GENERAL ATOMIC

DIVISION OF GENERAL DYNAMICS

GA-3376

# A PROGRAM OF BASIC RESEARCH ON MECHANICAL PROPERTIES OF REACTOR MATERIALS

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QUARTERLY PROGRESS REPORT

FOR THE PERIOD ENDING JUNE 30, 1962

Contract AT(04-3)-167
Project Agreement No. 4

U.S. Atomic Energy Commission

July 25, 1962

## DISCLAIMER

**GENERAL ATOMIC**

DIVISION OF

**GENERAL DYNAMICS**

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## PREVIOUS REPORTS IN THIS SERIES

GA-2502—April, May, June, 1961

GA-2691—Summary Report, January through September, 1961

GA-2848—October, November, December, 1961

GA-3076—January, February, March, 1962

# I. DEFORMATION MECHANISMS IN BCC METALS

## DEFORMATION AND FABRICATION CHARACTERISTICS OF REACTOR MATERIALS

### Tension Experiments

Past work on bcc metals has revealed significant differences in both yielding and flow behavior between room temperature and 77°K. The well-known sharp increase of yield or flow stress with decreasing temperature is an indication of a change in the mechanism of deformation. Current speculations concerning the yielding of bcc metals at low temperatures describe the phenomenon in terms of various dislocation interactions.[1][2]

Results obtained from slip-line studies in this investigation have also revealed large differences in the type of slip-band pattern observed for specimens tested at 295°K and at 77°K. A higher density of slip lines is observed for the room-temperature tests; in addition, for the particular orientation of the single-crystal samples used, abundant conjugate slip is also observed at room temperature. These results indicate in a qualitative manner that sources generate dislocations more easily at room temperature than at 77°K.

In view of the difference in behavior, the effect of modifying the dislocation structure by room-temperature prestraining on the subsequent yielding behavior at 77°K is being studied.

The tantalum single crystals were obtained from Materials Research Corporation and have an orientation about in the middle of the stereographic triangle.[3] These crystals have a residual resistivity ratio, $\Gamma (R_{300^\circ K}/R_{5^\circ K})$, of about 300 and normally exhibit a slight yield-point effect during a room-temperature test. The single crystals are generally tested in the annealed condition at 77°K to a strain of not more than $250 \times 10^{-6}$ in order to provide a base line for comparison with further tests. Following this test, they are given a room-temperature prestrain and immediately tested at 77°K again.

Figure 1 illustrates the observed behavior of sample Ta-5s for a room-temperature prestrain of 3%. As can be noted, the effect is quite large and indicates that a considerable decrease in the early yield stress for a given strain is effected by prestraining at room temperature.

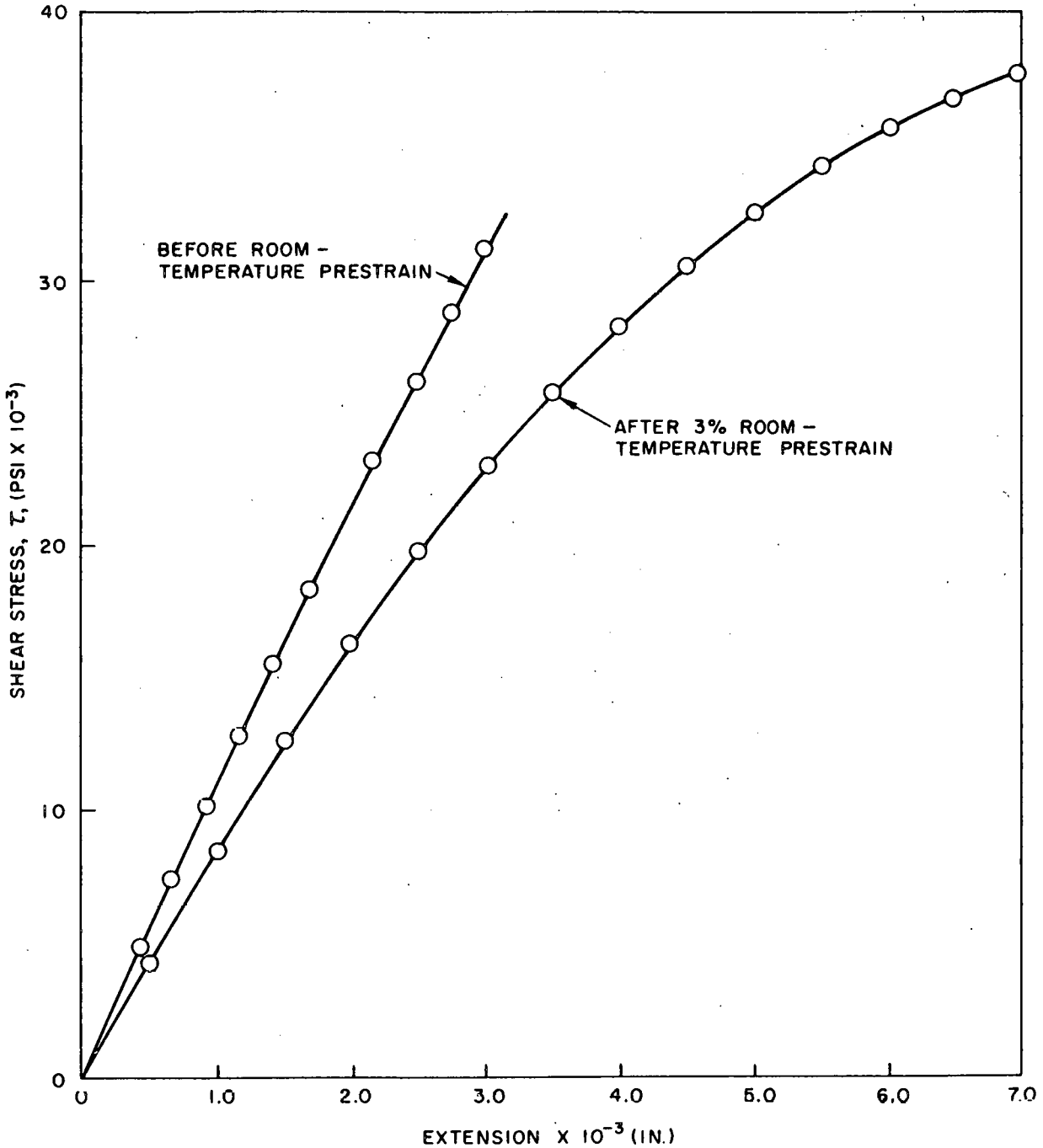


Fig. 1--Stress-strain curves for tantalum single crystal showing effect of room-temperature prestrain on yielding behavior at 77°K

The effect appears to reach a maximum at 3% to 6% prestrain and decreases thereafter. Experiments are in progress to obtain more accurately the prestrain effect.

