INFLUENCE OF TEMPERATURE UPON DISLOCATION MOBILITY
AND ELASTIC LIMIT OF SINGLE CRYSTAL HgI_2 *

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ABSTRACT

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The practical importance of studying mechanical properties and dislocation structure of HgI_2 is reviewed briefly. Specifically, the performance of single crystal HgI_2 radiation detectors is evidently sensitive to crystalline imperfections; the dislocation structure, in turn, can be altered during detector fabrication, depending upon the mechanical properties of the crystal and the stresses to which the crystal is subjected. The influence of temperature upon dislocation mobility and plasticity in vapor-grown crystals of mercuric iodide is examined. Dislocation mobility is determined by measuring the lengths of the longest arms of dislocation etch pit rosettes on (001) surfaces following microhardness indentation and chemical etch. Measurements were made in the range from room temperature to the phase transition temperature of 127°C . Dislocation mobility was found to be an increasing function of temperature, with the effect accelerating as the phase transition is approached. Increasing temperature was also found to lower the critical resolved shear stress for plastic deformation on slip on (001) planes. In these contexts, the vapor-grown crystals are clearly "softer" at their elevated growth temperatures. The results are discussed in terms of a dislocation model involving "soft" and "hard" glide dislocations.

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Abstract

The practical importance of studying mechanical properties and dislocation structure of HgI_2 is reviewed briefly. Specifically, the performance of single crystal HgI_2 radiation detectors is evidently sensitive to crystalline imperfections, including dislocations. The dislocation structure, in turn, ~~can be~~ ^{may be} altered during ^{crystal growth and} detector fabrication; the nature of such alterations depends upon the mechanical properties of the crystal and the stresses to which the crystal is subjected. This work examines the influence of temperature upon dislocation mobility and plasticity in vapor grown single crystals of mercuric iodide. Dislocation mobility is determined by measuring the lengths of the longest arms of dislocation etch pit rosettes on (001) surfaces following microhardness indentation and chemical etch. Measurements were made at temperatures ranging from room temperature to the phase transition temperature of 127°C . Dislocation mobility was found to ~~be an increasing function of~~ ^{increase} ~~127°C~~ with temperature; with the effect accelerating as the ^{127°C} phase transition is approached. Increasing temperature was also found to lower the critical resolved shear stress for plastic deformation ^{or} slip on (001) planes. In these contexts, the vapor grown crystals are clearly "softer" at their elevated growth temperatures. The results are discussed in terms of a dislocation model involving "soft" and "hard" glide dislocations.

I. Introduction

Electronic properties of semiconductor crystals are known to be very sensitive to crystal defects, including dislocations. No doubt the energy resolution and response uniformity of mercuric iodide detectors to nuclear radiation are dependent upon dislocation density and distribution in the crystal [1], although specific dependencies evidently have yet to be determined quantitatively. In recent years, a number of investigators have studied dislocation structures and crystalline perfection in single crystal mercuric iodide using a variety of experimental techniques.[1-11]

Here we examine the influence of temperature upon dislocation mobility and plastic deformation in vapor grown mercuric iodide crystals. Particular attention is focused upon measurements carried out near the phase transition temperature of 127°C (since vapor grown crystals are formed near that temperature). We present microhardness and compressive yield strength data that demonstrate a considerable softening of the crystals as the phase transition temperature is approached. This work was carried out as part of an ongoing, comprehensive study aimed at characterizing the mechanical properties of single crystal mercuric iodide as they relate to crystal growth parameters, temperature, and dislocation structure. Since mechanical properties such as hardness or yield strength are also highly sensitive to dislocation structure, mechanical properties measurements can be useful in revealing the nature of dislocations in single crystal mercuric iodide. For example, although verification of dislocations that lie in (001) planes evidently has not yet been achieved in vapor grown mercuric iodide crystals by direct observation (i.e. using etch pit formation or related "direct observation" techniques), James and Milstein [11] were

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able to induce their existence and high mobility characteristic from plastic deformation-work hardening behavior. Furthermore, measurements of mechanical properties may provide convenient means of characterizing the crystals (e.g. a low yield stress can imply a low dislocation density). Finally, it is important to determine the influence of deformation upon dislocation structure for the practical reason that internal or external stresses, to which the crystal is subjected during growth or detector fabrication, can induce changes in dislocation structure which in turn can affect detector efficacy.

