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M.G. Tabache, E.D. Bourret,
and A.G. Elliot

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Lawrence Berkeley Laboratory
University of California
Berkeley, California 94720

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Measurements of the critical resolved shear stress for indium doped and undoped GaAs single crystals

M. G. Tabache and E. D. Bourret

Center for Advanced Materials, Lawrence Berkeley Laboratory, University of California, Berkeley, CA 94720

A. G. Elliot

Optoelectronics Division, Hewlett-Packard Corporation, San Jose, CA 95131

ABSTRACT

Direct measurements of the critical resolved shear stress of undoped- and indium-doped GaAs single crystals at high temperatures have been performed using dynamical compression tests. At the melting point the critical resolved shear stress of GaAs:In is only twice that of undoped GaAs. At low temperatures, the activation energy for the motion of dislocations is not affected by indium doping. From these findings we conclude that crystallographic glide is not the only cause of dislocation formation in these GaAs crystals and solution hardening is not responsible for the reduction in dislocation density in GaAs:In.

It has been suggested that dislocations in GaAs originate from crystallographic glide induced by excessive thermal stress associated with crystal growth.[1-3] Critical resolved shear stress (CRSS) values at the melting point on which these theories are based were estimated, by extrapolation of measurements at low temperatures, and vary in a broad range (from 7 to 60g mm^{-2})[3,4] for undoped GaAs crystals. It has also been found that dislocation densities can be drastically reduced by intentional additions of dopants, in particular, indium.[5] However, the effects of isoelectronic dopants on the CRSS of GaAs have not been experimentally studied. It was suggested that doping with In increases the CRSS of the crystals. Duseaux and Jacob[6] were the first to report experimental results from crystals grown under known thermal stresses. They were able, in some instances, to grow undoped and doped GaAs crystals with low dislocation densities under stresses 30 to 200 times higher than the published[3,4] estimated values of the CRSS. These results point to an important need for precise values of the CRSS at the melting point for doped and undoped GaAs crystals in order to understand the mechanisms for dislocation formation in GaAs and the role played by dopants.

This letter reports the first direct experimental measurements of the CRSS at high temperatures (350°C to 1100°C) for undoped and indium doped GaAs single crystals. The crystals used in this study were grown by the liquid encapsulated Czochralski technique. All were grown under identical conditions in order to minimize differences in native defect concentration, stoichiometry, and residual stress that might influence the deformation behavior. The characteristics of the crystals used (including dopant concentration, average electrical resistivity and etch pit density) are shown in Table I.

The specimens for the deformation tests were cut into prisms with square bases. The long axis oriented along the <321> direction was used as the compression axis so that deformation occurs by glide in a single slip system, in this case $(1\bar{1}1)$ $\langle 10\bar{1}\rangle$. The Schmid factor for this slip system is $\mu = 0.4667$. The samples were carefully checked for perfect polish of the side faces and parallelism of the end faces, both being important factors for the uniformity of the deformation.[7] A schematic of the experimental setup is shown in Figure 1. The fixtures were built to fit a universal testing machine (Instron 1122). For $T < 800°C$, the sample rests directly on the fixed Al_2O_3 rod. For $T > 800°C$, the sample is placed in an Al_2O_3 crucible and held strain-free in the upright position by a boron nitride ring. Sample and ring were then encapsulated with B_2O_3 to prevent As loss. The use of liquid B_2O_3 with a high hydroxyl content (1200 ppm), which lowers its viscosity, was found to have no effect on the deformation tests. A nominal strain rate of $\dot{\varepsilon} = 10^{-4}s^{-1}$ was used for all tests.