Slip-line Studies

In addition to the above observations on Ta-5s, metallographic studies were made after ~10% strain. During the initial 77°K straining a series of drops in flow stress were observed (see Fig. 2) accompanied by audible clicks, which are indicative of twinning. Figure 3 is a representative area of the crystal surface near the minor axis of the slip ellipse. In this region, crystallographic slip is not occurring on a scale gross enough to be resolved at 1000×. Of particular interest is the twin in the middle of the micrograph, with the profusion of slip emanating from the sides of the area of accommodation. These slip lines, along with the slip trace initiated at the terminus of the twin, start in an apparently unfavorable orientation, as a shift to a more favorable direction is accomplished in a short distance. This type of slip initiation in a bcc metal has been observed by Hull[4] in a thin crystal of $\alpha$ Fe.

More detailed metallographic observations have been made on Ta-4s, which has two flat surfaces at 90°, parallel to the tensile axis. The first deformation markings appear to be fine twins (see Fig. 4) and occur as early as 0.01% strain. These markings have the usual twin characteristics of limited length, sharp terminations, and straightness; also, after additional straining, the difference in appearance of the twins and the slip traces is quite evident. In addition, subsequent chemical polishing results in the twins persisting after all vestiges of the slip traces have been removed.

These observations of twinning in tantalum at 77°K and normal rates of strain ($\dot{\epsilon} = 10^{-4}$/sec) are, to the authors' knowledge, unique, as the only other evidence of twinning in tantalum has resulted from hammering at 77°K[5] and at room temperature.[6] The greater ease of twinning in the current work may be due to the lower impurity content (<100 ppm) of the electron-beam zone-melted crystals used.

Figure 5 shows two adjacent areas along the same edge of Ta-4s after it had been inadvertently bent and then strained at 77°K sufficiently to be straightened. A twin is evident in Fig. 5a, while Fig. 5b shows evidence of slip. Each slip line can be seen to be made up in part of short crystallographic segments, as observed on previous crystals. This area will be replicated for more detailed study by electron microscopy. Further straining combined with optical and electron microscopy will be undertaken to ascertain in more detail the point of onset of twinning and slip and the systems operating.

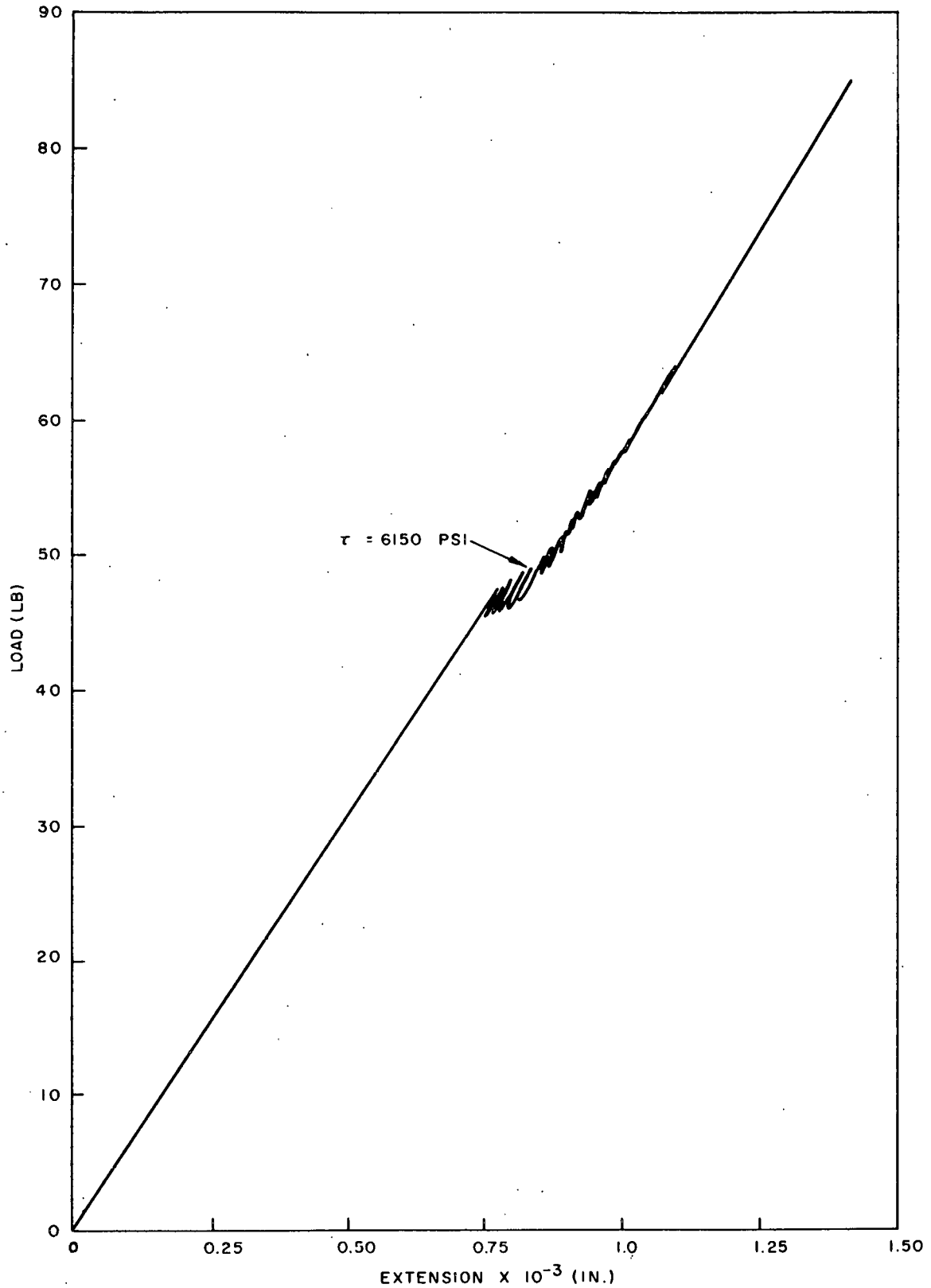


Fig. 2--Load-elongation curve for tantalum single crystal Ta-5s showing serrations indicative of twinning

