JOURNAL OF MATERIALS SCIENCE 24 (1989) 328–332

Directional dependence of *c*-plane slip in single crystals of mercuric iodide

FREDERICK MILSTEIN, GARY GEORGESON
Departments of Materials and Mechanical Engineering, University of California, Santa Barbara, California 93016, USA

An experimental study of plastic deformation in thin single crystals of mercuric iodide (HgI_2) subjected to (0 0 1) [1 0 0] and (0 0 1) [1 1 0] shear loadings has established a directional dependence to the phenomenon of (0 0 1) or *c*-plane slip in HgI_2. The average stresses s_c for the "onset of yielding" were 12.6 psi (86.9 kPa) for (0 0 1) [1 0 0] shear and 16.5 psi (113.8 kPa) for (0 0 1) [1 1 0] shear; the average values of the deformation parameter s_0, interpreted as a "bulk yield stress", were 24.7 psi (170.3 kPa) for (0 0 1) [1 0 0] shear and 37.7 psi (259.9 kPa) for (0 0 1) [1 1 0] shear. These values are in agreement with the theoretical relations $s_c(\theta) = s_c(0)/\cos\theta$ and $s_0(\theta) = s_0(0)/\cos\theta$, where θ is the smallest angle between $\langle 1\,0\,0\rangle$ crystallographic axis and the direction of shear stress applied in the (0 0 1) plane.

1. Introduction

The response of single-crystal mercuric iodide (HgI_2) to various modes of compressive loading has been characterized by James and Milstein [1], who observed that

". . . for all crystallographic orientations wherein a non-zero shear stress component acts on the (0 0 1) planes, the crystals are easily plastically deformed by slip of the (0 0 1) planes; furthermore, the (0 0 1) planes are the only planes that exhibited slip in these tests. We also found this mode of plastic deformation to exhibit work hardening (i.e. the critical resolved shear stress for slip of the (0 0 1) planes increases as plastic deformation progresses). Within the limits of accuracy and repeatability of our tests, we could not observe any anisotropy of slip direction within the (0 0 1) planes; thus, we simply speak of (0 0 1) slip, without reference to a specific slip direction parallel to the (0 0 1) plane. In cases where the shear stress on the (0 0 1) planes is zero (i.e. when the crystals are loaded either parallel or perpendicular to the [0 0 1] direction), under sufficient load, failure occurs by brittle fracture (prior to any macroscopically observed plastic deformation)."

Subsequent to the work of James and Milstein [1], various refinements have been made [2–4] in the techniques of measuring the response of single-crystal HgI_2 to load and in the methods of data analysis; the latest of these, described by Georgeson and Milstein [4], has provided the means by which we have determined a directional dependence to (0 0 1) or *c*-plane slip in single-crystal HgI_2.

The crystal structure of mercuric iodide is illustrated in Fig. 1. (Here we are concerned only with the α phase [5, 6], which is the stable phase at room temperature.) The structure of α-HgI_2 can be described as a layered sequence of monoatomic *c*-planes in the order . . . Hg I I Hg I I Hg . . . ; bonding between two successive iodine layers (thought to be mainly van der Waals) is weak and thus plastic deformation by *c*-plane slip (i.e. by slip between two successive iodine layers) occurs readily; with reference to Fig. 1, easy slip can occur between Planes 2 and 3 and between Planes 5 and 6. A "top" view of the *c*-planes is shown in Fig. 2. Analysis of Figs 1 and 2 suggests the existence of a directional dependence to *c*-plane slip; e.g. if slip of the (0 0 1) planes were to occur in the [1 1 0] direction (i.e. if (0 0 1)[1 1 0] slip were to occur) by simple shear between a 2-plane and a 3-plane, say, iodine atoms in the 2-plane would pass directly over iodine atoms in the 3-plane (at a closest I–I approach of 0.274 nm); whereas in (0 0 1)[1 0 0] slip, the iodine atoms in the 2-plane pass "between" the iodine atoms in the 3-plane (with a closest I–I approach of 0.350 nm in simple shear). Similarly, in *c*-plane slip that occurs via a dislocation mechanism (rather than by simple shear), the distance between nearest iodine atoms in successive iodine planes must become smaller during slip via a dislocation with a [1 1 0] Burgers vector than with a [1 0 0] Burgers vector. This analysis of structure, augmented with the logical assumption that the repulsion between nearest-neighbour I–I atoms (in successive iodine planes) increases as the interatomic spacing decreases (from its equilibrium value of 0.412 nm) leads to the hypothesis that, for *c*-plane slip, the easiest slip direction is [1 0 0] and the hardest is [1 1 0]. If *c*-plane slip in HgI_2 follows the usual Schmid law, and if $s_{crit}(0)$ is the shear stress in the (0 0 1) plane in the [1 0 0] direction required to initiate (0 0 1)[1 0 0] slip, then a shear stress $s(\theta)$ applied in the (0 0 1) plane at an angle θ, with respect to the [1 0 0] axis, will initiate slip when it reaches the critical value $s_{crit}(\theta)$ given by

