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# Dislocation relaxation in silver single crystals at megahertz frequencies

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**Abstract.** The Bordoni peak in high-purity silver single crystals has been investigated by measuring the attenuation of longitudinal waves of 5, 10 and 30 MHz, using a conventional ultrasonic pulse technique with single quartz transducer. The crystals, of  $\langle 100 \rangle$ ,  $\langle 110 \rangle$  and  $\langle 111 \rangle$  orientations, were deformed by compression, and the temperature-dependent ultrasonic attenuation was measured after applying resolved shear stresses ranging up to  $60 \text{ N mm}^{-2}$ . The results lead to the following conclusions: (i) the temperature of the Bordoni peak in silver single crystals is essentially independent of the orientation; (ii) the temperature of the Bordoni peak in silver single crystals decreases as the prior deformation is increased up to resolved shear stress near  $28 \text{ N mm}^{-2}$ , and then increases for higher prior stresses; (iii) the Bordoni peak height decreases as the frequency increases and (iv) the Bordoni peak height increases with increasing the amount of deformation.

## 1. Introduction

The internal friction peak in deformed FCC metals, which was first observed by Bordoni [1] has been extensively investigated at frequencies in the kilohertz range, but there have been relatively few measurements of it at megahertz frequencies.

The principal features of the Bordoni peak have been reviewed by a number of authors, Niblett [2], Seeger [3], Nowick and Perry [4], De Batist [5], Burdett and Queen [6] and Fantozzi *et al* [7]. It is now generally agreed that the peak is caused by thermally activated formation of pairs of kinks on dislocation. This was first suggested by Seeger [8] and modifications of the theory have been made by Paré [9], Seeger and Schiller [10], Alfield [11], Engelke [12], Schlipf and Schindlmayr [13] and Ensouf and Fantozzi [14]. Work on polycrystalline and single crystals of different orientations by Grandchamp [15], Mecs and Nowick [16], Niblett and Zein [17], Zein [18], Zein and Alnaser [19] and Zein and Alnaser [20], shows that for a given metal at a particular frequency, the dislocation relaxation peaks always occur at the same temperature, apart from variation of a few degrees for specimens given different mechanical and thermal treatments. However, Mongy *et al* [21] investigated a silver single crystal at frequencies of 10, 20 and 50 MHz and found a strong dependence of the temperature of the peak on the orientation of the crystal with respect to the direction of propagation of the ultrasonic pulses. For example, at 10 MHz the peak occurred at 149, 137 and 175 K respectively when the attenuation was measured along  $\langle 100 \rangle$ ,  $\langle 111 \rangle$  and  $\langle 110 \rangle$  directions. They also found similar results for aluminium [22], gold [23] and copper [24] single crystals.

Any such anisotropic effects occurring at megahertz frequencies would also manifest themselves in measurements on single crystals at kilohertz frequencies. However, when Brown and Niblett [25] measured the internal friction of silver single crystals at kHz

frequencies, they found the peak occurred at approximately the same temperature as for polycrystalline specimens. Rather similar results were obtained for copper single crystals by Brown and Niblett [26] and for gold single crystal by Grandchamp [27]. This apparent absence of orientation dependence of the temperature of the Bordoni peak at kHz frequencies suggested the need for further measurements at MHz frequencies. The present work is an investigation of the ultrasonic attenuation at 5, 10 and 30 MHz, of silver single crystals of various orientation, after various amounts of deformation. Preliminary results have been reported elsewhere Zein and Alnaser [20].

## 2. Experimental procedure

Three specimens used in this investigation were high-purity 6N silver single crystals, supplied by Metal Research Ltd in the form of cylinders approximately 10 mm long and 12 mm in diameter. These crystals were deformed in a Monsanto tensometer at room temperature. The orientation of each crystal was checked after each deformation by the Laue back-reflection x-ray method; in each case the orientation was within  $3^\circ$  of the appropriate axis. The specimens were left for three days at room temperature after deformation, before the ultrasonic attenuation was measured. A conventional ultrasonic pulse technique was used, at frequencies of 5, 10 and 30 MHz. A short pulse of longitudinal wave, about  $1.5 \mu\text{s}$  in duration, was introduced into the specimen by means of an x-cut quartz crystal, and the same crystal acted as a receiver for reflected echoes. These were amplified and displayed on an oscilloscope. Nomaq stop cock grease was used as an acoustic bond between the transducer and the specimen. Measurements were made in the temperature range from 65 to 240 K. The temperature was measured by using two platinum sensors placed close to the specimen and connected to a digital thermometer. This is to assure temperature equilibrium, and also so that one of them will run in case of failure of the other during the experiment. The measurements were made with a rough linear warm-up rate of  $0.8 \text{ K min}^{-1}$ .