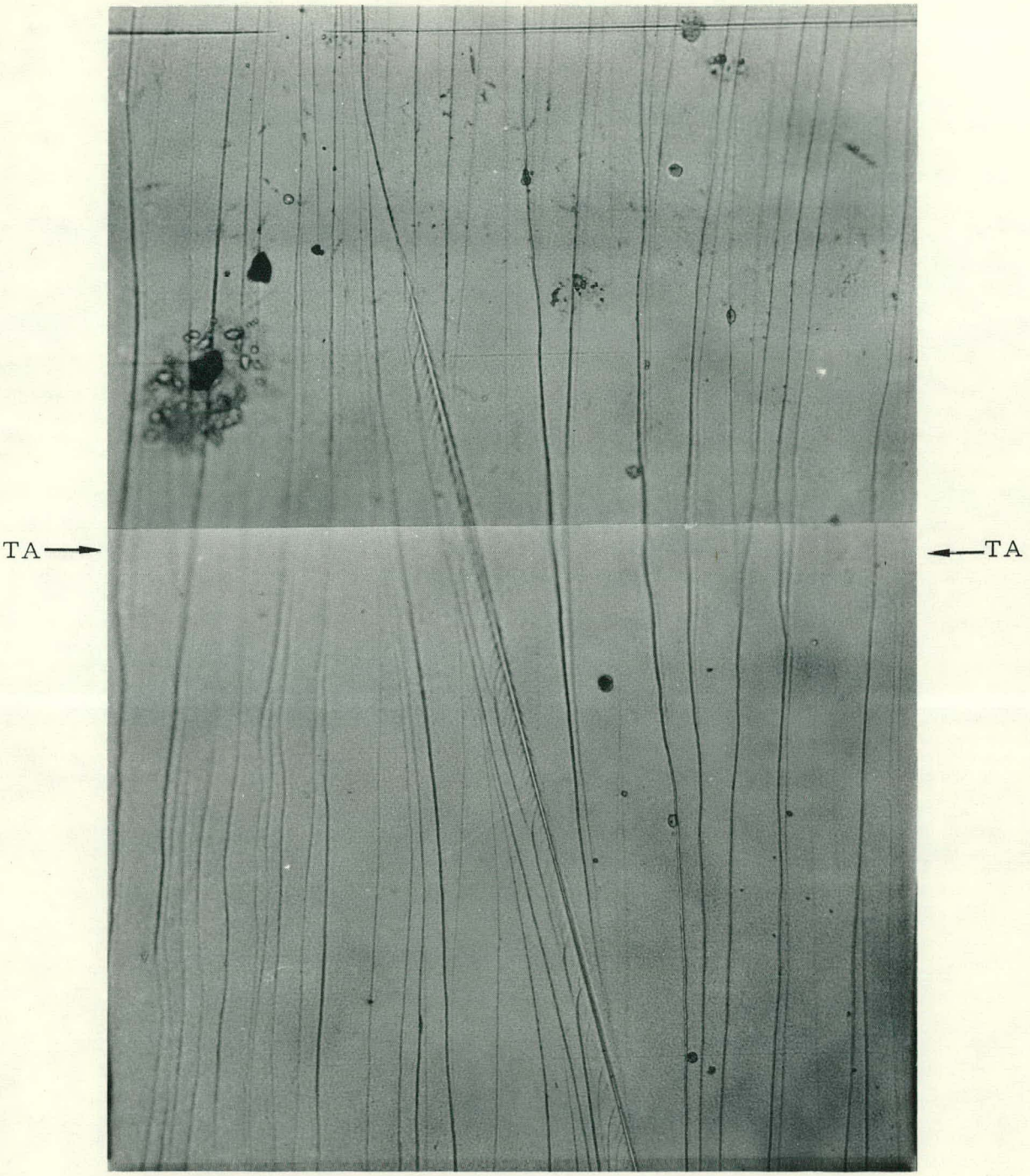


Fig. 3--Photomicrograph of Ta-5s after straining represented in Fig. 2

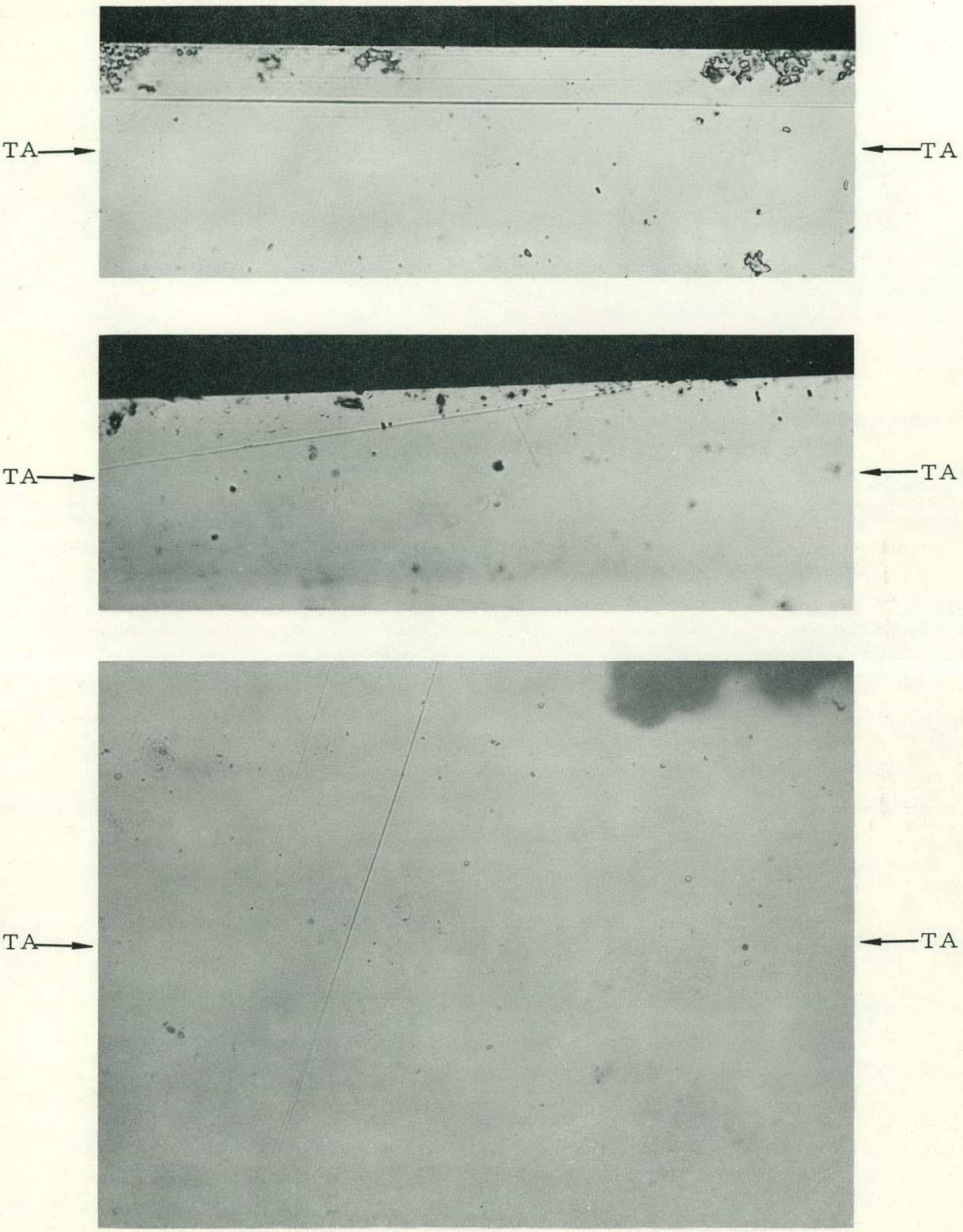


Fig. 4--Photomicrographs of Ta-4s after 0.01% strain

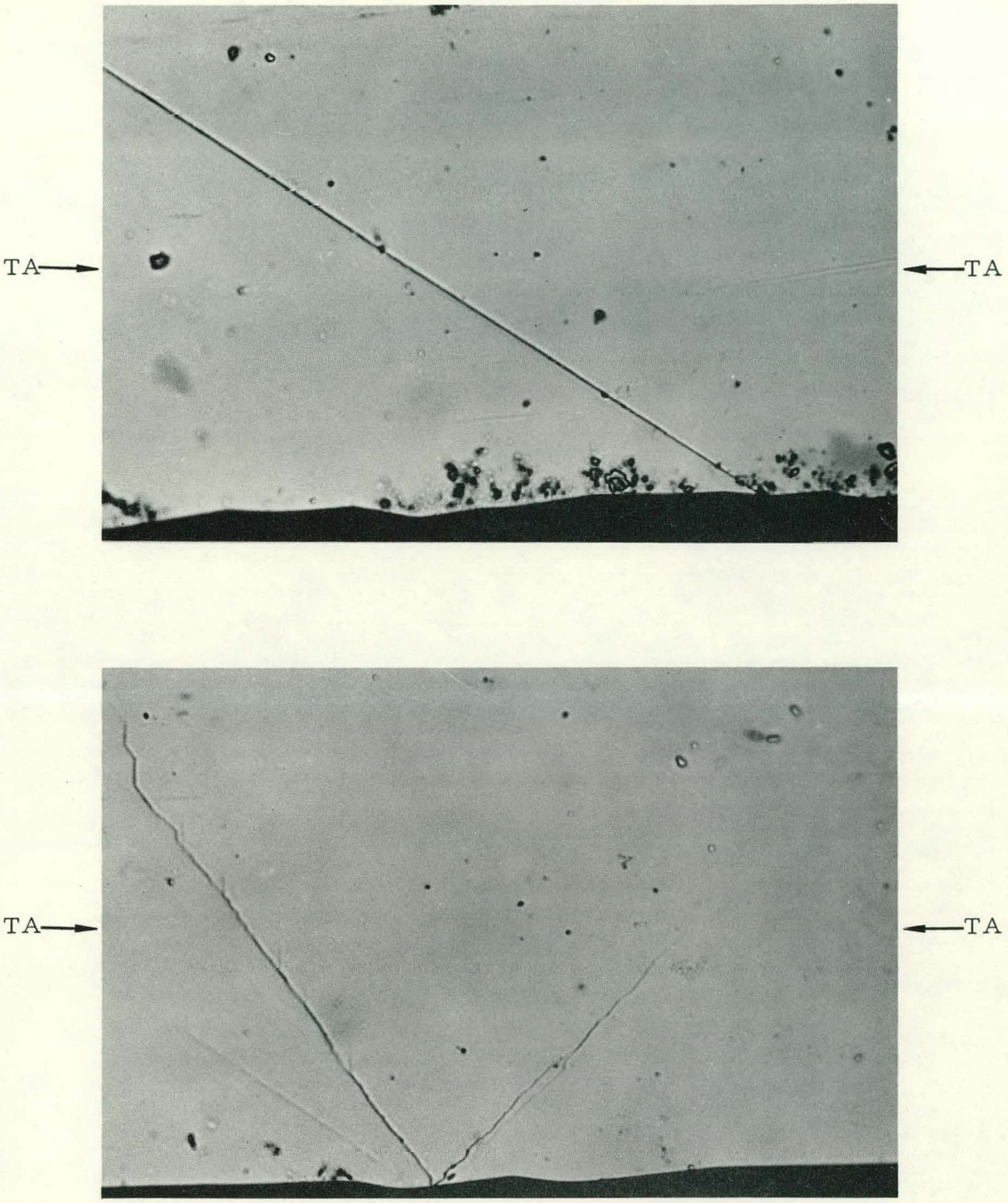


Fig. 5--Photomicrographs of adjacent areas of Ta-4s after inadvertent bending followed by straightening at 77°K

## DISLOCATION RELAXATION SPECTRA AND THE MEASUREMENT OF THE PEIERLS POTENTIAL IN BCC TRANSITION METALS

One of the most important questions in the physics of plastic deformation is whether there exists an intrinsic potential barrier (the Peierls potential) to dislocation motion in crystalline solids. To date all theoretical attacks on this problem have been frustrated by the lack of detailed knowledge of the atomic structure and interatomic forces associated with the region of maximum misfit in the dislocation--the "core" of the dislocation. Until more sophisticated theoretical methods are developed it appears that this question will be answered only by using whatever experimental "handles" may exist. Unfortunately, it has proved very difficult to obtain unambiguous proof that what is being measured is indeed the intrinsic lattice resistance to dislocation motion. This section will attempt to show how this potential can be measured in relatively pure bcc transition metals such as niobium, tantalum, molybdenum, and tungsten. Furthermore, it is thought that the experiment described below demonstrates that the intrinsic lattice potential is being measured and not the resistance to dislocation motion caused by an impurity interaction. Curiously enough, an impurity effect (pinning of dislocations) is very instrumental in making the measurement.

Historically, one of the earliest attempts to measure the Peierls potential was by means of the internal-friction phenomenon known as the "Bordoni peak." A number of workers[7-11] have constructed several dislocation models designed to explain this internal-friction relaxation peak (Bordoni peak[12]), which appears with plastic deformation of fcc metals. In these models the peak is considered to result from the thermally assisted jumping of segments of dislocation lines between wells separated by a potential barrier which is both periodic and intrinsic to the lattice (Peierls potential[13]).

It will be shown below that the temperature dependence of the amplitude dependence of the Bordoni peak of the fcc metals copper and aluminum is inconsistent with predictions based on these models, whereas data presented below on the temperature dependence of the amplitude dependence of the deformation peaks[14] in bcc metals <u>are</u> in accord with these predictions.

