

## Dislocation Relaxation Peaks in Aluminum

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Bordoni relaxation (BR) of internal friction peaks in fcc metals around liquid nitrogen temperatures, was investigated experimentally using a pure aluminum crystal by a resonant bar method of 51 kHz. Each time after slight deformations or subsequent anneals, we measured the BR at temperatures between 78 K and 250 K. Usually the Bordoni relaxation consists of two broad peaks, i.e., the Bordoni peak ( $B_2$  peak) at higher temperature ( $\sim 180$  K) and Niblett-Wilks peak or shoulder ( $B_1$  peak) at lower temperature ( $\sim 120$  K). It was not expected that after an anneal at 800 K in air, there appeared four sharp internal friction peaks for the temperature range of BR. The peak located at 120 K seems related with  $B_1$  peak. The remaining three peaks seem related with  $B_2$  peak. These sharp peaks are found probably for the first time.

Key words: Bordoni relaxation, internal friction, dislocations, fcc metals, Peierls stress

### 1. INTRODUCTION

Plastic deformation in metals has been well understood macroscopically and technologically. However, the Peierls mechanism in fcc metals, which is a microscopic mechanism for the resistance to the dislocation motion due to discrete arrangement of atoms, is not well understood. Most researchers studying plasticity of fcc metals believe that the Peierls stress in fcc metals should be negligibly small, i.e., less than  $10^{-5}$  G (G: shear modulus) from yield stress measurements at low temperature. On the other hand, from the Bordoni relaxation peaks of internal friction in fcc metals, the Peierls stress is estimated to be  $10^{-3}$  G by assuming the kink pair formation (KPF) theory for the mechanism [1,2]. Disagreement of the Peierls stress between estimated from flow stress measurements of plasticity and the Bordoni peak has not been solved since the KPF theory is proposed in 1956 [3]. Recently the similar dislocation relaxation peaks are found in bcc metals and ionic crystals, and the estimated Peierls stress from the KPF theory agrees well with that from flow stress at low temperatures for these crystals [4,5]. Further, in a high purity aluminum a low-temperature dislocation relaxation peak is found at 11 K for 53kHz [6]. If we assume that the 11 K peak is due to the KPF mechanism, the calculated Peierls stress is  $3 \times 10^{-5}$  G. This value is consistent with the flow stress of the plasticity measurements at low temperatures. If the 11 K peak is due to the KPF, what is the Bordoni relaxation?

Very recently we investigated the change of the peak height, the peak temperature and the peak shape on the 11 K peak and the BR against slight deformations or anneals in a high purity aluminum sample. While the 11 K peak satisfies all the features predicted from the KPF theory, the peak temperature change of the Bordoni peak ( $B_2$  peak) against anneals does not [7]. There is a consensus that the BR is due to an intrinsic motion of dislocations [8,9]. However, there might be other intrinsic mechanisms than the KPF, e.g., such as dislocation-dislocation interactions. Thus we planed to investigate the BR in the other aluminum sample with

different impurity concentration.

### 2. EXPERIMENTS AND DISCUSSION

Internal friction (IF) is measured by a resonance method in a composite oscillator made of a  $-18.5^\circ\text{X}$ -cut quartz bar and an aluminum sample bar. The size of the quartz is  $3.5 \times 3.5 \times 50$  mm and the resonant frequency is 51 kHz at room temperature [10].

5N aluminum (99.999% purity) is used for the sample. The sample is a single crystal bar ( $3.5 \times 3.5 \times 50$  mm) made by strain anneal. The axial direction is [2 -4 1]. The general features of the BR are summarized as follows [1]: The height of the BR increases with increasing plastic deformation from the annealed state. The BR consists of two broad peaks, i.e., the Bordoni peak or  $B_2$  peak located at higher temperature and the Niblett-Wilks peak or  $B_1$  peak as a subsidiary peak. The internal friction is larger for purer samples. Thus the BR is attributed to an intrinsic mechanism of dislocations. The height of BR will saturate by further deformations and even will decrease in the high temperature side of BR. By annealing the height decreases in the whole temperature range and the  $B_1$  and  $B_2$  peaks become unclear.

Last time, we measured the BR in addition to the 11 K peak in a very high purity aluminum (zone-refined Al: z.r. Al). The magnitude of BR in z.r. Al is rather large for the deformed state, i.e.,  $10^{-2}$  for IF [7]. The composite oscillator method we have been using is more suitable for relatively small IF, i.e.,  $10^{-5}$  to  $10^{-3}$  [10]. Thus we choose 5N Al (99.999% purity) for the sample.

To check the details of the change in the peak height, the peak temperature and the shape, internal friction was measured each time the sample was slightly deformed or annealed. The sample was first annealed in air at 770 K for 17 h by using a moving furnace to reduce the dislocation density and the internal stress. The aluminum oxide formed during the anneal was removed chemically. Then the sample was slightly deformed by a four point bending in both directions at room temperature (def.1:plastic strain  $\varepsilon_p \sim 5 \times 10^{-5}$ ).