The technique used here for studying dislocation mobility in mercuric iodide is that of James and Milstein [6]; they developed and used an etchant of 5% alcohol in trichlorethylene (TCE) to reveal dislocation motion under the influence of Knoop microhardness deformation on cleaved (001) surfaces. Their experiments were carried out at room temperature, with a constant indentation load. By etching the (001) face after indentation, they were able to observe arrays of dislocation etch pits emanating from the indentation. The etch pits were square (indicating dislocations perpendicular to the cleaved (001) surface) and their edges were parallel to the $\langle 100 \rangle$ directions. The arrays themselves were also parallel to the $\langle 100 \rangle$ directions.

James and Milstein [11] also carried out uniaxial compression tests on single crystals of mercuric iodide with various crystallographic orientations. For all orientations wherein a shear stress component on the (001) planes was nonzero, the crystals were easily plastically deformed by slip of the (001) planes; furthermore, the (001) planes were the *only* planes to exhibit slip (i.e. the only planes active in the macroscopically observable plastic deformation process). James and Milstein also found this mode of plastic deformation to exhibit work hardening [i.e. the critical resolved shear stress (the stress required to initiate slip of the (001) planes) increased as plastic deformation progressed]. In cases

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where the shear stress on the (001) planes was zero (i.e. when the crystals were loaded either parallel or perpendicular to the [001] direction), under sufficient load, failure occurred by brittle fracture (prior to any macroscopically observed plastic deformation).

From their experiments, James and Milstein [11] induced a dislocation model, for the plastic deformation of mercuric iodide, wherein two types of dislocations play important roles. One type of dislocation glides relatively freely in (001) planes and the movement of these dislocations gives rise to the easy slip of these planes. (Hence these dislocations are called "easy glide" dislocations.) The other type of dislocations, which James and Milstein have termed "hard glide" dislocations, are those observed in the etching of the (001) surface. N891 Although these dislocations are mobile under the relatively high stress caused by a hardness indentation and, as we show here, they become increasingly mobile at elevated temperatures, ^{However} they were not sufficiently mobile to produce observable (macroscopic) deformation in the compression tests. (That is, if these dislocations were sufficiently active during the compression tests, slip would have occurred on families of planes other than (001).) In the model of James and Milstein, the pinning of the easy glide dislocations (as they move on (001) planes) by the hard glide dislocations (that intersect these planes) is responsible for the observed work hardening. (The easy glide and hard glide dislocations thus serve, respectively, as "glissile" and "forest" dislocations during (001) slip, although the hard glide dislocations are glissile during the hardness indentations.)

In the present study we extend the work of James and Milstein [11] by measuring the influence of both temperature and indenter load upon the mobility of the hard glide dislocations (specifically upon the length of the etch pit arrays that emanate from the indentations). In addition, compression tests are

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performed at various temperatures to evaluate the influence of temperature on the yield stress of the material. Since the yield stress is the stress at which the crystal starts to flow plastically, it is a measure of the stress required to move the easy glide dislocations. To the extent that the easy glide dislocations are pinned by the hard glide dislocations, yield stress measurements provide an indication of the strength of the "pinning interaction" between the two types of dislocations.

II. Microstructural Characterization

Samples of seven different vapor grown crystals were made available for possible mechanical testing. Table 1 summarizes the growth conditions and the relative performance of detectors produced from these crystals. Crystal S2-20 was a particularly large, optically clear crystal that was dedicated specifically to mechanical testing. All of the compression tests and most of the hardness tests discussed in this paper were made on samples cut from crystal S2-20. We began by making microstructural examinations of the crystals using both reflected and transmitted light. A Leitz Laborlux 12 HL metallograph with NPL objectives was used. "Reflected light microscopy" consisted of conventional bright field illumination, i.e. light passes through the objective to the object surface, and is reflected back to the objective. In "transmitted light microscopy" illumination from the bottom light source passes through the transparent microscope stage and the HgI_2 sample. The transmitted light technique was particularly useful for examining inclusions and a low angle grain boundary in the interiors of the crystals. Cleaved, platelike crystals ranging in thickness from $150\text{ }\mu\text{m}$ to $450\text{ }\mu\text{m}$ were used as samples. Different patterns of inclusions were observed in different crystals. Also, variations in defect density and characteristics were found from region to region of the same crystal.

