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Investigation of the yield process by deformation luminescence of X-ray irradiated  
KCl:Ca<sup>2+</sup>

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**Abstract**

It is found that deformation luminescence gives us information about the microscopic yield process of X-ray irradiated KCl:Ca<sup>2+</sup>. The stress-strain curve has a macroscopic yield point. But we find that luminescence appears to start before the macroscopic yield. This means that dislocation begin to move before the macroscopic yield because deformation luminescence is attributed to radiation-induced dislocation motion. The beginning of luminescence is considered to be the microscopic yield. Investigating the dependence of microscopic yield stress on strain rate and impurity concentration gives us additional information. The activation volume obtained from the dependence of microscopic yield stress on strain rate is comparable to the value estimated from the concentration of impurity. Then the dislocation starts to move overcoming impurity-vacancy dipoles as obstacles to dislocation motion. The dislocation density starts to increase at the microscopic yield point and then sharply increases to the macroscopic yield.

*Keywords:* X-ray irradiation; Alkali halide; Yield; Dislocation; Interaction

**1. Introduction**

In materials science and condensed matter, the yield of materials is a complex function of many properties that can influence the initiation of dislocation motion, dislocation multiplication and plastic strain rate, which depend on mobile dislocation density as well as the average dislocation velocity. There are unresolved problems

though the yield of materials is of practical importance. There is substantial literature reporting on the dependence of yield stress on temperature, solute concentration and heat treatment (Dryden, et al., 1965; Chin, et al., 1973). However, there is relatively less literature on the microscopic yield of crystals where dislocation starts to move and to multiply without returning to its original site, i.e. plastic deformation starts from a microscopic point of view. We can investigate yield of materials by means of the stress-strain relation. Here, stress is defined as force per unit area and strain is expressed as the ratio of the total deformation to the initial dimension. But it is difficult to know how materials yield since the yield process depends on dislocation motion and its multiplication. The dislocation motion takes place through overcoming of obstacles commonly observed in irradiated materials. It is well known that luminescence occurs when a crystal is deformed or fractured. The former is called deformation luminescence and the latter is fractoluminescence. Recently, it has been reported that fractoluminescence of X-ray irradiated alkali halide crystals is attributed to dislocation motion (Ohgaku, et al., 2002). This phenomenon gives us a useful method to investigate yield process of crystals because we can detect weak light when dislocation begin to move. The aim of this study is to show that we can investigate the microscopic yield process of X-ray irradiated  $\text{KCl:Ca}^{2+}$  by means of deformation luminescence.

## 2. Experimental procedure

KCl single crystals doped with  $\text{Ca}^{2+}$  (0.05, 0.1 and 0.3mol% in melt) were grown from melt of superfine reagent powder by the Kyropoulos method in air. Samples were prepared by cleaving the grown ingot in size of  $2 \times 2 \times 5 \text{mm}^3$  and annealing at 1033K for 24hours. The samples were exposed to X-ray (W-target, 40kV, 30mA) for 2, 6, 20 and 120 min at a rate of 180J/kg/min. F-center concentration is shown in Table 1. Fig.1 shows a schematic illustration of the apparatus and experimental set up. The samples were deformed by compression in the  $\langle 100 \rangle$  direction along the longest axis of a crystal in the dark box of a testing machine (Instron 4465) after any after-glow was not detected. The compression speed was 0.003, 0.03, and 0.3 mm/min. Luminescence was detected by a photo-multiplier (Hamamatsu R928) through a lens and the data were recorded in a personal computer through an amplifier (TEAK SA34). The personal computer recorded load as well as the luminescence intensity. In order to measure

dislocation density, an etching technique was used. Dislocation density was obtained by averaging the number of etch-pits on new surfaces made by cleaving of the central region of a specimen after testing. All experiments were carried out at room temperature although the motion of dislocation and the stability of defects induced by irradiation depend on temperature.

### 3. Results and discussion

Fig.2 shows the relationship between luminescence and stress with strain for 20 minutes X-ray irradiated KCl:Ca<sup>2+</sup>(0.3mol%) at the crosshead speed of 0.03mm/min. Fig.2(b) shows the normal stress-strain curve. The elastic region, yield point and first stage can be seen. The yield stress is about 7Mpa. The yield point in the figure is named macroscopic yield. Fig.2(a) shows that luminescence starts before the macroscopic yield and gradually increases. The starting stress of luminescence is named microscopic yield stress. Fig.3 shows the relation between luminescence intensity and stress. It is seen from the figure that luminescence starts to increase clearly before macroscopic yield. The left arrow indicates the microscopic yield point and the right arrow the macroscopic one. This result indicates that dislocation starts to move overcoming obstacles and dislocation multiplication takes place before macroscopic yield.