The experimental curves, obtained as load on the sample versus time, were transformed into resolved shear stress versus percentage glide strain. Figure 2 represents typical stress-strain curves for GaAs:In tested at different temperatures. A yield elongation stage is sometimes observed at the lower yield point which is attributed to inhomogeneous slip.[8] Values of strains at the yield points decrease with increasing temperatures. The lower yield point of the stress-strain curves obtained under dynamical compression was chosen as a valid estimate of the CRSS for slip.[3,9,10] An expression for the lower yield stress ($\tau_{\ell y}$) has been derived from the yield point theory[8] as:

$$\tau_{\ell y} = C\dot{\varepsilon}^{(2 + m)^{-1}} \exp(E/(m + 2)kT) \qquad (1)$$

where C is a function of the strain rate, m is a kinetic factor characterizing the dependence of the dislocation velocity with stress and E is the activation energy for the motion of dislocations. Experimental values of the lower yield stress versus inverse temperature is shown in Fig. 3 for undoped and indium doped GaAs. The straight lines obtained by least-square fit were used to determine E/(m + 2) and the values of the CRSS at the melting point. The results are summarized in Table II. Statistical analysis of the experimental data obtained from a large number of samples have shown that the values for $\tau_{\ell y}$ (or CRSS) are reproducible within ±12%. The value found of CRSS for undoped GaAs, 41 gm mm^{-2}, is consistent with that previously reported.[1,11] For T > 390°C, the CRSS of GaAs:In is higher than that of undoped GaAs, while at lower temperatures it is lower. At the melting point, the CRSS of GaAs:In is only twice that of undoped GaAs. This increase is much too small to explain dislocation density reduction through reduction of crystallographic glide. It indeed indicates that crystallographic glide induced by thermal stresses is not the only cause for dislocation formation in GaAs. We suggest that indium doping inhibits dislocation formation by modifying the thermodynamic equilibrium of native defects. This modification, however, does not drastically increase the CRSS as previously suggested.[6] Rather, it appears to affect the formation of dislocations through the condensation and interaction of point defects.

From the slopes of $\log(\tau_{\ell y})$ versus 1/T, which equal E/(m + 2), we can obtain a value of the activation energy for the motion of the dislocation. The value of kinetic factor m for GaAs:In has not been reported; however, it can be estimated[12] to be 2.1 for a stress range 2-5

kg mm^{-2}. This corresponds to the stresses incurred for temperatures below 350°C. We then obtain for GaAs:In $E = 1.23$ eV. By using $1.2 < m < 1.6$ for the same stress range[7,13,14] for undoped GaAs, we calculate $1.18 \text{ eV} < E < 1.33$ eV. These approximate values indicate that In doping has very little effect, if any, on the average motion of freshly introduced dislocations at low temperatures. A similar conclusion was drawn in Ref. 11, where the authors found the same activation energies for the motion of α and β dislocations in both undoped- and indium-doped samples. Note that a high concentration of an isoelectronic dopant in silicon and germanium also resulted in no substantial change in the activation energy for dislocation motion.[15,16] It is not possible to determine the activation energy for the motion of dislocations at higher temperatures since the values of the parameter m have not been measured at low stresses. However, the present results for low temperatures confirm recent conclusions[17] that indium doping does not reduce dislocation formation by simple solution hardening as had been proposed.[18] Solution hardening involves pinning of the dislocations at the solute location and should reduce their velocity. Electron microscopy studies of the deformed samples as well as precise measurements of dislocation velocities at high temperatures are now underway for further investigations of these results.

In summary, dynamical compression tests at high temperature have been performed for the first time on indium doped and undoped-GaAs single crystals. The results show that the CRSS of indium doped GaAs samples ($[In] = 2.9 \times 10^{19}$ cm^{-3}) is twice that of undoped GaAs. Indium doping, at the concentration used for this study, does not affect the activation energy for the motion of dislocations at temperatures below 350°C.

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TABLE I. Characteristics of the GaAs crystals used for this study.

Crystal	Dopant concentration (cm^{-3})	Average Resistivity ($\Omega \cdot cm$)	Etch pit density (dislocation density)
undoped GaAs (RD2-301)	N/A	7×10^{6}	2000 - 3000
GaAs:In (H276)	2.9×10^{19}	1.8×10^{7}	0 - 100

TABLE II. Values of the CRSS at the melting temperature of GaAs and values of E/(M + 2).

Crystal	τ_{1y} (or CRSS) (g mm^{-2})	E/(m + 2) (eV)
undoped GaAs (RD2-301)	41 ± 5	0.37 ± 0.02
GaAs:In (H276)	82 ± 10	0.30 ± 0.01

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FIGURE CAPTIONS

FIG. 1. Schematic of the experimental set-up for the compression tests.