## 3. Results

The principle measurements were carried out on the three single crystals with orientations close to the  $\langle 110 \rangle$ ,  $\langle 111 \rangle$  and  $\langle 100 \rangle$  directions. The temperature dependence of the ultrasonic attenuation of each crystal was measured after each of a series of compressions for resolved shear stress, ranging up to  $60 \text{ N mm}^{-2}$ . The results of measurements of 10 MHz for the three crystals are shown in figures 1, 2 and 3.

All the measurements at 10 MHz show the Bordoni peak occurring in the range between 114 and 122 K. Figure 4 shows the variation in the temperature of the Bordoni peak with deformation for the three crystals at 10 MHz. This decrease is most marked for resolved shear stresses between 15 and  $28 \text{ N mm}^{-2}$ . The peak temperature then increases for stresses greater than  $28 \text{ N mm}^{-2}$ . The measurements reveal that the height of the Bordoni peak in the three specimens increased on increasing the amount of deformation, as shown in figure 5. Figures 6, 7 and 8 show the results of the measurements for the three crystals at 5, 10 and 30 MHz where the peak occurred at 107 K, 117 K and 127 K for the three orientations for a resolved shear stress of  $21 \text{ N mm}^{-2}$ . Table 1 summarizes the results for the three crystals.

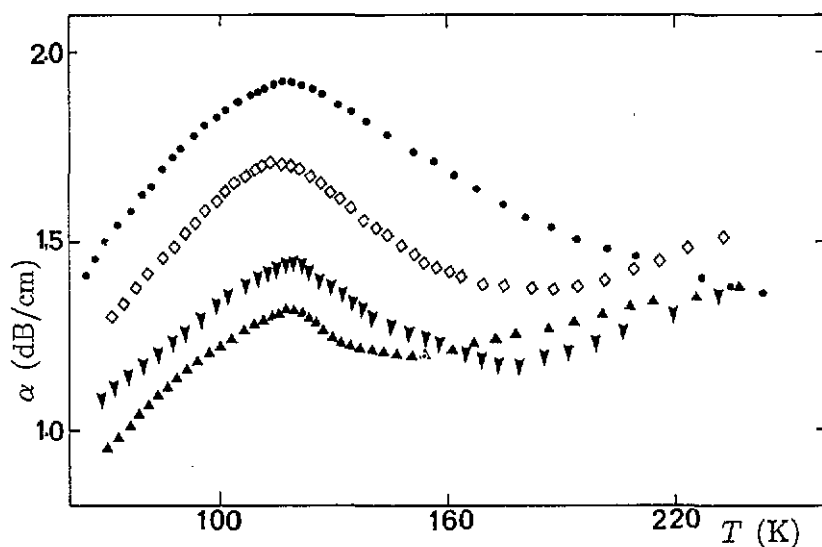


Figure 1. Ultrasonic attenuation of  $\langle 111 \rangle$  silver single crystal as a function of temperature: ▲,  $3 \text{ N mm}^{-2}$ ; ▼,  $15 \text{ N mm}^{-2}$ ; ◊,  $19 \text{ N mm}^{-2}$  and ●,  $50 \text{ N mm}^{-2}$ .