Three types of inclusions were observed in this study. The first consists of the bubble-like inclusions (so called because they ^{have} ~~had~~ the appearance of rows of small, dark ^{bubbles} ~~voids~~) seen in Figures 1a,b, which are photomicrographs of the same region of a thin specimen, taken with transmitted light. The focus of the microscope is adjusted to the top surface of the specimen in Fig. 1a and to the bottom surface in Fig. 1b. The inclusions are thus seen to pass through the crystalline sample, from the top to bottom surfaces. Figure 1c is also focused on the same region (top surface) as in Fig. 1a, but here reflected light is employed. The series of Figures 1a-c clearly demonstrates the benefits of using transmitted light when studying inclusions within the crystal. The Miller indices of the axes of the rows of inclusions in Figs. 1a,b were determined to be approximately $[1\ 2\ 1]$ by projecting the $[100]$, $[010]$, and $[001]$ components of the rows of inclusions onto the respective axes of mercuric iodide's unit cell. The $[100]$ and $[010]$ components were measured from the photomicrograph and the $[001]$ component is taken as the sample thickness (since the inclusions pass through the sample). This type of inclusion was observed in crystal S6-10 and S5-6, but was not seen in the other crystals studied in the present work. *The bubbles that comprise these inclusions are probably voids in the crystal.*

The second type of inclusion pattern is seen in Figure 2a. This photomicrograph shows inclusions in the form of short, straight line segments that are aligned with $\langle 100 \rangle$ type directions (and that apparently lie parallel to the (001) planes). Under higher magnification, each segment appears to be made up of a number of small ^{voids} ~~voids~~ in a straight line; these ^{voids} ~~voids~~ were smaller and more closely packed than the ^{"bubbles"} ~~voids~~ in the first type of inclusion pattern. In ~~Fig. 2a~~ 2a, some of these line segment inclusions are not in focus because they are not in "the same cluster" of (001) planes as those in focus. These inclusions tended to lie in particular (001) planes (or in clusters of planes that were close together); they were observed in crystals S4-25, S5-25, S3-31, and S2-20. Figure

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2b shows the same region of the same crystal as in Fig. 2a, but with reflected light; as in the case of the first type of inclusions, the inclusions of Fig. 2a are not evident under reflected light (Fig. 2b).

The third type of inclusion pattern is seen in the photomicrograph of Fig. 3a. This shows inclusions that are in the form of long, straight lines, which exist within the interior regions of the crystal and are parallel to a $[110]$ direction and to the (001) planes. These inclusions appear to be made up of arrays of small black "dots." ^{It is noted} ~~also~~ also that these inclusions are not evident under reflected light (Fig. 3b). A summary of which types of inclusions were found within ~~within~~ ^{each} ~~which~~ of the present crystals is given in Table 2.

In addition to the characterizations of the interior inclusions using transmitted light microscopy, examinations of dislocation etch pits on etched (001) surfaces of cleaved crystals were also carried out with reflected light microscopy. Here again a variety of microstructural features were observed, including ^{randomly distributed} ~~randomly distributed~~ etch pits (as have been reported by previous workers [1-3,5,6,10,11]), intersecting linear arrays of etch pits (as previously observed by James and Milstein [6]), and a few low angle grain boundaries (the nature of which we elucidate with the aid of transmitted light microscopy; see next paragraph for discussion). It is difficult to ascribe a dislocation density to the crystals since significant variations in etch pit formations were found from region to region of the same crystals, although (as with the inclusions) certain etch pit patterns were characteristic of certain crystals. For example, the intersecting linear arrays of etch pits were found only in crystals S6-10 and S2-20; an example of such intersecting arrays is shown in Figure 4.

As mentioned above, transmitted light microscopy was found to be particularly revealing in examining the nature of a low angle grain boundary. Figure 5a

shows an etched (001) face of a 430 μm thick wafer of mercuric iodide under reflected light; a "line" of dislocation etch pits (running from A to A') is readily apparent; such lines of etch pits are generally interpreted as edges of low angle grain boundaries. The grain boundaries themselves are considered to be made up of rows of edge dislocations. Figure 5b is a reflected light photomicrograph of the reverse face of the same sample (which was etched on both "top" and "bottom" faces); the presence of the lines of etch pits on both faces ~~gives good~~ indicates ~~indication~~ that the low angle grain boundary passes through the crystal; but Figures 5a and 5b ~~provide no~~ ^{lack} details about the nature of the boundary in the interior of the crystal. Using transmitted light, however, it is possible to see (Figure 5c) that the low angle grain boundary apparently consists of a row of decorated dislocations, oriented along a $\langle 111 \rangle$ direction in the crystal (their orientation was determined as described previously for the orientation of the bubble-like inclusions). Switching the focus alternatively from the top surface to the bottom surface of the sample (under transmitted light) provides a clear proof that the boundary passes through the sample, from top to bottom. Finally, one can estimate the mismatch θ between the crystallographic orientations on opposite sides of the boundary using the well known formula $\theta \approx b/D$ where b is the magnitude of Burger's vector and D is the distance between neighboring etch pits. This yields a mismatch of about 10^{-4} radians.