It is reported that deformation luminescence intensity is proportional to strain rate or can be expressed as an index of function of strain rate with the power of 0.6-1 (Hayashiuchi, et al. 1990). It is assumed that luminescence intensity is proportional to plastic strain rate because deformation luminescence intensity is considered to be proportional to the area that dislocation sweeps per unit time. This assumption leads to the Johnston-Gilman relation (Johnston and Gilman, 1959).

$$I = A \left( \dot{\epsilon} - \frac{1}{E} \frac{d\sigma}{dt} \right), \quad (1)$$

where  $I$  is the luminescence intensity,  $A$  is a constant,  $\dot{\epsilon}$  the strain rate,  $E$  the effective Young's modulus of the machine and specimen,  $\sigma$  the stress and  $t$  time. Fig.4 shows that the luminescence begins at the maximum value of the time derivative of stress and this point corresponds to the microscopic yield and that the specimen macroscopically yields at the upper bending point near the luminescence intensity of 0.5. The straight line in the figure indicates eq.(1) and agrees with the experimental data between the

microscopic and macroscopic yields.

Dislocation has to multiply during yield process so that a crystal achieves stationary plastic deformation. Fig.5 shows the dependence of dislocation density on stress for the 6 minutes X-ray irradiated KCl:Ca<sup>2+</sup>(0.3mol%). The figure shows that the dislocation density starts to increase at the microscopic yield point and that sharply increases to the macroscopic yield point. This means that dislocation multiplies in the macroscopic elastic region and reaches the macroscopic yield point.

The increase of yield stress with F-center concentration is not large in comparison with its increases with strain rate and with Ca<sup>2+</sup>-concentration (hereafter [Ca<sup>2+</sup>] is used). This is because [Ca<sup>2+</sup>] in a specimen is estimated to be larger than F-center concentration. Additionally, no supralinearity effect in deformation luminescence is reported as far as we know. But we can calculate activation volume from the dependence of yield stress on strain rate. The activation volume reflects the average separation of obstacles on dislocation. In order to obtain the activation volume  $V$ , the following equation was used (Evans and Rawlings, 1969).

$$V = kT \left( \frac{\Delta \ln \dot{\epsilon}}{\Delta \sigma} \right), \quad (2)$$

where  $k$  is the Boltzmann constant and  $T$  absolute temperature. Table 2 shows the activation volume calculated from the dependence of the microscopic yield stress on the crosshead speed. We cannot obtain a reliable value of activation volume from the macroscopic yield stress because of its low dependence on crosshead speed and data spread. It is found from the table that the activation volume is inversely proportional to [Ca<sup>2+</sup>]<sup>1/2</sup> but is not inversely proportional to the square root of F-center concentration with the exception of the case of low [Ca<sup>2+</sup>]. This means that the microscopic yield stress is influenced more by the doped calcium than by the F-center. Then, we averaged the data in order to investigate the dependence of macroscopic and microscopic yield stress on [Ca<sup>2+</sup>] and on strain rate.

Fig.6 shows the linear relation of macroscopic and microscopic yield stress with [Ca<sup>2+</sup>]<sup>1/2</sup>. Six straight lines reflecting the relation seem to converge at a stress of 1.6 MPa. This indicates that the value of 1.6MPa corresponds to the yield stress of a pure crystal. The value of stress is considered to be internal stress or the stress needed to multiply dislocation. The slope of straight line increases with increasing logarithmically with crosshead speed. The relation of the slope with the logarithm of crosshead speed

has the straight line both for macroscopic yield and for microscopic one. The two straight lines intersect at the crosshead speed of 2.2mm/min. This means that if the crosshead speed is over 2.2mm/min the microscopic yield stress becomes larger than macroscopic yield one. Then, yield hump may appear.

#### **4. Conclusion**

It is found that deformation luminescence of X-ray irradiated KCl:Ca<sup>2+</sup> crystals is useful method for investigation of microscopic yield process of crystals. It gives us detail information for yield of materials.

Dislocation moves and multiplies before macroscopic yield because deformation luminescence starts before macroscopic yield.

Taking account of the activation volume obtained from the dependence of microscopic yield stress on crosshead speed, microscopic deformation starts when dislocation overcome Ca<sup>2+</sup> and multiply.

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## Figure captions

Fig.1. Schematic illustration of apparatus.

Fig.2. Relations between luminescence and stress with strain for 20 minutes X-ray irradiated KCl:Ca<sup>2+</sup>(0.3mol%).

Fig.3. Relation between luminescence and stress. The left and right arrows show the microscopic and macroscopic yield stress.

Fig.4. Luminescence intensity versus rate of change of stress for 2 min X-ray irradiated KCl:Ca<sup>2+</sup>(0.1mol%) at the crosshead speed of 0.3 mm/min. Different symbols correspond to another specimen. The straight line shows the Johnston-Gilman relation.