$$s_{crit}(\theta) = s_{crit}(0)/\cos\theta \qquad (1)$$

for $|\theta| \leqslant \pi/4$.

0022–2461/89 $03.00 + .12

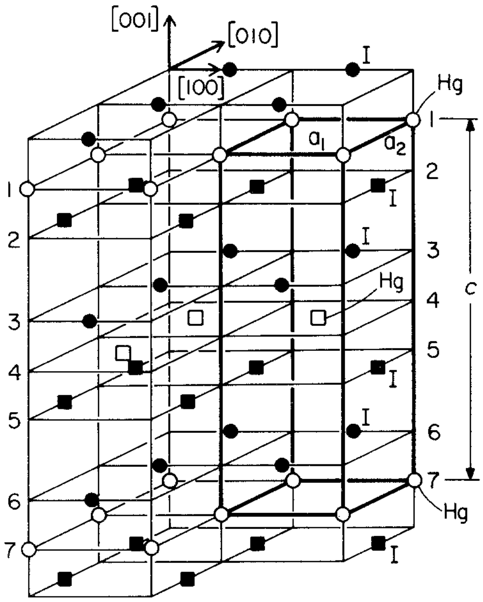

Figure 1 Crystal structure of HgI_2; crystallographic axes are shown in terms of Miller indices; iodine atoms are indicated as solid squares and circles and mercury atoms as open centred squares and circles; a tetragonal unit cell is shown with bold line segments; successive (0 0 1) or c planes are numbered (1 to 7) to facilitate discussion (Planes 7 and 1 are equivalent).

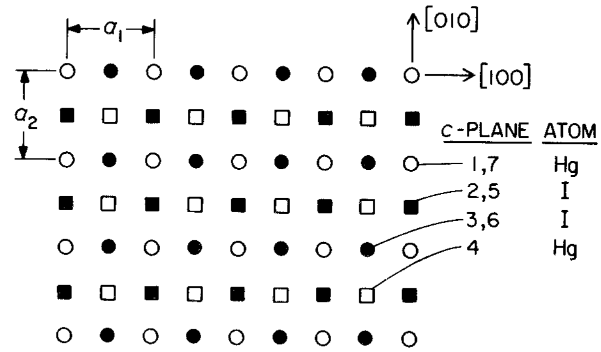

Figure 2 Planar view of (0 0 1) or c planes of HgI_2 (corresponding to "top" view of Fig. 1); lattice parameters a_1 and a_2 are of equal magnitude.

Evidently a directional dependence of c-plane slip in HgI_2 has not been confirmed in prior experiments owing to (i) the use of axial loading techniques that were not conducive to determining relatively small differences in the c-plane shear stress against shear strain response, (ii) difficulties in determining "clear-cut" values of $s_{crit}(\theta)$ for stress–strain curves exhibiting gradual yielding (rather than a "sharp" yield point), and (iii) quantitative variations in the stress–strain response of different samples loaded in the same directions. In the present work these respective difficulties are overcome as follows.

(i) Measurements of shear stress against shear strain are made using a shear testing apparatus specially designed for the purposes of applying shear stresses directly to the (0 0 1) planes of thin HgI_2 single-crystal specimens and of measuring the shear stress–strain response; this apparatus and the experimental technique employed are described in detail elsewhere [4].

