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Amplitude dependent internal friction during plastic deformation of KCl doped with KBr, KI, or $SrCl_2$

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Flow stress decreases by superimposition of ultrasonic oscillatory stress during plastic deformation. The amplitude dependent internal friction is investigated together with the stress decrement due to the superimposition of oscillation during the plastic deformation of KCl single crystals doped with KBr, KI or $SrCl_2$. The relation between the amplitude dependent internal friction and the stress decrement is divided into three regions for KCl doped with KBr or KI. The internal friction is proportional to the stress decrement in the first region up to the first bending point at the lower stress amplitude. The stress decrement at the first bending point is proportional to the square root of KBr or KI concentration. The internal friction of KCl–$SrCl_2$ has the amplitude independent part. The stress decrement at the onset of increasing of internal friction is independent of $SrCl_2$ concentration. The amplitude dependent internal friction of KCl–KBr and KCl–KI consists of the part due to the impurity and that due to forest dislocation. The amplitude dependent internal friction of KCl–$SrCl_2$ is only due to the forest dislocation.

1. Introduction

It is well known that flow stress decreases when oscillatory stress is superimposed during plastic deformation. The phenomenon is called the Blaha effect in honor of the discoverer [1] and is principally attributed to the superimposition of oscillatory stress [2–5]. It is however reported that the cause of the Blaha effect is the increase in activation volume because the strain rate sensitivity decrease when the strain rate change test is carried out under application of the oscillations [6]. Moreover, the internal friction during the plastic deformation increases proportionally to the stress decrement by the application of oscillatory stress. This proportional relation of the internal friction and the stress decrement is explained by the breakaway of dislocation from weak obstacles under the application of oscillatory stress. On the other hand, the stress decrease is mainly reported to be the decrease in internal stress by application of oscillation [7].

If the mechanism that flow stress decreases under application of oscillatory stress affects the internal friction during plastic deformation, the impurity concentration is expected to influence the amplitude dependent internal friction. In this paper, we will investigate the influence of impurity concentration on internal friction during the plastic deformation of KCl single crystals doped with KBr, KI, or $SrCl_2$.

2. Experimental procedure

The specimens are KCl single crystals containing various impurity KBr (0.5, 1 and 2 mol% in melt), KI (0.2, 0.5 and 1 mol% in melt) or $SrCl_2$ (0.035, 0.05 and 0.065 mol% in melt). Br^- or I^- ion is substituted for Cl^- in KCl crystal. In KCl including higher concentration of Br^- or I^- the double kink formation rather than the elastic interaction between the dislocation and Br^- controls the dislocation motion [8]. Those concentrations of Br^- or I^- are therefore chosen. Br^- or I^- in KCl mainly interacts with edge dislocation while Sr^{2+} forming impurity-vacancy dipole interacts with screw as well as edge dislocations [9]. Accordingly, smaller concentration of Sr^{2+} than Br^- or I^- ion can strengthen KCl crystal. This is one of the reasons why the concentration of Sr^{2+} is chosen. The other is that the plastic deformation region of KCl doped with higher concentration of $SrCl_2$ is too short to carry out the experiment. Specimens of size of $5 \times 5 \times 15$ mm^3 were obtained by cleaving the ingots grown by the kylopoulos method in air. The cleaved specimens were annealed immediately below the melting point for 24 h, followed by cooling to room temperature at the rate of 40 K/h. The $SrCl_2$ doped specimens were held at 773 K half an hour and quenched to room temperature immediately before the tests.

The specimens were compressed in the direction of $\langle 100 \rangle$ along the longest axis at the strain rate of

1.1×10^{-5}/s by the testing machine, Shimadzu DSS-500. The oscillatory stress with the frequency of 20 kHz was intermittently superimposed for one or two minutes in the same direction as the compression. The internal friction was estimated by the temperature rise due to the superimposition of oscillatory stress during plastic deformation. The temperature rise was measured by the copper-constantan thermocouple stuck on the specimen. The energy loss of oscillation is obtained by the comparison of the temperature rise of the specimen with that of the dummy specimen, which includes a heater inside, and then the internal friction is estimated by the ratio of the energy loss to the oscillatory energy. Fig. 1 shows the variation of flow stress and the temperature when the oscillatory stress is applied intermittently. The stress drop and the temperature rise are shown to increase with the increasing stress amplitude in order from the left. The temperature rise is approximately proportional to a square of the stress decrement. This square relation indicates that the internal friction keeps constant because the energy of oscillation is proportional to a square of the stress amplitude and the stress decrement is roughly proportional to the stress amplitude [6]. Fig. 2 shows the strain dependence of the stress decrements and the internal frictions obtained from the temperature rise. The relations between the internal friction and the stress decrement at a given strain were obtained for each specimen from a figure such as this one.

