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사파이어(α-Al_2O_3) 단결정에 있어 basal slip (0001)1/3<11$\bar{2}$0>전위 Part I : 재결합거동

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Basal slip (0001)1/3<11$\bar{2}$0> dislocation in sapphire (α-Al_2O_3) single crystals Part I : recombination motion

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초 록 사파이어(α-Al_2O_3) 단결정에 있어 basal slip (0001)1/3<11$\bar{2}$0>의 부분전위의 재결합거동을 알아보기 위해 prism plane (11$\bar{2}$0)의 사파이어 재료를 사용하여 4점 곡강도 시험을 행하였다. 이 굽힘시험은 온도 1200℃~1400℃에서 그리고 응력은 90MPa, 120MPa, 150MPa에서 행하여졌다. 굽힘시험 동안 basal 전위가 이동하기 위해 잠복기가 필요하였다. 실험온도 범위내에서 잠복기의 활성화에너지는 5.6-6.0eV이었으며, 이 잠복기는 자체-상승운동으로 분해된 부분전위들이 재결합하는데 필요한 시간인 것으로 추정되었다. 한편, 이 활성화에너지는 Al_2O_3에 있어 산소의 자체 확산을 위한 에너지 (대략 6.3eV)와 거의 일치하였다. 이 결과를 통하여, 두 부분전위들의 재결합은 부분전위사이 적층결함으로 산소 자체확산에 의해 제어되는 것으로 여겨진다.

Abstract The recombination motion of partial dislocations on basal slip (0001)1/3<11$\bar{2}$0> in sapphire (α-Al_2O_3) single crystals was investigated using the four-point bending test with the prism plane (11$\bar{2}$0) samples. These bending experiments were carried out in the temperature range from 1200℃ to 1400℃ at various engineering stresses 90MPa, 120MPa, and 150MPa. During these tests it was shown that an incubation time was needed for basal slip to be activated. The activation energy for the incubation time was 5.6-6.0eV in the temperature range from 1200℃ to 1400 ℃. The incubation time is believed to be related to recombination of climb dissociated partial dislocations via self-climb. In addition, these activation energies are nearly same as those for oxygen self-diffusion in Al_2O_3 (approximately 6.3 eV). Thus, the recombination of the two partial dislocations would be possibly controlled by oxygen diffusion on the stacking fault between the partials.

Key words : Basal slip, Sapphire single crystals, Recombination motion

1. Introduction

Basal slip in sapphire single crystals is the easy slip system at elevated temperatures.[1~3] The stress-strain curve of a sapphire single crystals deformed by basal slip includes an elastic region, distinct upper and lower yield points and a significant work-hardening region.[4~9] Firestone and Heuer[6] showed that the presence of and the magnitude of the initial yield drop in sapphire undergoing basal slip depends on the initial density of mobile dislocation sources. And also Conrad *et al.*[7] suggested that the yield point in sapphire could be described in terms of a dislocation multiplication mechanism based on the motion of dislocation through the lattice rather than the tearing of dislocations from a pinning atmosphere. This mechanism is similar to that suggested by Johnston and Gilman[10] for LiF and by Conrad[11] and Hahn[12] for LiF and BCC metals. Furthermore, Pletka *et al.*[13] and Lagerlöf *et al.*[14] explained the stress-strain curve in sapphire single crystals on the basis of dislocation motions, which are based on the interaction between mobile dislocations and stationary dislocation debris such as edge-trapped dipoles and prismatic dislocation loops.

After self-climb, the equilibrium separation distance (~7nm) between two partial dislocations has been measured using TEM (Transmission Electron Microscopy),[15] and the distance (~8nm) was estimated by computer simulation.[16] However, no detailed discussion of how the two partials may recombine after self-climb to resume its motion on the glide plane has been carried out. Therefore, the aim of the present paper is to ana-

lyze the behavior of the basal dislocations (*e.g.* how the two partials recombine) using 4-point bending test.

2. Experimental procedure

High-purity ingots of Czochralski-grown undoped sapphire single crystals (Union Carbide Co.) with very low grown-in dislocation densities ($<10^2 cm^{-2}$) were used for this study. Specimens (25x3x1.5mm^3) suitable for 4-point bending were oriented using the Laue back-reflection X-ray techniques and cut using a Bueher isomet diamond saw. Specimen had face parallel to (11$\bar{2}$0) with the long axis of the 4-point sample parallel to [$\bar{8}$80$\bar{5}$] as shown in Fig.1. Four sides of the specimen were polished using 30㎛, 15㎛, and 3㎛ diamond pastes, down to 1㎛ and syton (the colloidal silica) as a final step.

