2
JUN 14 1962

MASTER

GENERAL ATOMIC

DIVISION OF **GENERAL DYNAMICS**

GA-3076

A PROGRAM OF BASIC RESEARCH ON
MECHANICAL PROPERTIES OF REACTOR MATERIALS

QUARTERLY PROGRESS REPORT
FOR THE PERIOD ENDING MARCH 31, 1962

Contract AT(04-3)-167
Project Agreement No. 4
U.S. Atomic Energy Commission

May 1, 1962

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

GENERAL ATOMIC
DIVISION OF
GENERAL DYNAMICS

JOHN JAY HOPKINS LABORATORY FOR PURE AND APPLIED SCIENCE

P.O. BOX 608, SAN DIEGO 12, CALIFORNIA

GA-3076

Copy No.

A PROGRAM OF BASIC RESEARCH ON
MECHANICAL PROPERTIES OF REACTOR MATERIALS

QUARTERLY PROGRESS REPORT
FOR THE PERIOD ENDING MARCH 31, 1962

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

U.S. Atomic Energy Commission

Work done by:

R. H. Chambers
W. D. Crie
P. W. Flynn
L. C. Foster
K. Koyama
G. Schoeck
T. A. Trozera
J. L. White

Report written by:

T. A. Trozera
R. H. Chambers
J. L. White

May 1, 1962

I. DEFORMATION MECHANISMS IN BCC METALS

DEFORMATION AND FABRICATION CHARACTERISTICS OF REACTOR MATERIALS

Tension Experiments

Measurements of the variation of flow stress with changes in both strain rate and temperature have been obtained for two Materials Research Corporation single crystals over the temperature range 170° to 360° K. The method of obtaining the stress dependence of the activation energy or activation volume is based on rapid changes in strain rate during a tensile test. The activation energies may be obtained by measuring the temperature dependence of the stress during a tensile test and combining the two measurements as indicated below. The rate equation is

$$\dot{\epsilon} = \dot{\epsilon}_0 e^{-H(\sigma, T)/kT}, \quad (1)$$

where $\dot{\epsilon}$ = strain rate,
 $\dot{\epsilon}_0$ = pre-exponential constant,
 $H(\sigma, T)$ = activation energy,
 k = Boltzmann's constant,
 T = absolute temperature.

By differentiation of Eq. (1), and assuming zero variation of the pre-exponential with stress, σ , one obtains

$$kT \left(\frac{\partial \ln \dot{\epsilon}}{\partial \sigma} \right)_T = - \left(\frac{\partial H}{\partial \sigma} \right)_T = V, \quad (2)$$

where $(\partial H / \partial \sigma)_T = V$ is defined as the activation volume. In addition, one can measure experimentally the quantity $(\partial \sigma / \partial T)_{\dot{\epsilon}}$ by simply changing the temperature during a constant-strain-rate test.

Differentiation of Eq. (1) with respect to temperature yields

$$\left(\frac{\partial \ln \dot{\epsilon}}{\partial T} \right) = \frac{H}{kT^2}, \quad (3)$$

which, since the activation volume has been previously determined, simplifies to

$$H = V \times T \times \left(\frac{\partial \sigma}{\partial T} \right), \quad (4)$$

where H is the activation energy.

Using the equations given above, measurements of both V , the activation volume, and H , the activation energy, have been obtained over the temperature range 78° to 360° K. Figure 1 presents the results obtained to date in the form of activation energy, H , versus shear stress, τ . The measured values of activation volumes have also been plotted on this graph and are represented by short straight lines through the experimental points. These slopes are arrived at directly as a result of Eq. (2), which indicates that the variation of activation energy with stress should equal the activation volume. It is indeed fortunate that nature has provided this method of checking the internal consistency of the results obtained experimentally. Reference to Fig. 1 clearly reveals that the data are not self-consistent in the higher stress range, since the slopes, or activation volumes, measured experimentally do not lie on a curve joining the measured values of H . And indeed it can be rationalized that the values of H may be in error, since they were obtained directly by means of the activation volume. The cause of this apparent discrepancy may be traced at least in part to the assumption that the pre-exponential, $\dot{\epsilon}_0$, does not change with either stress or temperature. Introduction of this term into Eq. (2) then indicates that the measured quantity $(\partial \ln \dot{\epsilon} / \partial \sigma)_T$ may be composed of both $(\partial \ln H / \partial \sigma)_T$ and $(\partial \ln \dot{\epsilon}_0 / \partial \sigma)_T$. One experimental technique for minimizing the additional term $(\partial \ln \dot{\epsilon}_0 / \partial \sigma)_T$ is to work over extremely small ranges of strain, and this is the technique practiced by R. Chambers, utilizing the torsion pendulum.