3. Results and discussion

3.1. Concentration dependence of KBr or KI

The average dislocation velocity is 5×10^{-6} cm/s in our tests, if the dislocation density is 10^8/cm^2. The diffusion velocity of Cl^-, Br^- and I^- in KCl at the room temperature is of the order of 10^{-26}–10^{-25} cm/s when it is estimated from the data of Refs [10] and [11]. This is much smaller than the dislocation velocity. Therefore, the impurities, Br^- or I^- ion in KCl single crystal are considered to be an immobile obstacle for dislocation motion and not to form atmospheres around the mobile dislocation.

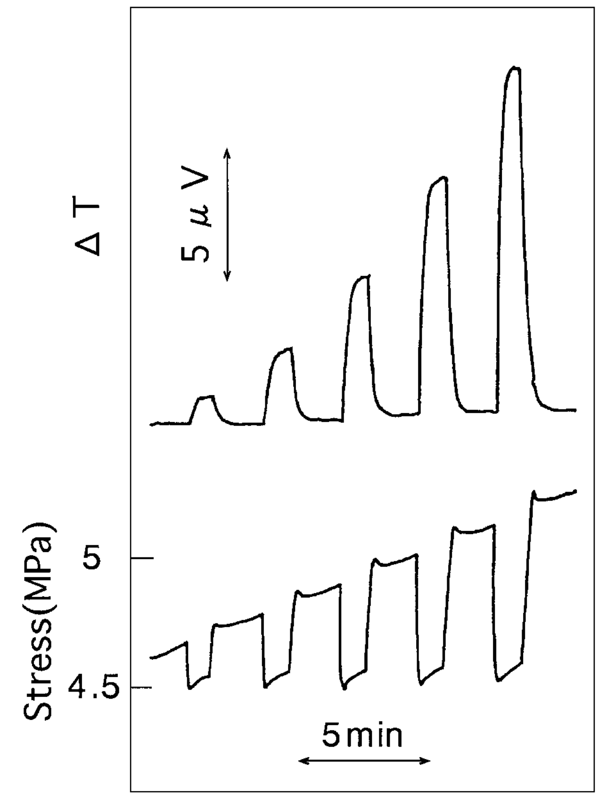


Figure 1 Variation of flow stress and temperature of the specimen under intermittently application of ultrasonic oscillation.

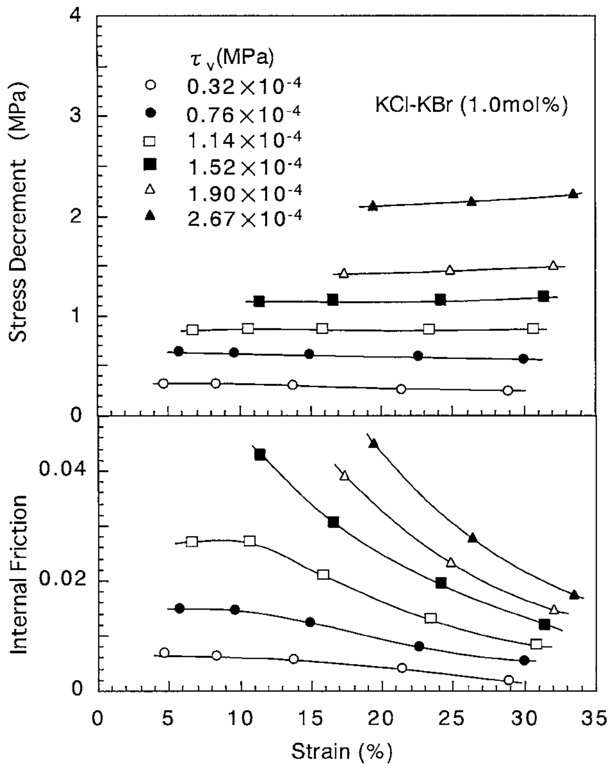


Figure 2 Dependence of the stress decrement and the internal friction on the strain with various stress amplitude τ_V.

