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Ultrasonic Treatment of GaP and GaAs

By

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Mobile dislocations are introduced into samples of GaP:S and GaAs:Zn by uniaxial compression (ε_{pl} = 1 to 5%) at 820 K. As TEM investigation shows, the resulting irregular dislocation network shows a tendency to rearrangement after a long term ($N \approx 7 \times 10^8$) ultrasonic treatment at a frequency of 100 kHz and a temperature between 400 and 600 K. The mechanical damping and modulus defect are in good agreement with the Granato-Lücke theory. Under fatigue conditions applied here, the dislocation structure can be changed by comparably low ultrasound stresses ($\sigma \approx$ 25 to 56 MPa) at low temperatures.

In Proben aus GaP:S und GaAs:Zn werden durch einachsige Kompression (ε_{pl} = 1 bis 5%) bei 820 K bewegliche Versetzungen erzeugt. Elektronenmikroskopische Untersuchungen zeigen, daß das entstehende unregelmäßige Versetzungsnetzwerk nach längerer ($N \approx 7 \times 10^8$) Ultraschallbehandlung mit einer Frequenz von 100 kHz bei Temperaturen von 400 bis 600 K eine Tendenz zur Umordnung zeigt. Die mechanische Dämpfung und der Moduldefekt stimmen gut mit der Granato-Lücke-Theorie überein. Mit der hier angewendeten Ermüdungsmethode kann die Versetzungsstruktur bei relativ geringen Spannungen des Ultraschalls ($\sigma \approx$ 25 bis 56 MPa) verändert werden.

1. Introduction

Predeformation of $A^{III}B^{V}$ compounds at relatively high temperatures is known to be a good means to introduce mobile dislocations into a crystal. If the dislocation content is sufficiently high, plastic deformation afterwards is possible at relatively low temperatures.

Cyclic deformation permits a fatigue study as well as the measurement of material properties by internal friction and modulus defect. In materials of high dislocation mobility plastic deformation can be enforced by application of ultrasound of high amplitude only [1, 2]. In an earlier investigation, some of the authors studied propagation of cracks resulting from stresses $\sigma > 30$ MPa during ultrasonic treatment of as-grown GaP:S crystals at room temperature [3]. In this study, we tried to avoid fracture by the introduction of mobile dislocations and by slightly higher temperatures during ultrasound treatment.

2. Experimental

Predeformed samples cut with a wire saw from LEC GaAs:Zn crystals (carrier density $n_0 = 5 \times 10^{18}$ cm^{-3}) or from LEC GaP:S crystals ($n_0 = 3 \times 10^{17}$ cm^{-3}) were mechanically polished. The cross section of samples was $\approx 2.5 \times 2.5$ mm^2 for the side faces (110) and (001). The length was matched to 1/2 wavelength of ultrasound (105 kHz) in the material

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in the appropriate direction of sample axis [$\bar{1}$10] (GaP: 28 mm; GaAs: 22.5 mm). Predeformation was carried out in an argon atmosphere of 0.1 MPa by uniaxial compression at 820 K at plastic deformation rates $\varepsilon_{pl} = 1$ to 5%. In any case, predeformation was stopped after passing the lower yield point.

With an experimental equipment very similar to that described earlier [3], an ultrasonic treatment was performed. For this purpose, samples were cemented to a $-18.5°$ X-cut quartz crystal. Gold or chromium layers on X faces served as electrodes. With a fm generator and amplifier (ENI 1040L) driving voltages up to 60 V could be applied to the quartz electrodes, yielding amplitudes of vibration in the samples up to $A_0 \approx 5 \times 10^{-4}$ and normal stresses up to $\sigma \approx 60$ MPa, if the oscillator was kept in resonance. Such stresses often lead to fracture of undeformed samples, whereas predeformed samples normally did not break. The stress by ultrasound was parallel to the stress by predeformation.

The ultrasonic treatment was performed in argon (0.1 MPa) at temperatures ranging from 300 to 600 K. The amplitude of vibration could be obtained from measuring the electric current I_r flowing through the quartz crystal [4]. The treatment by ultrasound was performed by stepwise increasing the driving voltage to a maximum value, leaving up to 2 h time to reach saturation of the plastic strain amplitude. At frequencies of about 10^5 Hz, this means as much as 7×10^8 cycles. At each step, the damping loss/cycle was determined by letting the quartz–sample unit oscillate at resonance frequency f_r and antiresonance frequency f_a and by measuring appropriate currents I_r or I_a [4],

$$Q^{-1} = \frac{2(f_a - f_r)}{f_r} \sqrt{\frac{I_a}{I_r}}\,. \tag{1}$$

By standard cutting and thinning techniques, thin foils for TEM investigations were prepared. Investigations were carried out with {111} foils in electron microscopes operating at 90 and 1000 kV.

