Study on Dislocation-Dopant Ions Interaction during Plastic Deformation by Combination Method of Strain-Rate Cycling Tests and Application of Ultrasonic Oscillations

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Abstract

Strain-rate cycling tests associated with the ultrasonic oscillation were conducted for the purpose of investigation on the interaction between dislocation and dopant ions during plastic deformation of seven kinds of single crystals: NaCl doped with Li⁺, K⁺, Rb⁺, Cs⁺, F⁻, Br⁻ or I⁻ ions separately. Relative curves between the stress decrement ($\Delta \tau$) due to ultrasonic oscillatory stress and strain-rate sensitivity ($\lambda$) of flow stress under superposition of the oscillation are obtained by the original method (combination method of strain-rate cycling tests and application of ultrasonic oscillations) at 77 K to room temperature and have stair-like shapes for the specimens at low temperatures. The Gibbs free energy for overcoming of the dopant ion by dislocation at absolute zero is calculated from the data analyzed in terms of $\Delta \tau$ vs. $\lambda$. As a result, the obtained energies are found to be varied linearly with the isotropic defect around it in the each specimen.

Keywords: dislocation, ultrasonic oscillatory stress, activation energy, monovalent ion, isotropic strain

1. Introduction

Dislocation (linear defects in crystal) motions are related to the plasticity of crystal in a microscopic viewpoint. It is well known that the solution hardening depends on dislocation motion hindered by the atomic defects around impurities in crystals and is namely influenced by the dislocation-point defects interaction, which has been widely investigated by various methods. For instance, measurements of yield stress (e.g., [1–7]) and proof stress (e.g., [8, 9]), micro-hardness tests (e.g., [10–14]), direct observations of dislocation (e.g., [15–21]), internal friction measurements (e.g., [22–27]), or stress relaxation tests (e.g., [28, 29]) have been carried out so far. Nevertheless, it is difficult to obtain such information on the motion of the dislocation which moves by overcoming the forest dislocations and the weak obstacles such as impurities during plastic deformation of bulk. A large
number of investigations have been conducted by the separation of the flow stress into effective and internal stresses on the basis of the temperature dependence of yield stress, the strain rate dependence of flow stress, and the stress relaxation. Yield stress depends on dislocation velocity, dislocation density, and multiplication of dislocations [30]. On the other hand, the effect of heat treatment on the micro-hardness is almost insensitive to the change of atomic order of point defects in a specimen. As for direct observations, electron microscopy provides the information on dislocation motion for a thin specimen but not for bulk, and also light scattering method is useful only for a transparent specimen. X-ray topography is the lack of resolution in the photograph, so that the specimen is limited to the low dislocation density below $10^4$ cm$^{-2}$. Internal friction measurements concern the motion of the dislocation which breaks away from the weak obstacles between two forest dislocations by vibration [31]. Stress relaxation tests are generally assumed that internal structure of crystals does not change, i.e., dislocation density and internal stress are constant. Above-mentioned methods cannot provide the information on dislocation-obstacles interaction in bulk during plastic deformation.

In this chapter, the study on interaction between a dislocation and dopant ions is made by the strain-rate cycling tests during the Blaha effect measurement. The original method (strain-rate cycling tests associated with the Blaha effect measurement) is different from above-mentioned ones and would be possible to clear up it. The Blaha effect is the phenomenon that static flow stress decreases when an ultrasonic oscillatory stress is superimposed during plastic deformation [32]. Ohgaku and Takeuchi [33, 34] reported that the strain-rate cycling under the application of oscillation can separate the contributions arising from the interaction between a dislocation and dopant ions and from the dislocations themselves during plastic deformation at room temperature. Using ionic single crystals of KCl doped with Br$^-$ (0.5, 1.0, and 2.0 mol%) or I$^-$ (0.2, 0.5, and 1.0 mol%) [35] and of NaCl doped with Br$^-$ (0.1, 0.5, and 1.0 mol%) [36], they discussed temperature dependence of the effective stress due to monovalent dopants (i.e., Br$^-$ or I$^-$) and found that the measurement of strain-rate sensitivity under the ultrasonic oscillatory stress provides useful information on a mobile dislocation-the dopant ions interaction [35, 36]. The information on the dislocation motion breaking-away from dopant ions [37–40] and also X-irradiation induced defects [41] with the ultrasonic oscillatory stress has been successively provided by the original method, which seemed to separate the contributions arising from the dislocation-the point defects interaction and from dislocations themselves during plastic deformation of crystals.

