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GENERAL ATOMIC
DIVISION OF GENERAL DYNAMICS CORPORATION

A PROGRAM OF RESEARCH ON MECHANICAL METALLURGY
AS RELATED TO FUEL-ELEMENT FABRICATION

A SUMMARY REPORT OF WORK THROUGH SEPTEMBER, 1959,
INCLUDING REPORT OF PROGRESS FOR SEPTEMBER

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

RELEASED FOR ANNOUNCEMENT

IN NUCLEAR SCIENCE ABSTRACTS

November 10, 1959

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GENERAL ATOMIC

DIVISION OF GENERAL DYNAMICS CORPORATION

JOHN JAY HOPKINS LABORATORY
FOR PURE AND APPLIED SCIENCE

SAN DIEGO 12, CALIFORNIA

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Work done by:

T. Trozera
R. Chambers
J. White

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Report written by:

T. Trozera
R. Chambers
J. White

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I. INTRODUCTION

The objective of the program on mechanical metallurgy as set forth in the original proposal was to bring the disciplines of solid-state physics, thermodynamics, mathematics, elasticity, and plasticity to bear on the problems of deformation of metals as related to fuel-fabrication processes. Following discussions among members of the scientific research staff concerned with this program, the following three related areas of investigation were selected in an effort to utilize this multidisciplinary approach to the solution of fabrication problems. The knowledge gained by cooperative interplay between these fields should ultimately provide a broader and less empirical basis for the solution of the problems of formability and general mechanical behavior of reactor materials.

DEFORMATION AND FABRICATION CHARACTERISTICS OF REACTOR MATERIALS

Work on the mechanical metallurgy aspect has been concentrated on the deformation and fabrication characteristics of reactor materials. Studies were initiated on the rod-drawing process and work to date has been concerned with various problems and analyses of this forming operation. In an analysis of the rod-drawing problem, it has been convenient to consider the total work required to draw a rod as arising from the sum of three additive factors--the work necessary to cause the homogeneous deformation, the work required to cause the attendant inhomogeneous deformation, and the work necessary to overcome the frictional effects between the die land and the metal being drawn.

Although these work terms are interrelated, this separation of work is a useful tool for analysis of the process, and a method for separating the components of work due to each of the above factors has been developed

and presented;^{(1)*} the method utilizes a technique of curve matching. A general mathematical analysis of rod-drawing has been performed and presented.⁽²⁾ Incorporated in this analysis is an empirical stress-strain relationship which represents the actual plastic-flow characteristics of the material being drawn. The successful incorporation of the stress-strain relationship into the drawing expression represents a contribution in one area of rod-drawing hitherto considered deficient.⁽³⁾ Of importance for both the curve-matching technique⁽¹⁾ and the mathematical analysis⁽²⁾ is an experimental knowledge of the stress-strain curve for the material being tested. Considerable effort is currently in progress to obtain a stress-strain curve corrected for the effects of necking, which is necessary for the curve matching technique,⁽²⁾ and an empirical expression of the form obtained by Hockett⁽⁴⁾ for this curve. Preliminary data have been obtained by the photographic technique outlined in a previous report,⁽²⁾ and a least squares fit of this data is currently being obtained with the IBM 650 at General Atomic. A complete experimental evaluation of the concepts developed will be forthcoming.

THE STORED ENERGY OF COLD WORK

The thermodynamic approach to an understanding of the formability characteristics of reactor materials has been concerned with the design, construction, and development of a calorimeter for measurements of the stored energy of cold work. The storage of energy in a material during a deformation process is directly related to the imperfection structure, which, to a large extent, is responsible for the mechanical behavior of the material. The purposes of this thermodynamic investigation are to measure the energy stored in reactor materials as a function of material and process variables and to determine the temperature spectrum of the release of the stored energy on annealing. By combining the information on the energetics of

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*References are listed on page 55.

the imperfection structures with the details of dislocation density and orientation that are being obtained by low-temperature acoustical spectrometry, a basis will be provided for a more fundamental understanding of the mechanisms of deformation and work-hardening in materials at the strain levels employed in practical fabrication processes.

The calorimetric method employed is that of differential (or twin) calorimetry in which the apparent heat capacity of a cold-worked specimen is compared directly with the heat capacity of a fully annealed specimen. The basic design and construction of the calorimeter have been completed, and the developmental phase is believed to be near completion. The first set of specimens, bars deformed in torsion, have been prepared, and measurements of the stored energy of cold work will be undertaken, employing the method of quantitative differential temperature analysis.

THE RELATION OF FORMABILITY TO IMPERFECTION STRUCTURE

The solid-state physics approach to the problem of formability has centered on the relation of formability to imperfection structure. The plastic properties of a deformed metal are strongly dependent on the details of the interaction between certain line-type structural imperfections known as dislocations as well as many point-type defects such as vacancies, interstitials, and impurities.

The purpose of the solid-state effort is to make use of various detection techniques in an effort to define the density and distribution of dislocations and associated defects in a deformed metal.

Much of the solid-state physics effort to date has centered on the design, construction, and operation of a new detection apparatus--a low-temperature acoustical spectrometer. This spectrometer can measure the acoustical energy absorption spectrum of a plastically deformed metal; it is particularly well suited for the detection of absorption peaks (Bordoni peaks), which are believed to be due to specific classes of dislocations.

The first stage of a series of acoustical studies using the spectrometer has resulted in the discovery of a strong absorption peak in polycrystalline niobium deformed in extension

Considerable effort has been spent in verifying the dislocation origin of this peak. A detailed investigation of the dependence of the structure of the peak on the amount and kind of deformation is now underway.

II. THE DEFORMATION AND FABRICATION CHARACTERISTICS OF REACTOR MATERIALS

INTRODUCTION

The objective of the program of mechanical metallurgy as related to the deformation and fabrication characteristics of reactor materials has been to examine various theories of deformation processes and to compare these theories with experiment. Effort has also been devoted toward the development of methods for simplifying analyses of deformation processes. During the past year, emphasis has been placed on problems concerned with and analysis of the rod-drawing process. Reliable experimental techniques have been developed and initial results have been compared with relatively simple theoretical predictions.⁽⁵⁾

BACKGROUND AND CURRENT STATUS

Figure 1 shows drawing forces obtained in early experiments on Type 1100 aluminum compared with the minimum forces required (homogeneous deformation) and forces predicted by an equation due to Sachs,⁽⁶⁾ which accounts simply for the components of work due to drawing homogeneously and the frictional forces at the die-wire interface. In attempting to make correlations with various theories, it soon became apparent that many assumptions were necessary relative to such items as the frictional coefficient between the die land and the metal being drawn, the proper yield stress to employ in the equations, and the separation of the total work required to draw a metal into its various components.

It will be convenient here to define these different components of work in drawing. Consider the total work required to draw a rod as arising from the sum of three additive factors--the work necessary to

Fig. 1--Drawing force (lb); reduction of area for annealed Type 1100 aluminum

cause the homogeneous deformation (planar cross sections remain planar under the die), the work required to cause the attendant inhomogeneous deformation (nonuniform shearing), and the work necessary to overcome the frictional effects between the die land and the metal being drawn. Although these work terms are interrelated, this separation of work is a useful tool for analysis of the rod-drawing process.

A consideration of the assumptions necessary for comparison of experimental results with existing theories led to the development of the following method for separating the components of work due to each of the above factors.

The total work to draw can be analyzed as follows:

$$W_T = W_H + W_R + W_F, \quad (1)$$

where W_T = total work to draw which can be obtained by integration of the drawing force-time relationship,

W_H = homogeneous work of deformation,

W_R = nonhomogeneous work of deformation,

W_F = frictional work.

