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# Deformation behavior of undoped and In-doped GaAs in the temperature range 700–1100 °C

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Compressive deformation of undoped and In-doped GaAs single crystals has been carried out in [001] and [123] orientations in the temperature range 700–1100 °C. Indium additions, at levels of $1–2\times10^{20}$ atoms $cm^{-3}$, result in critical resolved shear stress (CRSS) values that are about twice as large as the undoped crystals in the temperature range of 700–1100 °C. The CRSS was weakly dependent on temperature in the temperature range investigated as expected for a model of athermal solid solution hardening. The CRSS value of 3.3 MPa for the In-doped crystal is sufficient to eliminate profuse dislocation formation in a 75-mm-diam crystal on the basis of current theories for the magnitude of the thermal stress experienced during growth. The results also suggest that the process of dislocation climb is slowed appreciably by In doping.

## I. INTRODUCTION

Doping of GaAs with In, at a level of about $5\times10^{19}$–$1\times10^{20}/cm^3$, in single crystals grown by the liquid-encapsulated Czochralski process reduces the dislocation density from $10^4$–$10^5/cm^2$ to $\leqslant 10^2/cm^2$.[1] The generation of dislocations during LEC growth in GaAs and other III-V compounds is believed to occur when the thermal stress imposed on the crystal during growth exceeds the critical resolved shear stress (CRSS).[2] Minimization of radial thermal gradients during growth and enhancement of the inherent strengths of the crystal by the addition of isovalent as well as Group IV or VI elements can result in low dislocation density GaAs crystals.[3–5] Of the possible dopants, In has been found to be very effective and desirable because of its minimal influence on the electrical behavior of GaAs. Thermal stress calculations suggest that the maximum stress experienced is much larger than the extrapolated CRSS value of an undoped crystal.[6] Therefore, the In doping must result in a large increase in high-temperature strength. It has been suggested that hardening akin to solid solution hardening occurs in $Ga_{1-x}In_xAs$ with an $InAs_4$ tetrahedral cluster being the solute unit that causes strengthening.[7] However, experimental data on high-temperature deformation of GaAs and the influence of dopants are scarce and the role of In is still not well understood.

In a preliminary study, we first measured the temperature dependence of the hardness of undoped and In-doped GaAs, which was reported earlier.[8] In this subsequent study, we have evaluated the flow stress in the [001] orientation for which multiple equivalent slip systems of {111} ⟨110⟩ type operate and in the [123] orientation suitable for the operation of only one slip system, $(\bar{1}11)$ [101]. The results of the study made in the temperature range 700–1100 °C are reported here, compared with other reported results,[6,9–13] and discussed in terms of models for the high-temperature deformation of diamond cubic materials.[14,15]

## II. EXPERIMENTAL PROCEDURE

The 75-mm-diam single crystals of semi-insulating GaAs and $Ga_{0.99}In_{0.01}As$ used in the present study were obtained from the Westinghouse R&D Center. Crystals were grown in [001] axial orientation. The undoped crystal had a dislocation density of $10^4$–$10^5$ $cm^{-2}$. The indium-doped crystal had an In concentration of about $1–2\times10^{20}$ atoms $cm^{-3}$. The boron concentration in both doped and undoped crystals was about $5\times10^{17}$ atoms $cm^{-3}$. Dimensions of specimens for compressive deformation in the [001] orientation were 5.3 mm$\times$5.3 mm$\times$10.6 mm in size with (110) and $(1\bar{1}0)$ lateral faces. The dimensions for deformation in the [123] orientation were 2.75 mm$\times$2.75 mm$\times$5.5 mm. The lateral faces were parallel to $(11\bar{1})$ or $(\bar{5}4\bar{1})$ planes. The specimen faces were mechanically polished to 0.3-$\mu$m alumina followed by chemical polishing in a 1% bromine-methanol solution. The specimen faces were parallel and orthogonal to within 0.5°.

The compression testing was done on an INSTRON 1322 servohydraulic machine. A schematic diagram of the experimental setup is shown in Fig. 1. Tests were performed at temperatures of 700, 900, 1000, and 1100 °C at a strain rate of $1\times10^{-4}$ $s^-1$ in UHP argon. The furnace used in the study had a low heat capacity with a time of heat up to the test temperature of about 20 min. For tests at temperatures of 900 °C and above the specimen was immersed in $B_2O_3$ liquid. The compression rams used were made of high-purity alumina, and a special fixture made of alumina was used for the alignment and positioning of the specimen. The elongation was measured on the alumina rams by means of an extensometer and was corrected for the fixture compliance, at a temperature of $\sim$7.25 kN/mm, in converting the data to strain.

