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취성-연성 전이 model을 이용한 사파이어 단결정의 prism plane slip $\{11\bar{2}0\}<1\bar{1}00>$ 전위속도에 대한 활성화에너지 계산

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The Estimation of Activation Energy for Prism Plane Slip $\{11\bar{2}0\}<1\bar{1}00>$ Dislocation Velocity in Sapphire Single Crystals using Brittle-to-ductile Transition Model

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초 록 사파이어 단결정의 취성-연성전이에 대한 실험을 행하였다. 사파이어 단결정의 취성-연성전이온도는 변형율 3.3×10^{-5}/sec에서 1000±25℃ 그리고 변형률 3.3×10^{-6}/sec에서는 1100±25℃이었다. 취성-연성전이모델을 이용하여 prism plane slip $\{11\bar{2}0\}<1\bar{1}00>$ 전위속도의 활성화에너지를 계산하였으며, 그 결과 활성화에너지는 4.6±2.3eV의 범위를 가졌다. 이 활성화에너지는 에치-피트법을 이용하여 전위속도측정으로부터 구한 결과치 3.8eV와 유사하였다.

Abstract Experimental studies of the brittle-ductile transition (BDT) for pre-cracked sapphire single crystals were carried out. The BDT temperature in sapphire single crystals were 1000±25℃ and 1100±25℃ at constant strain rate 3.3×10^{-5}/sec and 3.3×10^{-6}/sec, respectively. With aid of the BDT model, the activation energy for prism plane slip $\{11\bar{2}0\}<1\bar{1}00>$ dislocation velocity was in the range of 4.6±2.3eV. This activation energy for dislocation velocity with BDT model was compatible with the result of the dislocation velocity (3.8eV) using the etch-pit techniques.

Key words: Sapphire single crystals, Brittle-ductile transition, activation energy

1. Introduction

The brittle-ductile transition (BDT) of materials which fail in a brittle manner below a critical temperature is of great practical importance, since key structural materials such as ferrite steels fall into this category. The criterion for ductility was proposed by Kelly, Tyson, and Cottell[1] that a material would be ductile if the theoretical shear stress (τ_{max}) of the material is exceeded at crack tip before the theoretical tensile strength (σ_{max}) is reached even at absolute zero temperature. This model predicts the ductility of the fcc metals (σ_{max}/τ_{max}) and the brittle nature of diamond ($\sigma_{max}=\tau_{max}$), but it is not sufficient for the crack to blunt because the shear stress near a crack is not everywhere constant on the shear plane as it would have to be to cause the atoms to shear past one another uniformly. Later, Rice and Thomson[2,3] suggested that ductility in materials occur when a crack tip spontaneously emits dislocations before stable crack growth and also calculated the activation energy to nucleate a stable dislocation loop at a loaded crack tip in various materials which is related to the various forces operating between a crack and dislocation.

On the other hand, in order to understand the basic processes that occur at a sharp crack tip the brittle-ductile transition in silicon was studied experimentally by St. John.[4] He found the relationship between stress intensity and the activation energy for the controlling process equal to the activation energy for the dislocation mobility as shown in Eq. (1):

$$\exp\left[-\frac{U_{BDT}}{kT_c}\right] = C\dot{K} \qquad (1)$$

where $\dot{K}$ is stress intensity, C is the constant, and U_{BDT} is the activation energy controlling the strain-rate dependence of T_c.

A model due to Hassen[5], following an earlier treatment of St. John[4], in which it is assumed that dislocation nucleation occurs relatively easily at the crack tip,

and that the rate of blunting is controlled by the rate at which dislocations can move away from the crack tip, the relationship between the dislocation velocity and the stress intensity ($\dot{K}$) was predicted as follows:

$$V = Ar^m \exp\left[-\frac{U}{kT_c}\right] = V_o\tau^m \qquad (2)$$

where V is dislocation velocity, A is a constant, m is a stress exponent, τ is applied shear stress for moving the dislocation, U is the activation energy controlling dislocation velocity. With both relations Eqs. (1) and (2), the stress intensity ($\dot{K}$) at T_c is represented by

$$\dot{K} \propto V_o \propto \exp\left[-\frac{U_{BDT}}{kT_c}\right] \qquad (3)$$