Past solutions of general stress and strain tensors have yielded the following equations which have been found experimentally to represent satisfactorily the work-hardened state of an isotropic homogeneous material for which the σ_{ij} 's, τ_{ij} 's, ϵ_{ij} 's, and γ_{ij} 's are accurately known:

$$\tau_{II}^2 = \frac{(\sigma_{xx} - \sigma_{yy})^2 + (\sigma_{yy} - \sigma_{zz})^2 + (\sigma_{zz} - \sigma_{xx})^2}{2} + 3(\tau_{xy}^2 + \tau_{yz}^2 + \tau_{zx}^2) \quad (2)$$

$$\gamma_{II}^2 = \frac{4}{9} \left\{ \frac{(\epsilon_{xx} - \epsilon_{yy})^2 + (\epsilon_{yy} - \epsilon_{zz})^2 + (\epsilon_{zz} - \epsilon_{xx})^2}{2} + \frac{3}{4}(\gamma_{xy}^2 + \gamma_{yz}^2 + \gamma_{zx}^2) \right\} \quad (3)$$

where σ_{xx} , σ_{yy} , σ_{zz} ; ϵ_{xx} , ϵ_{yy} , ϵ_{zz} are the normal stresses and

normal strains, respectively, and τ_{xy} , τ_{yz} , τ_{zx} ; δ_{xy} , δ_{yz} , δ_{zx} are the shear stresses and shear strains, respectively. In general, these quantities are known precisely only for simple deformation processes.

For the case of simple tension, it can be readily verified that

$$\tau_{II} = \sigma_{xx} , \quad (4)$$

$$\delta_{II} = \epsilon_{xx} . \quad (5)$$

Hence, the $\tau_{II} - \delta_{II}$ relationship is the true stress-true strain curve in simple tension (see Fig. 2).

A specimen deformed homogeneously, as in simple tension (for the case of rod-drawing this is approximately true for a low die angle), to a value of $\delta_{II} = A$, corresponding to a finite reduction in area, would then exhibit a stress-strain curve such as ABC, and the area OAB = W_H is the homogeneous work of deformation. This may be more clearly seen by inspection of Fig. 3A, where cross sections originally planar have remained planar, and minimum work has been expended against internal material forces resisting deformation. A second specimen deformed to the same reduction of area, however at a larger die angle, would upon tensile straining yield a stress-strain curve now matching at A'B'C' (see Fig. 2) corresponding to a more cold-worked state. Figure 3B represents qualitatively the appearance that originally planar cross sections would assume under this condition.

If the reduction of area is the same for the two conditions, inspection of Eq. (3) reveals that the value of δ_{II} obtained only by consideration of normal strains would be increased because of the shear strains, δ_{ij} , which are now present, with the net result that the strain-hardened state of this material is represented by A'B'C. The difference between these two states is caused only by the shear strains which have been introduced and hence the area AA'B'B represents the additional work which must be performed because of the nonhomogeneous shearing.

Fig. 2--The τ_{II} - δ_{II} relationship

Fig. 3A--Material drawn through a die showing uniform deformation

Fig. 3B--Material drawn through a die showing nonuniform deformation

Since the $\tau_{II} - \delta_{II}$ relationship, OBCB 'C', can be experimentally obtained, the quantities W_H and W_R are readily known. The total work, W_T , may be obtained from direct integration of the drawing force-time relationship and from Eq. (1) by difference

$$W_F = W_T - W_H - W_R, \quad (6)$$

yielding the contribution of work due to friction at the rod-die land interface without recourse to assumed coefficients of friction. Indeed, one may calculate by quite independent means the coefficient of friction, μ .

Since efforts to validate the above concept were based on performing tensile tests of specimens following drawing to various reductions, consideration was given to the possible influence of strain rate on the desired properties. This was considered for the following reason: a usual strain rate employed in drawing the specimens is 5.0 in./min; however, the subsequent tensile tests are usually performed at 0.05 in./min. Although the effect of strain rate on strain hardening of a metal is generally neglected for cold working, the strain-hardened state of a metal is not uniquely determined by the strain, since it is also dependent on strain rate. Hence, it was decided to perform experiments to clarify this matter in order to determine whether the data obtained by using this testing procedure could be used subsequently for purposes of correlation.⁽⁷⁾

Eight specimens of annealed Type 1100 aluminum were given an initial prestrain of 10% in tension followed by drawing and testing as follows:

- 2 specimens, drawn 19.1% reduction of area (RA) at 5.0 in./min
- 2 specimens, drawn 19.1% RA at 0.05 in./min
- 2 specimens, drawn 50.5% RA at 5.0 in./min
- 2 specimens, drawn 50.5% RA at 0.05 in./min.

All drawn specimens were then tensile tested at a strain rate of 0.05 in./min.

If a significant difference in structure exists between specimens drawn at one rate and tensile tested at a considerably lower rate, and

those drawn and tensile tested at the same rate, it should manifest itself by differences in the stress-strain curves which are highly structure sensitive.

Figure 4 presents the results of the tensile tests following drawing, as outlined above, for both the 19.1% RA and the 50.5% RA. As can be seen by inspection, no significant differences in structure exist between specimens drawn at a speed of 5.0 in./min and those drawn at 0.05 in./min for both reductions of area. Specimens drawn 50.5% RA at 5.0 in./min appear to be somewhat stronger than those drawn the same amount at the slower rate; however, the data are well within the scatter encountered and may be adequately represented by a single curve, as shown.

The results of these tests indicate that within the range of drawing and strain rates employed, any effects due to structural differences caused by the one hundred to one ratio between rate of drawing and subsequent tensile test are smaller than the typical scatter experienced and hence may be neglected.

Following verification that there was no effect due to strain rate, a series of test specimens were reduced by drawing various amounts and were tested in tension. The results of the tension tests are shown in Fig. 5. Also included in Fig. 5 for comparative purposes is a true stress-true strain curve for the starting material (annealed and prestrained 10% in tension). These relationships are necessary for the calculation of the work distribution and coefficient of friction by the curve-matching method, as outlined previously. The stress-strain curve for the specimen drawn to 50.5% RA exhibits softening relative to that drawn to 46.3% RA. Since all testing was conducted at room temperature, at which temperature aluminum does not normally anneal, an approximate calculation of the temperature rise associated with the work to draw a specimen to 50.5% RA was made. These calculations were made by assuming adiabatic conditions and yielded a $\Delta T \approx 40^{\circ}\text{C}$. This ΔT would be lowered by consideration of the actual experimental conditions, but does give an order

Fig. 4--Stress-strain curves for Type 1100 aluminum following drawing at different rates

Fig. 5--Stress-strain curves for Type 1100 aluminum following drawing to various reductions

of magnitude. It is believed that this temperature rise in combination with the highly worked state is responsible for the softening effect.

Attempts to match the stress-strain curves of the drawn specimens with that of the starting material (annealed and prestrained 10% in tension), in order to determine the distribution of work and coefficient of friction as outlined, were successful for small reductions of area. Figure 6 is a plot of the stress-strain curve, $\tau_{II} - \delta_{II}$ relationship, for the starting material (master curve), and the initial portions of the stress-strain curves of the drawn materials in which the strain $\xi = \ln (A_o/A_f)$ corresponds to the reductions of area as shown. The apparent decrease in strength of the drawn material at reductions of area greater than 30.1%, as compared with the master curve ($\tau_{II} - \delta_{II}$), is anomalous and the cause of this discrepancy has been determined. The tensile flow stress, as computed in the usual manner, does not account for hydrostatic components of stress which arise when necking occurs. Bridgman⁽¹⁰⁾ has arrived at an approximate solution for the stress distribution in the neck of a tensile bar which shows the measured stress to be higher than that effectively causing plastic flow. The over-all effect of this correction is to lower the stress-strain relationship as shown by the dashed line corrected curve in Fig. 6. Since the technique of curve matching is quite sensitive to the exact position of the master curve, experiments of two types were initiated:

1. Tension-stress-strain experiments designed to take into account the correction factor by Bridgman.
2. Compression tests as an additional check.