The relations of the internal friction with the stress decrement for KCl single crystals doped with KBr or KI are shown in Figs 3 and 4. The curves shift down with increasing strain. This is probably because the average separation between forest dislocations, which act as a strong obstacle, decreases with increasing strain. And the curves also shift down with increasing impurity concentration because of the decreasing average separation between impurities which act as weak obstacles. There seem to be an amplitude independent part of internal friction for KCl–KBr (0.5 mol%) at the strain of 5%. The amplitude independent internal friction is too small to appear for the other curves.

The curves may be divided into three regions by two 'bending points.' This is also shown in Fig. 5 for the undoped KCl crystal and other crystals [12]. The three regions are designated I, II and III in the figure. The relations between the internal friction and flow stress at a given stress decrement in region I and III are shown, using logarithmic scale, in Fig. 6. In region III the relations have straight lines with a slope of about minus two for the three types of crystal. The plots can be put on the curves for KCl–KBr (0.5%), multiplying both the internal friction and the flow stress by an appropriate value, as shown in Fig. 7. There are two curves for region I and region III respectively. This suggests

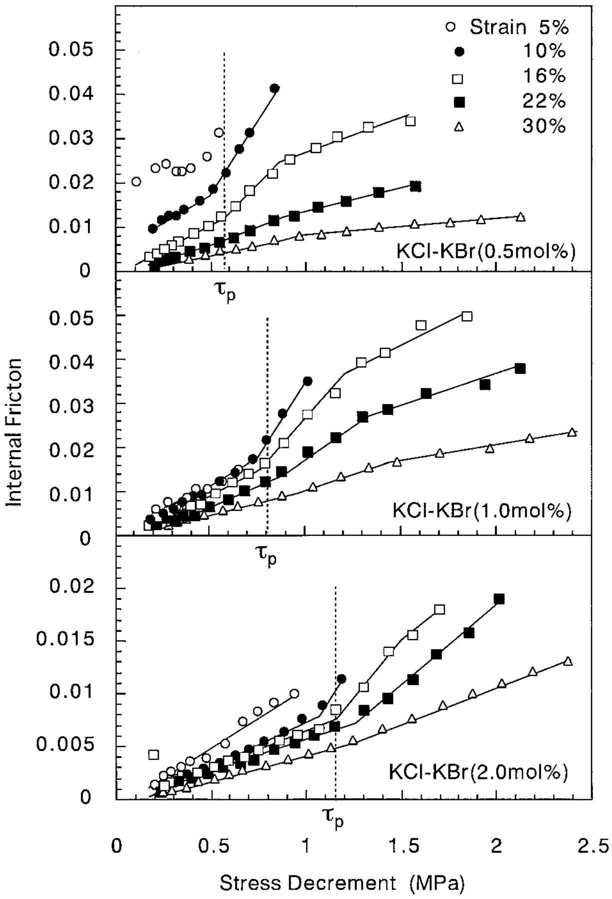


Figure 3 Relation between the internal friction and the stress decrement at several strains for KCl–KBr (0.5 mol%), KCl–KBr (1.0 mol%) and KCl–KBr (2.0 mol%).

that the mechanism in region I is different from that in region III.

The proportionality between the internal friction and the stress decrement is shown in region I up to the first bending point τ_p (see Figs 3 and 4). In this region, it is considered that the dislocation overcome the weak obstacles such as impurities lying on a dislocation segment between two strong obstacles such as forest dislocations with the aid of oscillation and begins to oscillate between the two strong obstacles. The fraction of dislocation segments that they break away from weak obstacles and oscillate between strong obstacles increases proportionally to amplitude dependent internal friction with increasing stress amplitude [13]. This means that the average length of dislocation segment increases with increasing stress amplitude. And it is seen that the internal friction is proportional to the stress decrement [6]. The fraction is saturated at the bending point τ_p, where all the weak obstacles are overcome by the oscillating dislocations and they are no longer obstacles to dislocation motion. Therefore, the value τ_p must be inversely proportional to the average separation between weak obstacles. That is to say, τ_p should be proportional to the square root of impurity concentration. This is shown in Fig. 8a and b by the logarithmic scale. The figures include the impurity concentration dependence of yield stress and half of σ_p in Fig. 6 of Ref. [14]. When the oscillation is applied to the specimen during deformation the average length of dislo-