III. Dislocation Mobility under Microhardness Indentation

Knoop microhardness indentations were made on cleaved (001) surfaces of single crystal HgI_2 at various loads and temperatures (between room temperature and the transition temperature of 127°C) using a Tukon microhardness tester equipped with a temperature controlled oil bath. Considerable care was taken in the selection of the specific regions of the crystals used for microhard-

ness testing. All of the microhardness tests were performed in regions that were microscopically observed to be free from the inclusions discussed in the previous section. Also, regions containing relatively low densities of "background" dislocation etch pits were sought. In all cases, the samples were oriented with the [110] direction aligned with the long diagonal of the Knoop indenter (this orientation was found to be best for obtaining reproducible hardness and dislocation mobility measurements).

Before doing the elevated temperature tests in the oil bath, a series of Knoop microhardness tests was conducted, in air at room temperature, under a 10 gram indentation load, on (001) surfaces of samples of all of the crystals except S3-31 (which was in short supply). Under these conditions, each crystal had an average value of Knoop microhardness H_k that was in the range of 16 to 18; this result displays a rather remarkable degree of reproducibility for this property. [By definition, $H_k = 14230 p / d^2$, where p is the indentation load in grams and d is the length of the long diagonal (of the resulting indentation) in μm .] Next, a series of room temperature tests was made using indentation loads that varied from 1 to 50 grams, in order to determine the influence of indenter load upon dislocation mobility. For these tests, one piece of the S2-20 crystal was successively cleaved parallel to (001) planes to provide a fresh (001) surface for each successive set of indentations. The sample surfaces were etched before and after each set of indentations. Most indentations produced etch pit arrays that emanated from the indentation along the $\langle 100 \rangle$ type directions. An example of the results of a 10 gram Knoop microhardness indentation at room temperature is shown in Figures 6a,b. The photomicrograph of Fig. ^{6a} shows an etched (001) face of a freshly cleaved vapor grown crystal containing a Knoop microhardness indentation; in this figure, the surface was etched before (but not after) the indentation was made. A random "background" distribution of

etch pits (from dislocations that existed prior to indentation) is evident. Figure 6b shows this same crystal face after re-etching to reveal changes in dislocation structure induced by the indentation. The presence of new etch pit arrays emanating from the indentation is evident. Figure 7 shows an example of dislocation etch pit arrays formed under the highest indentation load of 50 grams. As indicated by comparison between Figs. 6 and 7, the size of the indentation and the number and length of etch pit arrays increases as the indentation load increases.

To evaluate quantitatively the mobility of the dislocations revealed by these etch pits, for each indentation the lengths of the three longest arrays were measured and averaged; this average value is called l . Approximately 20 indentations were made at each load, and the average values of l (i.e. of the average of the three longest arrays) were computed over the population of indentations at each load and plotted versus indentation load in Figure 8; the error bars in this figure represent standard deviations for the population of indentations at each load. Under indentation loads of 1 or 2g, a few individual etch pits were formed on the edges of the indentations, but no etch pit arrays were produced. This indicates that there is a "critical" load in the range of about 2-5g required to produce the arrays of dislocations that emanate from the indentation. The average (taken over indentations) of the three longest etch pit arrays increases linearly with increasing load in the range of 5g to 50g. A similar linear dependence was reported by Tu and Yizhen [12] for silicon crystals.

can argue, as follows, that l should vary,

Theoretically, one might expect l to vary as the square root of indentation load P . ^{First assume} ~~provided~~ that the stress distribution under the indenter, $\sigma(r)$, as a function of position r in the crystal (measured from the tip of the indenter) "scales" according to a dimension of the indentation d ; i.e. $\sigma_1(r_1) = \sigma_2(r_2)$ when $r_2 = d_2 r_1 / d_1$, where σ_1 and σ_2 are the stresses under two different indentation

loads, p_1 and p_2 , and d_1 and d_2 are the respective values of the "lengths" of the resulting indentations. If the values of l scale as σ , they would then be expected to be proportional to d , and if the hardness (which is proportional to p/d^2) is independent of load, l would vary as $\sqrt{p}$. We note that the curve described by the equation $l=34\sqrt{p}$ (in which the coefficient "34" is chosen "arbitrarily" to fit the average value of l at 50g) also passes within the standard deviation error bars in Fig. 8, although the linear fit to the data is better.