The premise used in the following observations is the acceptance of the class of models[15, 16, 17] for amplitude-dependent internal friction based on the hysteretic unpinning and repinning of dislocation loops from impurity pinning points when the oscillating measuring stress exceeds a certain value, the breakaway stress; with the application of any of the aforementioned relaxation processes to the pinned dislocation model, the mobility of the loop lying between pinning points becomes strongly temperature dependent in the vicinity of the temperature of the peak. Thus, at temperatures above the peak the loop can move unimpeded by the lattice;

and if the oscillating measuring stress is of sufficient magnitude, the loop can pull free of the impurities and be recaptured on the return cycle, thus producing hysteretic amplitude-dependent internal friction. On the other hand, at temperatures below the peak, the loops are "frozen" behind the lattice potential barriers and therefore are unable to contribute to hysteretic internal friction. Thus, as the temperature is lowered through the region of the peak, the amplitude-dependent internal friction should, according to these models, show a relatively sharp drop, the size of which should depend on the dislocation density and the network length associated with a given peak.

Figure 6a shows the combined $\alpha$ and $\beta$ deformation peaks[14] in plastically deformed high-purity tantalum. The specimen was first deformed at 300°K to 50% torsional strain, and then measurements were made at 6 cycles/sec at various oscillating shear strain amplitudes. In Fig. 6b is plotted the amplitude-dependent internal friction, $\Delta Q^{-1}$, obtained by subtracting the amplitude-independent curve, represented by the measurement made at an amplitude of $1 \times 10^{-7}$, from the other curves in Fig. 6a. Note that as the temperature is lowered, $\Delta Q^{-1}$ for all amplitudes decreases monotonically, with a particularly sharp drop occurring in the vicinity of the group of $\beta$ peaks near 160°K; another smaller drop is just resolvable in the region of the group of $\alpha$ peaks (120°K). Analogous results have been obtained for niobium, molybdenum, and tungsten and will be reported elsewhere. The deformation peaks of the refractory bcc metals thus exhibit the temperature dependence of the amplitude-dependent internal friction predicted by the aforementioned models.