Stress relaxation tests were performed following the [001] specimen compression tests. At the end of compressive deformation, the decrease in stress at constant specimen

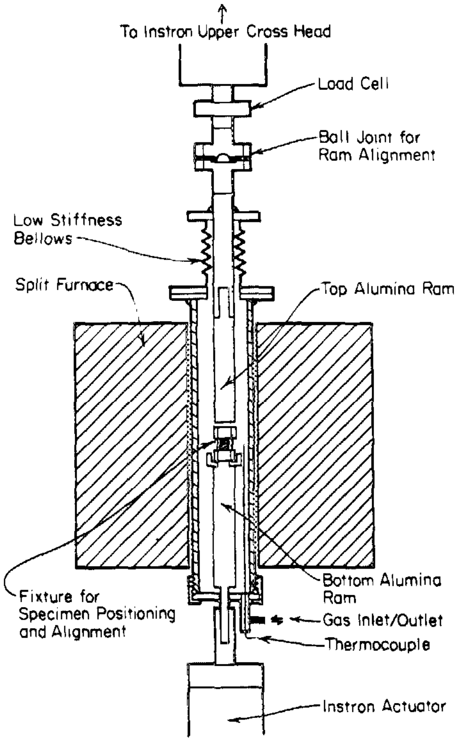


FIG. 1. Schematic of the compression testing jig.

length was measured as a function of time and was converted to plastic strain as a function of time. The total amount of strain during stress relaxation is very small and the total time of testing is only a few minutes.

## III. RESULTS

### A. [001] orientation

Figures 2(a) and 2(b) show the results of compression tests in [001] orientation for undoped and In-doped GaAs. No easy glide, stage I deformation is observed for these crystals, oriented for multiple slip. Also, only at 700 °C for the In-doped case is there an indication of the yield point phenomena characteristic of a similar material[6] tested at 350–590 °C. The tendency for a less pronounced yield point with increasing temperature is consistent with results in other studies,[6,10] and the absence of well-defined yield points at high temperatures agrees with the work of Djemel and Castaing.[11] We note that the slope below the yield point at 700 °C for the In-doped case is less than the reported elastic modulus so that microyield and plastic flow are already occurring. This characteristic has been observed in other measurements,[6,10,11] including those at lower temperatures. Indeed, *in situ* x-ray topographic measurements made during tensile testing of In-doped and undoped GaAs at 450–700 °C demonstrate directly that dislocation formation and propagation occur at stresses well below the upper yield point.[16,17] Such phenomena are well known for metals where microyield also occurs well below the macroscopic yield[18] with sufficient mobility and multiplication rates for dislocations. The microyield regime A (Ref. 19) merges smoothly into stage II deformation behavior.

The stress-strain curves, with the exception of the In-doped 700 °C case, are characterized by a stage II linear work hardening regime of flow, followed by stage III characterized by a decrease in work hardening rate, and the beginning of the stage IV linear hardening regime characteristic of diamond cubic materials.[14,15,20]

The results indicate hardening effects by indium in all stages of deformation. The stage A slopes increase with decreasing temperatures and are larger at all temperatures for the In-doped crystals. The onset of stage III occurs at larger stresses in the In-doped case, and the slope in stage IV is greater for the In-doped case. A yield point, indicative of insufficient dislocation mobility/multiplication, occurs for the In-doped crystals at 700 °C but not for the undoped crystals. Also for the 700 °C case, the microyield flow region A, below the yield point, has a larger slope (larger work hardening) than the postyield, stage II, linear work hardening regime.

Values for the 0.2% offset yield strength from the present work and that of others are presented in Table I and plotted in Fig. 3. The data are all converted to critical resolved shear stresses for later comparison with single slip crystals. The agreement between investigations shown in Fig. 3 is seen to be quite good.