Regarding the assumptions upon which the above analysis rests, we mention: (i) the hardness was not found to be strictly load-independent at room temperature, although it was reasonably constant over an appreciable range of loads, as is discussed below; (ii) the "scaling assumption" for stress distributions under differing loads may not hold for mercuric iodide over the range of loads studied here, owing to possible complications caused by fracturing and bending of (001) planes during indentation; and (iii) the assumption that l scales as stress may not be valid owing to complexities involved with dislocation generation near the crystal-indenter interface or dislocation pile-up interactions near the outer extremities of the arrays. Perhaps vastly greater numbers of tests (through which smaller standard deviation bars can be developed) will be able to further resolve the specific functional dependence of l upon p .

Knoop microhardness H_k was also measured as a function of load for indentations on cleaved (001) surfaces of crystal S2-20 at room temperature. The results are summarized in Table 3. In the range of loads between 5 and 25g, the microhardness was fairly constant at about $H_k=18$. However, the values of microhardness measured under lower loads were much higher and appeared to increase rapidly as the load decreased. The higher hardness values at the lower loads are likely associated with the aforementioned absence of dislocations pro-

pagating from the indentations at these loads. That is, at the higher loads, the movement of dislocations away from the indenter evidently allows the indenter to penetrate further, which, in turn, results in a softer Knoop hardness reading than would otherwise occur. The H_k value under the 50g load was somewhat lower; this may have been a result of excessive cracking or fracture of the (001) planes under the higher loads. Since, ideally, hardness should be independent of indenter load, these data suggest that loads in the range of 5g to 25g are most suitable for ^{Knoop} microhardness testing on mercuric iodide single crystals.

Indentations at higher temperatures were carried out in the oil bath under a constant indentation load of 10g using the following procedure. A freshly cleaved sample of crystal S2-20 was affixed to the stainless steel sample holder (using HumiSeal, type 1B15, produced by Columbia Technical Corp.) in order to prevent the sample from sliding ~~after it was placed~~ in the oil. The sample and holder were then placed in the dry bath. After bringing the specimen surface into focus, and selecting the regions of the specimen to be indented, oil was carefully poured into the bath, covering the sample holder and samples, and the ^{assembly} ~~sample~~ was brought to the appropriate temperature. Two Chromel-Alumel thermocouples were used to measure the temperatures of the specimen and the surrounding oil. After temperature stability was achieved, the sample was indented; usually two or three indentations were made on each sample at each temperature. The sample was then removed from the oil bath and rinsed with TCE for 45 seconds in order to remove the oil. This rinse was found necessary, for otherwise the oil leaves a film that prevents microscopic observation of the surface. Finally, the sample was etched with the TCE-ethanol solution and etch pit arrays were observed and measured under the microscope.

Figures 9a,b show the results of 10 gram indentations at 80°C and 120°C, respectively. The etch pit arrays in these figures are considerably longer than those observed after room temperature tests under the same indentation load (Fig. 6b). Indeed, the average value of l (the average of the lengths of the three longest etch pit arrays) is larger for 10 gram indentations at 120°C than for 50 gram indentations at room temperature, although the 50 g indentations themselves are larger and produce more etch pit arrays; compare Figs. 7 and 9b.

Figure 10 shows the results of numerous measurements at temperatures between room temperature and 127°C. The dislocation mobility clearly increases with increasing temperature and the effect accelerates as the 127°C phase transition is approached. At room temperature, the average length of l is under 100 μm . At 120°C, l averages 340 μm . At 127°C, this average rises to over 400 μm . Although the edges of some of the 127°C samples had begun the phase transition, the regions where the indentations were made were ^{visually observed to be} untransformed red phase. The hardness values H_k remained approximately constant for these tests, as can be seen by comparing Figs. 6 and 9. This is an important consideration, since it shows that l is not simply "scaling" as the length of the indentation diagonal d , but is increasing with temperature because of increased dislocation mobility.