Further deformations were made by the same manner with different plastic strains, i.e., def.2:  $\epsilon_p \sim 2 \times 10^{-4}$  and def.3:  $\epsilon_p \sim 1 \times 10^{-3}$ . After the deformations, the sample was annealed in air at 323 K, 373 K, 423 K and 473 K, respectively for 1 h. Lastly the sample was annealed at 800 K in air by the moving furnace for 15 h.

Internal friction measurement is made from 78 K to 250 K with increasing temperature. The strain amplitude of the measurement is about  $10^{-7}$ , at which there is no amplitude dependent internal friction.

The results are shown in Fig.1.

(i) annealed: 773 K for 17h in air in a traveling furnace;

The IF is relatively small ( $\sim 10^{-4}$ ) and slowly increases with increasing temperature. The purer sample usually has a larger dislocation relaxation. The relaxation magnitude of this sample is as same as that of the Al-50 ppm Zn [6]. The impurity concentration of this sample is thought to be 10ppm or more.

(ii) def.1: plastic strain  $\epsilon_p \sim 5 \times 10^{-5}$ ;

The internal friction increased 3 to 5 times compared with the annealed state. There is a very broad peak with a maximum around 175 K and a shoulder around 115 K.

(iii) def.2:  $\epsilon_p \sim 2 \times 10^{-4}$ ;

The internal friction further increased. A clear

maximum at 175 K and a shoulder at 115 K are seen. The former and latter are the  $B_2$  peak (Bordoni peak) and the  $B_1$  peak (Niblett-Wilks peak), respectively. The decrease of internal friction with increasing temperature is clearly seen at the high temperature side of  $B_2$  peak

(iv) def.3:  $\epsilon_p \sim 1 \times 10^{-3}$ ;

The IF did not change between 78 K and 175 K. But the IF decreased between 175 K and 240 K. The saturation of the magnitude and the decrease of high temperature side of the BR are often reported [1]. Then we investigated the change with annealing.

(v) anneal at 323 K for 1h;

The internal friction decreased between 78 K and 175 K, especially between 120 K and 170 K. The magnitude did not change above 180 K. As a result, the  $B_2$  peak locates at 180 K.

(vi) anneal at 373 K for 1h;

The internal friction decreased much above 180 K and that resulted in the maximum of the  $B_2$  at 165K.

(vii) anneal at 423 K for 1h;

The internal friction decreased much over the whole measured temperatures. The magnitude is as low as initial annealed case, except a small peaks around 160 K and 210 K.