During the over-all course of the program, consideration was frequently given to the mathematical formulation of the rod-drawing problem, and specifically to the introduction into the drawing expression of an equation describing the stress-strain relationship for the material. A solution based on graphical integration of the stress-strain curve as a function of position of the metal in the die was accomplished; however,

Fig. 6--Comparison of stress-strain curves of drawn Type 1100 aluminum with the corrected and uncorrected master curve ($\tau_{II} - \gamma_{II}$ relationship)

this was not presented or used because of the difficulty of performing the calculations. It was believed that any worthwhile contribution would have to be of a type to simplify the drawing problem. Recently, however, an analytic expression relating stress and strain of the form proposed by Hockett⁽⁴⁾ has been successfully introduced into the drawing expression, and this analysis is presented in the Appendix. The importance of this accomplishment is that it allows one to calculate from an experimentally obtained $\tau_{II} - \delta_{II}$ relationship, the drawing stress, or force, due to the frictional plus homogeneous component or that due to the frictional component alone as a function of the experimental parameters.

It will be convenient to describe the work of drawing in terms of average stresses in the z-direction, i.e., along the axis of the die. Thus we define

σ_T = the average total axial stress,

σ_R = the average stress required to cause the inhomogeneous shearing resulting from the die angle slope and friction,

σ_H = the average axial stress necessary to cause homogeneous deformation,

σ_F = the average axial stress arising from friction.

If it is assumed that planar cross sections remain planar under the die, the principal directions are in the axial, z, radial, r, and circumferential directions. Under these simplifying conditions it has been shown that the differential equation for axial stress distribution under the die becomes*

$$A d\sigma_{zz} + (1 - C) \sigma_{zz} dA = -C \tau_{II} dA, \quad (7)$$

where A = cross-sectional area at any point under the die,

σ_{zz} = the average axial stress at "A" under the die,

$$C = \frac{1 + \mu \cot \alpha}{1 - \mu \tan \alpha}, \quad (8)$$

---

* See the Appendix.

α = the half angle of the die,

μ = the coefficient of friction,

τ_{II} = the quadratic invariant of the deviator stress $\tau_{II} = \tau_{II}(A)$.

It should be noted that $C \geq 1$ depends on the die angle and coefficient of friction. When the coefficient of friction is zero, $C = 1$, a point which will be considered later. The integration of Eq. (7) is accomplished by means of an integrating factor and yields the following:

$$d \left\{ \sigma_{zz} A^{(1-C)} \right\} = \int - C A^{-C} \tau_{II} dA . \quad (9)$$

The $\tau_{II} - \delta_{II}$ true stress-true strain curve is presently being evaluated and is expected to fit the form

$$\tau_{II} = B(1 - e^{-F\delta_{II}}) + D\delta_{II} , \quad (10)$$

where B, F, and D are experimental constants. τ_{II} , which is given by Eq. (10) can be expressed in terms of A, since under the assumptions made

$$\delta_{II} = \ln A_0 / A , \quad (11)$$

where A_0 is the cross-sectional area of the rod at the entrance to the die. Therefore,

$$\sigma_{zz} A^{(1-C)} - \sigma_0 A_0^{(1-C)} = -C \int_{A_0}^A \left\{ B A^{-C} - B A_0^F A^{-(C+F)} + D A^{-C} \ln \left(\frac{A_0}{A} \right) \right\} dA , \quad (12)$$

where σ_0 is the back-tension stress at the die entrance where $A = A_0$. Such back tension is introduced in many wire-drawing operations to reduce the pressure acting on the die, to reduce the frictional work of drawing, and thereby to reduce the die wear. For these reasons this term is retained in the analysis, although experiments are being performed with zero back tension. Integration of Eq. (12) reduces to

$$\begin{aligned} \sigma_{zz} = \sigma_o \left(\frac{A_F}{A_o} \right)^{(C-1)} &+ \left\{ \frac{BC}{C-1} - \frac{DC}{(C-1)^2} \right\} + \left\{ -\frac{BC}{C-1} + \frac{DC}{(C-1)^2} + \frac{BC}{(C-1+F)} \right\} \left(\frac{A_F}{A_o} \right)^{C-1} \\ &- \left(\frac{BC}{C-1+F} \right) \left(\frac{A_F}{A_o} \right)^{-F} - \frac{DC}{(C-1)} \ln \left(\frac{A_F}{A_o} \right). \end{aligned} \quad (13)$$

Equation (13) applies when $C \neq 1$. In the absence of friction, i. e., when the coefficient of friction is zero, $C = 1$, and the solution of Eq. (13) should then give only σ_H . But when $C = 1$ is introduced into Eq. (13), an impossible answer is obtained.

This arises from the fact that the integration as performed is valid only when $C \neq 1$. However, if one substitutes $C = 1$ into Eq. (12) and integrates for this case

$$\sigma_{zz} = \sigma_H = \sigma_o - B \ln \left(\frac{A_F}{A_o} \right) - \frac{B}{F} \left\{ \left(\frac{A_F}{A_o} \right)^{-F} - 1 \right\} + \frac{D}{2} \left(\ln \frac{A_F}{A_o} \right)^2; \quad (14)$$

hence the stress arising from frictional effects could be obtained from Eqs. (13) and (14), thus $\sigma_F = \sigma_H + \sigma_F - \sigma_H$. If one performs the indicated operation

$$\begin{aligned} \sigma_F = \left\{ \frac{BC}{C-1} - \frac{DC}{(C-1)^2} - \sigma_o - \frac{B}{F} \right\} &+ \left\{ \sigma_o - \frac{BC}{C-1} + \frac{DC}{(C-1)^2} + \frac{BC}{(C-1+F)} \right\} \left(\frac{A_F}{A_o} \right)^{C-1} \\ &- \left\{ \frac{BC}{C-1+F} - \frac{B}{F} \right\} \left(\frac{A_F}{A_o} \right)^{-F} + B - \left(\frac{DC}{C-1} \right) \ln \left(\frac{A_F}{A_o} \right) - \frac{D}{2} \left(\ln \frac{A_F}{A_o} \right)^2. \end{aligned} \quad (15)$$

It would perhaps be simpler for purposes of calculation to consider Eq. (13) for obtaining the coefficient of friction which can be done in the following manner. σ_T can be obtained from a knowledge of the experimental drawing force, σ_R can also be isolated by a method previously described; hence, from the relationship $\sigma_T = \sigma_H + \sigma_F + \sigma_R$ and

$\sigma_H + \sigma_F = \sigma_T - \sigma_R$ all the necessary quantities are known. Trial solutions of Eq. (13) have been performed on the IBM 650 at General Atomic with assumed values of the experimental constants B, D, and F. Families of curves can be readily plotted from the data so obtained, which represent graphically the variation of $\sigma_{zz} = (\sigma_H + \sigma_F)$, Eq. (13) with either the quantity C, which includes μ , or directly with μ , the coefficient of friction. Hence, the task of solving Eq. (13) for each condition of drawing in order to ascertain the friction coefficient is simplified to one of reading a graph.

Present work is concentrated on obtaining tension-stress-strain curves for Type 1100 aluminum, which have been corrected for the stress system introduced when necking occurs. (8, 9)

A photographic method has been developed to obtain a measure of the radius of curvature of the necked section and the diameter of the neck at various stages of deformation. The diameters can be measured to ± 0.001 in. and the radii are reproducible to $\pm 5\%$, as obtained from the photographs. Figure 7 is a plot of σ_*/σ versus a/R , where σ_* is the corrected true stress, σ is the measured true stress, R is the radius of curvature of the neck, and a is the specimen diameter at the necked section. The values of σ_*/σ are obtained graphically from Fig. 7. Figure 8 shows an uncorrected stress-strain curve and a corrected stress-strain curve of Type 1100 aluminum with actual photographs and pertinent data of the necked section at indicated intervals. As can be seen, there is greater than 5% difference in stress level between the two curves at the same strain of approximately 0.8. Six tests have been conducted employing the photographic technique, and these data have been submitted to the computer facilities at General Atomic to obtain a least-squares fit to Eq. (28) in the Appendix. In order to verify the $\tau_{II} - \sigma_{II}$ relationship

Fig. 7--Plot of σ_*/σ versus a/R

Fig. 8--Corrected and uncorrected true stress-true strain curves for Type 1100 aluminum

so obtained, compressive tests have been initiated. The results of these tests should be available soon. The constants obtained from the test data will then be introduced into Eq. (13) to yield graphical relationships between σ_{zz} , the reduction of area (A_o/A), and the coefficient of friction, μ .