IV. Compression Testing Experiments

Among the crystals available for mechanical tests, only S2-20 was sufficiently large to provide more than a few compression test specimens. For this reason, the compression test results reported here are those for samples cut from crystal S2-20. The crystal was first solution cut into five sections or slabs, using a string saw and potassium iodide solution. Samples cut from one

slab were used to gain familiarity with the experimental apparatus and the crystal's behavior. A second slab was then polished flat and parallel using a potassium iodide solution and crystal polisher (South Bay Technology, Inc., Model No. 460). The slab was then solution cut into 21 pieces, eleven of which were suitable for compression test samples. The samples were made small (about 4 millimeters tall with cross-sectional areas approximately 20 square millimeters) in order to conserve material. The samples were solution cut such that the [001] directions were at a 45 degree angle to the loaded faces. This provides a condition of maximum shear stress on the (001) slip planes. In order to minimize end effects, care was taken to maintain flat and parallel top and bottom surfaces on each sample and on the faces of the mandrels that came into contact with the sample during loading.

The compression tests were performed using a specially constructed crystal test apparatus, a schematic of which is seen in Figure 11. This apparatus allowed very slow and controlled loading of the crystal, with precise measurement of both load and deformation. The 5 lb Lebowe load cell enabled stress measurements of ± 0.2 kPa (0.25 psi) to be made on a sample of 12 mm^2 cross section and the Brown and Sharpe electronic indicators, coupled to an X-Y recorder, have a strain measurement capability of 10^{-6} . The compression tests were generated by moving the top mandrel down at a fixed rate (approximately $1 \mu\text{m}$ per second), under the action of a piston which is aligned by a guide bushing, and recording the resulting load and strain on the X-Y recorder.

The test sample and parts of the mandrels were immersed in a temperature ~~controlled~~ ^{controlled} oil bath, which consisted of oil in a pyrex container wrapped with heating wire and insulation. This assembly allowed the graphite mandrels to be aligned with their faces parallel. The graphite mandrels were fabricated in such a

manner that the conduction heat transfer along the mandrel axis was much less than that along the face, thus helping to ensure the temperature uniformity of the sample. ^[11] The temperatures of both the bath and the mandrel adjacent to the mercuric iodide sample were measured using Chromel-Alumel thermocouples (type K); the latter was taken to be synonymous with the sample temperature ~~to~~ within 1°. The apparatus is the same as that used by James and Milstein [11], with the major modifications being the addition of the heating bath and the replacement of a rather temperamental optical extensometer by the more stable electronic indicators.

Figure 12 shows an example of "stress-strain" curves for one of the samples. This sample had been previously work hardened in order to obtain a relatively clear, sharp yield stress. (The "virgin" or "as grown" crystals yield much more gradually.) In the first curve at the left, ^{for} the sample ~~was~~ loaded at 124°C, ~~the~~ the loading is essentially linear elastic for stresses below the ~~critical resolved shear stress (CRSS) of nearly 100 kPa at A;~~ ^{the "point A" is also called} the elastic limit. The ~~CRSS is the maximum shear stress on the (001) planes at the onset of yield; for~~ the samples with the [001] direction at 45° to the loading axis, the CRSS is just one-half the applied stress at the yield point. (We did not observe a preferred direction for slip within the (001) plane.) At point B, the loading was stopped; the recorder pen was lifted from the X-Y chart paper while the crystal was unloading (for clarity). The crystal was then reloaded. The CRSS occurs at the same value of stress (point B) at which loading was stopped on the initial curve (owing to work hardening that occurred during the initial loading). That value of resolved shear stress (i.e. at B), for convenience, is called the *expected* CRSS. At C, the loading was again stopped; this process yields a new *expected* CRSS (at C) ^{next} for the ~~CRSS~~ loading. The temperature of the specimen was then lowered to 90°C. Upon reloading, the crystal yielded at a point D, which is above the *expected* CRSS. This indicates that the crystal is softer at the higher

temperature. A repeat of the loading cycle at 90°C confirms that at a constant temperature, the crystal yields at the *expected* value of CRSS. Next, the temperature was lowered to 74°C. Again, one can see that the actual CRSS (at E) is above the *expected* CRSS.

The results of compression tests on another sample are seen in Figure 13. The first loading curve is at 124 °C, and has its CRSS at A. After unloading (at B), the *expected* CRSS has been raised by work hardening to the value at B. That is, if the crystal temperature were to be held constant, the reload yield stress value would be equal to the *expected* yield stress value at B. Here, however, the crystal temperature was lowered to 30 °C next. Upon reloading, the crystal yields at C, which is well above the *expected* yield stress value at B. After unloading, reheating the crystal to an intermediate temperature of 60 °C, and then reloading, the actual yield stress at E is considerably below the expected yield stress at D, thus demonstrating that the material softens significantly as the temperature is increased.