Values for H may be obtained directly from constant-stress creep tests in which the temperature is rapidly changed. An apparatus to allow creep testing in the present tensile machine is under construction and will allow the performance of tensile tests followed without measurable delay by creep tests.

Following the creep tests on tantalum single crystals, work will be extended to the ultrapure materials developed by this group.

Slip-trace Analysis

Slip-trace analysis has continued by means of both X-ray analysis and standard metallographic methods. Figure 2 shows a stereographic projection of sample Ta-2s, which was tested over the temperature range 273° to 359° K. The data indicate slip predominantly on $(\bar{1}01)\langle 111 \rangle$ and

Fig. 1--Plot of activation energy, H, versus shear stress, τ , for tantalum single crystals; straight lines correspond to measured activation volumes

possibly some on $(101)\langle\bar{1}11\rangle$. The matter of slip planes in bcc metals has been the subject of numerous investigations. (1-7) It is significant that there is at present a lack of uniformity of opinion concerning the active slip plane or planes in this class of materials. Several results have indicated the existence of slip on (112) , (123) -type planes. (1, 2, 7) Other investigators have concluded that slip occurs on (110) -type planes only. Indeed, work by Chalmers and Martius, (4) later amplified by Chen and Maddin, (3) has shown that it is energetically favorable for slip to occur in bcc metals on planes having a trace on (112) or (123) , but this is derived from composite slip on (110) planes. Unfortunately, our study to date does not completely support or reject either school of thought, but the evidence, as indicated in Fig. 3, seems to be against crystallographic slip. The final orientations of both Ta-3s and Ta-2s indicate that the average slip trace direction lies within 5° of (112) or (123) . However, the body of evidence to date shows that all primary slip lines or bands are composed of short segments of slip on other systems, which would indicate some average orientation, as shown.

Fig. 2--Stereographic projection of initial and final orientations for Ta-2s and initial and final orientations at various stages of straining for Ta-3s

Replicas of Ta-2s are presently in preparation for examination under the electron microscope. It is planned to perform slip-line studies on tantalum single crystals pulled in tension at temperatures from 78° to 320°K

LINE
OF TEXT

TA

CENTERED
HEADING

BINDING
EDGE

RIGHT -
HAND
MARGIN

LEFT-HAND
MARGIN FOR
LEFT HAND
PAGE

TA

GA-3076

Fig. 3

LINE
OF TEXT

TA

CENTERED
HEADING

BINDING
EDGE

RIGHT -
HAND
MARGIN

LEFT-HAND
MARGIN FOR
LEFT HAND
PAGE

TA

GA-3076
Fig. 4

Fig. 3--Early slip lines of Ta-3s tested at 77° to 90° K showing extreme waviness of slip (290 $\times$, enlarged 4 times for reproduction)

in an attempt to shed light on the problem of active slip planes in bcc metals. It is of interest to note the recent result of Stein and Gorsuch⁽⁷⁾ on Fe-3% Si single crystals; under special conditions these authors observed etch pits and slip lines which appeared to lie in the (112) plane.