(ii) The phenomenon of c-plane slip is characterized in terms of a semiempirical model for plastic yielding that has been shown [3, 4] to fit the experimentally determined yielding response extremely well, both for HgI_2 specimens loaded in the shear apparatus [4] employed in the present study and those loaded axially [3]; measurement of the model deformation parameters provides a convenient and accurate means of quantitatively describing (and comparing) the yielding behaviour of single crystals of HgI_2 that pass through a gradual transition from elastic to plastic deformation without exhibiting a sharp yield point or critical resolved shear stress; briefly, a normalized Gaussian function $f(s)$ is used to describe the slip process; $f(s)\mathrm{d}s$ is the number of c planes (relative to the steady state) that begin to slip when the applied c-plane shear stress is increased from s to $s + \mathrm{d}s$; the Gaussian deformation parameters s_0 (the applied c-plane shear stress at which the number of c planes that are slipping has reached half of its eventual steady state value) and σ (the standard deviation) are determined from a least-squares fit between theoretically computed and experimentally measured stress–strain relations (see Figs 1 and 2 in Milstein and Georgeson [3] and Figs 5 and 6 in Georgeson and Milstein [4] for examples); s_0 characterizes "bulk yielding" and $s_c \equiv s_0 - 2\sigma$ describes the "onset of yielding" (s_c is the c-plane shear stress at which the number of mobile c planes has reached approximately 2% of the steady-state value); for further details, including an interpretation of the model parameters in terms of dislocation mechanisms, see Georgeson and Milstein [4].

(iii) The yielding responses (specifically the deformation parameters s_0 and s_c) are measured for a sufficient number of well characterized, good-quality samples for the cases of (0 0 1)[1 0 0] and (0 0 1)[1 1 0] applied shear stresses (which are the two stress states expected to exhibit the greatest difference in yield stress).

2. Experimental results and discussion

Equation 1 suggests, as a reasonable working hypothesis, the following presumed variations of the model deformation parameters s_0 and s_c as functions of the angle θ between the direction of the applied c-plane shear stress and the [1 0 0] axis

$$s_0(\theta) = s_0(0)/\cos\theta \tag{2}$$

and

$$s_c(\theta) = s_c(0)/\cos\theta \tag{3}$$

with $|\theta| \leqslant \pi/4$. As mentioned above, in the present experiments, aimed at observing a directional response to c-plane slip in HgI_2, measurements are made of $s_0(0)$ and $s_c(0)$ (i.e. s_0 and s_c in (0 0 1)[1 0 0] shear loading) and of $s_0(\pi/4)$ and $s_c(\pi/4)$ (i.e. s_0 and s_c in (0 0 1)[1 1 0] shear).

In the first set of experiments, three different single crystals of HgI_2 were obtained and two single-crystal specimens were prepared from each of the three single crystals. The single crystals were vapour-grown [7, 8] and the test specimens were prepared from the single crystals as described elsewhere [1, 4]. Three specimens (one from each crystal) were subjected to (0 0 1)[1 0 0] shear stresses and the remaining three were loaded in (0 0 1)[1 1 0] shear. The deformation parameters

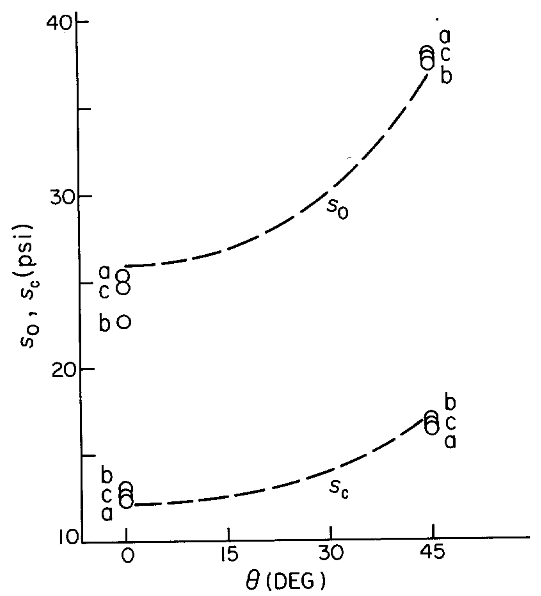

Figure 3 Average experimental values of s_0 and s_c for (0 0 1) [1 0 0] and (0 0 1) [1 1 0] slip (i.e. for $\theta = 0°$ and $\theta = 45°$, respectively). (a) Averages for detectors, (b) averages for non-detectors, (c) averages for all tests. The theoretical curves are "best fits" of the relations $s_0(\theta) = s_0(0)/\cos\theta$ and $s_c(\theta) = s_c(0)/\cos\theta$ to the points labelled (c). 1 psi = 6.895 kPa.

describing yielding, s_0 and s_c, were determined for each of these six tests. The experimental technique is described elsewhere [4]. The following observations were made.

