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## Unstable Internal Friction in Bismuth and Antimony Single Crystals

In the temperature range of 6 to 300 K an effect of the high amplitude ultrasound waves on amplitude dependences of decrement and modulus defects in the antimony and bismuth single crystals of different orientation is studied. The character of changes of the decrement and modulus defect amplitude dependences under the influence of the high amplitude ultrasound indicates that dislocation multiplication, obstacle break-through by dislocation pile-ups or deformation twinning may be observed in crystals depending on the ultrasound propagation direction and the temperature interval investigated.

В интервале температур 6—300 К изучено воздействие ультразвуковых волн высокой амплитуды на амплитудные зависимости декремента колебаний и дефекта модуля в монокристаллах сурьмы и висмута различной ориентации. Характер изменения амплитудных зависимостей декремента и дефекта модуля под воздействием высокоамплитудного ультразвука показывает, что в кристаллах может наблюдаться размножение дислокаций, прорыв дислокационных скоплений или механическое двойникование, в зависимости от направления распространения ультразвука и исследуемого интервала температур.

### 1. Introduction

The amplitude-dependent internal friction is a source of information on the dynamic behaviour of crystal lattice defects. Significant data may be obtained in particular from a study of the unstable interval friction in the relatively high vibration amplitude range (KRISHNAN et al.; PAL-VAL et al.; PAL-VAL, PLATKOV; VOINOVA et al.). The phenomenon is an involved one, but its essence is that an external impact causes the internal friction to change in time. There are several ideas as regards the mechanisms of the unstable internal friction, some of them incompatible (CHELNOKOV, KURPIN; LANDAU; SUPRUN). Further progress in understanding the phenomenon is however handicapped by deficiency in experimental evidences covering the wide temperature and vibration amplitude ranges, in anisotropic semimetal single crystals in particular. The effort reported here was designed to overcome the difficulty.

### 2. Experimental

For the experiment we used bismuth and antimony single crystals grown by the Bridgman method in a split graphite mould in argon atmosphere from 99.995% pure antimony and 99.9999% pure bismuth. The bismuth samples had the same orientation. The sample axes coincided with the crystallographic direction [111] in the face-centred rhombohedral unit cell, the angle between the axis of the specimens and the "basal" plane (111) was close to 90°.

The antimony samples had two orientations. In samples with orientation I the longitudinal sample axis coincided with the crystallographic direction [100] with the angle between the axis and the basal plane (111) being near  $56^\circ$ . In samples of orientation II the axis was roughly parallel to [110] direction in a rhombohedral primitive cell, with the axis — basal plane angle being  $3^\circ$ . Note that in case of a stress applied along the sample longitudinal axis, orientation I was favourable to slip in the basal plane, while orientation II to twinning.

Impurities in samples are listed in Table 1. The integral purity was checked by the electric resistance ratio  $R_{300}/R_{4.2}$ .

The internal friction and the modulus defect were measured by the composite oscillator technique with the longitudinal vibration frequency of 88 kHz in the temperature range between 6 and 300 K. Temperature stabilisation was no worse than to within  $10^{-4}$  (PALVAL et al. 1981).

Table 1

		Bi	Sb — I	Sb — II
Impurities wt %	Cd	1.0	0.0001	0.0001
	Bi	98.99	0.1	0.001
	Fe	—	0.0001	0.005
	Pb	0.005	—	—
	Mg	0.002	0.0001	0.0001
	Cu	0.0001	0.0001	0.0001
	Ag	0.0001	—	—
$R_{300}/R_{4.2}$		34	150	900

### 3. Results and discussion

We measured the dependences of the decrement and the modulus defect  $\Delta M/M = 2(f_{s2} - f_{s1})/f_{s1}$ , (where  $f_{s1}$  and  $f_{s2}$  are the resonant sample frequencies measured for the minimum and given strain amplitude  $\epsilon_0$ ). The measurements were carried out at constant temperature during  $\epsilon_0$  increase (direct run of the amplitude dependence measurement) and then decrease (reverse run). Figure 1 and 2 show the results