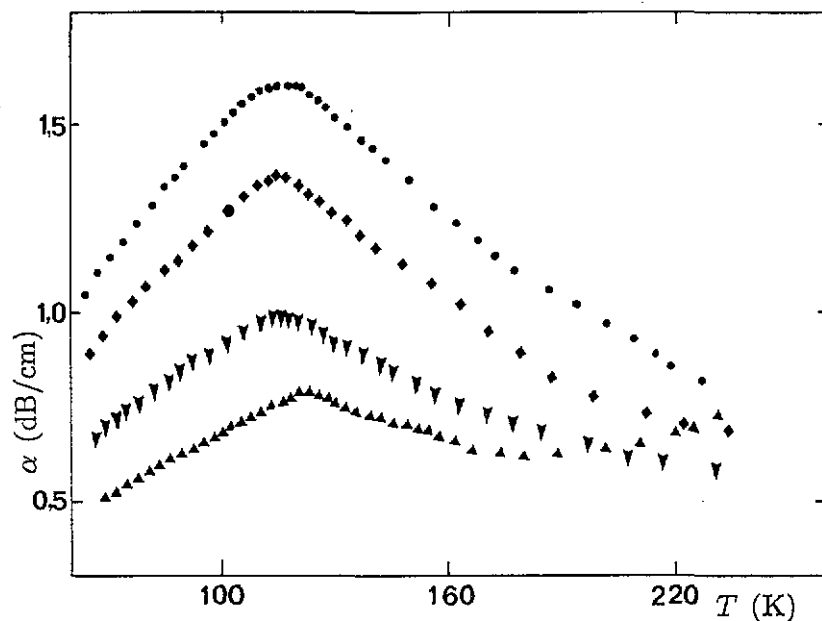


Figure 2. Ultrasonic attenuation of  $\langle 110 \rangle$  silver single crystal as a function of temperature: ▲,  $3 \text{ N mm}^{-2}$ ; ▼,  $15 \text{ N mm}^{-2}$ ; ◆,  $28 \text{ N mm}^{-2}$  and ●,  $42 \text{ N mm}^{-2}$ .

#### 4. Discussion

The results described here are consistent with those obtained by Brown and Niblett [25] for silver single crystals. In particular, the temperature of the Bordoni peak is essentially

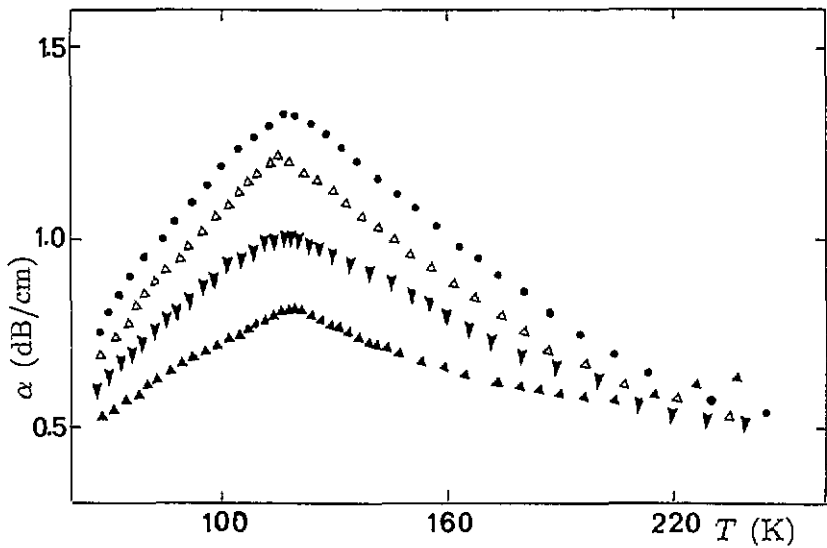


Figure 3. Ultrasonic attenuation of {100} silver single crystal as a function of temperature: ▲, 3 N mm<sup>-2</sup>; ▼, 11 N mm<sup>-2</sup>; △, 28 N mm<sup>-2</sup> and ●, 50 N mm<sup>-2</sup>.

Table 1. The variation of the Bordoni peak height and its temperature in three single crystals of silver with different crystallographic orientations, at frequency of 10 MHz.