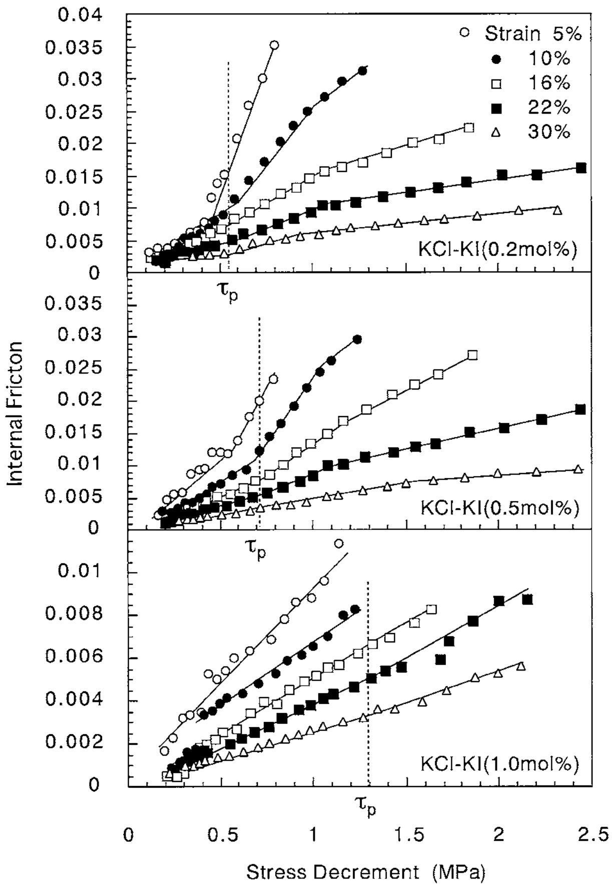


Figure 4 Relation between the internal friction and the stress decrement at several strains for KCl–KI (0.2 mol%), KCl–KI (0.5 mol%) and KCl–KI (1.0 mol%).

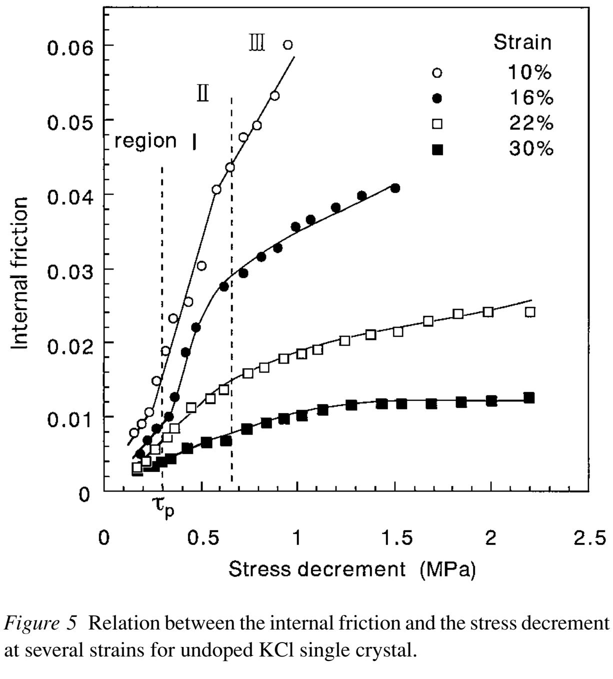
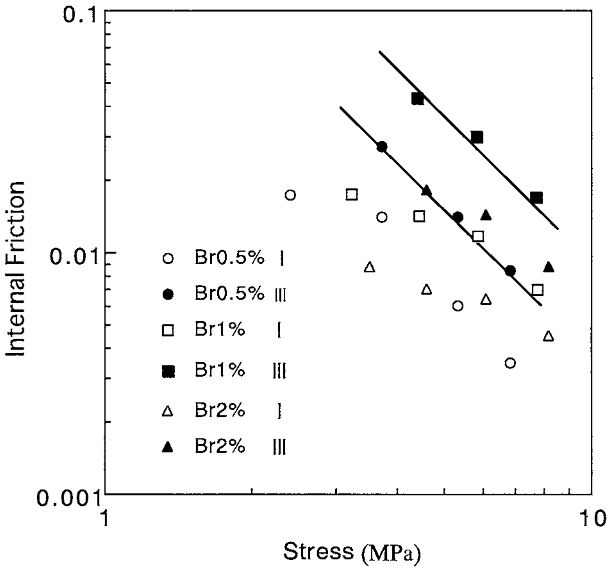
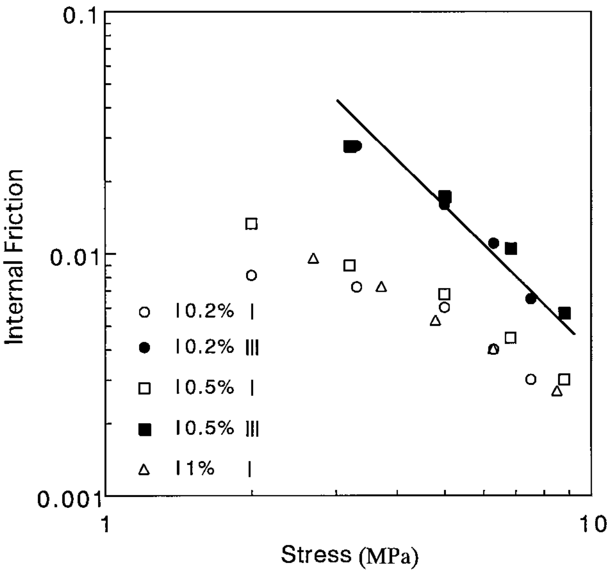