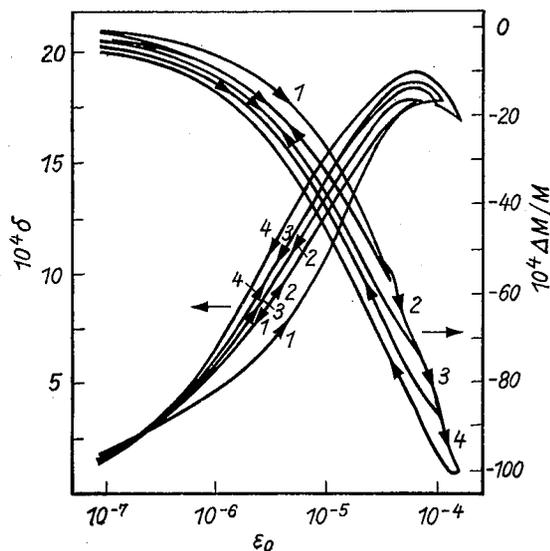


Fig. 1. The amplitude dependences of decrement (left axis) and modulus defect (right axis) in bismuth at 6 K. The figures indicate numbers of the successive measurement runs at which the maximum measurement amplitude is gradually increased

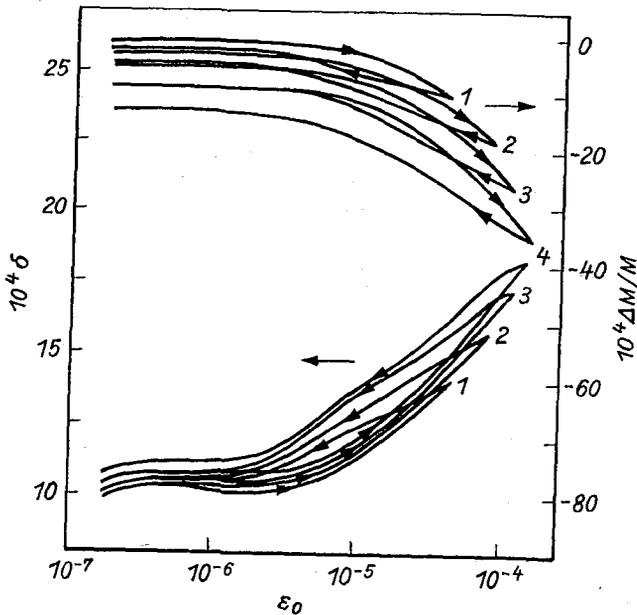


Fig. 2. The amplitude dependences of  $\delta$  and  $\Delta M/M$  in bismuth at 300 K

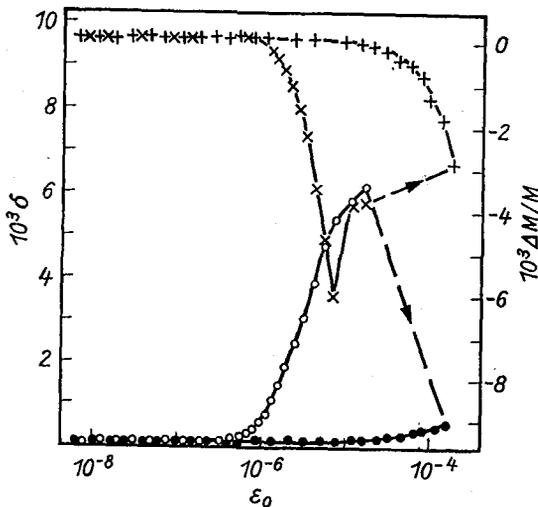


Fig. 3. The dependences  $\delta(\epsilon_0)$  and  $\Delta M/M(\epsilon_0)$  in the antimony single crystals of the orientation I at 300 K. O,  $\times$  - direct runs,  $\bullet$ ,  $+$  - reverse runs

measured in bismuth for 6 and 300 K. The maximum amplitude  $\epsilon_{0max}$  in the end of the direct run was gradually increased. Over the whole temperature range the direct and reverse branches for measurements of bismuth decrement and modulus defect did not coincide, as hysteresis loops. As  $\epsilon_{0max}$  increased, the total hysteresis loop area was growing, the decrement and the modulus defect in the reverse runs being larger than these in the direct runs. In the low temperature range, in the amplitude range used, the amplitude-dependent internal friction attained the maximum at high  $\epsilon_0$ , whereas

the dependence of the modulus defect on  $\varepsilon_0$  displayed only a slight variation of the  $\Delta M/M(\varepsilon_0)$  curve slope.