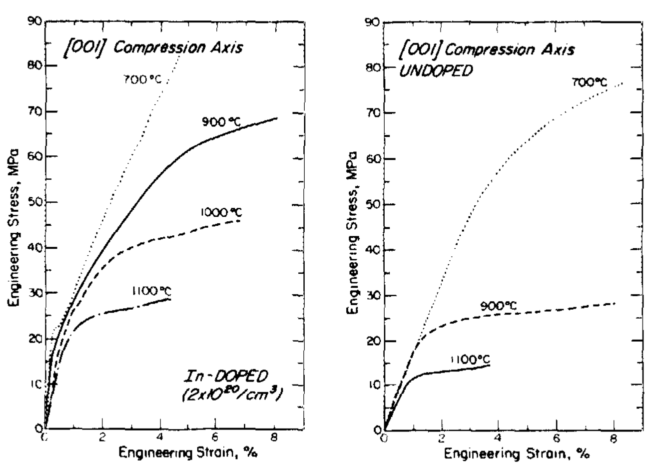


FIG. 2. Engineering stress-strain curves at different temperatures for (a) In-doped GaAs and (b) undoped GaAs specimens tested in the [001] orientation.

### B. [123] orientation

The specimens tested in [123] orientation showed sharp yield points. Flow stress-strain curves for undoped and In-doped specimens at 700 °C are shown in Fig. 4. The single slip orientation crystals showed extensive stage I deformation with a small yield drop at 700 °C (Fig. 4), but not at 900 or 1100 °C. The preyield slopes in the microyield region A varied just as in the [001] case, with smaller slopes for the undoped case at each temperature and with a decrease in slope with increasing temperature.

In comparison, the suppression of yield point phenomena at temperatures above 700 °C for [001] crystals (present work, Ref. 11) indicate a strong impediment to dislocation intersection in these crystals. This impediment, favored by the relatively low stacking fault energy of $\sim 45$ mJ/m$^2$ in these materials,[21] suppresses easy glide and contributes to

TABLE I. Resolved shear stress values for undoped and In-doped GaAs. $\sigma_Y$ is upper yield point, $\sigma_{0.002}$ and $\sigma_{0.04}$ are offset yield strengths at strains of 0.002 and 0.04, respectively, and $H/6$ is an estimate from data for hardness $H$. Numbers in parentheses are references to other work.

| | [123] orientation | | [001] orientation | | | |
|---|---|---|---|---|---|---|
| Material | $T$(K) | $\sigma_Y$(MPa) | $\sigma_Y$(MPa) | $\sigma_{0.002}$ (MPa) | $\sigma_{0.04}$ (MPa) | $H/6$ (MPa) |
| | 973 | 4.64 | ··· | 9.3 | 27.8 | 66.7 |
| In-doped GaAs | 1173 | 3.33 | ··· | 8.8 | 23.1 | 39.9 |
| | 1273 | ··· | ··· | 8.5 | 17.2 | ··· |
| | 1373 | 3.27 | ··· | 7.8 | 11.6 | ··· |
| | 973 | 2.55 | ··· | 2.8 | 21.8 | 51.7 |
| Undoped GaAs | 1173 | 1.91 | ··· | 2.45 | 10.0 | 28.6 |
| | 1373 | 1.83 | ··· | 2.45 | 6.0 | ··· |
| | 1253 | 1.4(10) | | | | |
| | 1353 | 1.05(10) | | | | |
| In-doped GaAs (LEC) | 873 | | 8.5(6) | | | |
| | | | 5.5(6) | | | |
| | 800 | | | 9.6(11) | | |
| | 900 | | | 7.6(11) | | |
| | 1053 | | | 7.2(11) | | |
| | 1053 | 1.22(10) | | | | |
| | 1253 | 1.0(10) | | | | |
| | 1353 | 0.53(10) | | | | |
| Undoped GaAs | 873 | | 6.9(6) | | | |
| (LEC) | 800 | | | 2.2(11) | | |
| | 900 | | | 2.1(11) | | |
| | 1053 | | | 2.2(11) | | |
| | 773 | 3.9(13) | | | | |
| Undoped GaAs (non LEC) | 823 | | 2.4(9) | | | |

the large stage II slope for the [001] crystal. Nevertheless, the dislocation mobility in stage II is sufficient to prevent macroscopic yield point behavior.

The values for the upper yield point as a function of temperature, together with results from other work, are listed in Table I and plotted in Fig. 5. Lower temperature data for [100] specimens from the work of Hobgood *et al.*[6] and Swaminathan and Copley[9] are included because their specimens also exhibited sharp yield points with little yield drop. This [001] yield point behavior below 700 °C is associated with the reduction in dislocation multiplication/mobility by the presence of the Peierls barrier and the attendant mechanism of double-kink dislocation motion.