| Resolved shear stress (N mm <sup>-2</sup> ) | Peak temperature (K) |       |       | Peak height (1/Q × 10 <sup>-4</sup> ) |        |       |
|---|----------------------|-------|-------|---------------------------------------|--------|-------|
|   | {111}                | {110} | {100} | {111}                                 | {110}  | {100} |
| 3   | 120                  | 122   | 119   | 3.14                                  | 3.58   | 3.24  |
| 6   | 119                  | —     | 118   | 3.5                                   | —      | 3.6   |
| 11  | 119                  | 119   | 117   | 3.68                                  | 4.63   | 4.93  |
| 15  | 118                  | 119   | 117   | 3.83                                  | 5.23   | 5.24  |
| 18  | 116                  | —     | 116   | 4.65                                  | —      | 5.43  |
| 19  | 115                  | 118   | —     | 5.2                                   | 5.89   | —     |
| 21  | 115                  | 117   | 115   | 5.7                                   | 6.27   | 5.89  |
| 24  | —                    | —     | 115   | —                                     | —      | 6.21  |
| 25  | —                    | 116   | —     | —                                     | 6.92   | —     |
| 28  | 114                  | 115   | 114   | 6.04                                  | 8.5    | 6.73  |
| 34  | 116                  | 117   | 117   | 6.15                                  | 8.964  | 7.03  |
| 42  | 118                  | 119   | 117   | 6.76                                  | 9.41   | 7.32  |
| 50  | 119                  | 119   | 118   | 7.02                                  | 10.3   | 7.57  |
| 60  | 119                  | 120   | 118   | 7.25                                  | 10.896 | 7.66  |

independent of the orientation of the crystal. Figure 9, which is an Arrhenius plot of the frequency against the reciprocal of the absolute temperature at which the peak occurs, shows that the results of the present work fit well with the measurements of Brown and Niblett [25], for specimens has given similar mechanical treatment. Our results are in full agreement with the results obtained by Besson *et al* [28] in silver specimens.

This observation leads us to conclude that the temperature at which the Bordoni peak occurs is essentially independent of the orientation of the crystal. This is in a marked contrast to the results obtained by Mongy *et al* [21], which show a high degree of anisotropy. The differences between our results and those obtained by Mongy *et al* are much too great to

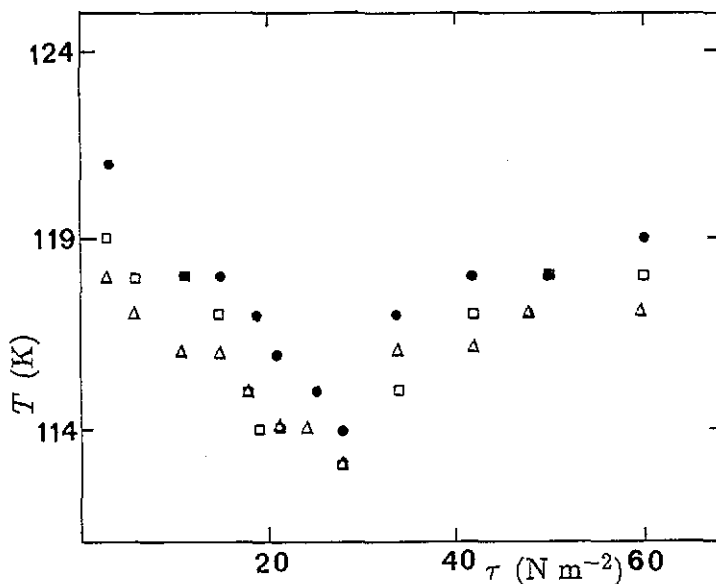


Figure 4. Variation of the Bordoni peak temperature for the three silver single crystals with resolved shear stress:  $\Delta$ ,  $\langle 110 \rangle$ ;  $\square$ ,  $\langle 111 \rangle$  and  $\bullet$ ,  $\langle 100 \rangle$ .

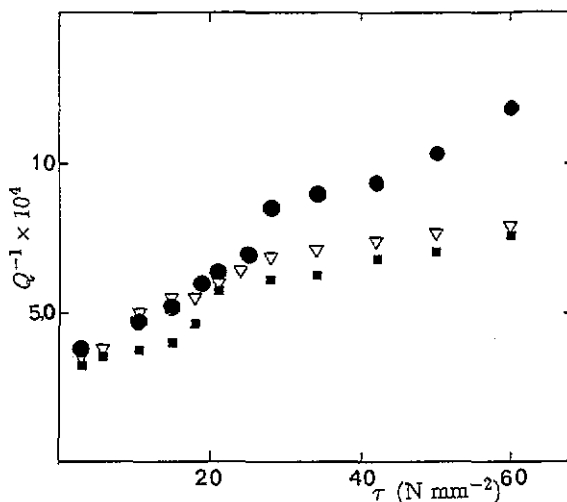


Figure 5. Variation of the Bordoni peak height for the three silver single crystals with resolved shear stress:  $\bullet$ ,  $\langle 110 \rangle$ ;  $\nabla$ ,  $\langle 100 \rangle$  and  $\blacksquare$ ,  $\langle 111 \rangle$ .

be explained by error in the measurements of temperature, e.g., there is a discrepancy of more than  $65^\circ$  in the case of the  $\langle 110 \rangle$  orientation at 10 MHz. It is difficult to explain the discrepancy between these results and those obtained by Mongy *et al* [21].