Therefore, on the basis from eqs (1) - (3) the activation energy for the dislocation velocity can be predicted indirectly through the measuring of the brittle-ductile transition temperature at different strain rate. The object of this experiment is to investigate the BDT behavior of sapphire single crystals and estimate the activation energy for prism plane slip dislocation velocity. Finally, the activation energy obtained from BDT model was compared with the activation energy which was obtained by etch-pit technique (direct method).[6]

2. Experimental procedure

2.1 Specimen

A high-purity ingots of Czochralski-grown undoped sapphire (α-Al_2O_3) single crystals with $(1\bar{1}02)$ orientation and very low grown-in dislocation density ($<10^2$ cm^{-2}) were obtained from a commercial source (Union Carbide, USA). Specimens (25x3x0.4mm^3) were oriented using Laue back-reflection X-ray techniques and cut using a diamond saw. Specimen orientation is shown in Fig.1. Four sides of specimens were polished with various diamond pastes, finishing with a 1 ㎛ grade. The final mirror-like surface finish was achieved by using a colloidal silica slurry (Syton, USA).

2.2 Fracture test

To investigate the brittle-to-ductile transition temperature, pre-cracked sample was used as shown in Fig.1. Samples were deformed by four-point bending around $\{11\bar{2}0\}$ axis in Ar atmosphere from 800℃ to 1300℃ in material testing machine (MTS system Co.) using bending jigs made from high quality sintered SiC. Temperature was controlled by a thermocouple mounted near the sample and was maintained constant within ±2℃. Stress was calculated using the equation given by Timosenko[7] for beams in four-point bending, the

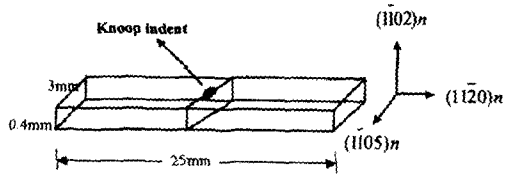

Fig. 1. Specimen orientation for investigating the brittle-to-ductile transition temperature of sapphire single crystals.

outer fiber stress (σ) is given by

$$\sigma = \frac{3p(L-l)}{2wh^2} \qquad (4)$$

where p is the applied load, w is the specimen width, h is the specimen thickness, L is the separation of outer supports and l is the separation of the inner supports. The precrack was introduced by Knoop indentation at room temperature. The cracks formed by Knoop indentation were subjected to a residual tensile stress from the mismatch between the plastic zone of the indentation and the surrounding elastic matrix. The load used during indentation was 500g. Cracks produced by 500g loads were approximately 20 ㎛ deep and 60 ㎛ in length at the surface. To remove the residual stress, Frett[8] used physical grinding which removed the indentation plastic zone, and Petrovic *et al.*[9] removed the residual stress by annealing the specimens at high temperature so that dislocations in the plastic zone were able to move. In our experiments, the annealing method was used. The precracked specimens were annealed at 1450℃ for 24 hrs.[10]

To convert values of load and displacement into values of stress and strain, the Eq. (5) was used. The bend tests were performed using two different cross-head speeds, corresponding to strain rates of the outer fiber of the specimen of $3.3\times10^{-6}s^{-1}$ and $3.3\times10^{-5}s^{-1}$, respectively. The relationship between strain rate and cross-head speed is given by

$$\dot{e} = \frac{\left[\frac{S}{L_o}\right]}{\left[\frac{A_o E}{KL_o}\right]} \qquad (5)$$

where S is the cross-head speed, L_o the length of specimen, A_o the cross-section area, K the stiffness of machine, and E the modulus of elasticity.