1. Although there was some scatter in the measured values of s_0 and s_c, (0 0 1) [1 0 0] slip was definitely easier than (0 0 1) [1 1 0] slip; s_0 varied from 15.5 to 28.5 psi (106.8 to 196.5 kPa) for (0 0 1) [1 0 0] slip and from 26.5 to 49.4 psi (182.7 to 340.6 kPa) for (0 0 1) [1 1 0] slip; s_c varied from 10.3 to 15.3 psi (71.0 to 105.4 kPa) for (0 0 1) [1 0 0] slip and from 14.5 to 18.4 psi (100.0 to 126.9 kPa) for (0 0 1) [1 1 0] slip.
2. When the data were evaluated in a "pairwise" manner (wherein comparisons were made within each pair of specimens prepared from the same crystal), each value of s_0 was greater in (0 0 1) [1 1 0] slip than in (0 0 1) [1 0 0] slip; likewise s_c was always greater in (0 0 1) [1 1 0] slip than in (0 0 1) [1 0 0] slip.
3. The average values of s_0 and s_c for the three specimens in (0 0 1) [1 0 0] shear (i.e. $\theta = 0$) and for the three in (0 0 1) [1 1 0] shear ($\theta = \pi/4$) are reasonably well represented by Equations 2 and 3; these average values are plotted in Fig. 3 as points labelled (b) (average for non-detectors).

It might be noted that a given specimen can be used to determine $s_0(\theta)$ and $s_c(\theta)$ for a given value of θ; during determination of these deformation parameters, the specimen is plastically deformed; this work-hardens the specimen, which alters the values of s_0 and s_c that would be determined in any subsequent measurements; in the present study, previously undeformed specimens were used in all determinations of s_0 and s_c. If a given specimen were to follow the behaviour indicated in Equations 2 and 3, but if $s_0(0)$ and $s_c(0)$ were to vary somewhat from specimen to specimen, owing to microstructural imperfections in the specimens, the average values of s_0 and s_c would also be expected to obey Equations 2 and 3, provided sufficient numbers of specimens were used in determining the average values of $s_0(\theta)$ and $s_c(\theta)$ and of $s_0(0)$ and $s_c(0)$. That is, if $s_0^i(\theta)$ and $s_c^i(\theta)$ are the respective values of $s_0(\theta)$ and $s_c(\theta)$ for the ith specimen, then the average values of $s_0(\theta)$ and $s_c(\theta)$ based upon n measurements, are, respectively, $(1/n)\ \Sigma_{i=1}^n\ s_0^i(\theta)$ and $(1/n)\ \Sigma_{i=1}^n\ s_c^i(\theta)$. If $s_0^i(\theta) = s_0^i(0)/\cos\theta$, then

$$\frac{1}{n}\sum_{i=1}^{n} s_0^i(\theta) = \frac{1}{n}\left[\sum_{i=1}^{n} s_0^i(0)\right]\Big/\cos\theta$$

and similarly for $s_c(\theta)$. If for a given value of θ, the quantities $(1/n)\ \Sigma_{i=1}^n\ s_0^i(\theta)$ and $(1/n)\ \Sigma_{i=1}^n\ s_c^i(\theta)$ are essentially independent of n (as would be expected for a sufficiently large number n of tests on similarly prepared specimens), and if Equation 2 holds for individual test specimens, it then follows that this equation also relates the average value of $s_0(\theta)$ to that of $s_0(0)$ (wherein the average value of $s_0(\theta)$ is determined for a group of specimens, each of which is subjected to c-plane shear at an angle θ with respect to the [1 0 0] axis and the average value $s_0(0)$ is measured for a different group of (similarly prepared) specimens, each of which is sheared at $\theta = 0$); similarly for the averages of $s_c(\theta)$ and $s_c(0)$. This suggests that the agreement between the experimental results and the stated hypothesis (that $s_0(\theta)$ and $s_c(\theta)$ are related to $s_0(0)$ and $s_c(0)$, respectively, by Equations 2 and 3 may be improved by additional measurements of s_0 and s_c on a greater number of similarly prepared specimens.