III. THE STORED ENERGY OF COLD WORK

INTRODUCTION

From the viewpoint of thermodynamics, the increase in the internal energy of a material during plastic deformation at temperatures below the recrystallization temperature is one of the most fundamental characteristics of any given process of cold-working. Earlier studies^(10, 11) have shown that a simple correlation exists between the energy stored and the work-hardening developed during the deformation process. In terms of crystal imperfections, the stresses developed during deformation lead to the creation of lattice defects of high energy, such as dislocations and vacancies, which accumulate to various extents in the cold-worked material. The work-hardening and, ultimately, the fracture of a material have been attributed to the interaction of the dislocations with other dislocations, with lattice impurities, with grain boundaries, and with the surface of the material.⁽¹²⁾

Since the crystal imperfections have characteristic energies associated with them, measurements of the amounts of stored energy at various stages of deformation provide fundamentally important quantities with which any suggested picture of the arrangement of defects in a material during deformation must be in agreement. During annealing, the defects of various kinds and in various configurations are eliminated and release their stored energy at temperatures characteristic of each type of defect. Thus, an investigation of the magnitude of stored energy as a function of material and process variables as well as the temperature spectrum of the release of stored energy on annealing will provide basic information on the mechanisms of deformation and work-hardening, which should lead to an improved understanding of the mechanical behavior of materials at various strain levels. The development of our experimental capability for the precise measurements of the stored energies of cold work is of particular value to the present

research program on the formability of reactor materials, since the techniques of measurement are directly applicable at the heavy strains encountered in most practical fabrication processes.

BACKGROUND

The general purpose of this thermodynamic investigation of the formability of reactor materials is to obtain, by the application of calorimetric methods, basic experimental facts to understand further the mechanical behavior of practical reactor materials that are subjected to forming processes during fabrication operations. Thus, the specific experimental objectives are to define, for selected simple processes of deformation, (1) the storage of energy as a function of cold work and (2) the temperature spectrum of the release of stored energy on annealing. Earlier studies,⁽¹¹⁾ largely confined to copper, nickel, and silver-gold alloys, have suggested the existence of two general similarities with respect to the storage and release of energy in materials subjected to various deformation processes. First, the stored energy tends to approach a saturation limit, i. e., a stage of deformation which may be pictured as an equilibrium between the generation and immediate annealing of imperfection structures or as a condition in which the various imperfection sources have ceased to operate. Second, the major release of stored energy occurs in a sharp peak at the recrystallization temperature. However, it appears that the more detailed characteristics of energy storage and release vary significantly for every material and every process of deformation.

In addition to such intrinsic characteristics of materials as crystal structure and the strength of interatomic bonding (e. g., as indicated by the melting point), the principal material variables affecting the work-hardening and storage of energy during a deformation process are the composition, including the distinctive cases of impurities and of alloying additions, and the microstructure. The process variables include the nature of the deformation process itself, e. g., extension, compression, torsion, drawing,

rolling, drilling, and other complex deformations, as well as such factors as the rate, temperature, and extent of deformation.

The mechanisms of energy storage⁽¹³⁾ in cold-worked materials are: (1) the generation and distribution of dislocations, including their interactions with themselves, with grain boundaries, and with impurities; (2) point defects, primarily vacancies; (3) stacking faults; (4) twins; (5) regions of reduced order, applicable only to alloys; and (6) elastically distorted regions. The limited experimental evidence presently available indicates that the relative contributions of these mechanisms, as well as their interactions, vary significantly even for a given metal deformed in various ways or to different extents. The temperature spectrum of the stored-energy release is valuable in providing a means for determining the relative contributions of these mechanisms.⁽¹⁴⁾ Parallel studies of hardness, density, x-ray diffraction, and electrical resistivity may also be employed to aid in the diagnosis of the imperfection structures corresponding to the characteristic regions of the release spectrum.⁽¹⁵⁾

It is felt that by providing a means of examining the variation in number and behavior of defects produced in various material by various deformation processes, calorimetric measurements offer a particularly promising approach to a further understanding of mechanical behavior at the high strain levels encountered in practical fabrication processes.

SELECTION OF CALORIMETRIC TECHNIQUE

Several techniques, including a number of minor variations, have been applied in earlier investigations of the stored energy of cold work.⁽¹¹⁾ The single-stage methods, in which the mechanical work and the heat generated during the deformation process are measured simultaneously,⁽¹⁶⁾ suffers from the disadvantage that the stored energy is given by the difference between two large quantities. Reaction calorimetry suffers from a similar disadvantage except in some special cases where the measured heats can be kept small, e. g., in the application of a liquid-tin solution calorimeter to cold-worked

and annealed silver-gold alloys.⁽¹³⁾ Anisothermal annealing studies have usually employed a single specimen and two successive runs to determine the stored energy as the difference in heat required to reach a temperature above the recrystallization temperature.^(17, 18) However, this technique is subject to difficulty in calibration owing to the problem of obtaining reproducible conditions of heat flow; a logical improvement is the use of a twin calorimeter in which two specimens, one an annealed reference specimen and the other the cold-worked specimen, are heated simultaneously.

The twin-calorimeter method has been selected for the present investigations of the stored energy of cold work because it is nondestructive to specimens, it is generally applicable to materials having annealing temperatures within the range of the calorimeter, and it yields the temperature spectrum of the release of the stored energy. This method is a differential technique for the comparison of the apparent heat capacity of a cold-worked specimen with the heat capacity of a fully annealed specimen. The instrument consists primarily of a calorimeter block which is uniformly heated in vacuo at a programmed rate of temperature rise and which encloses the two specimens (see Fig. 9). For measurements of the relatively small heat effects associated with cold work (of the order of 1% of the heat of fusion), a significant advantage of the double specimen method, as compared with the conventional single-specimen "heat-capacity-type" calorimeter, is that a base-line compensation for deviations from exactly identical specimens can be made by obtaining a second heating curve under the same experimental conditions.

In principle, the twin calorimeter may be operated by differential temperature analysis or by differential power analysis. In differential temperature analysis,^(19, 20) the temperature lags of the specimens relative to the block are measured by the amplified signals of differential thermocouples. In differential power analysis,⁽¹⁰⁾ the specimens are provided with internal heaters and the amplified signals are employed to control the power inputs to maintain equal temperatures on the specimens and on the

Fig. 9--Schematic diagram of calorimeter block and specimens

interior of the block. Both techniques have been previously applied to measurements of stored energy, but there has been no direct comparison of the relative advantages and precision of the two methods. The instrumentation for the two methods is identical except that differential power analysis requires additional control systems for the power inputs to the two specimens and a differential wattmeter to measure the differential-power input. Consequently, the present approach has been to test the method of differential temperature analysis experimentally and then to obtain the additional instrumentation for differential power analysis on the basis of this experience.

The operation of the twin calorimeter as a differential temperature device has been analyzed on the basis of the following assumptions:

(1) Newton's law of heating and (2) thermal exchange only by radiation.

The calorimeter block, specimens, and thermocouples are indicated schematically in Fig. 9, where T_W is the temperature of the inside wall of the calorimeter block, T_1 is the temperature of the reference (annealed) specimen, and T_2 is the temperature of the cold-worked specimen. If the specimens are identical in respect to geometry and emissivity, the same Newton's law constant, A , applies. Thus,

$$\frac{dT_1}{dt} = A(T_W - T_1) , \quad (16)$$

$$\frac{dT_2}{dt} = A(T_W - T_2) + \frac{1}{MC_p} \frac{dE_s}{dt} , \quad (17)$$

where M is the number of gram-atoms in each specimen, C_p is the atomic heat capacity, t is time, and E_s is the energy stored in the cold-worked specimen. Solving for $(1/MC_p)(dE_s/dT_1)$,

$$\frac{1}{MC_p} \frac{dE_s}{dT_1} = \frac{T_2 - T_1}{T_W - T_1} + \frac{d(T_2 - T_1)}{dT_1} . \quad (18)$$

Thus the stored-energy release as a function of temperature is given by the simple ratio of the thermal lags of the reference specimen with respect to the cold-worked specimen and the calorimeter block wall, plus a small correction for transients (the second term on the right-hand side of Eq. (18)).