Not enough work has been done on silver single crystals at megahertz frequencies for one to be able to resolve the contradiction between our observations and those of Mongy *et al*. However, in comparison with extensive results obtained from studying the Bordoni

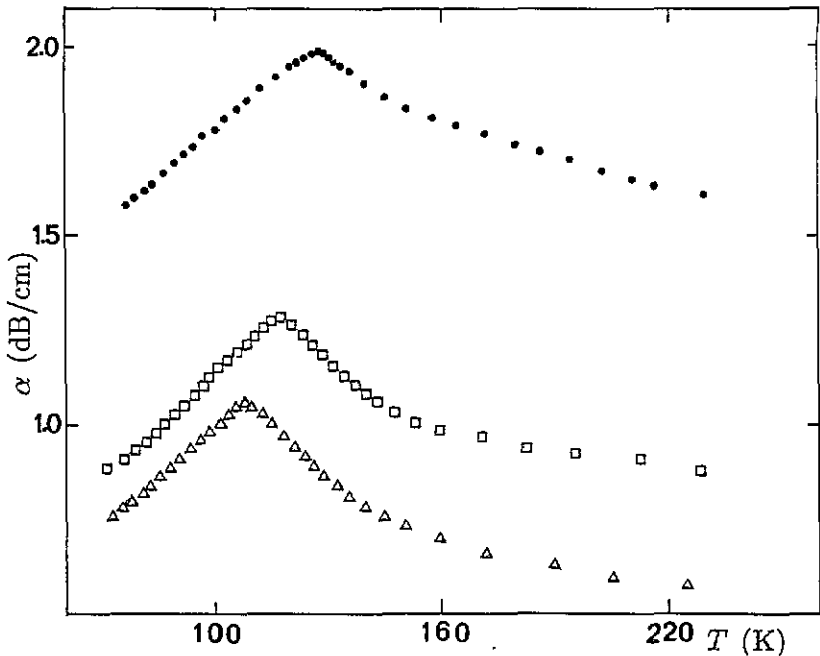


Figure 6. The ultrasonic attenuation of the  $\{110\}$  silver single crystal as a function of temperature:  $\Delta$ , 5 MHz;  $\square$ , 10 MHz and  $\bullet$ , 30 MHz.

peak in copper single crystals (Niblett and Zein [17], Alers and Thompson [29], Kayano, Kamigaki and Koda [30], Brown and Niblett [26]), and in aluminium single crystals [18, 20] one can believe that our conclusion is valid i.e. the dislocation relaxation in FCC is orientation independent. One possible source of the differences between the results of Mongy's work and ours is that we made our measurements on three different crystals of three different orientations, whereas Mongy *et al* [21] measured the attenuation in three directions on the same, specially shaped crystal. The Bordoni peak in silver reported by Mongy *et al* [21] occurred at the same temperature as in copper. Mongy *et al* [24], for the  $\{111\}$  orientation at a frequency of 10 MHz, in contrast with the findings of other workers reported in the review by Fantozzi *et al* [7]. It was clearly concluded by Fantozzi *et al* [7] that the temperature of the peak (and hence the activation energy) varies from one material to the other. The theory of the dislocation relaxation mechanism (Bordoni peak), which was first reported by Seeger [8] suggested that the main peak is caused by a thermally activated relaxation process involving a dislocation lying parallel to a close-packed direction (i.e. screw dislocation) in the crystal lattice. Dislocations that are oriented  $60^\circ$  to the close-packed direction produce another subsidiary peak at a lower temperature than the main peak i.e. the Niblett-Wilks peak (which was not clearly shown here). According to the latest published work by Seeger [31], the assignment of various types of dislocations to the damping appears arbitrary and not always supported by the experimental evidence. Dislocations that are not parallel to the close-packed direction will contribute to the background damping. In fact, the lowest background is found in the specimen of  $\{100\}$  orientation, while the highest background damping was found in the specimen of  $\{111\}$  orientation. The activation energy,  $w$ , and the attempt frequency,  $f$ , in the present work were calculated from the Arrhenius plot (figure 9) and found to be equal to 0.105 eV and  $3.8 \times 10^{11}$  Hz respectively. This value is