Figure 4 shows the slip-line structure of Ta-3s, which was tested at 77° to 90°K. Close inspection of the slip traces indicates that all the measurable strain is concentrated in a few bands which average ~100% shear strain per band. The effect of this observation on experimentally determined flow stress values in the temperature range 77° to 90°K is to raise the effective strain rate and hence the stress of a crystal tested at these temperatures. The amount by which the effective strain rate would be increased over that obtained using uniform extension as a basis has been calculated to be about 200 times. Hence, the actual strain rate of Ta-3s was 200 times that figured on a uniform-extension basis. An estimate of the change of stress due to a 200-fold change in strain rate has been made using Eq. (2) and an activation volume of 10 atomic volumes, obtained by Chambers,* for tantalum at 77°K. The change in stress is approximately 3000 psi and represents ~10% of the maximum shear stress for an ultrapure tantalum specimen tested at 77°K. It does not appear, therefore, that this effect will seriously alter the stress-temperature relationship for bcc metals. However, since the calculated 200-fold increase in strain rate is on the conservative side, the 10% change in stress level represents a minimum and could be higher.

DISLOCATION RELAXATION SPECTRA IN COLD-WORKED NIOBIUM SINGLE CRYSTALS

As part of the general program for the investigation of the rate-controlling mechanisms for plastic flow in body-centered-cubic metals, detailed data have been gathered concerning the effect of cold work on the internal friction due to dislocation relaxation oscillation. This internal friction is manifested as peaks in the internal friction versus temperature curves. The lowest temperature peak, or α peak, occurs near 175°K in cold-worked niobium measured at a frequency of 10 kc.

The purpose of this section is to attempt an interpretation of the α internal-friction peak in niobium in terms of the relaxation motion of kinks on dislocations in niobium. It is anticipated that if this model is essentially correct, a more complete interpretation of the higher temperature peaks will follow, which in turn should lead directly to an understanding of the various rate-controlling mechanisms responsible for the forces retarding dislocation motion in bcc metals. Implicit in this reasoning is

* This work will be described in a future report.

Fig. 4--Slip bands of Ta-3s tested at 77° to 90° K, showing slip offsets of about 100% (290 $\times$, enlarged 4 times for reproduction)

the assumption that at least some of the rate-controlling processes governing short-range dislocation motion (as observed by internal-friction experiments) are the same as the rate-controlling processes governing long-range dislocation motion (as measured in constant-strain-rate or creep experiments). Preliminary results, obtained using high-sensitivity torsion creep techniques, appear to be consistent with this assumption.

The experimental observations which strongly suggest that dislocation-kink relaxation oscillations are responsible for the α peak are illustrated in Figs. 5 and 6. Figure 5 shows both the α peak and the resonant frequency in a niobium single crystal deformed 10% in tension at room temperature at a strain rate of $\sim 1 \times 10^{-5}$ /sec. Included in Fig. 5 is the result of subsequent strain-aging of the specimen at 190°C for 15 min. Chambers and Schultz⁽⁸⁾ showed that such a strain-aging treatment leads to strong dislocation pinning by interstitial impurities such as oxygen. As dislocations are pinned, their contribution to the nonelastic component of the modulus is removed, resulting in a lowering of the α internal-friction peak and an increase in the resonant frequency of the sample on the high-temperature side of the relaxation peak. As is seen in Fig. 5, in the strain-aged state the peak height is lowered from 210×10^{-5} to $\sim 2.7 \times 10^{-5}$, or a ratio of ~ 75 , and the remaining peak is shifted down $\sim 50^\circ\text{K}$ in temperature. The upper frequency versus temperature curve is considered to represent the strictly elastic modulus dependence on temperature. This is the same reasoning employed earlier by Chambers and Schultz⁽⁹⁾ in comparable work on strain-aged molybdenum.

Figure 6 is a plot of the quantity $2\Delta f(T)/f [=(\Delta M/M)(T)]$, or modulus defect, for 9% and 10% prestrained niobium single crystals. The quantity $\Delta f(T)$ is the difference in resonant frequency between the cold-worked state and the strictly elastic state as a function of temperature. It is seen that $(\Delta M/M)(T)$ for niobium is essentially zero at liquid-helium temperatures. Included in this plot are earlier results found for cold-worked polycrystalline molybdenum deformed 11.7% in tension. In addition, the modulus-defect results obtained by Alers and Thompson⁽¹⁰⁾ for plastically deformed copper single crystals are plotted. Note that, contrary to the results found for niobium and molybdenum, the modulus defect in copper at liquid-helium temperatures is quite large.