FIG. 2. Resolved shear stress versus percentage glide strain obtained from dynamic compression of indium-doped GaAs at various temperatures.

FIG. 3. Experimental values of the lower yield stress versus inverse temperature for undoped and indium-doped GaAs.

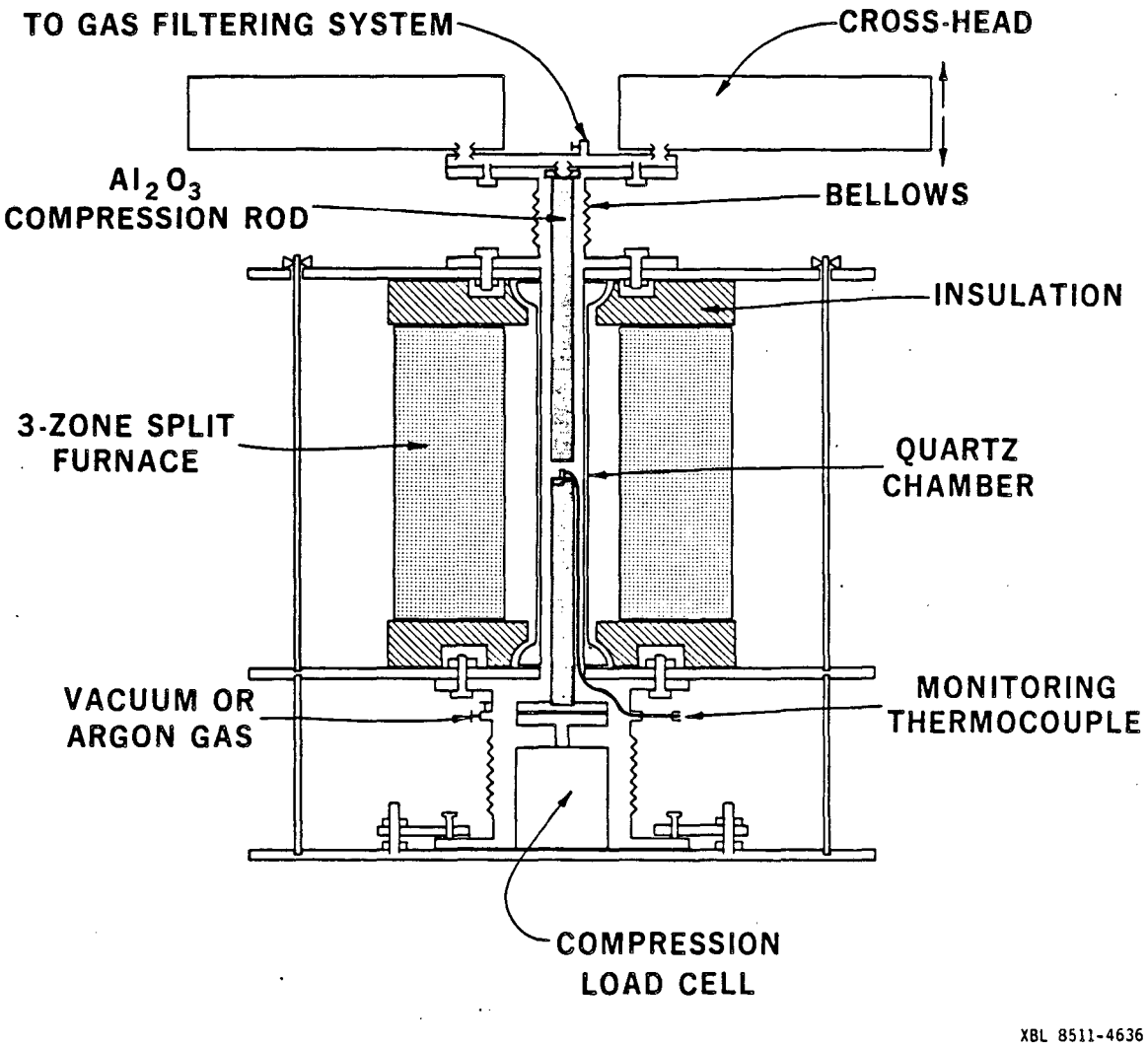
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Figure 1.