The Blaha effect was found by Blaha and Langenecker when the ultrasonic oscillatory stress of 800 kHz was superimposed during plastic deformation of Zn single crystals. The same phenomenon as Zn crystals has been also observed in many metals (e.g., [42–44]). Since this phenomenon has a significance as an industrial purpose, it has been widely made to apply to the plastic working technique: wire drawing, deep drawing, rolling, and another metal forming techniques (e.g., [45–53]).

The strain-rate cycling tests associated with ultrasonic oscillation were carried out here for NaCl single crystals doped with various monovalent ions separately. The monovalent ion is considered to have isotropic strain in the alkali halide crystal because its size is different from the substituted ion of the host crystal. Dopant ions are expected to cause the hardening due to the dislocation motion hindered by the defects around them at low temperature. Its force-distance profile between a dislocation and an atomic defect is expressed by Cottrell and Bilby [54]. This chapter refers to the energy supplied by the thermal fluctuations, when the dopant ions are overcome by a dislocation with the help of thermal activation during plastic deformation of crystals. This is estimated from the dependence of the effective stress
due to impurities on activation volume, which reveals the force-distance profile, given by the measurement of the stress decrement due to application of ultrasonic oscillatory stress and strain-rate sensitivity of flow stress under superimposition of ultrasonic oscillation. And further, it is presented that the difference in size of isotropic strain around the various dopants different from host ion has an influential factor of the energy for overcoming the dopant ion by a dislocation in several kinds of alkali-halide single crystals.

2. Combination method of strain-rate cycling tests and the Blaha effect measurement

Specimens used in this work were seven kinds of single crystals: NaCl doped with Li⁺, K⁺, Rb⁺, Cs⁺, F⁻, Br⁻ or I⁻ ions separately. Each concentration of the dopants was 0.5 mol% in the melt. The specimens were prepared by cleaving the single crystalline ingots, which were grown by the Kyropoulos method [55] in air, to the size of 5 × 5 × 15 mm³. Furthermore, they were kept immediately below the melting point for 24 h and were gradually cooled to room temperature at a rate of 40 K h⁻¹. This heat treatment was carried out for the purpose of reducing dislocation density as much as possible.

The schematic illustration of apparatus is shown in Figure 1. A resonator composed of a vibrator and a horn was attached to the testing machine, INSTRON Type 4465. The specimens were lightly fixed on a piezoelectric transducer and then cooled down to a test temperature. Each specimen was held at the test temperature for 30 min prior to the following test. The specimens were deformed by compression along the <100> axis at 77 K up to the room temperature, and the ultrasonic oscillatory stress was intermittently superimposed for 1 or 2 min by the resonator in the same direction as the compression. The temperature measurements of specimens were conducted by heater controlled using thermocouples of Ni-55%Cu vs. Cu. As for the tests at 77 K, the specimen was immersed in the liquid nitrogen. The stability of temperature during the test was kept within 2 K. The resonant frequency was 20 kHz from a multifunction synthesizer and the amplitude of the oscillatory...
stress was monitored by the output voltage from the piezoelectric transducer set between a specimen and the support rod, which was observed by an a.c. voltmeter or an oscilloscope. Since the wavelength, which is 226 mm on the basis of calculating from the data of ref. [56], is 15 times as long as the length of specimen, the strain of specimen is supposed to be homogeneous.

Strain-rate cycling tests made between the crosshead speeds of 10 and 50 μm min⁻¹ were performed within the temperatures. The strain-rate cycling test associated with the ultrasonic oscillation is illustrated in Figure 2. Superposition of oscillatory stress (τ₀) causes a stress drop (Δτ) during plastic deformation. When the strain-rate cycling between strain-rates of ε₁ (2.2 × 10⁻⁵ s⁻¹) and ε₂ (1.1 × 10⁻⁴ s⁻¹) was carried out keeping the stress amplitude of τ₀ constant, the variation of stress due to the strain-rate cycling is Δτ'. The strain-rate sensitivity (Δτ'/Δlnε) of the flow stress, which is given by Δτ'/1.609, was used as a measurement of the strain-rate sensitivity (λ = Δτ'/Δlnε). Slip system for rock-salt structure such as NaCl crystal is {110} <110> so that shear stress (τ) and shear strain (ε) calculated for the slip system were used in this study.

3. Relation between stress decrement (Δτ) and strain-rate sensitivity (λ)

Figure 3 shows the influence of temperature on Δτ vs. λ curve for the NaCl:Rb⁺ (0.5 mol%) single crystals at strain 6%. The variation of λ with Δτ has stair-like shape: two bending points and two plateau regions are on the each curve. That is to say, the first plateau region ranges below the first bending point at low Δτ and the second one extends from the second bending point at high Δτ. λ gradually decreases with increasing Δτ between the two bending points. The length of Δτ within the first plateau region is named τₚ as denoted in Figure 3. τₚ tends to be lower at higher temperature. Similar phenomena as Figure 3 are observed for all the other NaCl single crystals contained with the monovalent impurities (i.e. Li⁺, K⁺, Cs⁺, F⁻, Br⁻ or I⁻ ions).