This analysis may be readily extended to the practical case in which the specimens are not identical by introducing a factor accounting for the difference in the Newton's law constants for the two specimens. Although the algebra becomes more complex, the only experimental requirement is that a base-line run be made after the initial stored-energy release run. Lack of identicalness between specimens is inevitable in practice owing to the difficulty of controlling emissivity as well as exact specimen geometry.

The most serious theoretical problems with the method of quantitative differential thermal analysis are due to (1) the rapid change in the Newton's law constants with temperatures, (2) the difficulty of properly accounting for thermal interchange between specimens, and (3) the use of thermocouples for the critical measurements rather than as null-point indicators. For these reasons, the method of differential thermal analysis requires critical examination and may prove inferior to the method of differential power analysis despite more modest instrumentation requirements.

DESIGN AND CONSTRUCTION OF TWIN CALORIMETER

The twin calorimeter developed by Clarebrough, Hargreaves, Michell, and West⁽¹⁰⁾ was selected as the basis for the design of an instrument for measurements of the stored energy of cold work as well as of the temperature spectrum of stored-energy release on annealing. A recent paper by Clarebrough, Hargreaves, and Loretto⁽³⁾ has answered earlier criticisms of their technique, and their instrument has proved suitable up to at least 750°C. In view of the desirability of reaching higher temperature in order to study such reactor materials as niobium and zirconium, estimates were calculated for the coefficients of radiant-heat transfer in a calorimeter intended to reach 1100°C.

The results show that precise measurements of the surface temperatures of the specimens and the calorimeter block are essential and that care must be taken to achieve good temperature uniformity over these surfaces by use of heavy metal walls and by the optimum location of heating elements.

The instrumentation for the calorimeter has been developed on the basis of the analysis summarized by Eq. (18). For this purpose, it was necessary to define the sensitivity required in terms of the quantity $(1/MC_p)(dE_s/dT)$, i. e., the fractional instantaneous reduction in heat capacity of the specimen owing to the intrinsic release of stored energy. An analysis of earlier work on copper revealed that a reasonable sensitivity to be attained is $(1/MC_p)(dE_s/dT) = 0.04\%$, which should be compared with a typical peak heat release of 4% in a moderately deformed specimen. Some typical results of the calculations are given in Table 1. These data refer to the following conditions: (1) cylindrical copper specimens of 2-cm diameter and 5-cm high, (2) average total emissivity of 0.1, and (3) a heating rate of 6°K min^{-1} . These estimates indicate that low-level dc amplifiers of high stability are required to monitor the temperature differentials, particularly at higher temperatures.

A schematic diagram of the instrumentation for operation of the twin calorimeter by differential temperature analysis is given in Fig. 10. A general view of the calorimeter system, including the instrumentation for monitoring and control, is given in Fig. 11. The circuits for monitoring the temperature differences between specimens and between the calorimeter block and the reference specimen are identical and consist of a stabilized dc μv amplifier providing a signal to a conventional 5-0-5 mv recorder. The amplifiers have six scales ranging from -25/+25 to -1000/+1000 μv full-scale reading, with a limit of error of $\pm 0.4\%$ of reading.

The calorimeter block temperature is programmed by a program-control unit consisting of a motor-driven cam which acts to position a contact on a potentiometer slide wire. The control action is provided by a three-action, current-adjusting, recorder-controller which provides the

Fig. 10--Schematic diagram of instrumentation for operation of the twin calorimeter as a differential temperature analysis instrument

Component Identification for Fig. 11

- (A) Vacuum enclosure
- (B) 4 in. Vacuum valve
- (C) 4 in. Diffusion pump and cold trap
- (D) Helium tank
- (E)(E₁) Chopper amplifiers for differential thermocouple signals
- (F) Recorder for block temperature
- (G)(G₁) Recorders for differential temperatures
- (H) Control system for block temperature
- (J)(J₁) Control systems for internal heaters in reference and cold-worked specimens (for operation by differential power analysis)
- (K) Programmer for block temperature (saturable core reactor is out of sight below table)
- (L) Switch panel
- (M) Vacuum control and monitoring system for thermocouple and ionization gauges

Fig. 11--General view of calorimeter system

Table 1

TEMPERATURE DIFFERENTIALS BETWEEN DEFORMED SPECIMEN
AND REFERENCE SPECIMEN IN TWIN CALORIMETER
UNDER ASSUMPTION OF IDENTICAL SPECIMEN GEOMETRY

Temperature differential corresponding to sensi- tivity, 4×10^{-4} (in DTA or DPA experiments)			Temperature differential corresponding to peak release, 4×10^{-2} (in DTA experiment only)	
Temp. (°K)	Temp. (°K)	Chromel-alumel (μv)	Temp. (°K)	Chromel-alumel (μv)
300	0.960	38.4	96.0	3840
600	0.120	4.8	12.0	480
800	0.051	2.0	5.1	200
1000	0.025	1.0	2.5	100
1200	0.015	0.6	1.5	60
1400	0.009	0.4	0.9	40

control signal to a self-saturating reactor of 4-kva capacity.

Views of the internal parts of the calorimeter proper are given in Figs. 12, 13, and 14. The two specimens are placed on ceramic point supports to minimize thermal contact with the calorimeter block. Fully shielded chromel-alumel thermocouples are employed for both the differential temperatures and the block temperature. The calorimeter block, cubic in symmetry, can be dismantled in three pieces to facilitate mounting of specimens. The nichrome furnace consists of six sides of equal resistance wired in series and fed by low-resistance nickel leads. This furnace can be tapped to reduce power in appropriate sides in order to obtain a uniform temperature distribution. The furnace is surrounded by a series of five radiation shields. An extra shield is provided on the bottom to aid in balancing

Component Identification for Fig. 12

- Ⓐ Vacuum enclosure, raised from calorimeter base plate by means of differential chain hoist
- Ⓑ 4 in. Vacuum valve
- Ⓒ 4 in. Diffusion pump with cold trap
- Ⓓ Helium tank
- Ⓒ₁ Recorder for differential temperature
- Ⓙ₁ Control system for internal heater in specimen
- Ⓜ Vacuum control and monitoring system for thermocouple and ionization gauges
- Ⓝ Calorimeter block, demounted from base plate of block
- Ⓟ Side sections of nichrome furnace
- Ⓠ Radiation shields

Fig. 12--Calorimeter system dismantled for work on interior of block. Two cylindrical white dummies are placed to show location of cold-worked and annealed specimens

Component Identification for Fig. 13

- ① Calorimeter block, mounted on ceramic point supports
- ② Nichrome furnace
- ③ Radiation shields
- ④ Nickel furnace leads, insulated with ceramic tubing
- ⑤ Shielded thermocouples

Fig. 13--Calorimeter system dismantled to show interior of radiation shields; shielded thermocouples are mounted in a temporary arrangement for measurements of temperature distribution in the block

Component Identification for Fig. 14

- Ⓐ Vacuum enclosure
- ⒩ Calorimeter block
- Ⓟ Nichrome furnace
- Ⓠ Radiation shields

Fig. 14--Calorimeter system, viewed from above, with upper enclosure plate and upper radiation shields removed

the thermal losses through the supports and electrical leads. The entire calorimeter system is enclosed in a water-cooled vacuum enclosure, which can be lifted from the base plate by means of a chain hoist.

The calorimeter is operated in a vacuum to avoid thermal conduction in the gas phase. The 4-in. vacuum system of conventional design is monitored by Bourdon, thermocouple, and ionization gauges.