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This same general behavior was observed in all samples tested in this manner, including several tests in which sample temperatures were successively increased and several involving successive decreases. Table 4 tabulates the results for 5 different specimens; Figures 12 and 13 are for specimens 4 and 2, respectively.

It was not found possible to derive a general, *quantitative* relationship for the influence of temperature difference upon the change in the CRSS, owing to a lack of sufficient repeatability from sample to sample. This problem may be related to the nonuniform distributions of defects discussed in Section II. That is, since the compression test specimens are relatively large, different speci-

mens may have had different inclusion or dislocation structures in different regions of the specimen. ~~being to work hardening that occurred~~ [By contrast, in the hardness tests, it was possible to select regions of the specimen (for indenting) that were free from inclusions and relatively free of dislocation etch pits on (001) surfaces (since the hardness indentation is very localized)]. Nevertheless, without exception, when the temperature was lowered, the observed value of yield stress of the work hardened specimens was higher than the expected value. Similarly, when the temperature was raised, the observed value fell below the expected value. This is consonant with the results of the hardness tests, which showed an increased dislocation mobility at elevated temperatures.

A possible explanation for the decrease in yield stress with increasing temperature can be given in terms of the increased mobility of the hard glide dislocations. It can be supposed that as slip along the (001) planes proceeds, the easy glide dislocations pile up on the hard glide dislocations. If the yield stress is considered to be the stress at which the easy glide dislocation pile-ups actually cause the hard glide dislocations to begin slipping, the increased mobility of the hard glide dislocations at the higher temperatures could allow the "easy glides" to push the "hard glides" at lower stress values. On the other hand, the yield stress is no doubt a complicated phenomenon, and the stress, at which the easy glide dislocations become "unpinned from," or move past, the hard glide dislocations, is likely to be important. Thus, the softening of the yield stress can also result from the influence of temperature upon this "unpinning stress."

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List of Figures

- 1a Bubble-like inclusions are seen with transmitted light. The microscope is focused on the top surface [a (001) face] of a freshly cleaved (unetched), 200 μm thick wafer of crystal S6-10. Cleavage steps ~~present~~ on the top and bottom surfaces provide convenient points of reference for comparison with Figs. 1b and c.
- 1b Photomicrograph focused on the bottom surface of the same specimen as in Fig. 1a, and in the same region; taken with transmitted light.
- 1c Photomicrograph of the same region as in Fig. 1a, observed with reflected light. The bubble-like inclusions seen with transmitted light in Figs. 1a,b are not evident under reflected light.
- 2a Inclusions in the form of short "line-segments" that are parallel to the (001) plane and $\langle 100 \rangle$ directions. The photomicrograph is taken with transmitted light and the focus is near the top surface of the crystal. The sample is a freshly cleaved (unetched), 150 μm thick wafer of crystal S5-25.
- 2b Same region of same specimen as in Fig. 2a, but with reflected rather than transmitted light. (The focus is on the top surface of the crystal.)
- 3a Inclusions in the form of dark lines that are parallel to the (001) plane and [110] direction. The focus is in the interior of the crystal; transmitted light is employed; the sample is an etched 180 μm thick wafer of crystal S4-25. The bright crosses (also present in Fig. 3b) are dislocation etch pits.
- 3b Etched surface of crystal S4-25 (same region as in Fig. 3a, but in reflected light, focused on the surface).

- 4 Intersecting linear arrays of etch pits on an etched (001) surface of crystal S6-10.
- 5a Etched (001) face of a 430 μm thick wafer of crystal S3-31; the photomicrograph is taken with reflected light. The edge of a low angle grain boundary (passing from **A** to **A'**) is demarcated by etch pits that form where the dislocation lines that comprise the low angle grain boundary emerge at the (001) surface.
- 5b Etched (001) face of the reverse side of the specimen of Figure 5a, under reflected light; the low angle grain boundary is seen passing from **B** to **B'**.
- 5c Photomicrograph of the same region as shown in Figure 5a, but with transmitted light. Here both the top edge (running from **A** to **A'**) and the bottom edge (**B** to **B'**) of the low angle grain boundary are visible simultaneously. The grain boundary is seen to be made up of what are evidently decorated dislocations, which appear as "dotted lines," each of which passes from the top surface (from a point on the trace **AA'**) to the bottom surface (at a point on **BB'**), and oriented at about 45° to an edge of the figure.
- 6a Knoop microhardness indentation made on a pre-etched (001) surface at room temperature under a 10g indentation load.
- 6b Etching of the specimen shown in Figure 6a (i.e. etching after indentation) reveals linear arrays of dislocation etch pits emanating from the indentation.
- 7 Dislocation etch pit arrays emanating from an indentation performed at room temperature under a 50 g indentation load on a (001) surface.