In Fig. 7a the data of Paré[18] show the amplitude dependence of the Bordoni peak of copper. In Fig. 7b is plotted $\Delta Q^{-1}$ taken from the data in Fig. 7a. As the temperature is lowered, $\Delta Q^{-1}$ is seen to rise in the vicinity of the Bordoni peak, contrary to the prediction mentioned above. This behavior is apparently not confined to copper alone, since recently published data[19] on the Bordoni peak in 99.995%-pure aluminum show a similar anomaly in the temperature dependence of $\Delta Q^{-1}$.

Thus, if there are dislocations in copper and aluminum which can move only with the aid of thermal activation, they are either (1) pinned as discussed above but their numbers are less than ~1% of those dislocations that interact only hysteretically with impurities, (2) able to remain free of pinning points, or (3) pinned as discussed above but restrained by elastic internal stresses from producing breakaway from pinning points. It is suggested that (3) is the most probable alternative in view of the existence of the anomalous rise in $\Delta Q^{-1}$ with decreasing temperatures. A particularly simple example of such an elastically bound, thermally activated defect is Gilman's edge dislocation dipole with a separation of one or two lattice spacings. The activation energy for jumping should be determined by its

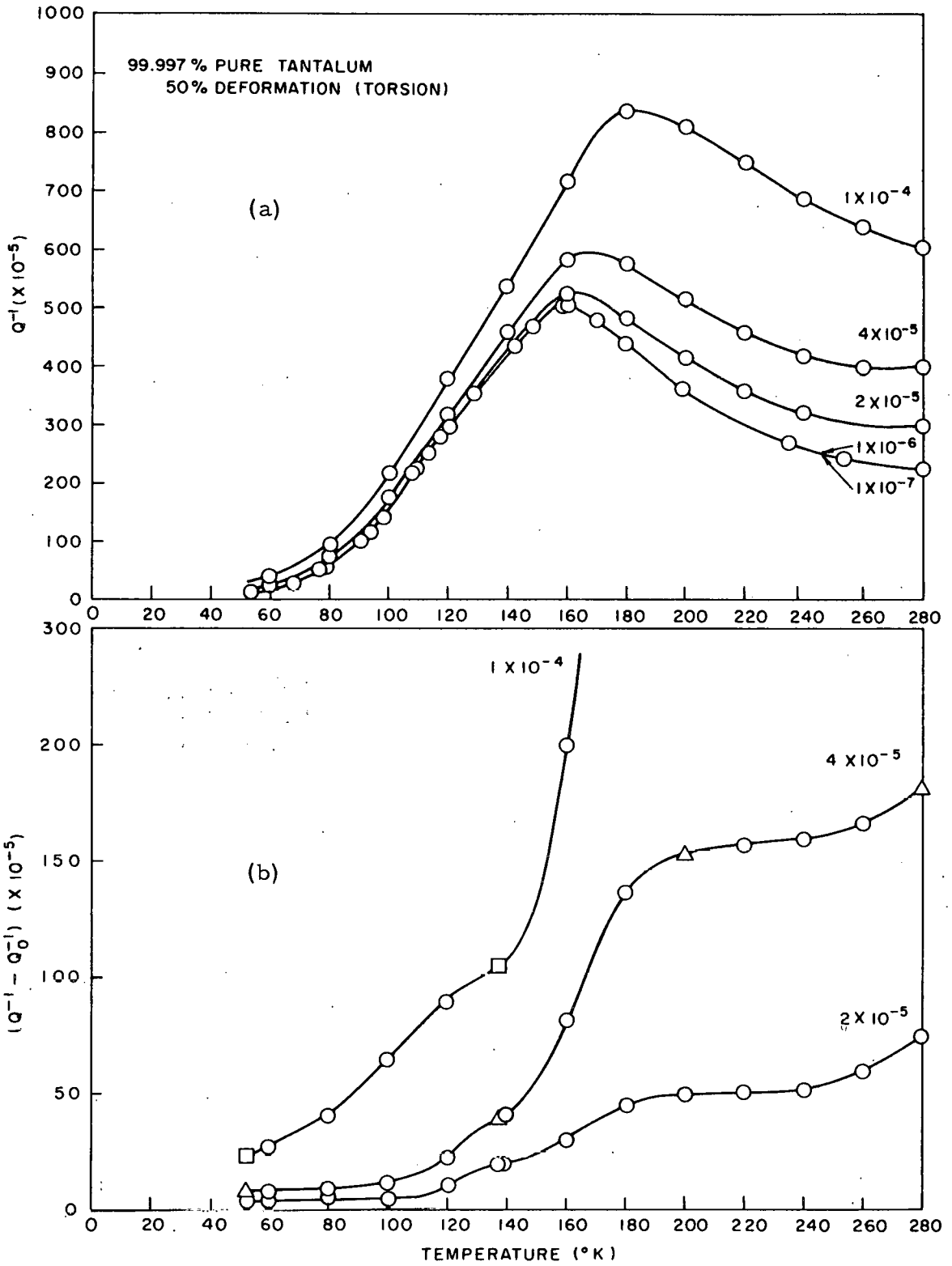


Fig. 6--(a) Internal friction, $Q^{-1}$, versus temperature for 99.997 at.-% pure Ta deformed to 50% in torsion at 300°K; measurements made at 6 cps at various shear strain amplitudes. (b) Amplitude-dependent internal friction, $Q^{-1} - Q_0^{-1}$, versus temperature for various amplitudes, from data in (a)