As was discussed in some detail in the last quarterly report,⁽¹¹⁾ the existence of a modulus defect at liquid-helium temperatures implies the existence of a finite length of dislocation line free to move in response to the applied measuring stress. It was concluded in that report that the size of this free length was less than about 50 to 100 atomic distances in the bcc metals studied so far. The more recent results for niobium suggest that this limit is perhaps too generous. Average free lengths of less than 10 atomic distances are required to fit the new niobium data. This size of

Fig. 5--Temperature dependence of internal friction, Q^{-1} , and resonant frequency of cold-worked niobium single crystals as a function of annealing

Fig. 6--Modulus defect, $\Delta M/M$, as a function of temperature for various prestrained metals

free length is to be compared with a free length of about 3000 atomic distances implied by Alers and Thompson's data for copper. Thus, we are forced to conclude that dislocations in the bcc metal niobium (and perhaps the other refractory bcc metals as well) are essentially "frozen in" at liquid-helium temperatures, whereas dislocations in copper are effectively free to move, even at 4.2°K. If all dislocations in cold-worked niobium are constrained to lie behind energy barriers at 4.2°K, then even dislocations which are lying at an appreciable angle to the Peierls potential troughs are unable to move. This suggests that even the geometrical kinks in dislocations connecting various segments of dislocation remaining in the potential troughs are unable to move at 4.2°K, which implies that there exists an effective Peierls-type barrier to even the motion of kinks. Thus, as the temperature is raised to where thermal energy can supply the energy necessary to surmount these barriers to the kink motion, the dislocation can begin to move in response to the applied stress. As the temperature is raised still higher, the phase angle between the applied stress and the resulting dislocation displacement or strain is reduced to zero. At that temperature where the phase angle is 90°, a maximum amount of energy will be dissipated as heat in the specimen, producing the maximum in the internal friction, or the top of the internal-friction peak. In the model now under consideration, the α peak in niobium is identified with these relaxation oscillations of the kinks in the dislocations of cold-worked niobium.

A detailed quantitative model which considers the diffusive motion of these kinks is in the completion stage. The main assumptions connected with this model are outlined below.

It is assumed that the usual stress-modified rate equations hold for the motion of the kink; i. e., we write the kink velocity, S , as

$$S = S_0 \exp \left(- \frac{H}{kT} \right) \sinh \frac{v\sigma}{kT}, \quad (5)$$

where S_0 is the kink velocity at very high temperature (limited by the velocity of sound), H is the free energy of kink activation, v is the activation volume associated with the elementary diffusion jump of the kink, σ is the effective shear stress doing work on the kink as it traverses the energy barrier, k is the Boltzmann constant, and T is the temperature. In the limit of very small stresses, the term $v\sigma$ is small compared with kT near the temperatures of the α peak, so that Eq. (5) can be rewritten as

$$S = S_0 \exp \left(- \frac{H}{kT} \right) \frac{v\sigma}{kT}, \quad (6)$$

since

$$\sinh \frac{v\sigma}{kT} \rightarrow \frac{v\sigma}{kT} \quad \text{when } v\sigma \ll kT .$$

Equation (6) can now be written as

$$S \times B(T) = \sigma , \quad (7)$$

where

$$B(T) \equiv \frac{kT}{S_0 v} \exp \left(\frac{H}{kT} \right) ;$$

thus, effective shear stress and the resulting kink velocity are linearly related through a temperature-dependent coupling coefficient $B(T)$. As a kink moves down a dislocation which is constrained between fixed pinning points, there is developed an elastic restoring force which is proportional to the average distance, x , moved by the whole dislocation. Although this proportionality coefficient varies with the number of kinks originally on the dislocation line, and with the length of the line, we shall assume that there is an average restoring force given by the term $-Cx$.