3. Results

3.1 The temperature dependence of load-displacement curve

Fig.2 shows the typical load-displacement curve at

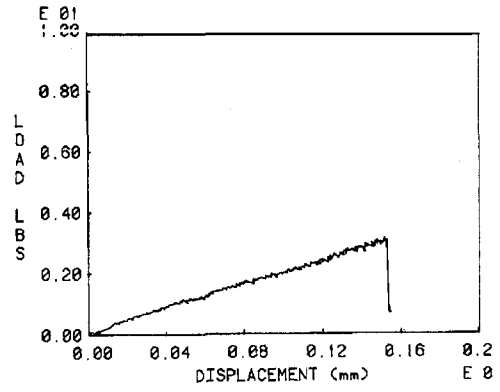

Fig. 2. Typical graph of load-displacement curve at below BDT temperature(975℃ at strain rate $3.3x10^{-6}s^{-1}$).

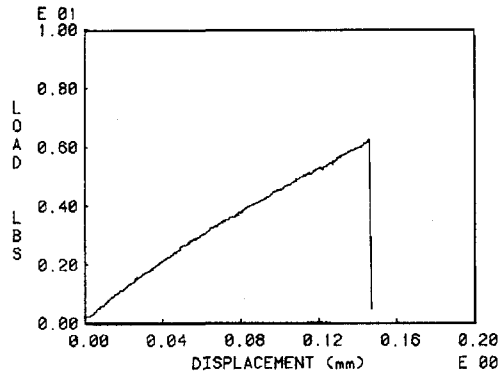

Fig. 3. Typical graph of load-displacement curve at near BDT temperature(1000℃ at strain rate $3.3x10^{-6}s^{-1}$).

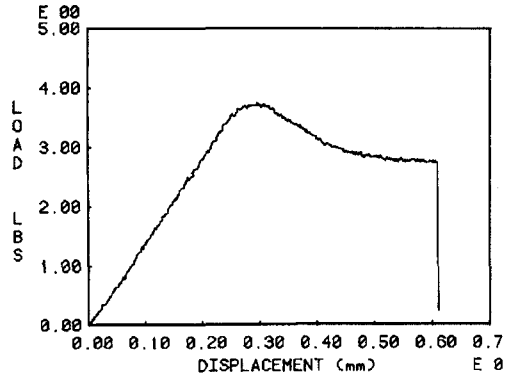

Fig. 4. Typical graph of load-displacement curve at above BDT temperature.(1050℃ at strain rate $3.3x10^{-6}s^{-1}$).

below BDT temperature. Below the BDT temperature (T_C), the load-displacement curve is linear indicating elastic behavior. Around T_C, the load-displacement curve was still linear but fracture does not occur until a considerably higher stress (418MPa) which is 80% higher than that below T_C (Fig.3). Fig.4 shows the load-displacement curve at above T_C, the specimens no longer fractured but deformed plastically. The load-displacement curve was no longer linear. The applied load first increase and then passes through a maximum as

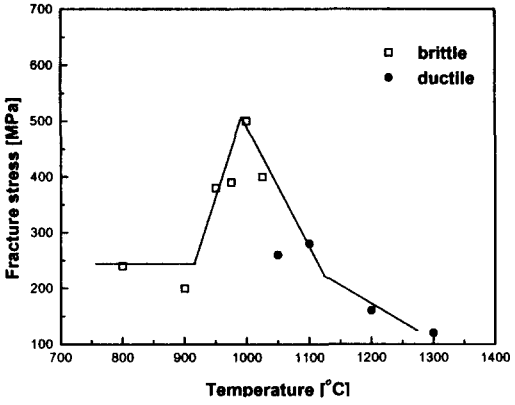

Fig. 5. Fracture stress as a function of temperature at constant strain rate $3.3x10^{-6}s^{-1}$.

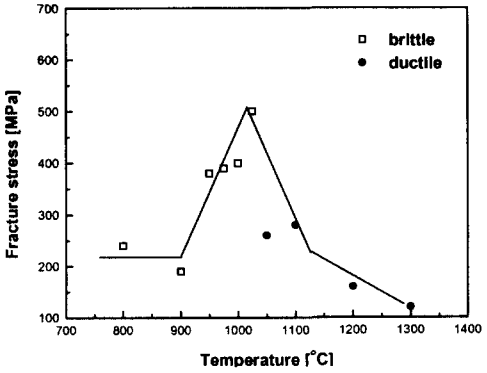

Fig. 6. Fracture stress as a function of temperature at constant strain rate $3.3x10^{-5}s^{-1}$.

the specimen exhibits general yielding behavior.