The present results align well with the other work on the same material.[6,9,10] All results are in agreement with regard to the general shape of the curves of flow stress versus temperature. However, the present results are consistently higher in stress, by about a factor of 2, than the other extensive

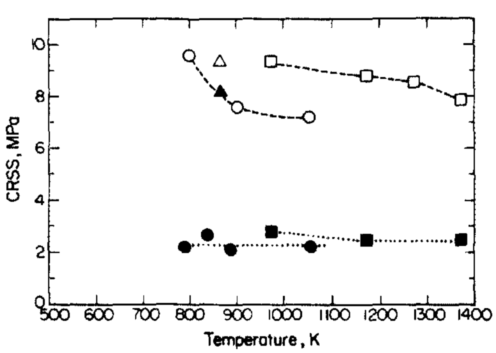


FIG. 3. Comparison of critical resolved shear stress values of undoped and In-doped GaAs from present and other work in the [001] orientation as a function of temperature. Open squares: In-doped (this study); solid squares; undoped (this study); open triangle: In-doped (Ref. 6); solid triangle: undoped (Ref. 6); open circles: In doped (Ref. 11); solid circles: undoped (Ref. 11).

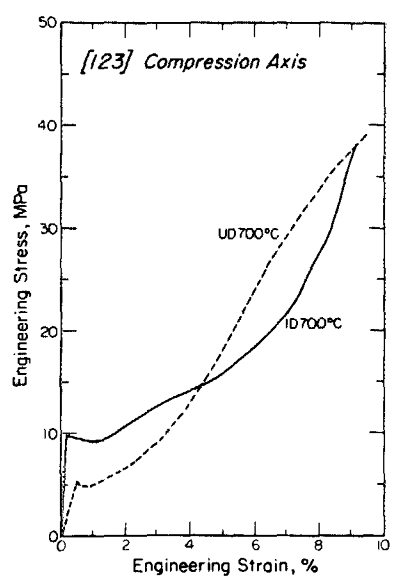


FIG. 4. Engineering stress-strain curves for undoped and In-doped GaAs in the [123] orientation.

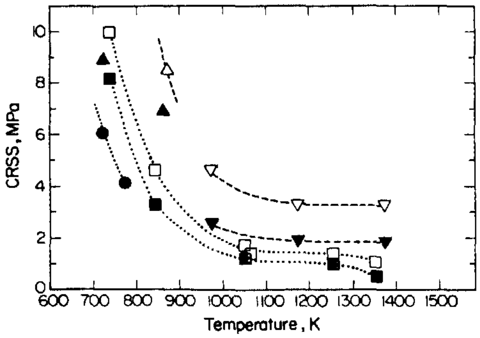


FIG. 5. Comparison of critical resolved shear stress values of undoped and In-doped GaAs from present and other work as a function of temperature. Inverted open triangle: In doped (this study); inverted solid triangle: undoped (this study); open triangle: In doped (Ref. 6); solid triangles: undoped (Ref. 6); open squares: In doped (Ref. 10); solid squares: undoped (Ref. 10); solid circles: undoped (Ref. 9).

work on single slip crystals.[10] A possible cause for this difference, as discussed previously,[8] is the presence of $\sim 5\times 10^{17}$ $cm^{-3}$ of boron in the present samples, a solute that would also be expected to produce strong solution hardening.

## IV. DISCUSSION

### A. Compression tests

The overall form of the stress-strain curves with stages II–IV for [001] crystals, and stage I, as well, for [123] crystals, is consistent with the behavior at elevated temperatures of the much more extensively studied Si and Ge crystals.[14,15,20] The analogy suggests that glide processes are important in determining the flow stress in stages I and II while recovery processes are important, in addition, in stages III and IV. The relative temperature independence of the critical resolved shear stress above 700 °C (Figs. 3 and 5) implies plateau-type behavior[22] characteristic of athermal solid solution hardening. Hence, the hardening indicated at yield and in stage I is consistent with the suggestion[7] that In should provide strong solid solution hardening in $Ga_{1-x}In_xAs$

Below ~700 °C, the sharp rise in flow stress with decreasing temperature is related to a change in the mechanism of glide to one controlled by double-kink nucleation and propagation, again analogous to Si and Ge. The hardening effect in this regime could also be related to the elastic field of an $InAs_4$ unit,[7] in this case effective through an impediment to the propagation of kinks along a dislocation line. Because of the apparent change of mechanism below about 700 °C, results obtained below this temperature, e.g., on dislocation velocity,[23,24] may not be applicable to behavior above 700 °C.