TESTS AND DEVELOPMENT OF TWIN CALORIMETER

Following the construction and assembly of the various components of the calorimeter system, a program of tests and development was undertaken to test the general high-temperature performance, to obtain temperature uniformity and control in the calorimeter block, and to develop the optimum monitoring system for the temperature differentials.

The initial tests have shown that adequate power is available from the saturable-core reactor to permit heating of the block to a temperature of 765°C at heating rates of at least $6^{\circ}\text{K min}^{-1}$. However, the thermal isolation of the block is so effective that excessive times are required to cool the calorimeter after a heating cycle. Since a complete measurement of stored energy involves two runs to high temperature, one for the initial stored-energy release and the second to determine the base-line temperature differentials, it is essential to expedite the cooling of the calorimeter, particularly at low temperature where thermal exchange by radiation is small. To meet this requirement, the vacuum system was redesigned to permit the introduction of helium as a thermal exchange gas. Furthermore, the thermal inertia has been decreased by using reduced wall thickness for the calorimeter block and radiation shields, and the enclosure has been provided with water-cooling coils on all sides.

The original calorimeter furnace, consisting of a nichrome-wire resistor wound on a cubic frame of vitreous silica, was found to have only a short life owing to shifts of the wire on the frame, which resulted in

breakage of the frame during the cooling portion of the thermal cycle.

The furnace currently in use consists of a nichrome network-heating element mounted on an Inconel frame by means of ceramic insulators. Care is taken to obtain uniform spacing of the windings and equal resistances in each face of the cubic furnace.

Differential thermocouples were inserted at geometrically symmetrical points in the calorimeter block to test the uniformity of temperature. The results of several tests showed that (1) above about 200°C , spurious potentials (having dc as well as ac components) appeared on all thermocouples, and (2) appreciable thermal gradients exist in the block, particularly between the bottom and the top. The spurious potentials were traced to inductive effects by the ac field generated by the furnace current and to unbalanced thermionic emission from the furnace windings to exposed portions of each thermocouple circuit. These effects have been effectively eliminated by the use of fully shielded lead wires.

The tests and development currently in progress concern the improvement in temperature uniformity of the calorimeter block and the optimization of the program-control system for the heating rates. Copper has been tested and found suitable as a material for the calorimeter block, provided the temperature does not exceed about 700°C . The high thermal conductivity of copper permits a reduction in block weight and thermal inertia and simultaneously improves the temperature uniformity.

PROGRAM AND CURRENT STATUS

The initial phases of calorimeter design and development are almost complete, and preliminary measurements by differential thermal analysis of the energy stored in relatively large copper bars deformed in torsion will be undertaken soon. Specimens have been prepared of a 7/8 in. bar of oxygen-free high-conductivity copper.

The experimental program has been given in detail in GACP-954.⁽³⁾

The immediate experimental work to be pursued is summarized as follows:

1. Completion of calorimeter development, including improvement of the temperature uniformity of the calorimeter block and optimization of the control systems.
2. Calibration of the differential temperature analysis instrument, including comparison with results reported in the literature.
3. Development of the differential power analysis instrument.
4. Study of the effects of impurities on the storage and release of deformation energy, involving a simple deformation process and a material obtainable in high purity and easily alloyed, e. g., aluminum.
5. Extension of the measurements to materials such as zirconium, stainless steel, and niobium.

IV. THE RELATION OF FORMABILITY TO IMPERFECTION STRUCTURE

INTRODUCTION

The progress made during the past year, including the month of September, in the study of the relation of formability to imperfection structures in metals is summarized here. From the beginning it was decided that most of the existing techniques for detecting and measuring the imperfection structures that are responsible for controlling the progress of plastic flow in metals were inadequate for the present tasks. The relatively unexplored method of acoustical spectrometry was considered to be promising for understanding the details of the interactions between dislocation arrays and point defects--details that are of great importance in constructing theoretical models in dislocation mechanics. To investigate this method, a high-resolution acoustical spectrometer was constructed. About two-thirds of the effort to date has been devoted to the design, construction, and operation of this new apparatus.

EXPERIMENTAL METHODS AND APPARATUS

The principle of the operation of the spectrometer depends on several considerations. Most spectrometers measure energy absorption as a function of frequency of the input energy source. Since it is very difficult to design a single type of acoustical transducer that will operate over the many decades of frequency desired for the present studies, the frequency of energy absorption is controlled by varying the temperature of the absorber, in this case, the plastically deformed metal. Since most absorbers of acoustical energy behave as linear relaxation oscillators, the relation between the angular measuring frequency, ω , and the relaxation period, τ , of maximum energy absorption is given by⁽²¹⁾

$$\omega\tau = 1 \quad (19)$$

From statistical-mechanical considerations,⁽²⁴⁾ τ is related to temperature, T , by

$$\tau = \tau_0 \exp H/RT, \quad (20)$$

where H is the activation energy of the process, R is the molar gas constant, and τ_0 is an intrinsic time period for the unit relaxation mechanism, e.g., the Debye frequency, 10^{13} cps, for diffusion processes. It can be seen from Eq. (20) that τ is very sensitive to temperature, and hence many decades of τ can be covered by varying the temperature only tens of degrees.

The principal relaxation process of interest in this acoustical study of plastic deformation is the Bordoni mechanism.⁽²²⁾ The Bordoni relaxation mechanism is believed to be related to thermally activated glissile jumps of segments of dislocation lines.⁽²³⁾ For most metals, the activation energies for the Bordoni process are of the order of 2000 or 3000 cal/mole,⁽²⁴⁾ which means that the peak in energy absorption occurs in the vicinity of 50° to 100°K for acoustical input frequencies of tens of kilocycles. To be able to find such acoustical absorption peaks over as wide a band as possible, it is clear that it is desirable to be able to span a temperature range as large as possible. Furthermore, to be able to measure activation energies with precision, a range of measuring frequencies of at least a decade is necessary. The amount of acoustical power that can be coupled into the specimen should be variable over a large range if the absorption strengths of the peaks vary considerably and if certain nonlinear effects⁽²⁵⁾ are to be measured. In its latest modification, the Mark II, the low-temperature acoustical spectrometer, is capable of operating over the temperature range 4°K to 600°K , a frequency range from about 10 kc/sec to 100 kc/sec, and depending on the sample, an oscillating strain amplitude range of from 10^{-8} to 10^{-4} .

The problem of measuring with high precision the acoustical energy absorption due to only one mechanism is complicated by competition from other absorbers of a continuous nature, e.g., scattering from specimen walls, grain boundaries, etc., which can provide a high noise level. This problem is solved by restricting the measurements to low enough frequencies so that the acoustical wavelength is much larger than the specimen diameter. This generally means restricting frequencies to less than 1/10 Mc. The problem of coupling energy into a specimen in this range of frequencies over such a large range of temperature, while maintaining high precision in the measurement of energy absorption, is accomplished by using a resonant-bar technique.⁽²⁶⁾ The specimen is in the form of a long thin rod that is suspended at its midpoint and excited electromagnetically to vibrate in the longitudinal mode. Strong acoustical coupling is accomplished by cementing thin ferromagnetic end faces onto the specimens and bringing the electromagnetic transducers to within 0.007 in. of the specimen end faces. The design of the electromagnetic transducers is essentially the same as a telephone-type transducer.⁽²⁶⁾

Figure 15 shows the relative positions of the electromagnetic transducers and of the specimen. The top aluminum shield cylinder houses the driver core. The specimen is shown suspended between the two transducers, the suspension includes the nodal contact thermocouple, which measures the temperature of the specimen. Both transducers are separated by rods of the same material as the specimen to reduce fluctuations in transducer-specimen separation.