The relation between Δτ and λ reflects the effect of ultrasonic oscillation on the dislocation motion on the slip plane containing many weak obstacles such as impurities and a few forest dislocations during plastic deformation [40]. Δτ vs. λ curve is divided into three regions as shown in Figure 3. Within the first plateau region of relative curve (i.e. region 1 in Figure 3), the application of oscillation with low stress amplitude cannot influence the average length of dislocation segments (l) and l is considered to remain constant. All weak obstacles act as impediments to the dislocation motion there. In region 2, the dislocation begins to break-away from the weak ones between the forest dislocations by applying oscillation with high stress amplitude. As a result, l begins to increase and the λ of flow stress starts to decrease at the stress decrement Δτ of τₚ. This is because λ is inversely proportional to l [57].
Some weak obstacles stop acting as impedimenta in the region. The weak obstacles are supposed to be monovalent dopants (Li$^+$, K$^+$, Rb$^+$, Cs$^+$, F$^-$, Br$^-$ or I$^-$ ions) and not to be vacancies here, since the vacancies have low density as against the dopants in the specimen. When the specimens were plastically deformed, it is imagined that a dislocation begins to overcome from the dopants which lie on the dislocation with the help of thermal activation. Then, $\tau_p$ is considered to represent the effective stress due to the ions. Accordingly, $\tau_p$ is expected to decrease with increasing temperature. $\Delta \tau$ vs. $\lambda$ curves shown in Figure 3 correspond to this. As the temperature becomes larger, $\tau_p$ shifts in the direction of lower $\Delta \tau$. $\tau_p$ depends on type and density of the weak obstacle [36, 38]. Applying still larger stress amplitude during plastic deformation of the specimens, the second plateau region within stage 3 becomes to appear on the relative curves in Figure 3. In stage 3, the dopants are no longer act as the impedimenta to mobile dislocations and the dislocations are hindered only by forest dislocations. Then, $\bar{l}$ becomes constant again. This leads to the constant $\lambda$ of flow stress. $\lambda_p$ denoted in Figure 3 is introduced later.

4. Model overcoming the thermal obstacle by a dislocation

A dislocation will encounter a stress field illustrated schematically in Figure 4 as it moves through on the slip plane containing many weak obstacles and a few strong ones. In the figure, the positive stress concerning axis of the ordinate opposes the flow stress (applied stress $\tau$) and the negative stress assists it. Extrinsic resistance to the dislocation motion has two types: one is long-range obstacle (the order of 10 atomic diameters or greater) and the other short-range obstacle (less than about 10 atomic diameters). The former is considered to be forest dislocations, large precipitates or second-phase particles, and grain boundary, for instance, and the latter impurity atoms, isolated and clustered point defects, small precipitates, intersecting dislocations, etc. Overcoming the latter type of obstacles (bname, thermal obstacles) by a dislocation, thermal fluctuations play an important role in aid of the flow stress above the temperature of 0 K. Then the aid energy, $\Delta G$, supplied by the thermal fluctuations is given by the shaded part in Figure 4. Thus the dislocation can move through below $\tau_0$ (i.e. effective stress $\tau^*$ due to short-range obstacles and internal stress $\tau_i$).
due to long-range ones in Figure 4). \( \tau_0 \) is the value of \( \tau \) at 0 K. As for the long-range obstacles (by name, athermal obstacles), the energy barrier is so large that the thermal fluctuations play no role in overcoming them within the temperature range.

The representation of Figure 5 is concerned with a common type of thermal activation barrier. The free energy \( (G) \) varies with the distance \( (x) \) between a

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**Figure 4.**
Stress fields encountered by a dislocation moving through the crystal lattice [57].

**Figure 5.**
The process for thermal activated overcoming of the short-range obstacle by a dislocation. Variation in (a) the Gibbs free energy of activation and (b) the force acted on the dislocation with the distance for a dislocation motion [57].
dislocation and the obstacle as given in Figure 5(a). When a dislocation overcomes the short-range obstacles, the free energy becomes high on account of the work ($\Delta W$) done by the applied stress. Then the resistance ($F$), where it can be defined by the differentiation of free energy with respect to $x$ (i.e. $\partial G/\partial x$), to the dislocation motion is revealed as Figure 5(b) in accord with the abscissa of Figure 5(a). $F$ value is maximum at position $x_1$. Figure 5(b) corresponds to typical force-distance curve for short-range obstacle among those in Figure 4. Shape of this curve represented by $F(x)$ means the model overcoming the obstacle by a dislocation. $G_0$, which is taken as the shaded area under $F(x)$ between saddle-point positions $x_0$ and $x_2$ in Figure 5(b), is the Gibbs free energy of activation for the breakaway of the dislocation from the obstacle in the absence of an applied stress (in this case it is equivalent to the Helmholtz free energy for the dislocation motion).