Figure 5 Relation between the internal friction and the stress decrement at several strains for undoped KCl single crystal.

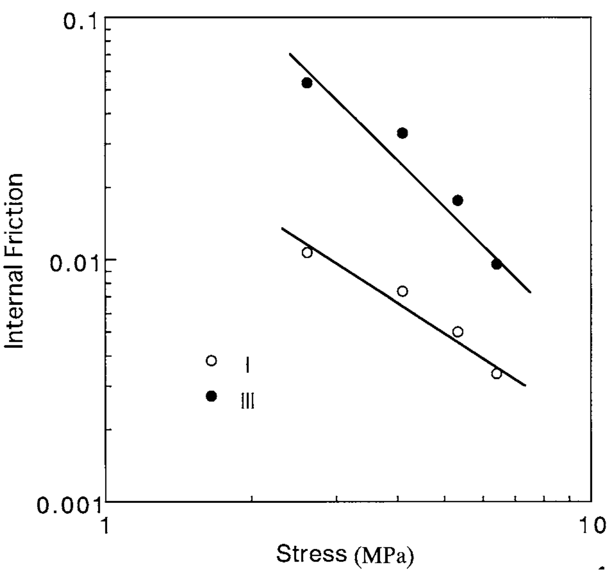
cation segment increases and the strain rate sensitivity decreases. And when the fraction of dislocation segments that they break away from weak obstacles and oscillate between strong obstacles saturates the strain rate sensitivity stops to decrease at the applied stress decrement σ_p and become to be constant with increasing stress decrement. Every relation lies on the straight



(a)



(b)



(c)

Figure 6 Relation between the internal friction and flow stress in region I and III for (a) KCl–KBr, (b) KCl–KI and (c) undoped KCl.

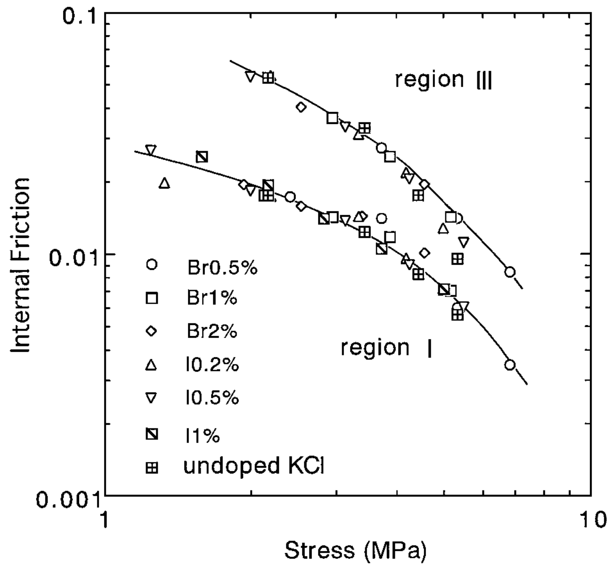


Figure 7 Superposition of the curves on the one for KCl–KBr (0.5 mol%) by translation.

line with the slope of 1/2. The line shift upward with increasing strain. This is provably because the separation between weak obstacles slightly decreases with increasing strain. The dependence of τ_p on impurity concentration indicates that the impurity affects the internal friction during plastic deformation as well as the Blah effect [14].