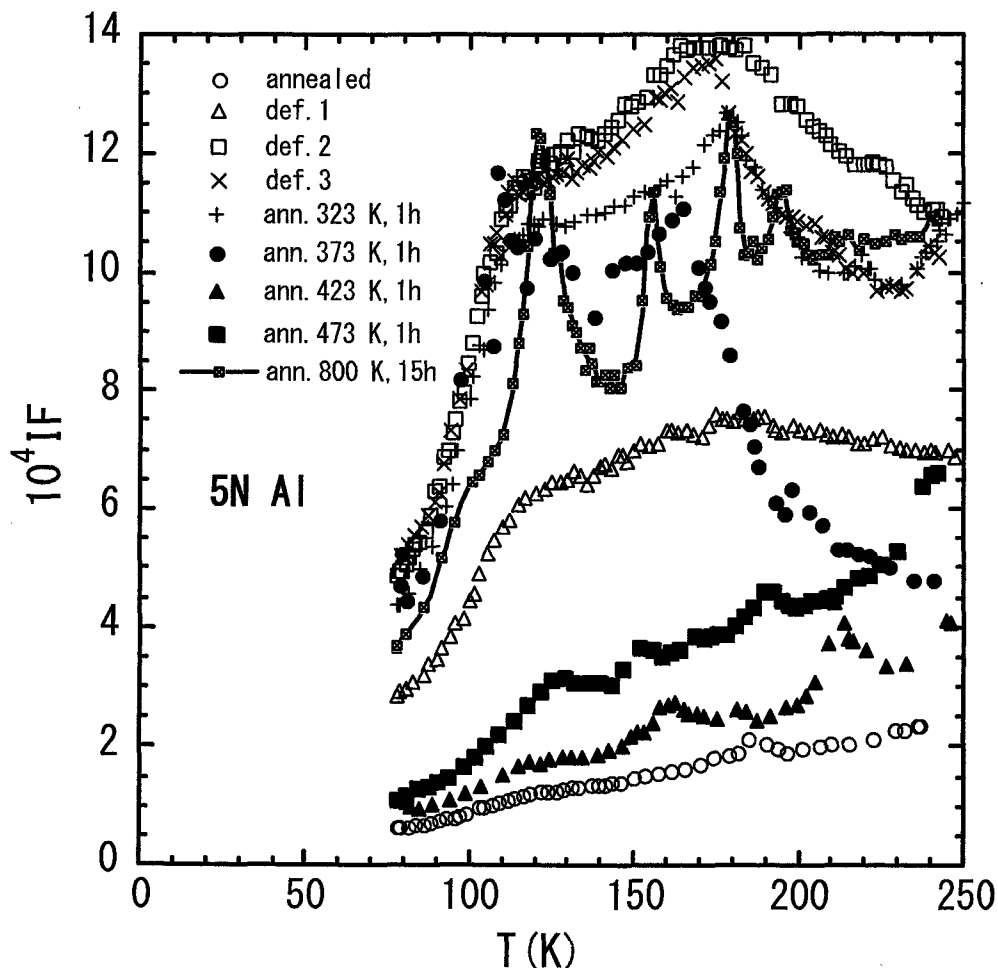


Fig.1 Internal friction of 5N aluminum. The frequency is about 51 kHz. annealed: 773 K for 17h in air; def.1: plastic strain  $\epsilon_p \sim 5 \times 10^{-5}$ ; def.2:  $\epsilon_p \sim 2 \times 10^{-4}$ ; def.3:  $\epsilon_p \sim 1 \times 10^{-3}$ ; ann.: annealed in air.

(ix) anneal at 473 K for 1h;

There was an accident during the measurement that the vibration condition of the composite oscillator was not good enough for taking data. After the oscillator being fixed, re-measurement was made for the annealed state. It was noticed that internal friction increased compared with the previous measurement. There might be some handling effect on the sample during the preparation of composite oscillator. There are small peaks at 125 K, 152 K, 170 K and 190 K. Then we decided to anneal the sample at a high enough temperature to finish the measurements.

(x) anneal at 800 K by using a moving furnace in air for 15h;

We expected to reduce the IF as small as that of the initial annealed state. Against our expectation, the internal friction became very large as the deformed states. The spectrum is rather different from that of the BR which is usually composed of two broad peaks  $B_1$  and  $B_2$ . There are four sharp peaks located at 120 K, 155 K, 180 K and 195 K respectively. The magnitude, however, is similar to that of def.3 or the annealed state at 323 K over the measured temperatures. These peaks should be due to dislocation relaxation as the BR. The peak located at 120 K is as sharp as a Debye peak. Other three peaks located at higher temperatures look sharper than a Debye peak. The appearance of these sharp dislocation peaks seems unreported before. A possible reason for the appearance, as far as we know, is a plastic deformation of the sample during cooling it to liquid nitrogen temperature. After the anneal the surface of the sample was oxidized. If the removal of oxidized surface from the aluminum sample is not complete enough, the difference of the thermal expansion between the aluminum oxide and the bulk may produce a plastic strain when the sample is cooled down to liquid nitrogen temperature. The thermal expansions are  $25 \times 10^{-6} \text{ K}^{-1}$  and  $8 \times 10^{-6} \text{ K}^{-1}$  for aluminum and aluminum oxide (alumina), respectively.

The difference of the strain between aluminum and alumina would be  $4 \times 10^{-3}$  when the sample was cooled to liquid nitrogen temperature. It could have been that at the boundary of aluminum and alumina film the stress was produced and aluminum was plastically deformed.

As a result the sharp peaks were found for the first time. The peak at lowest temperature ( $\sim 120$  K) seems closely related with the  $B_1$  peak. The rest three peaks seem related with the  $B_2$  peak. If these peaks are attributed to elementary processes in the Bordoni relaxation, further investigation on these peaks is very important. We firstly need to know the experimental condition to reproduce the sharp peaks. Then we would like to study the details of the properties of the sharp peaks.

#### References

- [1] G. Fantozzi, C. Esnouf, W. Benoit and I.G. Ritchie, *Prog. Mater. Sci.* 27, 311-451 (1982).
- [2] "Mechanical Spectroscopy  $Q^{-1}$  with Applications to Materials Science" Ed. by R. Schaller, G. Fantozzi and G. Gremaud, Trans. Tech. Publications, Ueticon, Zurich (2001).
- [3] A. Seeger, *Philos. Mag.* 1, 651-662 (1956).
- [4] H. Schultz, *Mater. Sci. Eng. A*, 141, 149 (1991).
- [5] T. Kosugi and T. Kino, *Mater. Sci. Eng. A*, 164, 368-372 (1993).
- [6] T. Kosugi and T. Kino, *J. Phys. Soc. Jpn.*, 58, 4269-4272 (1989).
- [7] T. Kosugi, K. Sakieda and Y. Kogure, *Mater. Sci. Eng. A*, 442, 147-150 (2006).
- [8] A.S. Nowick and B.S. Berry, "Anelastic Relaxation in Crystalline Solids", Academic Press, New York (1972).
- [9] R.De. Batist, "Internal Friction of Structural Defects in Crystalline Solids", North Holland, Amsterdam (1972).
- [10] T. Kosugi, *Jpn. J. Appl. Phys.*, 33, 2862-2866 (1994).

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