Fig.5. Dependence of dislocation density on stress for the 6 minutes X-ray irradiated KCl:Ca<sup>2+</sup>(0.3mol%). Dislocation density starts to sharply increase at the microscopic yield stress.

Fig.6. Relation of yield stress with [Ca<sup>2+</sup>]<sup>1/2</sup>. Open symbol corresponds to microscopic yield stress and solid symbol to macroscopic one. Circle, square and triangle correspond to the crosshead speeds of 0.003, 0.03 and 0.3 mm/min, respectively.

Table 1. F-center concentration

Table 2. Activation Volume

Table1

Concentration(mol%)	Irradiation time(min)	F <sup>-</sup> center conc.(cm <sup>-3</sup> )
0.05	2	1.7x10 <sup>16</sup>
	6	3.3x10 <sup>16</sup>
	20	7.9x10 <sup>16</sup>
	120	1.3x10 <sup>17</sup>
0.1	2	2.0x10 <sup>16</sup>
	6	5.9x10 <sup>16</sup>
	20	1.1x10 <sup>17</sup>
	120	1.3x10 <sup>17</sup>
0.3	2	2.6x10 <sup>16</sup>
	6	4.6x10 <sup>16</sup>
	20	1.3x10 <sup>17</sup>
	120	2.2x10 <sup>17</sup>

Table2

Impurity conc.(mol%)	Irradiation time(min)	Activation vol.(m <sup>3</sup> )
0.05	2	1.6x10 <sup>-26</sup>
	6	1.1x10 <sup>-26</sup>
	20	5.8x10 <sup>-27</sup>
	120	5.2x10 <sup>-27</sup>
0.1	2	6.8x10 <sup>-27</sup>
	6	6.4x10 <sup>-27</sup>
	20	4.2x10 <sup>-27</sup>
	120	3.3x10 <sup>-27</sup>
0.3	2	4.3x10 <sup>-27</sup>
	6	4.1x10 <sup>-27</sup>
	20	3.8x10 <sup>-27</sup>
	120	2.1x10 <sup>-27</sup>