Thus, the equation of motion of the pinned dislocation can be written as

$$m^* \frac{ds}{dt} + BS + Cx = \sigma \cos \omega t , \quad (8)$$

where $S = a(dx/dt)$, m^* is the effective mass of the dislocation, σb is the maximum driving shear stress acting on the dislocation, and ω is the angular frequency of oscillation of the driving stress.

It is easy to show that one can neglect the inertial term. Let $m^* \rightarrow 0$. Thus, Eq. (8) becomes

$$a B \frac{dx}{dt} + Cx = \sigma \cos \omega t . \quad (9)$$

The internal friction, Q^{-1} , can be calculated by standard methods from Eq. (9) to give

$$Q^{-1} = D \times \frac{B\omega}{C^2 + B^2\omega^2} . \quad (10)$$

This relation has a maximum, $Q_{\max}^{-1}$:

$$Q_{\max}^{-1} = \frac{D}{C} , \quad (11)$$

when

$$B\omega = C ; \quad (12)$$

or, combining Eqs. (11) and (12),

$$Q_{\max}^{-1} B\omega = D ; \quad (13)$$

or, replacing B with its definition,

$$Q_{\max}^{-1} \frac{kT}{S_0 v} \exp\left(\frac{H}{kT}\right) \omega = D ; \quad (14)$$

or, if we consider Eq. (14) at constant frequency,

$$Q_{\max}^{-1} T \exp\left(\frac{H}{kT}\right) = \text{const.} \quad (15)$$

This means that if the α peak is reduced in height from $Q_{\max_1}^{-1}$ to $Q_{\max_2}^{-1}$ by impurity pinning, the peak temperature will be lowered from T_1 to T_2 , corresponding to a temperature shift, ΔT , amounting to

$$\ln \left(\frac{Q_{\max_1}^{-1} T_1}{Q_{\max_2}^{-1} T_2} \right) = \frac{H}{k} \frac{\Delta T}{T_1 T_2} .$$

From the niobium data given above, the temperature shift observed is $\sim 50^\circ \text{K}$, whereas the computed shift amounts to about 40°K . In view of the simplifications made in this treatment, it is felt that the agreement between experiment and this simple theory of the diffusing kink is encouraging.

II. THE STORED ENERGY OF COLD WORK

During the period covered by this report, efforts were made to extend the technique of quantitative differential thermal analysis (DTA) to cold-worked iron, which was selected as the most readily treated representative of the body-centered-cubic structural metals. Refinements were made in the calorimetric technique itself, leading to an improved procedure of data reduction. The earlier results on high-purity copper were recomputed, and some further data on the grain-size effect were obtained. Progress in these areas is described in greater detail below.

CALORIMETER DEVELOPMENT

The cell technique employed to ensure stable thermal-absorption surfaces for the calorimeter specimens was described in the last quarterly report. (11) Further experiments with the lead calibration specimens have shown that cells involving internal tungsten springs to ensure good thermal contacts are unnecessarily complex. Simple cells consisting of a copper cylinder closed with a lid to which the thermocouple junction is attached by crimping a small tab have been found satisfactory without special loading of the lid to maintain contact with the specimen.

In previous work with the method of quantitative DTA both here (12) and elsewhere, (13)(14) thermal coupling between the test specimen and the reference specimen has been ignored in order to simplify the process of data reduction. In view of the availability of the IBM-7090 computer, these simplifications are no longer necessary. Therefore, the question of thermal coupling between specimens was investigated, commencing with the following expressions derived by applying Newton's law of heating to the specimens within the calorimeter block:

$$\frac{dT_r}{dt} = \frac{\alpha}{1 + \beta'} (T_w - T_r) + \frac{\alpha f'}{1 + \gamma} (T_s - T_r), \quad (16)$$

$$\frac{dT_s}{dt} = \alpha(T_w - T_s) - \alpha f'(T_s - T_r) - \frac{n_s}{\sum_s n_i C_i} \frac{dE_s}{dt}, \quad (17)$$

where T_s , T_r , and T_w are the temperatures of the test specimen, the reference specimen, and the calorimeter wall, respectively; E_s is the stored energy; t is the time; α is the general thermal-coupling constant between the wall and the specimens; n_s is the number of gram-atoms in the test specimen; and $\sum_s n_i C_i$ is the total heat capacity of the test cell and thermally associated parts. β' and γ account for the thermal imbalance between specimens, and the terms in $\alpha f'$ account for the thermal interaction between specimens.