Dislocations were introduced on the sample faces using a Vickers diamond indentor mounted in a hot hardness tester (Model QM, Nikon, Inc.) and an atmosphere of 10^{-5} torr vacuum with 0.5N load using a dwell time of 15sec at 1000℃. An array of indents was made on both the tensile and compressive faces of the four-point bend sample. To reveal the dislocation substructure around the indentation site the indented surfaces were etched in saturated solution of KOH between 380℃ and 400℃ for 10-15 min.

After indentation, the 4-point bend sample was subjected to a static load in the Ar atmosphere in material testing machine (MTS System Co.) using bending jigs made from high quality sintered SiC. The temperature was controlled using a thermocouple mounted near the sample and was maintained constant within ±2℃. During 4-point bending the applied shear stress was calculated using the equation given by Timosenko[17] for beams in 4-point bending, the outer fiber stress (σ) is given by

$$\sigma = \frac{3P(L-l)}{2wh^2} \quad (1)$$

where P is the applied load, w is the specimen width, h is the specimen thickness, L is the separation of the outer supports and l is the separation of the inner supports.

3. Results and discussion

In Fig.2 the bending displacement rate as a function of time was observed at constant shear stress (120MPa). Of interest, as shown by Fig.2, is the fact that an incubation time was needed before the bending displacement (a macroscopic plasticity) occurred. During the experiments at different temperatures, it was shown that the incubation time decreased with increasing temperature. In particularly, there was no (or very short) incubation time at 1400℃. In order to understand the effect of stress on the incubation time, additional bending deformation tests were carried out at two different stresses (90 MPa and 150 MPa, respectively) at three different temperatures, 1200, 1300, and 1350℃, respectively. The results are shown in Table 1.

During the bending displacement experiments, an incubation time was needed to activate. This is believed to be due to the fact that the basal plane has high stacking fault energy (~2.1 J/m^2) in comparison with the prism plane (~0.17 J/m^2).[18,19] In order to combine the

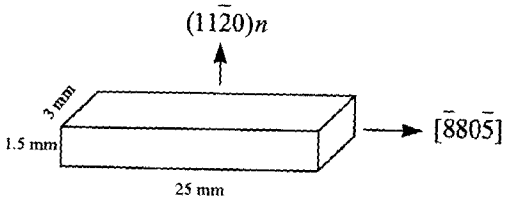

Fig. 1. Orientation of specimen for bending displacement experiments.

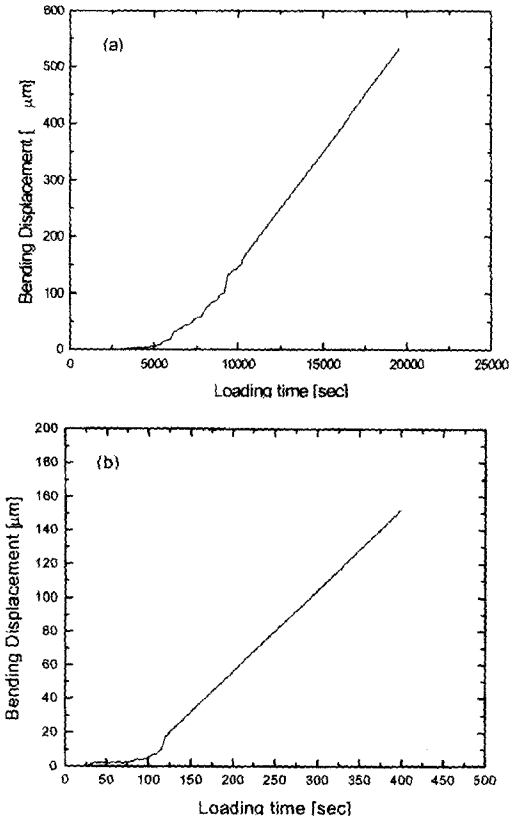

Fig. 2. Typical bending displacement graph as a function of loading time at given conditions: (a) 90MPa applied stress at 1200℃ and (b) 90MPa applied stress at 1350℃.

Table 1. The incubation time during the bending displacement experiments at different temperatures and stresses.