- 8 Dislocation etch pit array length l versus indentation load p at room temperature for Tukon microhardness indentations on (001) surfaces of single crystal HgI_2 . Each datum point represents an average value of l for about 20 indentations.
- 9a Dislocation etch pit arrays emanating from an indentation performed in a 80°C oil bath under a 10 g indentation load on a (001) surface.
- 9b Same as Fig. 9a, but in a 120°C oil bath.
- 10 Dislocation etch pit array length l versus temperature T for 10 g Tukon microhardness indentations on (001) surfaces of single crystal HgI_2 . Each datum point represents the average for about 10 different indentations.
- 11 Compression test apparatus.
- 12 Resolved Shear Stress versus cumulative strain for a pre-work hardened single crystal specimen of HgI_2 at three different, descending temperatures; the yield stress increases as the temperature decreases.
- 13 Resolved Shear Stress versus cumulative strain for a pre-work hardened single crystal specimen of HgI_2 ; the yield stress increases and decreases, respectively, as the test temperature is lowered and raised. Temperature is also seen to have an influence on the rate of work hardening.

Table 1 Growth data and detector grade for crystals.

Crystal Number	Growth rate (mm/day)	Material*	(001) plane angle to pedestal	Number of detectors** in the grade of			
				A	B	C	D
S6-10	-	30x	90°	-	-	-	-
S5-6	1.5	30x+Te	10°	-	-	-	-
S4-25	0.36	30x+I ₂ M+30x	90°	-	4	14	2
S5-25	0.63	90x+I ₂ M+30x	0°	-	-	4	-
S3-29	0.3	90xMS+S	90°	-	-	4	-
S3-31	0.82	120xMS	90°	-	-	4	-
S2-20	0.31	120xMS	10°	-	-	1	-

*30x: open tube sublimation (once)
 90x: open tube sublimation (three times)
 120x: open tube sublimation (four times)
 M: melted
 S: closed tube sublimation
 I₂M: iodine melt
 Te: Tellurium is added

** detector grade: A: excellent
 B: good
 C: Average
 D: poor

Table 2-- Summary of Observed Inclusions

The symbol X denotes the presence of the particular type of inclusion. For each case where an inclusion is indicated, the inclusions were not present homogeneously throughout the entire crystal; in particular, each crystal also contained ~~some~~ regions that were free from such inclusions.

Crystal Number	Bubble-Like Inclusions Along [1 2 1] Direction Figure 1a,b	Line-Segment Inclusions Along <100> Directions Figure 2a	Linear Inclusions Along [110] Direction Figure 3a
S6-10	X		
S5-6	X		
S4-25		X	X
S5-25		X	
S3-29			
S3-31		X	
S2-20		X	X

Table 3 Knoop microhardness on the cleaved (001) surface of HgI_2 under various loads of indentation.

p (g)	d (μm)	H_k
1	20	35.5
2	31.5	28.7
5	62	18.5
10	89	18.0
25	144	17.2
50	220	14.7

p: indentation load.

d: average length of long diagonal of Knoop indentation.

H_k : Knoop microhardness (each value in the table represents the average of approximately 20 tests).

Table 4		
Temperature Effects on CRSS		
Sample	Temperature Rise Between Successive Tests, °C	%Change from Expected Value
1	30° to 60°	-6.5%
2	30° to 60°	-13%
3	30° to 80°	-4%
	80° to 120°	-6%
	Temperature Drop Between Successive Tests, °C	
4	124° to 90°	+26%
	90° to 74°	+10%
	74° to 39°	+6%
2	124° to 30°	+20%
5	70° to 50°	+7%

Figure 1a

S-216-TP

Figure 1b

S-216-TP

Figure 1c

S-216-TP

Figure 2a

S-216-TP

Figure 2b

S-216-TP

Figure 3a

S-216-TP

Figure 3b

S-216-TP

Figure 4

S-216-TP

Figure 5a

S-216-TP

Figure 5b

S-216-TP

Figure 5c

S-216-TP

Figure 6a

S-216-TP

Figure 6b

S-216-TP

Figure 7

S-216-TP

Figure 8

S-216-TP

Figure 9a

S-216-TP

Figure 9b

S-216-TP

Figure 10

S-216-TP

Figure 11

S-216-TP.

Figure 12

Figure 13