5. Relation between the effective stress due to impurities on activation volume

When the dislocation breaks-away from the defects on a slip plane with the aid of thermal activation during plastic deformation, observations of $\tau_p$ and $\lambda_p$ would provide information on the dislocation-defect interaction in the specimen. $\lambda_p$ is the difference between $\lambda$ at first plateau place and at second one on $\Delta \tau$ vs. $\lambda$ curve as presented in Figure 3, which has been regarded as the component of strain-rate sensitivity due to dopant ions when a dislocation moves forward with the help of oscillation [40].

Figure 6 shows the relation between $\tau_p$ and activation volume ($V$) for NaCl:Rb⁺ (0.5 mol%). The activation volume has been expressed as [57).

$$V = kT\left(\frac{\partial \ln \dot{\varepsilon}}{\partial \tau}\right)$$

(Figure 6. Relation between $\tau_p$ and activation volume ($V$) for NaCl:Rb⁺ (0.5 mol%) (reproduced from Ref. [58] with permission from the publisher).
where $k$ is the Boltzmann constant and $T$ is the absolute temperature.

Here, the $\left(\frac{\partial \ln \varepsilon}{\partial \tau}\right)$ in Eq. (1) is obtained from $\lambda_p$. Eq. (1) is namely replaced by

$$V = \frac{kT}{\lambda_p}.$$  \hspace{1cm} (2)

This dependence ($\tau_p$ vs. $V$) also represents the force-distance profile between dislocation and Rb$^+$ ion. The $\tau_p$ vs. $V$ curve gives the value of $G_0$ for the specimen. The $G_0$ values for the other specimens (i.e. NaCl: Li$^+$, K$^+$, Cs$^+$, F$^-$, Br$^-$ or I$^-$) are similarly estimated and are listed in Table 1.

Figure 7 shows the obtained energies $G_0$ with the isotropic defect size ($\Delta \varepsilon$), which is estimated from the difference between the lattice constants of host crystal and dopant, around ion doped in the each specimen. The ions beside each plot represent the dopants in NaCl single crystals. $G_0$ values vary linearly with $\Delta \varepsilon$ in the specimens. The intercept of the straight line is 0.36 eV, which is considered to be the interaction energy between dislocation and inherent obstacle of the host crystal because $\Delta \varepsilon$ is zero.

### 6. Conclusions

The following conclusions were derived from the data analyzed in terms of the $\Delta \tau$ vs. $\lambda$ curves for NaCl: Li$^+$, K$^+$, Rb$^+$, Cs$^+$, Br$^-$, I$^-$, F$^-$, Br$^-$ or I$^-$ single crystals.

| Specimen        | $G_0$ (eV) |
|-----------------|------------|
| NaCl:Li$^+$ (0.5 mol%) | 0.55       |
| NaCl:K$^+$ (0.5 mol%)  | 0.60       |
| NaCl:Rb$^+$ (0.5 mol%) | 0.61       |
| NaCl:Cs$^+$ (0.5 mol%) | 0.82       |
| NaCl:F$^-$ (0.5 mol%)  | 0.69       |
| NaCl:Br$^-$ (0.5 mol%) | 0.47       |
| NaCl:I$^-$ (0.5 mol%)  | 0.53       |

Table 1. Values of energy $G_0$. 

Figure 7. Variation of the interaction energy ($G_0$) between dislocation and the dopant ion with the defect size (reproduced from Ref. [58] with permission from the publisher).
1. The relation between $\Delta \tau$ and $\lambda$ has stair-like shape for the specimens at a given temperature and strain. There are two bending points and two plateau regions. $\lambda$ decreases with $\Delta \tau$ between the two bending points. The measurement of $\tau_p$ and $V$ calculated with $\lambda_p$ provides information on the interaction between mobile dislocation and the dopant ion in the specimens during plastic deformation.

2. The Gibbs free energy $G_0$ for the overcoming of dislocation from the dopant is obtained for each of the specimens and increases linearly with increasing the defect size $\Delta \varepsilon$. This result leads to the conclusion that the dopant ion as weak obstacle to dislocation motion becomes slightly stronger with larger defect size around the dopant in NaCl single crystal.

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Conflict of interest

The author declares no conflict of interest.

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