The amplitude-dependent internal friction and modulus defect of the antimony single crystals were qualitatively different in comparison with the bismuth single crystals. For relatively small  $\varepsilon_0$  the internal friction and modulus defect reverse runs coincided with the direct runs. It was, however, found that in some temperature ranges, increasing amplitude led to the rapid jumplike change of the decrement and modulus defect to magnitudes, which were not subsequently reached during the direct run, even with the same  $\varepsilon_0$  (Figs 3 and 4). Such unstable internal friction behaviour has not been previously reported. As follows from Figures 3 and 4, there was a qualitative difference in the jumps observed in crystals of different orientations. In samples with orientation I, i.e. that favorable to slip, the decrement, on having reached the amplitudes of  $10^{-5}$ , decreased jumplike (Fig. 3). After that the critical amplitude  $\varepsilon_{0c}$  of the dependence of the decrement on  $\varepsilon_0$  rose more than an order of magnitude, while the amplitude dependence proper was little pronounced. In the direct measurement run, the modulus defect,  $\Delta M/M$  was very closed indeed to the amplitude-dependent part of the decrement,  $\delta$ . In the vicinity of the jumplike internal friction decrease amplitude the modulus defect also changed, though without the strict correspondence to the decrement jump. A certain value of  $\Delta M/M$ , having been reached the modulus defect sharply dropped, and then, at the amplitude of the preceding decrement jump, there was a second and smaller decrease in the modulus defect. After the jump the modulus defect values were larger by an order of magnitude. These measurements were made at 300 K. While during the preceding measurements, at 300 K and below,