In region III the relation between the internal friction and flow stress have a slop of about minus two by the logarithmic scale, irrespectively of specimen (see Fig. 6 and Ref. [12]). This means that the internal friction in region III is in principle independent of impurity and type of crystal but is considered to depend on deformation, i.e. forest dislocation density. The internal friction in region II and III correspond to the amplitude dependent part and saturated one of internal friction due to forest dislocation. Consequently the amplitude dependent internal friction during deformation of KCl crystals consists of the part due to the impurity and that due to forest dislocation.

3.2. KCl doped with $SrCl_2$

The diffusion coefficient of Sr^{2+} in KCl is estimated to be 1×10^{-7} cm^2/s at room temperature [15]. This means that the diffusion velocity of Sr^{2+} in KCl is 2×10^{-10} cm/s. The velocity is thus much smaller than the dislocation velocity in our tests. Hence, Sr^{2+} in KCl is regarded as an immobile obstacle as well as Br^- or I^-. However, Sr^{2+} in KCl forms the I–V dipole combining the cation vacancy. Then, the I–V dipole may rotate by a dislocation moving near.

Fig. 9 shows the amplitude dependent internal friction for KCl–$SrCl_2$ (0.035, 0.05 and 0.065 mol%). It seems that there are amplitude independent parts of internal friction, except that the internal friction at the small strain and smaller stress amplitude decreases with amplitude. The curves shift downward parallel to the ordinate axis with increasing strain. The curves were also observed to shift down with $SrCl_2$ concentration.

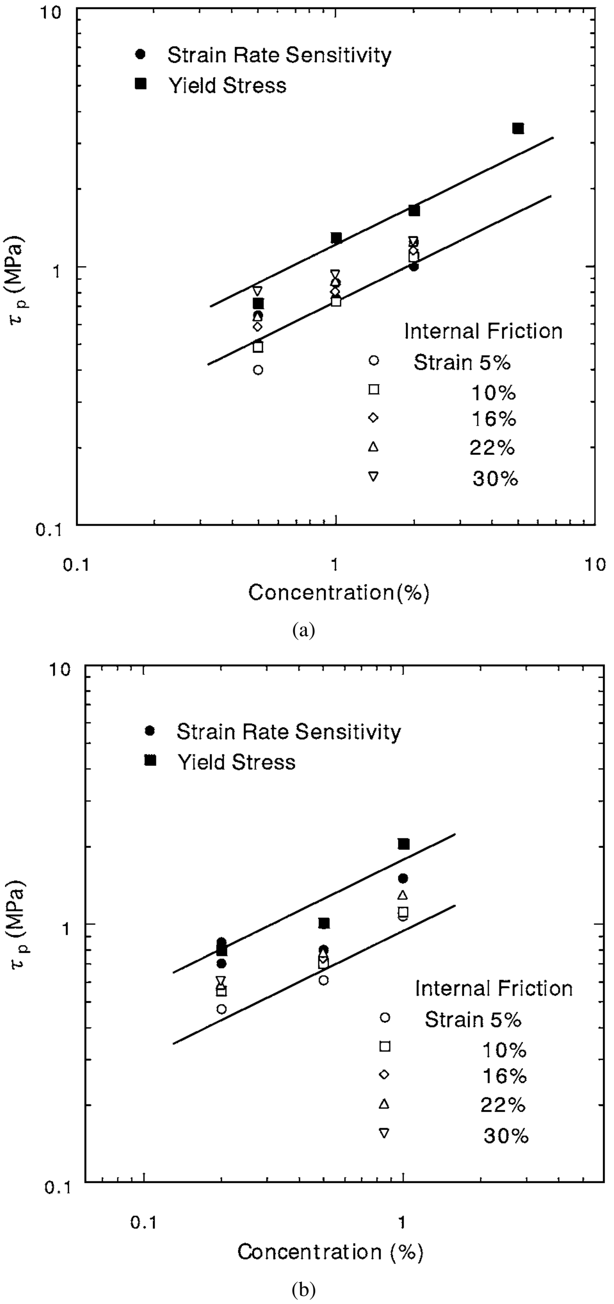


Figure 8 Impurity concentration dependence of τ_p obtained from strain rate sensitivity measurement and from internal friction one, and the yield stress. Impurities are (a) KBr and (b) KI.