	1200℃	1250℃	1300℃	1350℃
90 MPa	5560 sec (5060-6060)		215 sec (180-250)	102 sec (90-115)
120 MPa	4700 sec (4400-5000)	450 sec (300-600)	145 sec (140-150)	75 sec (50-100)
150 MPa	3030 sec (3000-3060)		105 sec (100-110)	45 sec (40-50)

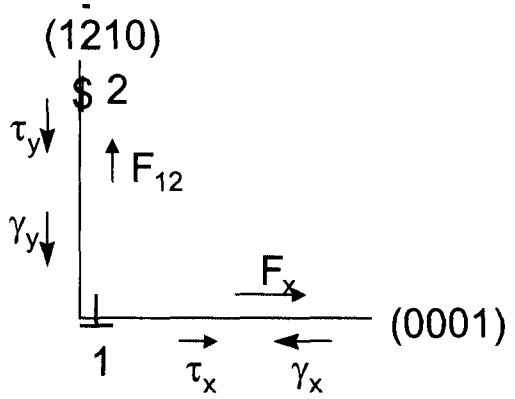

Fig. 3. Schematic diagrams after self-climb dissociated partials on basal slip dislocation. 1;edage dislocation, 2;screw dislocation, stacking fault energy:γ_x=2.1 J/m², γ_y=0.17 J/m², respectively.

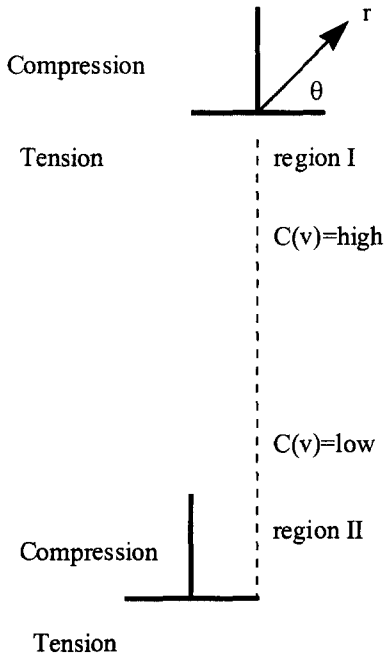

Fig. 4. Schematic diagram of vacancy concentration between two edge dislocations.

two partial dislocations as shown in Fig.3, one or both partial dislocations need to move (climb).

The force balance must be considered in order to understand how the two partial dislocations can recombine (Fig.3). The equilibrium force balance between two partial dislocations is given by

$$\frac{F_{12}}{L} = \gamma_y + \tau_y \tag{2}$$

where τ_y is the shear stress promoting recombination. Assuming constant stacking fault energy of 0.2 J/m²[19] and the interaction force (F_{12}/L) calculated from the Peach-Kehler equation[20], the external stress needed to combine two partial dislocations is around 56 GPa. This value exceeds the stress needed to climb dissociation to fracture sapphire before the onset of deformation. Thus, based on the above calculation, recombination of partial dislocations would never occur. However, the model does not take into account the fact that climb-dissociation via self-climb require localized transport of vacancies (and/or interstitial) against its concentration gradient. This will affect the localized force balance by the introduction of a chemical force related to the activity of the point defects required for the recombination of the partial dislocations.

To address this problem, a chemical force due to local vacancy concentration gradient around the dislocation was examined, following the method developed by Bullough and Newman.[21] The vacancy concentration near the dislocation (Fig.4) is represented by the equation (3).

$$C = C_0 \exp\left[-\frac{\Phi(\gamma,\theta)}{kT}\right] \tag{3}$$

where C is the vacancy concentration around the dislocation at the angle θ and distance γ. The total dislocation/vacancy interaction energy $\{\Phi(\gamma,\theta)\}$ is given by

$$\Phi(\gamma,\theta) = -\frac{\beta v_a \sin\theta}{\gamma} - \frac{\alpha}{\gamma^2} - \frac{\rho \sin^2\theta}{\gamma^2} \tag{4}$$

where $\alpha = \frac{5Gb^2\gamma^3}{\pi(1-\nu)(7-5\nu\nu)}$, $\beta = \frac{Gb(1+\nu)}{3\pi(1-\nu)}$, $\rho = \frac{Gb^2\gamma_0^2(1+6\nu-5\nu^2)}{\pi(1-\nu)(7-5\nu)}$, G is the shear modulus, b is the Burgers vector, γ_0 is the radius of vacancy, ν is Poissons ratio, and v_a is the vacancy volume. The vacancy concentration at the points shown as I and II, respectively, can be calculated by the equations (3) and

Table 2. Given parameters for calculating the chemical force in sapphire single crystals.