In view of the above considerations, an additional fourteen shear tests were carried out on single-crystal HgI_2 radiation detectors. Actual detectors were selected for the remaining tests because single-crystal specimens that are of high enough quality to serve as radiation detectors are likely to have a more uniform microstructural character than those rejected as detectors; detector fabrication is described by Schieber *et al.* [7] and Lamonds [8]. Of the fourteen tests, nine were in (0 0 1) [1 0 0] shear and five were in (0 0 1) [1 1 0] shear. The average values of s_0 and s_c for these tests also fit Equations 2 and 3 well, as seen in Fig. 3, i.e. the data points labelled (a) (averages for detectors); likewise the averages over all tests (i.e. the six non-detector specimens and the fourteen detectors) also fit these equations (see data points labelled (c) in Fig. 3).

It is of further interest to note that the single-crystal HgI_2 radiation detectors are given a subjective rating (A, B or C, with the detector quality decreasing from A to C) [9] and that the detector quality rating is also considered to be influenced by microstructural imperfections [9]. Thus the results of the measurements of s_0 and s_c for the individual detectors are organized separately according to detector grade and shown in Figs 4 and 5. Fig. 4 shows s_0 for detectors rated A, B, and C sheared in $\langle 1\,0\,0\rangle$ directions ($\theta = 0$) and in $\langle 1\,1\,0\rangle$ directions ($\theta = \pi/4$). Although there is some scatter in the data, it is clear that s_0 is greater for $\theta = \pi/4$ than for $\theta = 0$. For example, seven C

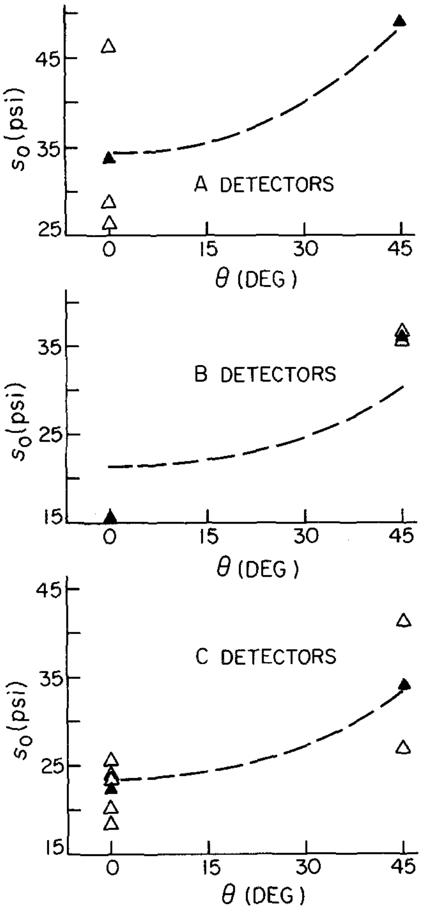

Figure 4 (△) Individual values of s_0 for (0 0 1)[1 0 0] slip ($\theta = 0°$) and (0 0 1)[1 1 0] slip ($\theta = 45°$) for single-crystal HgI_2 radiation detectors of Grades A, B, C. (▲) Average values for each detector grade and slip direction. The theoretical curves are "best fits" of the relation $s_0(\theta) = s_0(0)/\cos\theta$ to the average values. 1/psi = 6.895 kPa.

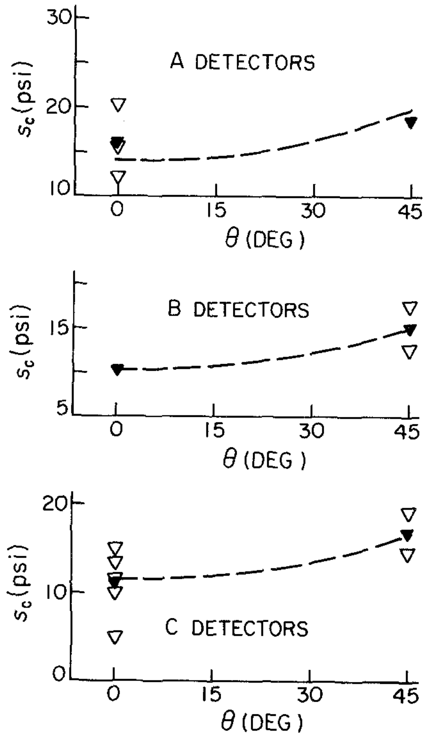

Figure 5 Same as Fig. 4, but with s_c substituted for s_0. 1 psi = 6.895 kPa.