The exact solution of Eqs. (16) and (17) is algebraically complex. In the present work it has been found satisfactory to employ a simplified expression which ignores second-order and higher-order corrections; the first-order Q' -function is thus defined as follows:

$$Q' = - \frac{1}{\left(1 - \frac{T_s - T_r}{T_w - T_r}\right)} \sum_s \frac{n_s}{n_i C_i} \frac{dE_s}{dT_r} + \beta' = \frac{(1 + 2f') \left(\frac{T_s - T_r}{T_w - T_r} \right) + \frac{d(T_s - T_r)}{dT_r}}{\left(1 - \frac{T_s - T_r}{T_w - T_r}\right)} \quad (18)$$

This first-order Q' -function differs from the zeroth-order Q -function used in earlier work⁽¹²⁾ only by inclusion of the term involving f' , which is essentially an apparatus constant dependent on the details of calorimeter geometry.

A series of calibration experiments (similar to those described previously⁽¹²⁾) employing the heat of fusion of lead as a known thermal effect was undertaken to evaluate f' . For those experiments in which cells were used, a typical value was $f' = 0.072 \pm 0.013$. Appropriate values of f' have been used in all calculations involved in obtaining the stored-energy results given in this report. The net correction in E_s depends on the shape of the release curve but generally amounts to an increase of 2% to 10%. A further significant improvement resulting from use of the first-order Q' -function is elimination of the "thermal after-effect" (described in a previous quarterly report⁽¹²⁾), which simplifies the problem of base-line placement and appreciably reduces the estimated error of each determination of stored-energy release.

It is felt that the development of these refinements marks a convenient stage of completion in the development of the method of quantitative DTA, and a topical report on this calorimetric technique is now in preparation.

STORED-ENERGY RELEASE IN COLD-WORKED IRON

Information on energy storage by deformation in the body-centered-cubic structural metals is limited to iron and steels,⁽¹⁵⁾ and the results on the stored-energy-release spectra^(16, 17, 18) are not fully consistent. Some preliminary experiments were therefore undertaken on an iron of good purity to test the applicability of the method of quantitative DTA to the temperatures necessary to observe complete energy release in this least refractory representative of the bcc structural metals.

The specimens were prepared from Westinghouse "Puron" iron (nominal purity of better than 99.95% Fe). An average grain size of approximately $50\ \mu$ was attained by swaging and vacuum-annealing in the ferrite range. The specimens were deformed in compression in an ice bath.

The release spectrum obtained in the first experiment with iron is shown in Fig. 7. The results are incomplete, and proper base-line runs are not available owing to repeated failures of the calorimeter furnace at high temperature. These failures are attributed to the materials of construction, and no essentially calorimetric limitation appears to exist to restrict the use of quantitative DTA for investigations of iron. The stored-energy-release spectrum appears to comprise (1) a complex region commencing at $\sim 500^\circ\text{C}$, $E_s \cong 7\ \text{cal/g-atom}$, with a structure suggesting two overlapping peaks, and (2) a lengthy and less clearly defined region, commencing near 590°C and apparently extending to temperatures above the Curie point. The validity of the higher-temperature release is doubtful in view of existing information on the annealing behavior of iron⁽¹⁹⁾ and the lack of adequate base-line data thus far in the present work.

These investigations will be continued using a more refractory furnace and calorimeter block. Metallographic studies will be made to define the steps of recovery and recrystallization.

THE EFFECT OF GRAIN SIZE ON STORED-ENERGY RELEASE IN HIGH-PURITY COPPER

The results of a continuing investigation of the effect of grain size on the stored-energy-release spectra of high-purity copper (99.999% Cu, American Smelting and Refining Company) have been reported previously.⁽¹¹⁾ These results have been recomputed using the Q' -function, and the slightly modified data appear in Fig. 8. This figure also contains several data points obtained during the present reporting period.