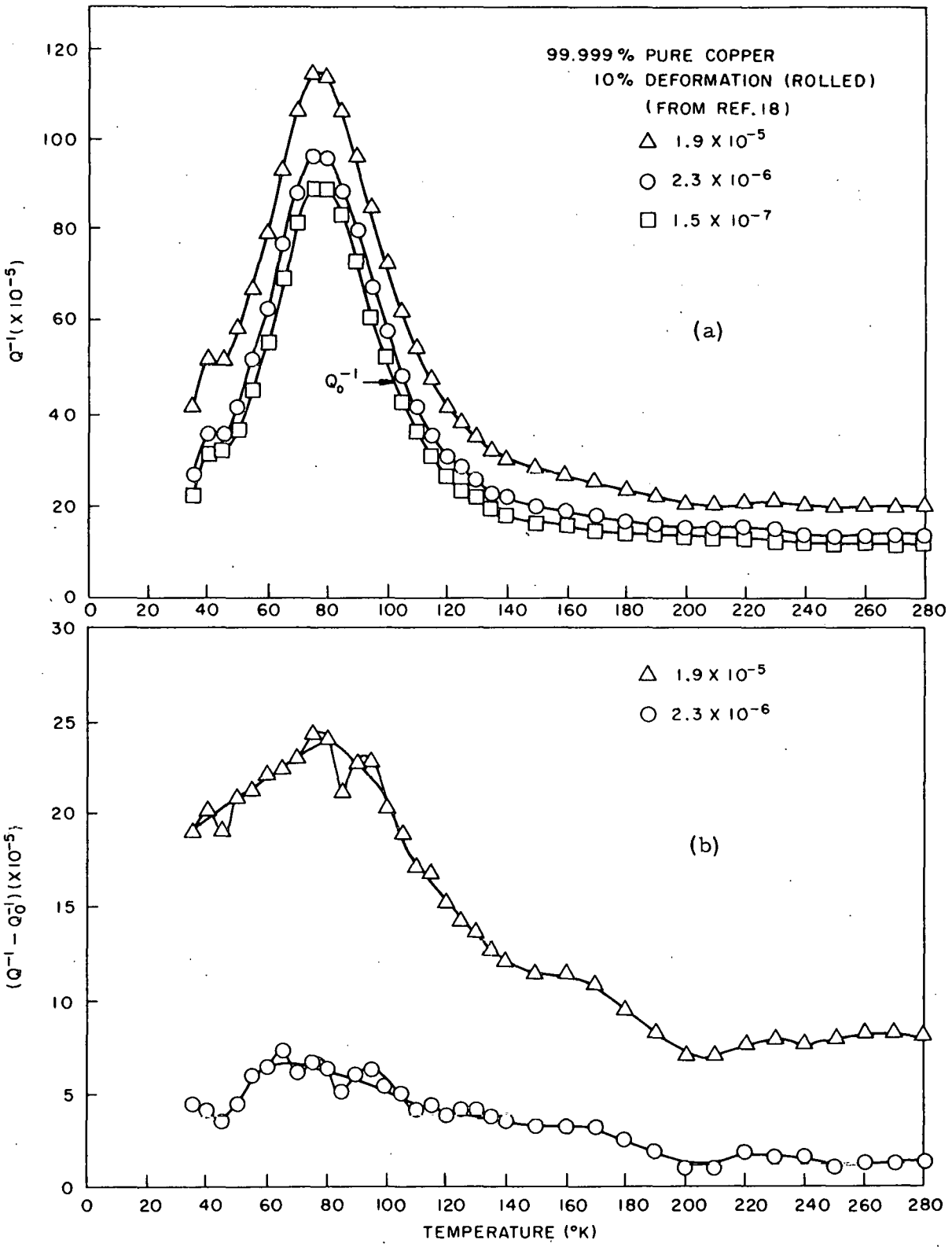


Fig. 7--(a) Internal friction, $Q^{-1}$, versus temperature for 99.999 at.-% pure Cu (from data of Ref. 12) deformed to 10% by rolling at 300°K; measurements made at 5500 cps at various strain amplitudes. (b) Amplitude-dependent internal friction, $Q^{-1} - Q_0^{-1}$, versus temperature for various amplitudes, from data in (a)

self-interaction energy.* Furthermore, the stress necessary to separate the arms of the dipole the distance required to produce breakaway should be much larger[20] than the stress employed in these measurements; therefore, the dipole should not be able to contribute to the hysteretic internal friction.

If these observations are valid, previous estimations[8] of the magnitude of the Peierls potential for copper and aluminum based on the Bordoni peak measurements would not be meaningful; in fact, the apparent lack of a peak with the required temperature dependence of $\Delta Q^{-1}$ suggests that the Peierls potential for both copper and aluminum is very small indeed. On the other hand, at least two different values of the Peierls potential corresponding to the activation energies[8] associated with the $\alpha$ and $\beta$ deformation peaks appear to exist in the bcc transition metals.

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*The author wishes to thank Professor Gilman for communicating his preliminary report dealing with the estimation of the activation energy for the flipping of the dipole.

## II. THE STORED ENERGY OF COLD WORK

The major aim of the current work is to extend the method of differential calorimetry to measurements of stored-energy-release spectra in deformed bcc metals. In view of the apparent discrepancies and inconsistencies in the past record of stored-energy measurements,[21, 22, 23] attention was given during the period of calorimeter development to problems concerning the stored energy of cold work in copper, which has been the most frequently studied metal. The results have provided some resolution, particularly in respect to the effects of grain size, strain, and impurities, and afford useful guidance for the investigations on the bcc metals, for which stored-energy-release data are practically nonexistent.[21]

### EXPERIMENTAL WORK

Some preliminary experiments on cold-worked iron, described in the last quarterly report,[3] indicated that the method of quantitative differential thermal analysis was applicable to stored-energy measurements on iron but that more refractory materials were required for the calorimeter block. Accordingly a calorimeter block of approximately cubic geometry was designed to permit the use of straight lengths of molybdenum or molybdenum-alloy wire distributed uniformly over the external surface of the block. Straight lengths of the resistor element are used to minimize the ductility required in the wire. The present block is made of OFHC copper; when all design details are settled and proved by experiment, a molybdenum block will be constructed.

Alterations in the power control system[24] have been made to accommodate the rapid change in resistance of the molybdenum heater with temperature. Some further tests are required to optimize the control parameters. Advantage of this opportunity is being taken to make improvements in the low-level dc circuitry[24] to minimize electronic contributions to base-line disregistry and to ensure stable calibration of the differential thermocouple signals. All bucking potentials are being placed in the preamplifier circuits, and thermal-free switches are being incorporated in order to permit direct checks of signals by the K-3 potentiometer with minimum delay.

A preliminary calibration experiment, using a lead calibration specimen similar to that previously employed,[24] has been run to test the

molybdenum-wound block and to determine the thermal intercoupling constant[3] for the new block design. The data appear satisfactory, and the data reduction will be made by existing computer programs.

A vacuum annealing furnace is being constructed to enable heat treatments of calorimeter specimens of bcc metals to temperatures of the order of 1600°C with good control over the furnace atmosphere. The furnace consists of a molybdenum sheet resistor; the amount of ceramic material is minimized to reduce degassing problems. An activated-alumina isolation trap of recent design[25][26] has been constructed for the vacuum system to provide good vacua over long periods with little attention from the operator. The furnace design includes means for temperature programming and specimen quenching in order to obtain metallography specimens representative of various points on the stored-energy-release curve. Observations of microstructure by conventional optical methods as well as of dislocation configurations by electron transmission microscopy are envisaged.

## STORED-ENERGY-RELEASE SPECTRA FOR COPPER

In previous reports the stored-energy-release data were generally presented in the form of the Q' plotting function,[3] since a direct indication of the base-line registry was given and the details of the release spectrum were qualitatively apparent. The development of the cell technique, in which the thermal effect is diluted by the heat capacity of the cell, as well as the interest in quantitative comparisons for interpretive purposes, has made it desirable to use the stored-energy-release spectrum (SERS) in the form

$$\mathrm{SERS} = -\frac{1}{c_s}\frac{dE_s}{dT_s} = \left(\frac{\sum_s n_i c_i}{n_s c_s}\right)\left(Q' - \beta'\right)\left(1 - \frac{T_s - T_r}{T_w - T_r}\right)\left(1 - \frac{d(T_s - T_r)}{dT_s}\right), \quad (1)$$

where $-dE_s/dT_s$ is the stored-energy release taking place at the specimen temperature $T_s$; $c_s$ is the heat capacity of the specimen, and the symbols on the right-hand side are as defined in the last quarterly report.[3] Accordingly, a computer program was developed for routine computation of the SERS as well as the total stored energy, and representative SERS for the previous work on high-purity copper and two lots of OFHC copper are given in Figs. 8, 9, and 10. In order to avoid suppression of the base-line problem, the calculations are made with smoothed values of the $\beta'$ function for the first base-line run, and any adjustments of the base-line positions are directly represented on the SERS plots.

