $(10\bar{1})$ on the (001) surface of NaCl crystals deformed with a strain rate of $5.5 \times 10^{-5} \text{ s}^{-1}$ at 523 K just above the transition temperature.

pendicular to the primary slip line, *i.e.* 90° . However, such cross slip was not observed in the present specimens oriented to a $[100]$ axis.

A slip line observed by an optical microscope essentially corresponds to a slip band which is the aggregate of fine slip lines. A low magnification AFM image of the slip band in a specimen deformed at 523 K at a rate of $5.5 \times 10^{-5} \text{ s}^{-1}$ is shown in Fig. 7, where the cross-slip band about $3 \mu\text{m}$ width exists besides the primary-slip band about $5 \mu\text{m}$ width. However, the fine structure in both slip bands is not resolved at this magnification. Figure 8 shows a high magnification AFM image of fine slip lines lying inside the cross-slip band of Fig. 7. Each fine slip line inside the cross-slip band is seen as wavy lines. Thus it is evident that cross slip operates on indefinite planes at above the temperature of brittle-to-ductile transition in NaCl crystals.

4. Discussion

In alkali-halide ionic crystals of rock salt structure, the main slip system is restricted to $\{10\bar{1}\}(101)$ slip systems, and the yield stress for the slip systems is generally low. The slip systems provide only two independent slip systems. To deform a crystal into any desired form, it is necessary to operate five independent slip systems according to the von Mises criterion.⁶⁾ Therefore, it is widely believed that the brittleness of ionic crystals such as NaCl comes from the limited number of independent slip systems. However, this concept does not provide any detailed mechanism for crack tip process in terms of the shielding/anti-shielding effect on fracture.⁵⁾

As described above, brittle-to-ductile transition is closely related to the fine cross slip on indefinite planes. The cross slip must be achieved by the successive cross slip of screw dislocations onto the different planes of high indices. Even if each screw dislocation splits into two partials, the partials are of screw-type so that the cross slip does not require the recombination of the partials. Strunk⁸⁾ suggests that cross slip operates not only on the (010) plane but also on higher index planes such as the $(21\bar{2})$, $(31\bar{3})$ and $(32\bar{3})$ planes. He estimates

Fig. 7 Primary and cross slip bands observed by an AFM at low magnification on the (001) surface of a NaCl crystal deformed at 523 K with a strain rate of $5.5 \times 10^{-5} \text{ s}^{-1}$.

Fig. 8 Fine slip lines within a cross slip band observed by an AFM at high magnification on the (001) surface of a NaCl crystal deformed at 523 K with the strain rate of $5.5 \times 10^{-5} \text{ s}^{-1}$.

that the shear stress necessary to move screw dislocations on those planes is at most about four times of the critical shear stress of NaCl crystals. Since the critical stress is quite low in NaCl, the cross slip may successively operate on high index planes at above the temperature of brittle-to-ductile transition.

When a dislocation half-loop is introduced from a crack tip to the hinge-type plane of $\{10\bar{1}\}$, it does relieve the stress concentration within the loop by crack tip shielding and suppresses the advance of the inner crack front. However, the outer part of the crack front suffers higher stress concentration by anti-shielding due to the loop.⁹⁾ As a result, the outer front is more forced to advance, as is shown in Fig. 9(a). Below the transition temperature, the loop can move only on a $\{10\bar{1}\}$ plane to be restricted from the self-extension, though it receives a force toward a higher stress field of the outer front. After both sides of the front are turned around the loop, the whole crack front will be advanced, leading to the brittle fracture. Above the transition temperature, cross slip operates on indefinite planes to promote sideways extension of the loop along the outer crack front, as is illustrated in Fig. 9(b).

Fig. 9 Crack tip process for dislocations on a hinge-type plane. (a) The generation of a dislocation half-loop and (b) the extension of dislocation half-loops along the crack front by cross slip.

Fig. 10 A crack ledge and dislocations. (a) Dislocation emission from a crack ledge and (b) the extension of a dislocation by cross slip along a crack front.

The extended dislocation loop can effectively reduce crack tip stress intensity to decelerate the crack advance.

Furthermore, dislocations on the shear-type plane intersected by a crack-front line are often observed near the crack tip besides those on the hinge-type plane.^{10,11} Such dislocations must be emitted from a crack ledge (crack jog) or from the free surface close to a crack tip. Figure 10(a) illustrates an example for the emission of dislocations from a crack ledge. On the way to dislocation bowing-out after the emission from the crack ledge, both ends of the dislocation may become a screw-type so that cross slip operates at above the transition temperature. The direction of shear stress for crack opening at the upper location from a crack plane is opposite to that at the lower location. Therefore, the dislocation near its upper and lower ends does cross-slip to opposite direction each other followed by the sideways extension along the crack front, as is shown in Fig. 10(b). The dislocation along the crack front reduces local stress intensities to impede the crack advance. It is noteworthy that dislocations effectively contribute to the crack tip shielding only through the cross slip on indefinite planes, because the dislocations can sideways pursue a moving crack-front.

5. Conclusions

Interrelation between slip-band structure and brittle-to-ductile transition has been examined using NaCl crystals oriented to a $\langle 100 \rangle$ axis. Slip band structure was observed using an optical microscope and an UHV-AFM for crystals deformed at temperatures from room temperature to 623 K. The results are summarized as follows.

(1) Brittle-to-ductile transition takes place at around 400 and 500 K with the respective strain rates of 5.5×10^{-6} and $5.5 \times 10^{-5} \text{ s}^{-1}$, where the transition in tensile tests is sharper than that in K_{IC} tests.

(2) The temperatures of transition well correspond to those for the abundant operation of cross slip.

(3) The cross-slip lines are not parallel to a specific crystallographic plane, but are widely distributed in the angles of $10^\circ \sim 40^\circ$ from the slip line of primary plane (101) on the (001) surface. In AFM images at high magnification, fine slip lines inside a cross-slip band are revealed as wavy lines, indicating the cross slip on indefinite planes.

(4) Dislocations emitted from a crack tip can extend sideways along a moving crack front through the cross slip on indefinite planes, and reduce local stress intensities to impede the crack advance by the crack tip shielding due to dislocations, thereby causing the brittle-to-ductile transition in NaCl crystals.

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