The larger stress for the onset of stage III and the larger slope in stage IV for In-doped crystals imply a decrease in recovery rate for the In-doped crystals. This could be caused, in principle, by suppression of cross slip or by retardation of climb. Several factors indicate that the latter climb effect is predominant. First, transmission electron microscopy studies[21] indicate that In doping, at the level studied here, does not affect the stacking fault energy, implying that there should be little influence on the cross slip probability. Second, our transmission electron microscopy studies[25] of [001] specimens strained into stage IV show well-developed dislocation networks similar to structures observed for stage IV deformaton of Si,[20] a case where climb is known to contribute to the recovery mechanism. Third, stress relaxation measurements in the present work[26] show consistently lower relaxation rates for the In-doped case at a given stress over the entire range of relaxation, implying a lower climb rate.

### B. Comparison with hardness results

Resolved shear stress values calculated earlier by us from values of the hardness $H$[8] are much higher than the measured flow stress values. Comparative data using the earlier estimate of $H/6$ together with 4% offset flow stresses are listed in Table I. The reason for the discrepancy is our earlier use of an empirical correlation of hardness and yield strength for engineering materials with characteristically low work hardening rates. If, instead, the strain hardening rates are taken into account using the empirical relationship developed by Cohoon, Broughton, and Kutzak,[27] $(H/\sigma_{7.4}) = 3\times 10^n$, where $n$ is the strain hardening exponent, and $\sigma_{7.4}$ is the flow stress at 7.4% strain, the values of flow stress calculated from hardness values approach the flow stress at 4% strain, but still appreciably exceed the 0.2% offset yield stress. The trends in hardness with temperature and the hardening increment provided by indium as revealed in the earlier work, however, do give an indication of the strengthening effect of In consistent with the present work.

### C. Crystal growth

Recent calculations by Jordan, Von Neida, and Caruso[28] indicate that dislocations can be completely removed in 75-mm-diam GaAs crystals grown under low thermal gradient conditions if the CRSS for plastic flow initiation is a factor of 4 larger than a GaAs base level value of 0.6 MPa, determined by extrapolation to the melting point. The basis for the extrapolation is the data for the yield point of undoped GaAs (Ref. 9, Fig. 3). The present results, Figs. 3 and 5, suggest a value of 3.3 MPa for the yield point extrapolated to the melting point for the present case of In at a level of 1–$2\times 10^{20}$ $cm^{-3}$. Hence, such a doping level of indium should prevent the profuse dislocation generation characteristic of stage I deformation according to the theory of Jordon *et al.*[28]

However, the present work and that of others[6,10,11] show that microyield occurs well below the yield point in compression tests above 700 °C and particularly at 1100 °C. These results, together with the *in situ* x-ray topographic studies in tension[16,17] at temperatures less than 700 °C, indicate that dislocations, once formed, are highly mobile at high temperatures. This suggests that, if GaAs crystals are grown in a dislocation-free condition, a major impediment to the presence of dislocations must be the nucleation process. In all of the compression tests (present work, Refs. 6, 9–11) the crystal end in contact with the loading platens is an easy site for dislocation nucleation because of stress intensification at contact asperities and because of the weak singularity in the stress field at the edge of the contact surface of the specimen. Hence, such data, while indicative of the yield point, may not correspond in microyield behavior to the crystal growth case where nucleation of dislocations would be more difficult.

There is some indication of such a possibility in the low-temperature tensile test observations of Tohno *et al.*,[16,17] a case where such end effects are absent and where dislocation nucleation does not occur at stresses of at least 20% of the yield stress.

## V. SUMMARY

(i) Addition of In at levels of $1\text{–}2 \times 10^{20}$ atoms/cm$^3$ results in CRSS values for flow that are about twice as large as the undoped crystals throughout the stress-strain curve and in the temperature range of 700–1100 °C.

(ii) The CRSS for a yield point or for the 0.2% offset yield strength was weakly dependent on temperature in the temperature range investigated, as expected for a model of athermal solid solution hardening.

(iii) The strength of In-doped crystal is sufficient to eliminate profuse dislocation formation in a 75-mm-diam crystal on the basis of current theories for the magnitude of the thermal stress during growth.

(iv) The onset of stage III occurs at much higher stress levels in the In-doped alloy. Together with observations of dislocation structures, this suggests that the process of dislocation climb is slowed appreciably by In doping.

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