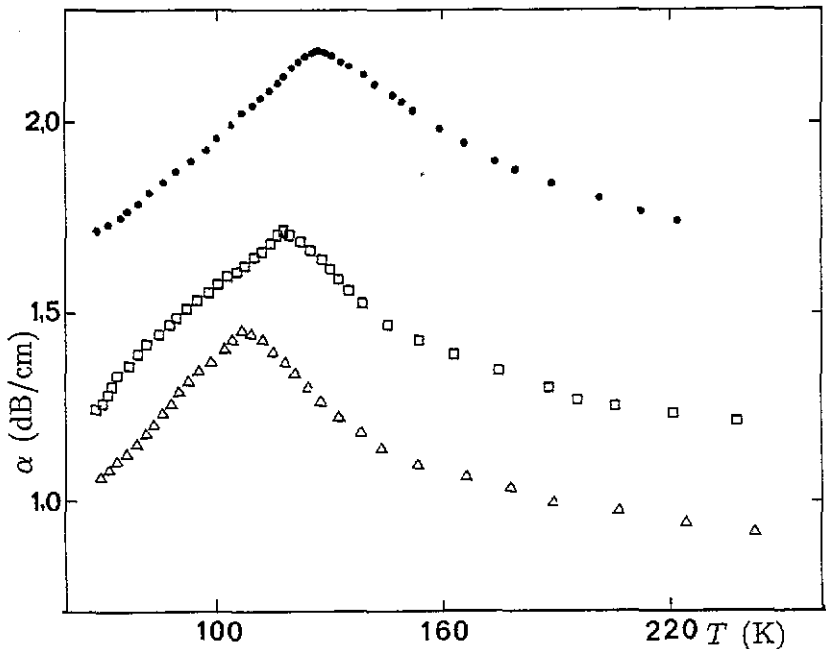


Figure 7. The ultrasonic attenuation of the  $\langle 111 \rangle$  silver single crystal as a function of temperature:  $\Delta$ , 5 MHz;  $\square$ , 10 MHz and  $\bullet$ , 30 MHz.

very consistent with that listed in the review by Fantozzi *et al* [7].

Previous workers have reported small shifts in the temperature of the Bordoni peak due to different amounts of plastic deformation. This observation has been studied in detail. We found that the Bordoni peak occurs at 122 K for lightly deformed crystals. This temperature decreased to about 114 K as the resolved shear stress was increased to about  $28 \text{ N mm}^{-2}$ . Further deformation caused the temperature of the peak to increase to about 119 K as shown in figure 4. The variation of the peak temperature with the resolved shear stress is in quantitative agreement with Seeger's theory as modified by Paré [9] and Engelke [12] to take into account the different internal stresses and dislocation loop lengths present at different deformations. The higher peak temperature observed in the lightly deformed crystals is believed to be due to longer dislocation loops occurring at these deformations, while the increase in the peak temperature at high deformation is attributed to a reduction in the internal stresses, caused by the formation of a cell structure, in which the majority of the dislocations are located in the cell walls.

In terms of the variation of the Bordoni peak height with deformation, the Bordoni peak increases monotonically with the flow of stresses through stage II of the plastic deformation as shown in figure 5. This is also supported by the kink-pair generation theory proposed by Seeger, which predicted an increase of the Bordoni peak height with increasing plastic deformation, unless some process intervenes to change the course of plastic deformation. This point means that the Bordoni peak height does not continue to increase indefinitely with increasing cold-work. Dislocation density and loop length are two factors which have been considered to determine the Bordoni peak height, a fact which is accepted by the pair-of-kink theories. An increase of the Bordoni peak height with deformation was studied in copper single crystals by Zein [32] and in aluminium single crystals by Zein and Alnaser [19].