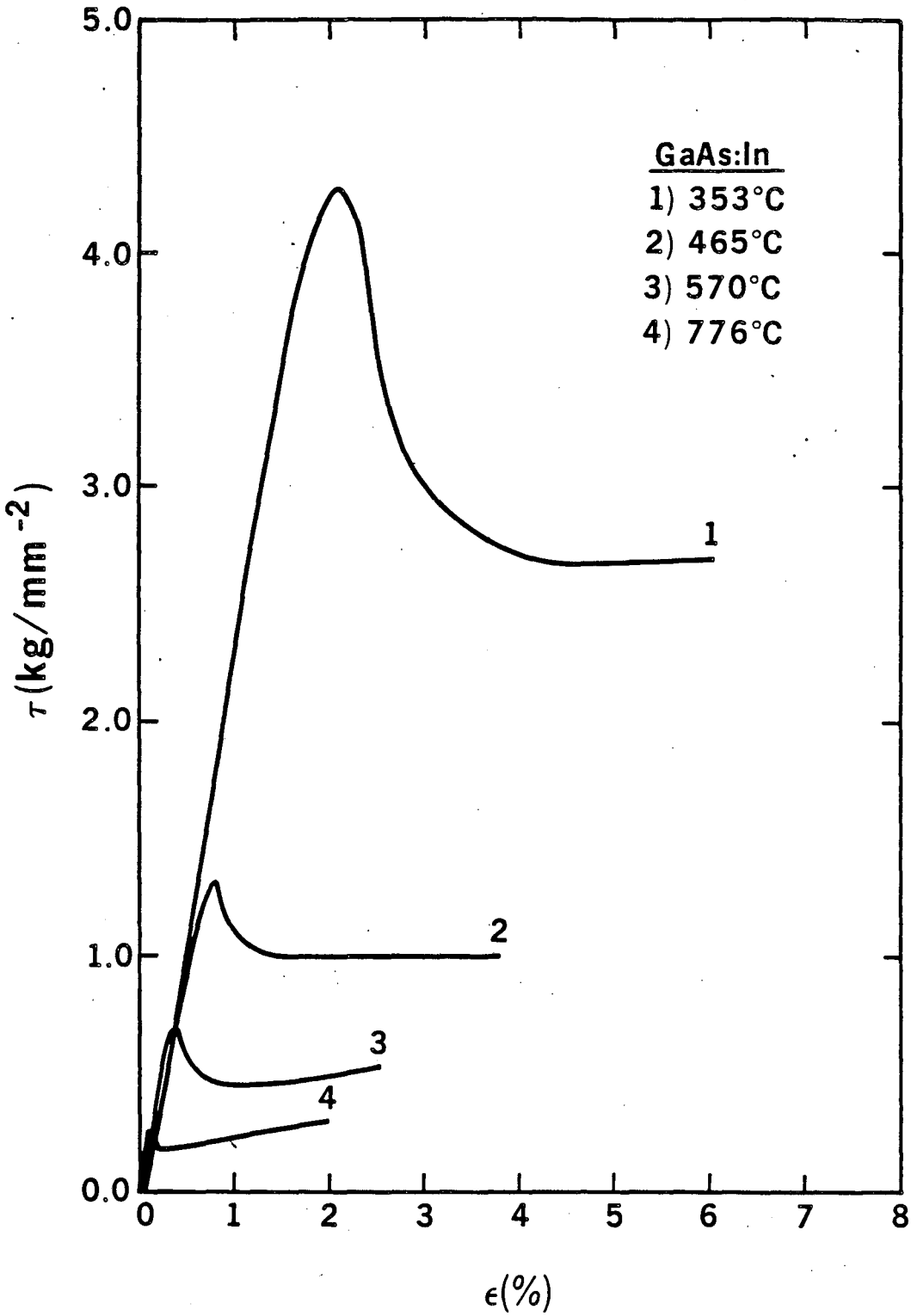

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Figure 2.