Figure 16 shows the specimen-holder-transducer assembly in relation to the cryostat. For ease of interchanging specimens, the counterweighted cryostat is designed to ride on vertical rails. The cryostat is simply a standard pyrex double dewar set with a thin-walled stainless-steel and copper tube inserted inside the inner dewar. A manganin wire is wound around the inner tube to serve as a heater when temperatures are to be raised above that of the liquid-gas bath. When it

Component Identification for Fig. 15

- Ⓐ Driver transducer shield
- Ⓑ Specimen
- Ⓒ Thermocouple at nodal position
- Ⓓ Pickup transducer shield showing laminated ferromagnetic core
- Ⓔ Inner thin-walled stainless-steel cryostat tube

Fig. 15--Electromagnetic transducers and specimen assembly withdrawn from cryostat

Component Identification for Fig. 16

- ① Vertical guiding rails for dewar assembly of cryostat
- ② Transducers and specimen on support, thin-walled down tube
- ③ Top of cryostat

Fig. 16--Cryostat lowered to permit mounting of specimen

is desired to lower the temperature, the furnace is turned off and helium transfer gas is let into the outer tube, thereby connecting the inner tube thermally with the bath. A low pressure of helium gas is always maintained in the inner chamber to connect the specimen thermally with the walls of the inner tube. By adjusting the furnace current and the outer tube exchange pressure, it has become a matter of standard procedure to control specimen temperatures to within 0.05°C for periods of a minute during a measurement. For all high-precision work, this steady-state technique is used as it was found to be necessary for reproducible results. For coarser and faster measurements an "on-the-run" warm-up method can be used. The warm-up rate which has been found satisfactory is not more than $1/3^{\circ}\text{K}/\text{min}$.

Both the specimen resonant response and the temperature are recorded simultaneously on a dual-pen recorder. The sweep oscillator that drives the specimen is shaft-coupled to the dual-pen recorder, thus mechanically tying together their time base. The thermocouple output is partially bucked out by a precision voltage source, and the difference signal is sent through a Leeds and Northrup chopper amplifier and displayed on one side of the dual-pen recorder. For all very low temperature work, gold-cobalt against silver-gold thermocouples are used because of their relatively large thermoelectric output at temperatures below 60°K .

Figure 17 shows the entire cryostat assembly in place with the specimen inserted inside the inner tube.

The Mark II has been operating on a regular basis since the middle of July.

EXPERIMENTAL RESULTS

Early work included several detailed runs on plastically deformed copper to verify the work of Thomson and Holmes⁽²⁷⁾ and to learn the capabilities of the instrument. The results of these runs showed that it

Fig. 17--General view of cryostat with dewars raised into regular position.
(A) Vacuum plumbing; (B) Outer glass dewar

is necessary to obtain a more stable sweep oscillator. This was done by substituting a standard negative-resistance oscillator with thermostated tank circuit for the Hewlett-Packard oscillator formerly used. Subsequent runs proved this oscillator to be very satisfactory. Stabilities of the order of 5 parts per million in a 10-min interval are possible.

Since late in July, work has been concentrated exclusively on using the spectrometer to study the effects of plastic deformation on 99.8% pure polycrystalline niobium. This work has been particularly interesting since, to our knowledge, it is the first time a body-centered cubic metal has been investigated for the purpose of studying the acoustical effects of plastic deformation.

Specimens were formed as follows: a 3/4-in. diameter bar of niobium was swaged down in over 20 passes to a nominal diameter of 0.160 in., cut into ten 6 in. lengths, sanded to remove surface contamination, wrapped in niobium foil, and high-vacuum annealed (10^{-5} mm Hg) in a quartz vessel at 1050°C for one hour. The grain size for the annealed specimens was still quite small (0.002 by 0.010 in.) and there was still evidence of the swaging structure. Eight of the specimens were pulled in tension on an Instron testing machine at a strain rate of 1% per minute to varying amounts of tensile strain ranging from 0.50% to 8%.

The stress-strain diagram of these specimens indicated an average stress at the proportional limit of $25,000 \text{ lb/in.}^2$, an ill-defined yield point for 0.2% strain, and the start of work-hardening at about 3% strain.

A typical acoustical spectrum is presented in Fig. 18, in which the acoustical energy loss measured in Q^{-1} units is plotted versus temperature of measurement. Figure 18 shows the spectrum for a specimen pulled 5%. A substantial absorption peak is centered at 166°K . The details of its shape suggest that it may not be a single relaxation peak but several peaks spaced closely together.

Figure 19 shows the behavior of the strength of the relaxation peak shown in Fig. 18 as a function of prestrain. The as-annealed

Fig. 18--Acoustical energy loss, Q^{-1} , versus absolute temperature of polycrystalline niobium prestrained 5% in tension

Fig. 19--Relaxation strength (peak height) versus prior tensile strain

specimen shows no evidence of a peak; but for prestrains below 3%, increasing strain increases the strength of the absorption, and for strains exceeding 3%, increasing the prestrain decreases the peak height. Both of the swaged specimens still show a small peak.

The dependence of Q^{-1} on the acoustical power input expressed in terms of the oscillating measuring strain amplitude is shown in Fig. 20. This figure also shows the effect of annealing on the amplitude dependence of the specimen. The behavior seen in Fig. 20 is typical of all the specimens measured in this investigation. It is to be particularly noted that the depression of the low-temperature peak is accompanied by a nearly tenfold increase in the strain amplitude necessary to produce nonlinear effects in the energy absorption. Analogous nonlinear effects were noted in the dependence of the Young's modulus on strain amplitude.

The niobium data discussed above may be summarized as follows:

1. A strong acoustical energy-absorption peak related to plastic flow exists in polycrystalline niobium.
2. The strength of the absorption peak depends markedly on the amount of prestrain in the metal.
3. The peak disappears on annealing at the relatively low temperature of 200°C .
4. There exists a close relationship between the disappearance of the peak and the beginning of nonlinearity in strain amplitude dependence.
5. Detailed examination of the absorption peak structure indicates that it may be a multiple peak; also, the position of the peak depends slightly on the amount and type of prestrain. Most of the peaks center around 167°K for tensile deformation. One specimen which had a predominate bending strain had an absorption peak centered at 173°K .

Fig. 20--Amplitude dependence of internal friction as a function of annealing

6. Preliminary measurement of dependence of the position of the peak on frequency of measurement indicates that the peak is due to a relaxation process that is characterized by the relation

$$f = f_0 \exp (-H/RT)$$

with the activation energy $H = 5500$ cal/mole and $f_0 = 10^{11}$ cycles per sec.

INTERPRETATION OF RESULTS

The above data suggest that the peak is the body-centered cubic lattice analog to the face-centered cubic lattice Bordoni peak phenomena; i.e., caused by the thermal relaxation of dislocation segments produced by plastic flow. The surprisingly low temperature and short time for the anneal of this peak (200°C for 5 min) is consistent with the assumption that dislocation segments are pinned by interstitial impurities in the sample. The above annealing conditions are almost identical with those which allowed the yield point to return in niobium pulled at low temperatures;⁽²⁸⁾ it is believed that interstitial oxygen is responsible for this yield-point return. This view of the gaseous pinning of dislocation segments is consistent with the observations of the change in the amplitude dependence of the energy absorption with annealing. Several current theories^(25,29-31) which take into consideration the interaction between dislocations and point defects predict that increased density of pinning points on dislocation lines will increase the strain amplitude necessary to produce nonlinear acoustical effects.

The value of 5500 cal/mole for the activation energy of the peak found in this investigation is also suggestive of the diffusion activation energy for hydrogen or perhaps divacancies produced by the deformation. That neither of these two point defects can be the cause of the peak is shown by the fact that if the peak is caused by the stress-induced jumping of either of these defects, then their diffusivity can be estimated from

the relation⁽³²⁾

$$D = D_0 \exp(-H/RT) ,$$

where $H = 5500$ cal/mole, from the above measurement, and D_0 can be considered to lie in the range from 10^{-2} to 10^{-3} cm^2/sec .⁽³³⁾ At room temperature, then, the mean free path for such a defect must be of the order of 10^{-2} to 10^{-1} cm. Therefore, such a defect would have ample opportunity to visit many traps many times and thus would have a very high probability of being captured. However, the peak did not show evidence of disappearing until the temperature exceeded 150°C . Furthermore, from the observations of Pfeiffer,⁽³⁴⁾ of the linear dependence of the residual resistivity of molybdenum on strain, one expects the concentration of point defects produced by plastic deformation to be a monotonically increasing function of strain. Therefore, if the absorption peak depends on the concentration of these point defects, the strength of the peak would also be expected to increase monotonically with the amount of prestrain, but Fig. 19 shows that this is not true. The observed depression of the peak for strains exceeding a certain critical strain is qualitatively explained by the dislocation theory,⁽²³⁾ since increasing the strain increases the density of dislocations and results in decreasing the average free dislocation segment length.