The internal friction begins to increase at the stress decrement of about 1 MPa independently of strain and impurity concentration. The curves for KCl–$SrCl_2$ (0.035 mol%) are considered to have region II and III. The other curves have only region II. No region I is shown in the figures. The relations between internal friction and the flow stress in amplitude independent region and in region III are shown by the logarithmic scale in Fig. 10. The plots for each region lie on the straight line with a slope of minus two. This means that the internal friction of KCl–$SrCl_2$ is mainly related to deformation. These phenomena are explained as follows.

The strain rate cycling tests of KCl–$SrCl_2$, which is carried out under superimposition of ultrasonic oscillatory stress, indicates that the strain rate sensitivity due

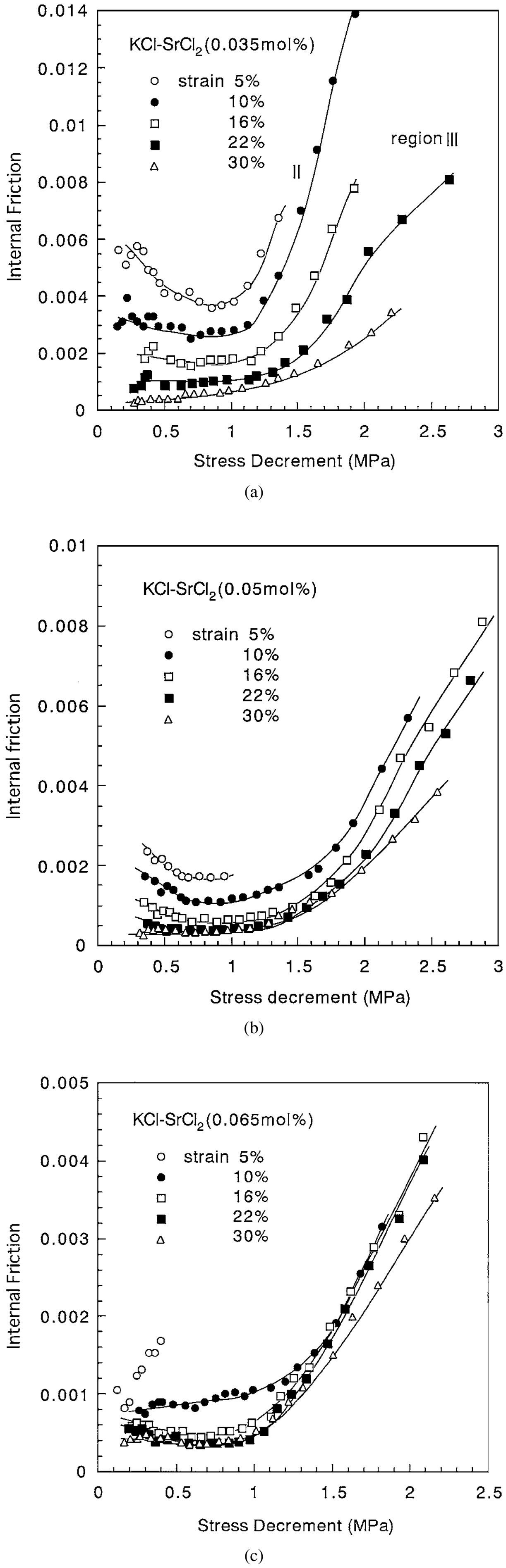


Figure 9 Relation between the internal friction and the stress decrement at several strains for KCl–$SrCl_2$. The concentrations of $SrCl_2$ are (a) 0.035 mol%, (b) 0.05 mol%, and (c) 0.065 mol%.