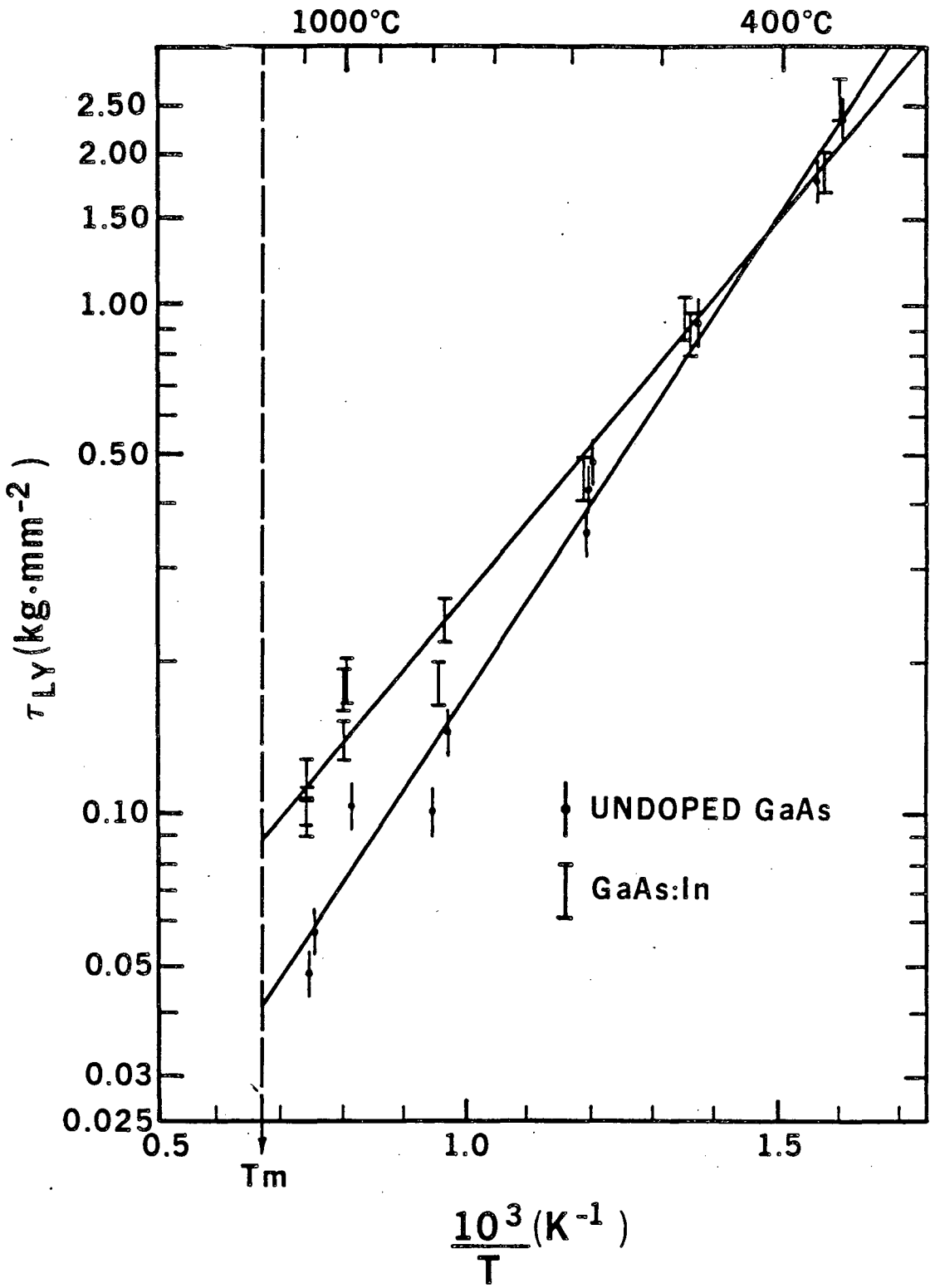

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Figure 3.

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