CONCLUSIONS

Now that the general character of this acoustical energy absorption has been explored, more detailed studies are needed to determine whether various types of plastic deformation are reflected in modifications of the structure of the peak. Some preliminary experiments (such as the bending deformation mentioned above) indicate that the details of the peak as well as its strength do in fact depend on the type of deformation.

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Appendix

In the following mathematical analysis of the rod-drawing problem, several simplifying assumptions have been made:

1. That the axial, radial, and tangential directions are the principal directions,
2. That the axial tension stress is distributed evenly over the cross-sectional area, and
3. That the planar cross sections remain planar under the die.

The problem concerns the drawing of a cylindrical rod through a conical die as shown in Fig. 21.

Fig. 21--Wire drawing

F_B = back force (usually zero, but occasionally introduced to reduce die wear),

F = drawing force,

$2R_o$ = original diameter of drawn material,

$2R_F$ = final diameter of drawn material

2α = die angle.

Under a sufficiently high force, F , the portion of the metal stock under the die land becomes plastic and "flows" through the die, resulting in a reduction in cross-sectional area as shown above.

If one considers an element of the metal passing through the die, as in Fig. 22,

Fig. 22--Element of drawn material

certain relations can be quickly drawn

$$F_p = 2\pi r ds P$$

where P = normal pressure on the die land, and

$$F_{-z} = \sigma_{zz} A$$

$$F_{+z} = \sigma_{zz} A + \frac{\partial}{\partial z} (\sigma_{zz} A) dz .$$

A summation of forces in the z direction yields, $\sum F_z = 0$ and

$$\frac{\partial}{\partial z} (\sigma_{zz} A) dz - 2\pi r ds P \sin \alpha - \mu 2\pi r ds P \cos \alpha = 0 \quad (21)$$

and from consideration of a simple element.

$$\frac{dz}{ds} = \cos \alpha; \quad \frac{dr}{dz} = -\tan \alpha .$$

From Fig. 22

$$\frac{dA}{dz} = 2\pi r \frac{dr}{dz} ;$$

hence, $dz = -dA/2\pi r \tan \alpha$ and Eq. (21) reduces to

$$\frac{\partial}{\partial z} (\sigma_{zz} A) + P(1 + \mu \cot \alpha) dA = 0 . \quad (22)$$

The correlation between P and σ_{zz} is obtained from consideration of the radial forces acting upon an element and leads to the following:

$$P = \frac{-\sigma_{rr}}{(1 - \mu \tan \alpha)} .$$

A strain analysis on an element of volume is as follows:

$$\epsilon_{\theta\theta} = \ln \frac{2\pi r}{2\pi R} = \ln \frac{r}{R} ;$$

also, from experiment (see Fig. 22)

$$\frac{r}{r_o} = \frac{R}{R_o} \quad \text{and} \quad \epsilon_{rr} = \ln \frac{r_o}{R_o} = \ln \frac{r}{R} ;$$

hence, $\epsilon_{rr} = \epsilon_{\theta\theta}$ and $\sigma_{rr} = \sigma_{\theta\theta}$. From a consideration of the effective strain defined as

$$\gamma_{II}^2 = \frac{2}{3} \{ (\epsilon_{rr} - \epsilon_{\theta\theta})^2 + (\epsilon_{\theta\theta} - \epsilon_{zz})^2 + (\epsilon_{zz} - \epsilon_{rr})^2 \}$$

one obtains

$$\gamma_{II} = \frac{2}{3} (\epsilon_{zz} - \epsilon_{rr}) = -2\epsilon_{rr} = -2 \ln \frac{R}{R_o} ;$$

hence, $\epsilon_{zz} = -2\epsilon_{rr}$.

The Von Mises criterion for yielding is employed in this derivation, τ_{II} , which is defined in the following manner.

$$\tau_{II} = \sqrt{\frac{(\sigma_{rr} - \sigma_{\theta\theta})^2 + (\sigma_{\theta\theta} - \sigma_{zz})^2 + (\sigma_{zz} - \sigma_{rr})^2}{2}} .$$

This reduces to

$$\tau_{II} = \sigma_{zz} - \sigma_{rr} \quad (23)$$

therefore

$$\tau_{II} - \sigma_{zz} = -\sigma_{rr}$$

and

$$P = \frac{\tau_{II} - \sigma_{zz}}{1 - \mu \tan \alpha} .$$

When expression (23) is introduced into the differential equation for equilibrium (22), one obtains

$$\partial(\sigma_{zz} A) + (\tau_{II} - \sigma_{zz}) \left\{ \frac{1 + \mu \cot \alpha}{1 - \mu \tan \alpha} \right\} dA = 0 . \quad (24)$$

Let

$$C = \frac{1 + \mu \cot \alpha}{1 - \mu \tan \alpha} ;$$

Eq. (24) then reduces to

$$\partial(\sigma_{zz} A) + C(\tau_{II} - \sigma_{zz}) dA = 0 . \quad (25)$$

When (25) is operated upon as indicated and terms collected,

$$\frac{d\sigma_{zz}}{dA} + \sigma_{zz} \frac{(1-C)}{A} = -C \tau_{II} \frac{1}{A} . \quad (26)$$

Equation (26) is the general equation for equilibrium, and when the integrating factor A^{1-C} is introduced into Eq. (26), a general solution of the form

$$d(\sigma_{zz} A^{1-C}) = - \int C \tau_{II} A^{-C} dA \quad (27)$$

is obtained. Now τ_{II} can be expressed empirically as a function of A in the following manner:

$$\tau_{II} = B(1 - e^{F\delta_{II}}) + D\delta_{II} \quad (28)$$

where $\delta_{II} = \ln(A_o/A)$. Therefore

$$\sigma_{zz} A^{(1-C)} - \sigma_o A_o^{(1-C)} = -C \int_{A_o}^A \left\{ B A^{-C} - B A_o^F A^{-(C+F)} + D A^{-C} \ln \left(\frac{A_o}{A} \right) \right\} dA \quad (29)$$

where σ_o is the back-tension stress at the die entrance where $A = A_o$.

Upon integration, Eq. (29) reduces to

$$\begin{aligned} \sigma_{zz} = \sigma_o \left(\frac{A_F}{A_o} \right)^{(C-1)} &+ \left\{ \frac{BC}{C-1} - \frac{DC}{(C-1)^2} \right\} + \left\{ -\frac{BC}{C-1} + \frac{DC}{(C-1)^2} + \frac{BC}{C-1+F} \right\} \left(\frac{A_F}{A_o} \right)^{C-1} \\ &- \left\{ \frac{BC}{C-1+F} \right\} \left(\frac{A_o^F}{A_o} \right)^{-F} - \frac{DC}{(C-1)} \ln \left(\frac{A_F}{A_o} \right) \quad (30) \end{aligned}$$

which is the desired analytic expression between drawing stress, materials constants, coefficient of friction, and die angle. The σ_{zz} of Eq. (30) is the sum of the homogeneous and frictional contributions to the total work to draw. As may be noted, Eq. (30) becomes indeterminate for the particular case $C = 1$, which corresponds to $\mu = 0$. For the particular case $\mu = 0$, it is necessary to return to Eq. (29). A solution of Eq. (29) for this particular case yields a $\sigma_{zz} (\mu = 0)$, which is the homogeneous contribution to the total work.