Several points may be derived from the release spectra of Figs. 8,

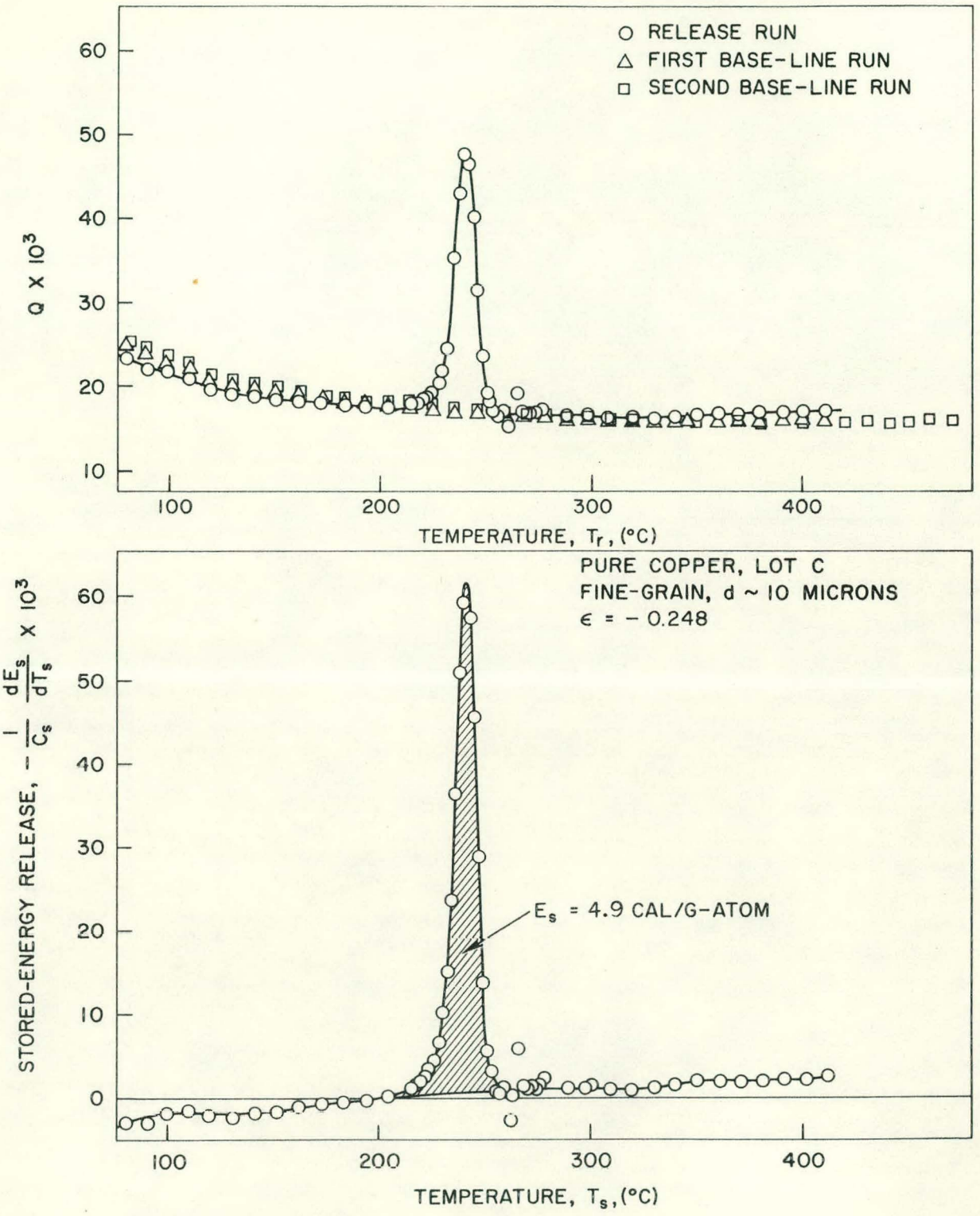


Fig. 8--Stored-energy-release data for high-purity copper (Lot C, 99.999 wt-% Cu) deformed in compression: upper plot, experimental Q' values for release and base-line runs; lower plot, SERS computed from Q' - β' by means of Eq. (1)