The results of this investigation (as of the issuance of the last quarterly report⁽¹¹⁾) were presented at the Symposium on Recovery and

Fig. 7--Preliminary data on the stored-energy release in "Puron" iron deformed in compression to a true strain of 50%; the deformation work was 282 cal/g-atom, and the average grain size was approximately 50- μ ; the base-lines are offset because the calorimeter furnace had to be rebuilt before the base-line runs could be made

Fig. 8--Stored energy of cold work in high-purity copper; the results of the present investigation for deformation in compression are compared with the results of Loretto and White (deformation in compression) and of Gordon (deformation in tension)

Recrystallization of Metals* during the discussion of the review paper by Clarebrough, et al. (20) A written discussion⁽²¹⁾ has been prepared for publication in the symposium proceedings.

The serious discrepancies between results for the stored-energy release in cold-worked copper have been discussed by Titchener⁽²²⁾ in terms of the dependencies on strain, mode of deformation, grain size, and purity. In response to Titchener's call for further measurements on similar materials subjected to similar deformation conditions, the present results go far to confirm the utility of stored-energy calorimetry for the study of these deformation variables and specifically shed further light on the effects of grain size, strain, and mode of deformation.

The present results for fine-grain copper (average grain size of approximately $35\ \mu$) deformed in compression are in good agreement with the results of Gordon⁽²³⁾ for a similar grade of copper having an average grain size of $15\ \mu$ and deformed in tension at room temperature. A qualitative confirmation of the grain-size effect reported by Loretto and White⁽²⁴⁾ is also clear; the present stored-energy-release spectra are similar in their characteristic shapes to those observed by these workers. It should be noted that this degree of consistency between results obtained in different laboratories is believed to be unique in the record of stored-energy calorimetry.

The results shown in Fig. 8 demonstrate the strong effect of grain size at low strains; the functional dependence of stored energy on strain appears to be concave downward for fine-grained material and concave upward for coarse-grained material. In the high-purity copper, practically the entire energy release occurs in a single recrystallization peak for which the release mechanism is primarily the annihilation of dislocations. Thus, assuming that the energy of dislocation interaction is small relative to the dislocation-line energy, these results indicate that the processes of dislocation multiplication and entanglement, which include the activation of new slip systems, depend strongly on the density of grain boundaries in a polycrystalline body. Appreciable, and calorimetrically measurable, energy storage will appear only when a sufficient number of slip systems have been activated in the majority of the grains to cause dislocation-dislocation interactions leading to the familiar tangled networks of the cell structures observed by electron-transmission microscopy. ⁽²⁵⁾⁽²⁶⁾

* At the annual meeting of the American Institute of Mining, Metallurgical, and Petroleum Engineers, New York, February 19-22, 1962.