G (Shear modulus)	150 GPa
b (Burger's vector)	2.74Å
ν (Poisson's ratio)	0.23
v_a (atomic volume)	$2.32\times10^{-29}m^3$
γ_v(vacancy radius)	0.65Å

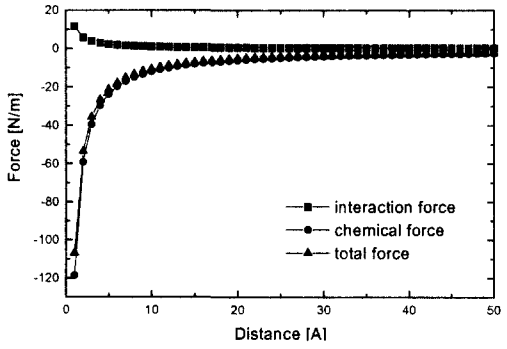

Fig. 5. Graph showing the total force as a function of distance with the interaction force and chemical force due to the vacancy concentration gradient.

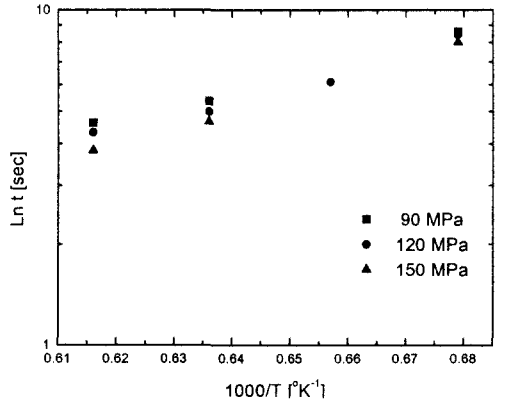

Fig. 6. Incubation time as a function of temperature during bending displacement experiment at different stresses.

(4). Due to local concentration gradient between I and II, the chemical force[20] is derived as shown in the eq. (5):

$$\frac{F_c}{L}=-\frac{\partial}{\partial\gamma}kTln\frac{C_1}{C_2}=-\frac{\partial}{\partial\gamma}kTln(C_1-C_2)=\frac{\partial}{\partial\gamma}\left[\frac{2\beta v_a}{\gamma}\right] \quad (5)$$

On the basis of the eq.(5) and the parameters (see Table 2), the chemical force which is applied to the two partial dislocations as a function of the distance can be developed as follows:

$$\frac{F_c}{L}=-\frac{118.5}{\gamma^2} \quad (6)$$

Thus, the total interaction force between the partial dislocations would be given by equation (7).

$$F_{total}=\frac{F_{12}}{L}+\gamma_y+\tau_y+\frac{F_c}{L} \quad (7)$$

At given stacking fault energy (γ_y) and applied shear stress (τ_y), the total force between two partial dislocations as a function of the distance is shown in the Fig.5. As shown in Fig.5, the total force is repulsive in the range between around 15Å and 40Å. However, for distances less than 15Å, the total interaction force is attractive which would counteract climb dissociation. In fact, consideration of the total force between the two partials suggest that dissociation must be nucleated after which the repulsive interaction force will separate the partials to their equilibrium separation distance. Since the climb dissociation must nucleate and grow, two possible configurations exist; one partial climb up and one partial climb down, and vice versa. Thus, since one partial may climb up along part of the dissociation and climb down along another part of the dislocation, a node at which the partial moves from "up" to "down" will form. The presence of such nodes has been observed in TEM.[22]

Now consider the temperature dependence of the incubation time. The values of log (incubation time) plotted against (1/T) are shown in Fig.6. The respective stresses at which the measurements were made are also indicated. At constant stress the incubation time as a function of temperature obeys the relation [equation (8)],

$$t=t_o\exp\left[\frac{U}{kT}\right] \quad (8)$$

where t_o is a constant and U is the activation energy for the incubation time. The activation energies (U) were obtained from the slope of the least square curve fit through the data points with equation (8). The values can be estimated to be in the range between 5.6 and 6.0eV at different applied stresses 90, 120, 150MPa. These activation energies are nearly same as that for oxygen self-diffusion in Al_2O_3 (~6.3eV).[23,24] On the basis of these results, during the bending displacement experiments, an incubation time was needed to activate dislocation multiplication sources. These sources are believed to be related to the recombination of climb dissociated stationary partial basal dislocations. Thus, the recombination of the two partial dislocations would be possibly controlled by oxygen diffusion on the stacking fault between the partials.

4. Conclusions

Throughout the four-point bending test with the prism plane ($11\bar{2}0$) samples, it was observed that an incubation time was needed for basal slip to be activated. The incubation time is believed to be related to recombination of climb dissociated partial dislocations via self-climb. The activation energy for the incubation time was 5.6-6.0 eV in the temperature range from 1200℃ to 1400℃ at different applied stresses 90, 120, 150MPa, suggesting that it would be controlled by oxygen vacancy diffusion on the stacking fault between the climb dissociated partials.

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