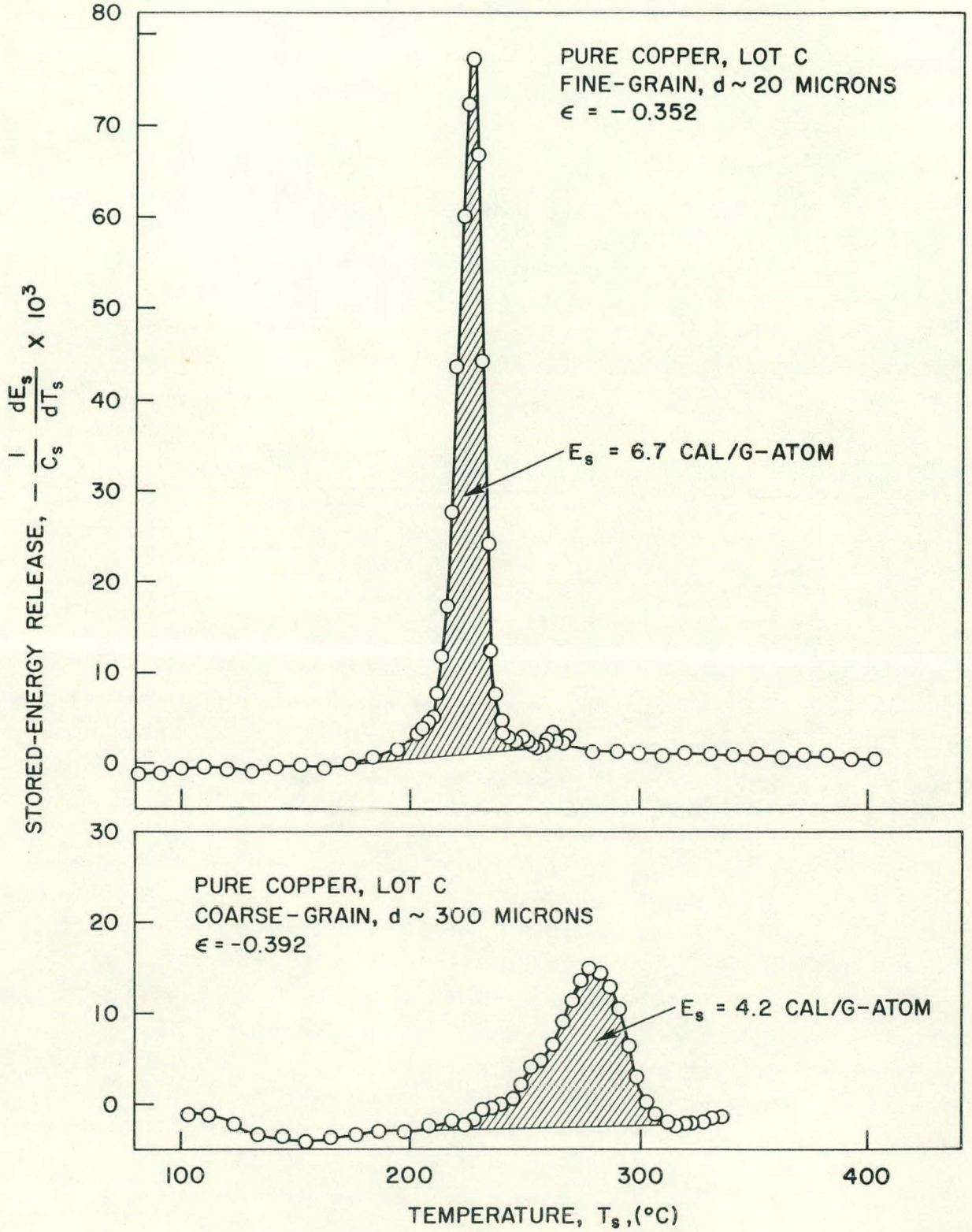


Fig. 9--Representative SERS for high-purity copper (Lot C, 99.999 wt-% Cu) of two grain sizes; deformation in compression

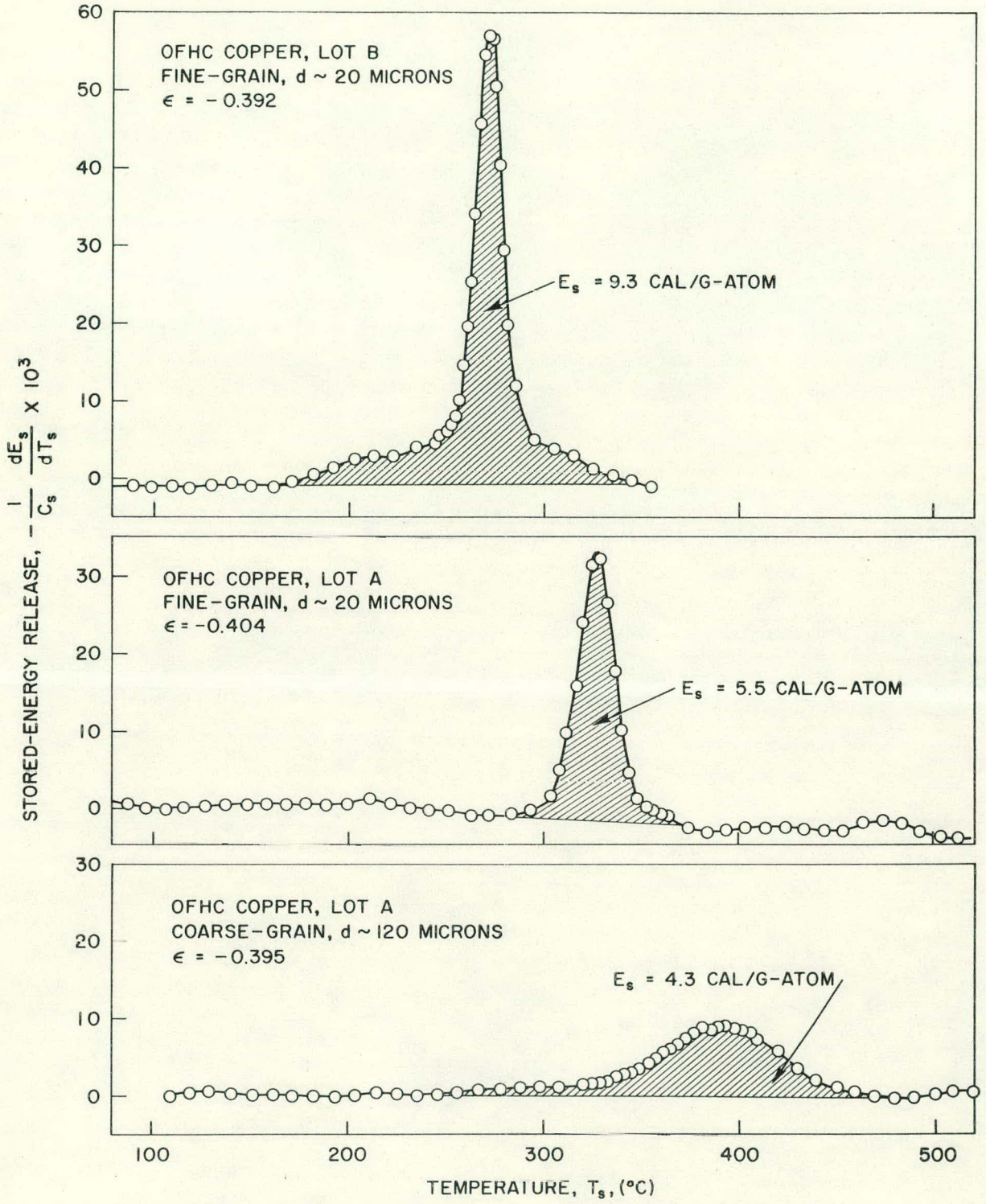


Fig. 10--Representative SERS for OFHC copper deformed in compression; analysis of Lot A indicated approximately 400 ppm Te plus 100 ppm of other impurities; analysis of Lot B indicated approximately 800 ppm Te plus 100 ppm of other impurities

9, and 10 and from the total stored energies (as a function of strain) presented in earlier reports.[3][27] The grain-size effect is operative in both high-purity copper and commercial copper containing impurity levels of the order of 500 to 1000 ppm. The impurities appear to have specific effects on the total stored energy, in some cases actually reducing the total stored energy below the values observed for high-purity copper. In all cases observed thus far, the presence of impurities makes the recrystallization kinetics more sluggish, as evidenced by higher temperatures for the recrystallization peaks and a general smearing-out of the release spectra.

An understanding of these observations is being sought by starting from the consideration that as the grain size is reduced, the amount of material in turbulent flow during macroscopic deformation is increased. Following the lines of consideration reviewed by McLean,[28] the imperfection structures generated by the complex stress conditions existing near a grain boundary will depend on the relative importance of the barrier-hardening effect and the multiple-slip effect. In pure fcc metals, the latter "complexity effect" should predominate because such metals contain so many slip systems that no neighboring grain can be very unfavorably oriented with respect to the applied stress. However, the formation of Cottrell atmospheres should increase the importance of the barrier-hardening effect in less-pure copper. Thus, the action of impurities will depend on their location in the microstructure, i.e., whether on grain boundaries, in dislocations, or in the undisturbed lattice; this consideration may account for the major portion of the discrepancies in stored-energy values for commercial materials. Further work on the interpretive points outlined above is under way, since these considerations are directly relevant to the investigations on the bcc metals.

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