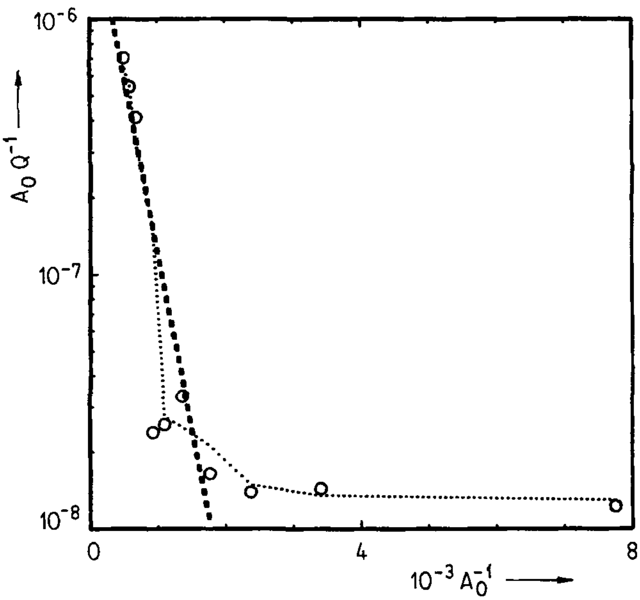

Fig. 1. Granato-Lücke plot of predeformed GaP during ultrasonic treatment at 560 K

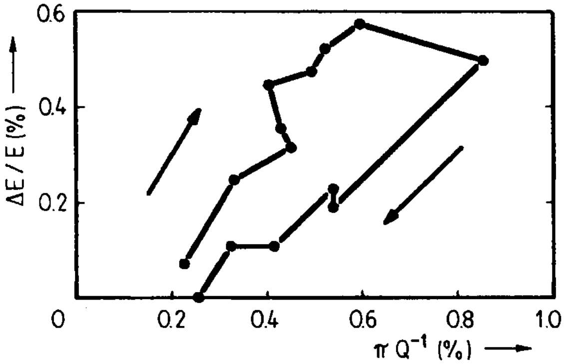

Fig. 2. Relative Young modulus defect vs. damping of predeformed GaP · $T = 560$ K, $\sigma_{max} = 34$ MPa, $\varepsilon_{max} = 2.4 \times 10^{-4}$

3. Results

Theory predicts, that oscillating dislocation segments should increase the modulus defect $\Delta E/E$ as well as the mechanical damping Q^{-1} of the sample, when the amplitude of vibration A_0 rises [5, 6]. Linear plots of lg $(A_0 Q^{-1})$ versus A_0^{-1}, obtained during ultrasonic treatment

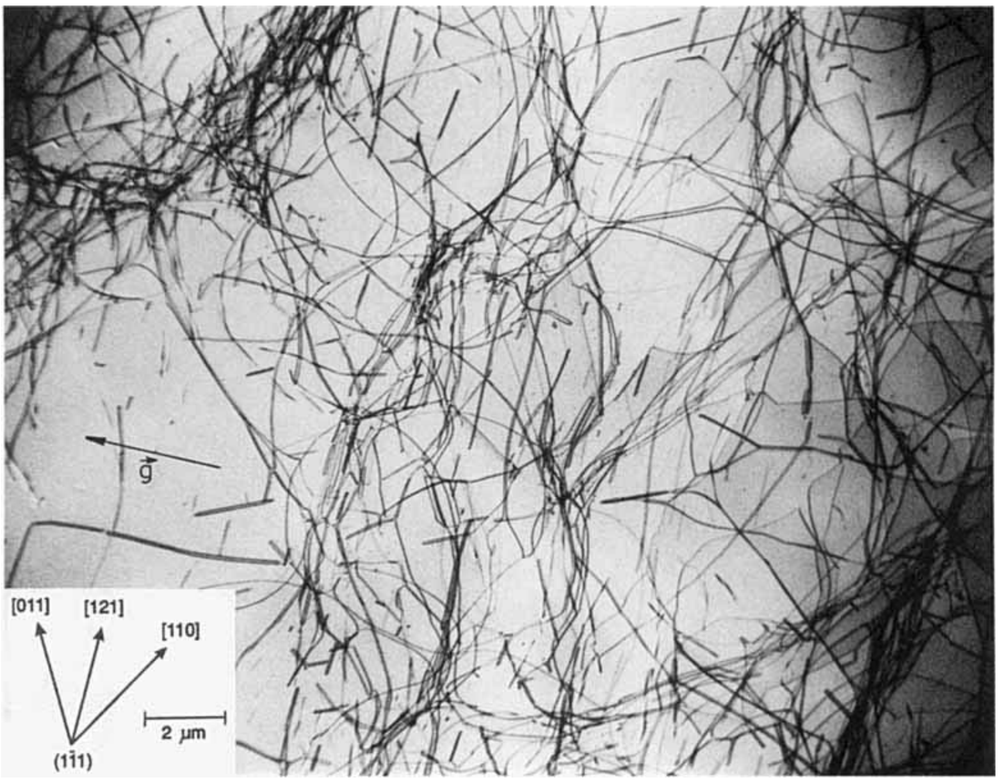

Fig. 3. Irregular dislocation network in p-GaAs after predeformation at 820 K

Table 1

Quantitative analysis of dislocation line directions in predeformed p-GaAs before and after ultrasonic treatment

	line direction $\langle 112 \rangle$	line direction $\langle 110 \rangle$
predeformed sample ($\varepsilon = 5\%$)	$(63 \pm 3)\%$	$(37 \pm 3)\%$
after ultrasonic treatment (470 K, $\tau = 12$ MPa)	$(52 \pm 2)\%$	$(48 \pm 2)\%$

of predeformed samples, show that the amplitude dependent internal friction is mainly due to a dislocation mechanism (Fig. 1). If the amplitude A_0 is raised to some maximum value of 2×10^{-4}, hysteresis occurs.