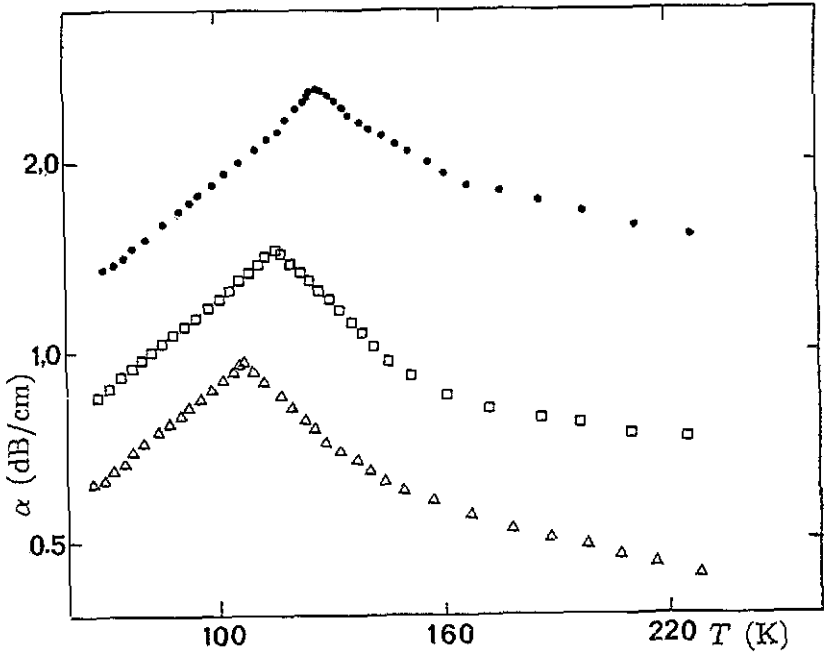


Figure 8. The ultrasonic attenuation of the {100} silver single crystal as a function of temperature:  $\Delta$ , 5 MHz;  $\square$ , 10 MHz and  $\bullet$ , 30 MHz.

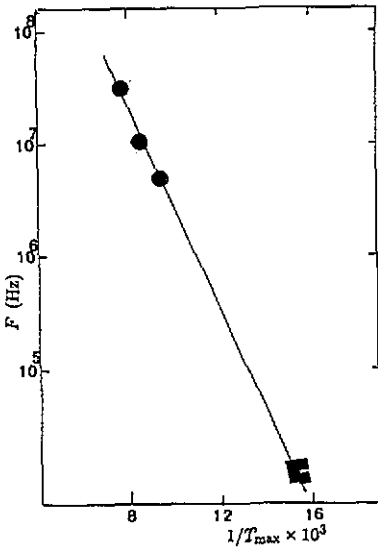


Figure 9. Frequency dependence of the temperature of the dislocation relaxation peak for the three silver crystals {100}, {110} and {111};  $\blacksquare$ , Brown and Niblett;  $\bullet$ , present work.



Figure 10. The relation between the Bordoni peak height and the frequency of measurement.

A further interesting observation is that the relaxation strength decreases as the frequency increases. This is in agreement with Seeger's modified theory Fantozzi *et al* [7] and Engelke [12]. Esnouf and Fantozzi [14] applied rate theory to kink-pair relaxation for the many-valley periodic potential. For the case of heavily deformed specimens, their numerical calculations showed that between 1 Hz and  $10^4$  Hz, the relaxation strength,  $\delta$ , of the Bordoni peak decreases with frequency according to the relations

$$\delta = \delta_1(1 - 0.04)w(f)$$

where  $\delta$  is the peak height ( $\delta = Q^{-1}\pi$ ) at frequency of 1 Hz. This equation seems to be valid for high frequencies, as illustrated in figure 10.

## 5. Conclusions

(i) The temperature of the Bordoni peak in silver single crystal is essentially independent of the orientation.

(ii) The temperature of the Bordoni peak in silver single crystals decreases as the prior deformation is increased up to a resolved shear stress near  $28 \text{ N mm}^{-2}$ , and then increases slightly for higher prior stresses.

(iii) The peak height increases with increasing amount of deformation.

(iv) The peak height decreases as the frequency increases.

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