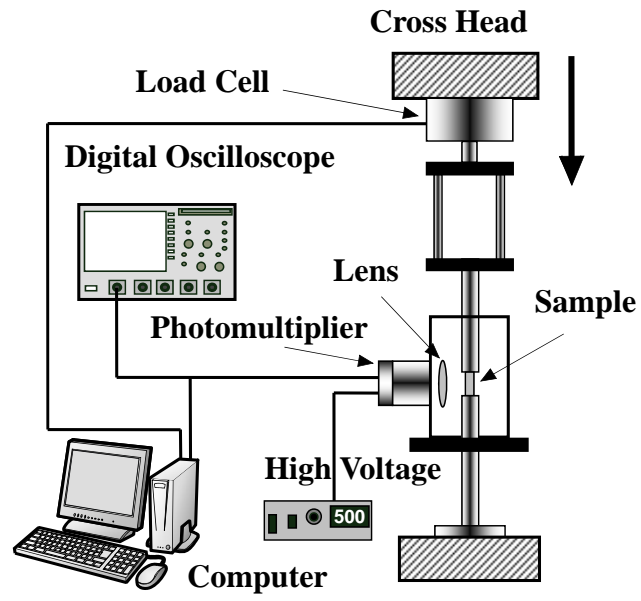


Fig.1

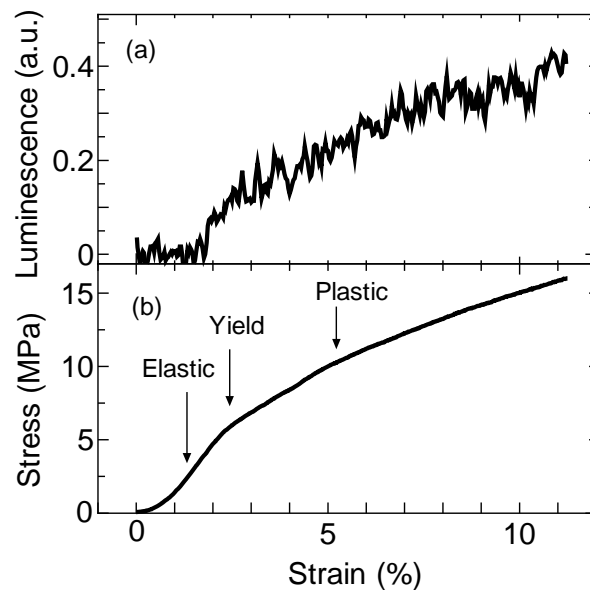


Fig.2

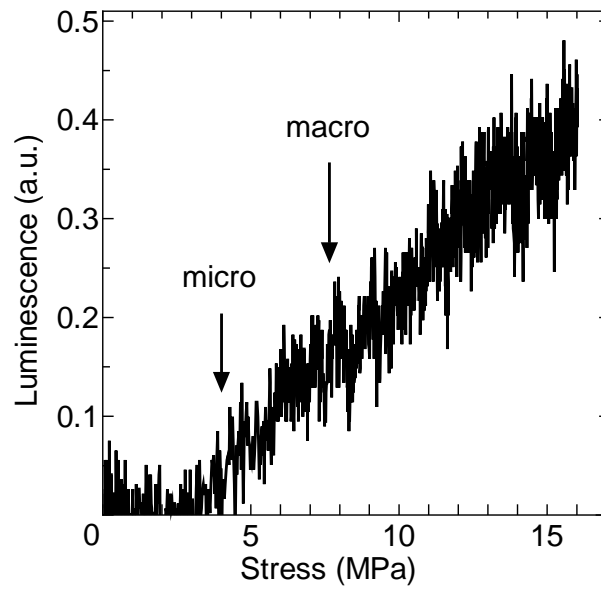


Fig.3

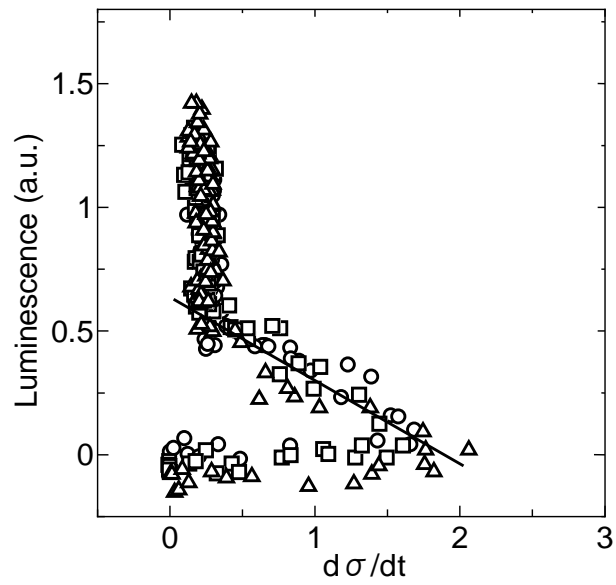


Fig.4

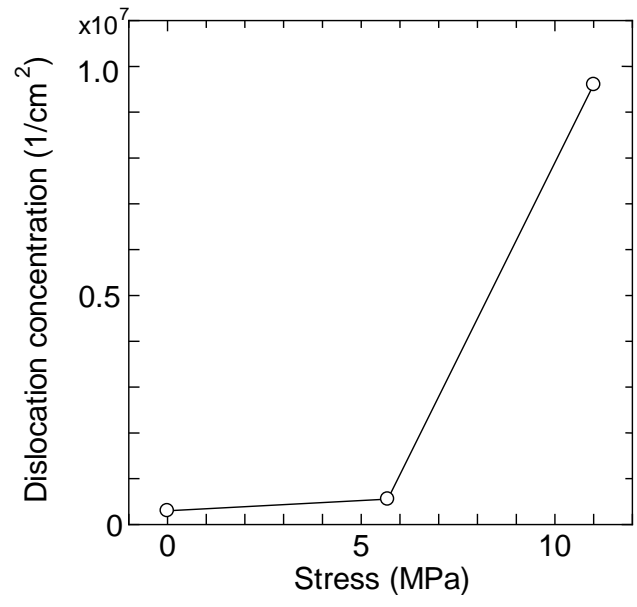


Fig.5

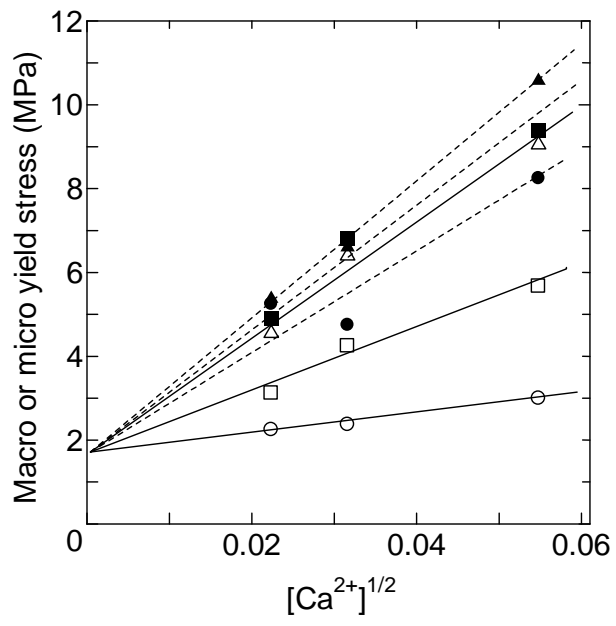


Fig.6