Hysteresis can be seen more clearly, if the modulus defect $\Delta E/E$ is plotted versus mechanical damping Q^{-1} (Fig. 2). According to the Granato-Lücke theory,

$$\frac{\Delta E}{E} \approx \pi Q^{-1} . \tag{2}$$

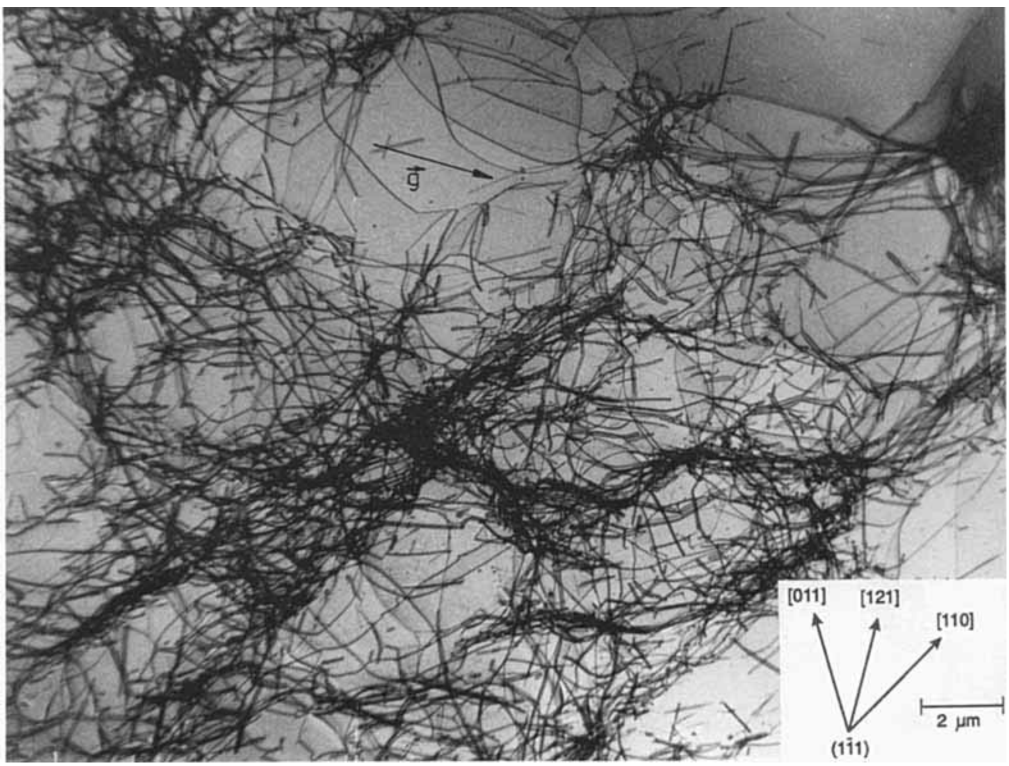

Fig. 4. Elongated cell structure of dislocations after subsequent ultrasonic treatment at 470 K and $\sigma = 30$ MPa

Indeed, the slope of the plot in Fig. 2 is nearly unity. The curve does not extrapolate to the origin, as amplitude independent damping occurs as well. At higher amplitudes, hysteresis occurs as there is a sudden increase of Q^{-1} for nearly constant $\Delta E/E$. However, when the amplitude of vibration is lowered subsequently, Q^{-1} drops nearly to the initial value.

TEM investigations after predeformation at equal conditions of predeformation show that the ratio of dislocation segments with line direction parallel to $\langle 110 \rangle$ or $\langle 112 \rangle$ in the glide plane is strongly dependent on the plastic deformation rate ε_{pl}. At low ε_{pl}, 60° dislocations with line segments along $\langle 110 \rangle$ predominate, whereas at $\varepsilon_{pl} > 3\%$ dislocations with line segments parallel to $\langle 112 \rangle$ are slightly in the majority. Fig. 3 shows an example of an irregular dislocation network, which is typical of large predeformation ($\varepsilon_{pl} = 5\%$). $\langle 112 \rangle$ dislocation line directions are slightly predominant. After ultrasonic treatment of this sample at 470 K by glide stress $\tau = 12$ MPa, a tendency to rearrangement of the dislocation line segments from $\langle 112 \rangle$ to $\langle 110 \rangle$ can be observed (Fig. 4, Table 1).

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