REFERENCES

1. Allen, N. P., B. E. Hopkins, and J. E. McLennan, "The Tensile Properties of Single Crystals of High-purity Iron at Temperatures from 199 to -253° ", Proc. Roy. Soc. (London), Vol. A234, 1956, pp. 221-246.
2. Opinsky, A. J., and R. Smoluchowski, "The Crystallographic Aspect of Slip in Body-centered Cubic Single Crystals. II. Interpretation of Experiments," J. Appl. Phys., Vol. 22, No. 12, 1951, p. 1488.
3. Chen, N. K., and R. Maddin, "Slip Planes and the Energy of Dislocations in a Body-Centered Cubic Structure," Acta Met., Vol. 2, 1954, p. 49.
4. Chalmers, B., and U. M. Martius, "Slip Planes and the Energy of Dislocations," Proc. Roy. Soc. (London), Vol. A213, 1952, p. 175.
5. Ferriss, D. P., R. M. Rose, and J. Wulff, "Deformation of Tantalum Single Crystals," submitted for publication in Trans. Met. Soc. AIME.
6. Schadler, H. W., "Deformation Behavior of Zone-melted Tungsten Single Crystals," Trans. Met. Soc. AIME, Vol. 218, 1960, p. 649.
7. Stein, D. F., and P. D. Gorsuch, "Etch Pit Evidence of (112) Slip in Iron," Acta Met., Vol. 9, 1961, p. 904.
8. Chambers, R. H., and J. Schultz, "Low-Temperature Stress-Relaxation Peaks in Plastically Deformed Polycrystalline Niobium," Acta Met., Vol. 8, 1960, p. 585.
9. Chambers, R. H., and J. Schultz, "Dislocation Relaxation Spectra in Plastically Deformed Refractory BCC Metals," Acta Met., Vol. 10, 1962, p. 296.
10. Alers, G. A., and D. O. Thompson, "Dislocation Contributions to the Modulus and Damping in Copper at Megacycle Frequencies," J. Appl. Phys., Vol. 32, 1961, p. 283.
11. Trozera, T. A., R. H. Chambers, and J. L. White, A Program of Basic Research on Mechanical Properties of Reactor Materials - Quarterly Progress Report for the Period Ending December 31, 1961, General Atomic, Report GA-2848, January 31, 1962.
12. Trozera, T. A., J. L. White, K. Koyama, and R. H. Chambers, A Program of Research on Mechanical Metallurgy as Related to Fuel-element Fabrication - Summary Report to December 31, 1960, General Atomic, Report GA-2235, June 21, 1961.
13. Henderson, J. W., and J. S. Koehler, "Low Temperature Release of Stored Energy in Cold Worked Copper," Phys. Rev., Vol. 104, 1956, p. 626.

14. Overhauser, A. W., "Stored Energy Measurements in Irradiated Copper," Phys. Rev., Vol. 94, 1954, p. 1551.
15. Titchener, A. L., and M. B. Bever, "Stored Energy of Cold Work," Prog. Metal Phys., Vol. 7, 1958, p. 247.
16. Sato, S., "Thermal Measurements of the Latent Energy in Cold-worked Metals and Alloys," Sci. Repts. Tohoku Imp. Univ., Vol. 20, 1931, p. 140.
17. Quinney, H., and G. I. Taylor, "The Emission of the Latent Energy due to Previous Cold Working when a Metal is Heated," Proc. Roy. Soc., Vol. 163A, 1937, p. 157.
18. Wang, T. P., and N. Brown, "The Energy Stored in Ingot Iron Deformed by Torsion at 25, -82, and -185°C," Trans. ASM, Vol. 50, 1958, p. 541.
19. Michalak, J. T., and W. R. Hibbard, Jr., "Effect of Rolling Procedure on the Kinetics of Recrystallization of Cold-rolled Iron," Trans. ASM, Vol. 53, 1961, p. 331.
20. Clarebrough, L. N., M. E. Hargreaves, and M. H. Loretto, Energy Releases Associated with Recovery and Recrystallization, AIME Symposium on Recovery and Recrystallization of Metals, presented February 20, 1962, New York; to be published.
21. White, J. L., Discussion of Paper by Clarebrough, Hargreaves, and Loretto on "Energy Releases Associated with Recovery and Recrystallization", General Atomic, Report GA-2987, March 1, 1962.
22. Titchener, A. L., "The Stored Energy of Cold Work in Relation to Grain Size and Other Variables," Acta Met., Vol. 9, 1961, p. 379.
23. Gordon, P., "Microcalorimetric Investigation of Recrystallization of Copper," Trans. AIME, Vol. 203, 1955, p. 1043.
24. Loretto, M. H., and A. J. White, "Influence of Grain Size on the Energy Stored in Deformed Copper," Acta Met., Vol. 9, 1961, p. 512.
25. Howie, A., "Quantitative Experimental Study of Dislocations and Stacking Faults by Transmission Electron Microscopy," Met. Reviews, Vol. 6, 1961, p. 467.
26. Darken, L. S., "Some Observations on Atoms and Imperfections," Trans. ASM, Vol. 54, 1961, p. 600.