3.2 The failure stress as the function of temperature

To obtain the effect of strain rate on the failure stress, the fracture tests performed at two different cross-head speeds 10 ㎛/min and 100 ㎛/min, respectively (corresponding to strain rates of $3.3x10^{-6}$/sec and $3.3x10^{-5}$/sec). The experimental results, plotted as stress to failure versus temperature for each strain rate, are shown in Fig.5 and 6. The points with a square indicate that the specimen fractured in a brittle manner, those with a sphere show ductile behavior and marked with a line indicate transition behavior. Below T_C, the fracture was entirely brittle and the fracture stress was independent of temperature.

4. Discussion

During the load-displacement experiments, the specimens fractured catastrophically below T_C. For example, the critical stress at 800℃ and $3.3x10^{-6}$/sec is approximated 235 MPa. Also, the fracture stresses were in the range of 180-320 MPa. These values correspond to a fracture toughness of 1.4-2.5 MPa, which are close to the value 2.4 MPa determined by Iwasa and Bradt.[11] In

Fig.5 and 6, the failure stress decreased, as the strain rate at a given testing temperature was decreased. It can be seen that there is an individual sharp transition curve for each loading rate. This is consistent with the observation of St. John[4] who found that an temperature increase in loading rate caused an increase in the brittle-ductile transition.

In both cases, a sharp increase in critical stress intensity at the transition was similar to the results in sapphire[10] and in silicon[4], which measured critical stress intensities at T_C of up to five times the low-temperature value. Particularly, at T_C, there is a sharp "jump" in the fracture stress. Similar behavior is observed in the experiments of Michot and George[12] and Brede and Haasen.[5] This sharp "jump" is believed to be due to shielding of the crack tip by dislocations, which is the basis for the dynamic dislocation model to explain the brittle-to-ductile transition.[10]

On the basis of these tests, it can be assumed that the BDT in sapphire which was used in this experiments is controlled by a thermally activated process (Eqs.(1)-(3)). The activation energy for the dislocation velocity was obtained from two different cross-head speed fracture tests. These results also had a sharp jump in stress to fracture in the BDT. With Eqs.(1)-(3), the results of two different strain rates in this sapphire are shown in Table 1.

Table 1. Activation energy for dislocation velocity with the BDT model

Strain rate [sec^{-1}]	BDT temperature	Activation energy [eV]
3.3×10^{-6}	1000 ± 25℃	4.6 ± 2.3
3.3×10^{-5}	1100 ± 25℃	

In Table 1, the results from these experiments suggest that the activation energy for dislocation velocity is very sensitive to the BDT temperature. The activation energy for the dislocation velocity is represented as a function of strain rate. From the variation of strain rate on T_C, the activation energy of the process controlling the BDT in this orientation $\{11\bar{2}0\}<\bar{1}100>$ of sapphire single crystals was in the range of 4.6±2.3eV. This activation energy for dislocation velocity with BDT model was compatible with the result of the dislocation velocity (3.8 eV) using the etch-pit technique.[6]

5. Conclusions

Experimental studies of the brittle-ductile transition (BDT) for pre-cracked sapphire single crystals were carried out. The brittle-to-ductile transition temperatures in sapphire single crystals were 1000±25℃ and 1100±25℃ at constant strain rate 3.3×10^{-5}/sec and 3.3×10^{-6}/sec, respectively. With aid of the BDT model, the activation energy for prism plane slip $\{11\bar{2}0\}1/3<1\bar{1}00>$ dislocation velocity was in the range of 4.6±2.3eV. This activation energy for dislocation velocity with BDT model was compatible with the result of the dislocation velocity (3.8eV) using the etch-pit techniques.

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