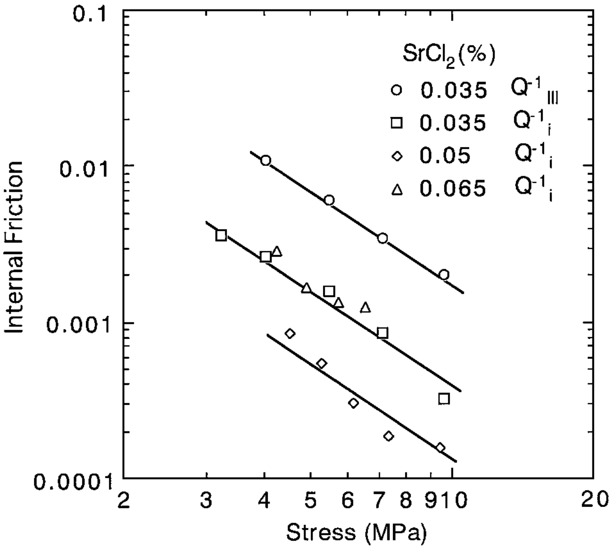


Figure 10 Relation of the internal friction (Q_i^{-1}) in the amplitude independent region and (Q_{III}^{-1}) in region III with the flow stress for KCl–$SrCl_2$ crystals with the Sr^{2+} concentration of 0.035, 0.05 and 0.065 mol%.

to $SrCl_2$ impurity does not appear at room temperature [16]. This should lead to the result that the amplitude dependent internal friction due to Sr^{2+} which corresponds to region I does not appear, as seen in Fig. 9. That is to say, it means that Sr^{2+} does not act as a short range obstacle to the oscillating dislocation at room temperature. Then, the amplitude dependence internal friction of KCl–$SrCl_2$ is chiefly attributed to the forest dislocation multiplying during deformation. The decrease of internal friction in amplitude independent region at the small strain and smaller amplitude may concern the rotation of I–V dipole by moving dislocation. Consequently, the influence of impurity concentration on internal friction during plastic deformation of KCl single crystals is dependent on type of impurity.

4. Conclusion

It is found that the amplitude dependent internal friction decreases with increasing strain and impurity concentration. The decrease concerning the strain is due to forest dislocations. The relation between the amplitude dependent internal friction and the stress decrement is divided into three regions for KCl doped with KBr or KI. The first bending point τ_p is proportional to the square root of impurity concentration. Therefore, the amplitude dependent internal friction in region I is due to an impurity. The amplitude dependent internal friction in region II and III is due to forest dislocation. And it is also found that the amplitude dependent internal friction of KCl–$SrCl_2$ is chiefly attributed to forest dislocation. This corresponds to the situation that the strain rate sensitivity due to impurity does not appear at room temperature. The influence of impurity concentration on internal friction during plastic deformation of KCl single crystals is dependent on type of impurity.

References

1. F. BLAHA and B. LANGENECKER, *Naturwissenschaften* **42** (1955) 556.
2. G. S. BAKER and S. H. CARPENTER, *J. Appl. Phys.* **38** (1967) 1586.
3. G. KAISER and W. PECHHOLD, *Acta Metall.* **17** (1967) 527.
4. T. ENDO, M. TASAKI, M. KUBO and T. SHIMADA, *J. Japan. Inst. Metals* **46** (1982) 773.
5. T. OHGAKU and N. TAKEUCHI, *J. Mater. Sci.* **20** (1985) 2245.
6. *Idem.*, *Phys. Status. Solidi. (a)* **105** (1988) 153.
7. K. V. SAPOZHNIKOV, S. N. GOLYANDIN, S. B. KUSTOV, Y. NISHINO and S. ASANO, *Phil. Mag.* **77** (1998) 151.
8. T. KATAOKA and T. YAMADA, *Japan J. Appl. Phys.* **18** (1979) 55.
9. R. L. FLEISCHER, *J. Appl. Phys.* **33** (1967) 3504.
10. S. PEHKONEN, *J. Phys. D* **6** (1972) 538.
11. J. C. BEAUMONT and J. CABANE, *C. R. Acad. Sci.* **252** (1961) 113 and 266.
12. T. OHGAKU and N. TAKEUCHI, *Phys. Status. Solidi. (a)* **111** (1989) 165.
13. A. V. GRANATO and K. LUCKE, *J. Appl. Phys.* **52** (1981) 7136.
14. T. OHGAKU and N. TAKEUCHI, *Phys. Status. Solidi. (a)* **118** (1990) 153.
15. M. F. DE SOUZA, *Phys. Rev.* **188** (1969) 1367.
16. Y. KOHZUKI, T. OHGAKU and N. TAKEUCHI, *J. Mater. Sci.* **30** (1995) 101.

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