detectors were tested; five were sheared in ⟨1 0 0⟩ and two in ⟨1 1 0⟩ directions; both values of s_0 at $\theta = \pi/4$ are greater than the highest value of s_0 at $\theta = 0$; the average values of s_0 for the C detectors at $\theta = 0$ and $\pi/4$ are described well by Equation 2. Two B detectors were tested at $\theta = \pi/4$ and one at $\theta = 0$; both values of s_0 at $\pi/4$ are considerably higher than the measured value at $\theta = 0$. Three A detectors were tested at $\theta = 0$ and one at $\pi/4$; each of the values of s_0 at $\theta = 0$ is less than the value of s_0 at $\pi/4$; the average values of s_0 for the A detectors at $\theta = 0$ and $\pi/4$ are also described well by Equation 2. Qualitatively similar behaviour is observed in Fig. 5 for the values of s_c of the A, B, and C detectors.

In summary, theoretical considerations suggest that *c*-plane slip in single-crystal HgI_2 should be easiest in a [1 0 0] direction and hardest in a [1 1 0] direction. Measurements of the deformation yielding parameters s_0 and s_c have substantiated this behaviour. The parameter s_0 is interpreted [4] as the shear stress at which either (i) the density of slip planes that are actively slipping has reached half of its steady state value (wherein the steady-state value is that which this density ultimately reaches as the stress s is arbitrarily increased), or (ii) the density of mobile dislocations on (0 0 1) planes has reached half of its eventual steady-state value. The parameter s_c has an analogous interpretation, but with about 2% of the ultimately active slip planes (or alternatively, ultimately active dislocations) participating (currently) in the slip process when the shear stress reaches s_c. The parameter s_c is thus a good measure of the "onset of yielding" whereas s_0 measures "bulk yielding". For (0 0 1)[1 0 0] slip, the average values of s_0 and s_c, respectively, were 24.7 and 12.6 psi (170.3 and 86.9 kPa); for (0 0 1)[1 1 0] slip these respective values were 37.7 and 16.5 psi (259.9 and 113.8 kPa). These average values are in good agreement with the theoretical relations of Equations 2 and 3.

Acknowledgements

The crystals tested in the present study were grown from the vapour phase at EG&G Inc., Santa Barbara Operations; the radiation detectors were prepared and their detection capabilities were evaluated at EG&G. We are grateful to the State of California MICRO program and EG&G Inc. for support of this work (NASA, H-78559B; DOE, DE-AC08-83 NV10282). Particular thanks are due to Mr W. Schnepple, Dr L. van den Berg, Ms C. Ortale and Dr M. Schieber for helpful discussions and invaluable assistance.

References

1. T. W. JAMES and F. MILSTEIN, *J. Mater. Sci.* **18** (1983) 3249.
2. G. GEORGESON and F. MILSTEIN, *Rev. Sci. Instr.* **54** (1983) 1026.
3. F. MILSTEIN and G. GEORGESON, in "Dislocations in Solids: Some Recent Advances", AMD Vol. 63, edited by X. Markenscoff (ASME, New York, 1985) p. 89.
4. G. GEORGESON and F. MILSTEIN, *J. Mater. Sci.* **23** (1988) 3792.
5. G. A. JEFFREY and M. VLASSE, *Inorg. Chem.* **6** (1967) 396.

6. R. W. G. WYCOFF, in "Crystal Structures", 2nd Edn. Vol. 1 (Interscience, New York, 1963) pp. 309–310.
7. M. SCHIEBER, W. F. SCHNEPPLE and L. van den BERG, *J. Cryst. Growth* **33** (1976) 125.
8. H. A. LAMONDS, *Nuc. Inst. Meth.* **213** (1983) 5.
9. M. SCHIEBER, C. ORTALE, L. van den BERG, W. SCHNEPPLE, L. KELLER, C. N. J. WAGNER, W. YELON, F. ROSS, G. GEORGESON and F. MILSTEIN, in Proceedings of 6th International Workshop– Compound Semiconductors for Room Temperature X-ray and Nuclear Detectors, Davos, September, 1987, in press.

Received 27 October 1987
and accepted 3 February 1988