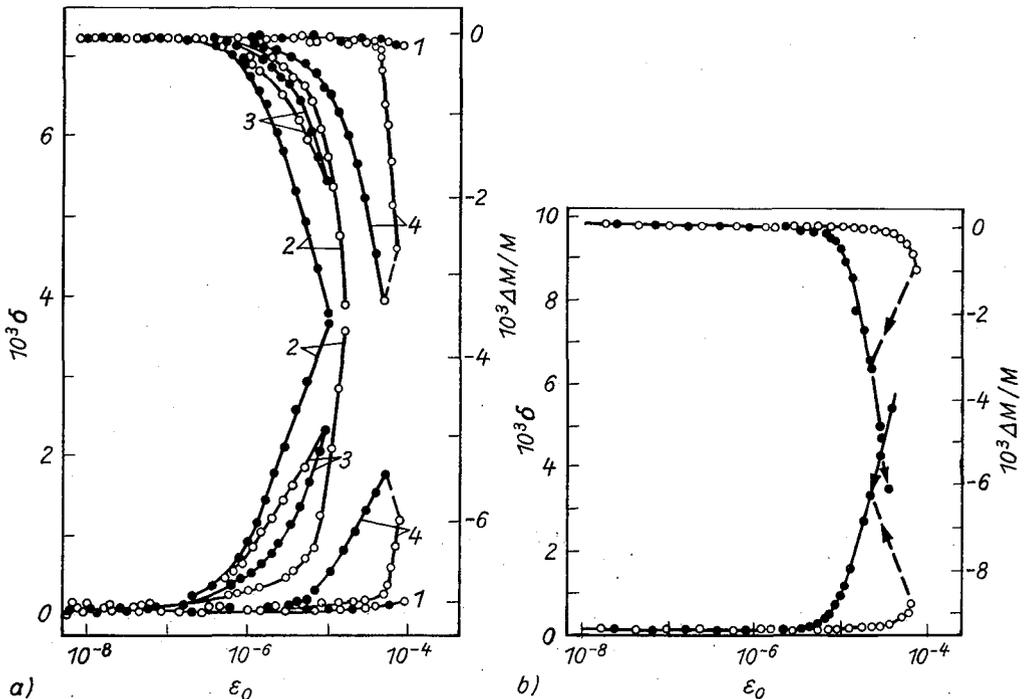


Fig. 4. The dependences  $\delta(\varepsilon_0)$  and  $\Delta M/M(\varepsilon_0)$  in the antimony single crystals of the orientation II. a) 1 — the direct (○) and reverse (●) runs at 300 K; 2 — the first measurement of  $\delta(\varepsilon_0)$  and  $\Delta M/M(\varepsilon_0)$  at 78 K; 3 — The hysteresis of the amplitude dependences at 130 K; 4 — the second measurement of  $\delta(\varepsilon_0)$  and  $\Delta M/M(\varepsilon_0)$  at 78 K. b) The amplitude dependences measured at 6.5 K

the amplitude  $\varepsilon_0$  was restricted to values at which the jump was not realized, the jump and the behaviour of the decrement and the modulus defect similar to that described above were, on the contrary, observed also at 6 K. Note that it was only once that the jump could be provoked in samples of this orientation.

In samples of orientation II at 300 K the direct and reverse measurement was yielded coincident amplitude dependences of the internal friction and the modulus defect (Fig. 4a). At the temperatures 78 K and below, with the amplitude increasing to  $\varepsilon_0 = 10^{-5}$ , there was a jump both the quantities,  $\delta$  and  $\Delta M/M$ . As a result, the decrement and the modulus defect increased, and the reverse branches of the amplitude dependences were markedly displaced to the smaller magnitudes of  $\varepsilon_0$ . The modulus defect and the decrement measured at the same amplitude appeared to be equal or nearly equal, both during the direct run and in the reverse run following the jump. During sample warming at a fixed temperature the direct and reverse branches did not coincide, the reverse branches displaced towards higher  $\varepsilon_0$ , so that there formed a closed loop (curves 3 in Fig. 4a).

After heating up the sample and holding it at 300 K the measurements were repeated at 78 K. The amplitude dependences of the internal friction and the modulus defect were in this case markedly displaced towards higher values  $\varepsilon_0$  than in the preceding measurements. At large amplitudes thereis again a jump. The amplitude at which it was observed this time was larger than during the first measurement by a factor of about 5.

The similar jumps of the quantities could be seen also below 78 K. Figure 4b presents results measured at 6.5 K. The jump and the alteration of the amplitude dependences look quite like those observed at 78 K.

The changes in the amplitude dependences of the internal friction and the modulus defect in the antimony single crystals of orientation I evidence that the total dislocation mobility are after the jump much smaller than before. We can propose the following mechanisms. Dislocations in a crystal make up pile-ups and are exposed to a complex field of internal stresses. Excitation of vibrations with large  $\varepsilon_0$  favours breakthrough of obstacles by dislocation pile-ups. As this takes place, the internal stresses are relaxed, dislocations stop where pinning is stronger. That leads to the increase of the critical amplitude of the amplitude dependence beginning as well as to a considerable decrease of the amplitude-dependent losses.

At the high-amplitude ultrasound excitation in the antimony samples of the orientation II a generation of unelastic and elastic twins takes place in crystals. The appearance of numbers of the twinning dislocations leads to the growth of the decrement and the modulus defect. The displacement of the amplitude dependences towards larger  $\varepsilon_0$  with growing temperature is due to blocking of the twinning dislocations of the fresh twins by impurity atoms.

As has been mentioned, the noncoincidence of the direct and reverse branches of the measured amplitude dependences of the internal friction and the modulus defect may be due to more than one reason. However the bismuth hysteresis loops that were not closed suggests that exposure to high amplitude ultrasound excitation, gave rise to dislocation multiplication. Increasing dislocation density should also result in that the internal friction and the modulus defect are in the reverse run larger than in the direct run, as was observed in the experiment. This mechanism is also supported by the fact that the total hysteresis loop area increases with the maximum amplitude in the end of the hysteresis loop measurement direct run.

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