Diffusional Creep of Multicomponent Systems

Progress Report

MASTER

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Abstract

The accomplishments this year are as follows:

1. The paper on the equilibrium distribution of mobile solutes around interface dislocations has been published in Materials Science and Eng. 16 261 (1974). ~~A copy of reprint is attached.~~
2. The method developed last year for the analysis of experimental creep data indicating multiple processes is applied to the creep data of uranium dioxide.
3. In the area of chemical equilibria involving crystal defects, some strain volumes are calculated and the interaction between prismatic dislocation loops is being studied.
4. The major effort is being spent on equipment building for penetration creep measurements. The following items have been built: a standard Bridgman crystal growing apparatus for growing molecular single crystals, a penetration creep apparatus for model systems, and an elaborate high temperature penetration creep system for metal and alloy crystals. Some creep data have been obtained already for single crystal succinonitrile. Other creep measurements will begin soon.

1. Interaction between Prismatic Loops

As part of study of elastic interaction of crystalline defects and solute atoms, the interaction between large prismatic loops was examined. The results may be applied to precipitate morphology in multiple component systems, arrangement of vacancy and interstitial loops, spinoidal decomposition, etc.

Some results are shown in Figs. 1 and 2. They give the free energy of formation of one loop in the vicinity of another. It is seen that there are several low energy configurations. These attractive interactions are the cause for orderly arrangements for vacancy or interstitial disks and precipitates. The interaction among several loops is being studied also.

2. Creep of Uranium Dioxide

Uranium dioxide is a nuclear material which demonstrates multiple processes in creep. Although such phenomena are recognized by workers in the field, no detailed analysis of experimental data is available to delineate these processes.

An attempt is being made in this project to apply a method developed recently (C00-2296-3) for such phenomena. Briefly two processes can operate simultaneously, coupled either in parallel or consecutively in series. The phenomenological nature of the observed activation parameters as well as various transition behavior have been established and readily comparable to experimental information.

One of the processes is identified as due to the motion of dislocations.

After separating out the linear part, the creep rate-stress dependence is shown in Fig. 3. However, the unusually large stress exponent (6) is attributed to periodic internal stress fields caused by strain centers such as impurity atoms and dislocations themselves. The role of internal stresses played in creep has not been recognized before, although the extremely large stress exponent in dispersed systems is explained recently in such terms.

It is hoped that creep of uranium dioxide will be understood more fully through these analyses.

3. Strain Volumes in Defect Equilibria

As part of study of chemical equilibrium under non-hydrostatic stresses, the concept of strain volumes has been introduced (see attached paper). Macroscopically the strain volume is defined as the stress derivative of equilibrium constant, K:

$$\Omega_{ij} = RT \left(\frac{\partial \ln K}{\partial \sigma_{ij}} \right)_{T, \sigma'} \quad (1)$$

In Eq. (1), R is gas constant, T is absolute temperature, and σ_{ij} is a stress component. Like stress, the strain volume has six components corresponding to the six stress components. Microscopically it is the strain component in a volume element, caused by a unit advance of the equilibrium reaction in question, integrated over a uniformly stressed region, or over a region in which the increment of the corresponding component of stress is uniform.

This connection between microscopic structural changes and macroscopic effects on measurable quantities enables us to analyze the stress effects on

many defect equilibria. Some examples are shown in Figs. 4 and 5.

4. Penetration Creep

The major effort of this project during the year is equipment building for the experimental study of Penetration Creep of multicomponent systems described in the last renewal proposal. Two students are working in this area, one concerns himself with model systems such as molecular crystal solid solutions for the purpose of a general understanding of the behavior and the other works on real metallic systems for comparison and for practical information. The progress so far is as follows:

Model Systems

Standard Bridgman crystal growing apparatus has been built for the purpose of growing single molecular crystals. As shown in Figure 6, the system consists of a driving device which has a lowering velocity of less than 2mm per hour and a standard two zone furnace with nichrome heating coils which can heat up to 500°C. The coils are powered by a stabilized A.C. current. By controlling the current in the two separate coils, various temperature gradients can be achieved.

Penetration creep apparatus, as shown in Figs. 7-8, has been built also. A steel rod guided by a pyrex tube of the same diameter to avoid wobbling can slide up and down vertically. The pyrex tube is held vertically by four Teflon blades which are adjustable. The thin rod used to penetrate into the crystal is held by a pin vise at the lower end of the steel rod. A weight holding plate is attached to the upper end of the rod. The core of the Hewlett-Packard displacement transducer 7DCDT is attached to the plate. The

entire weight is balanced by the weights on the weight holder through a pulley which has a ball bearing. The crystal used for creep test is placed on a metal disc with flat and smooth surface. A constant temperature furnace will be designed. The displacement transducer is connected to a transistor power supply. The output of the transducer is directly recorded on a Heath-Schlumberger strip chart recorder.

Penetration creep in a succinonitrile single crystal has been examined at room temperature. A cylindrical single crystal of size 1" diameter by 1.5" long was grown by zone melting method. A thin stainless steel rod of diameter 0.79 mm was used to penetrate into the crystal at various stresses. A log-log plot of the steady state velocity of penetration versus applied stresses (Fig. 9) shows a slope of about 4.1 which is close to the reported value of 4.6 in the literature (H. M. Hawthorne and J. N. Sherwood, Trans. Faraday Soc. 66, 1792, 1970).

Alloy Systems

The furnace acquired for this study is Marshall high temperature tubular furnace. It is equipped with a Platinum 40% Rhodium heating element and is designed to produce a maximum temperature of 1540°C (~2800°F) with a 2.5" uniform temperature zone in the middle. The dimensions of the furnace are 1 1/2" ID, 12" OD, and 12 1/2" in height. It has a viewing window at the center.

The furnace is accompanied by a stepdown transformer to convert the line voltage from 110 to 65 and a temperature controller. The power supplied into the furnace to reach the desired temperature is automatically established by a silicon rectifier in the temperature controller. The rectifier can vary the current supplied to the furnace over a range from 0 to 100% of the rated

line voltage in proportion to the signal from the controller. Once the approximate level has been established, a thermocouple in the furnace senses any deviation from the control point, and thus maintains the uniform temperature zone to within $\pm 1^{\circ}\text{C}$.

A rectangular framework was constructed with slotted angle steels. It was fastened on the top of a table. Two pieces of angle steels were attached to the mounting brackets on both sides of the furnace. The furnace was thus hung vertically with these angle steels to the framework just mentioned.

Two pieces of Al alloy 2024-T351 circular plates, 20" in diameter and 1/2" thick, were used to transfer forces inside the furnace chamber. One piece was fixed on top of the furnace, another piece was placed under the furnace and is capable of moving up and down. In order to be sure that the motion is vertical and as frictionless as possible, three pieces of stainless steel shafts, case hardened and ground, were used to connect these two plates through four super precision stainless steel ball bushings. The ball bushings were housed inside the adapters made with Al alloy 2024-T351, the adapters were fastened to the circular plate. The shafts were fastened vertically to a big piece of Al alloy 6061-T6, 21" x 36" x 1", which served as the base. In between the two circular plates there will be a series of high purity Al_2O_3 disks, tubes, and rods glued together by Al_2O_3 cement. The specimen will be located in the middle of the furnace between a disk and a thin rod; the forces are transferred from the plates through these ceramics to the specimen.

The applied force will be transferred through a ball bearing (3/4 " ID x 1 5/8" OD) on the end of a level arm to the center of the movable circular

plate. The level arm was supported by two ball bearings (1/2" ID x 1 1/8" OD), which served as fulcrum. Some weights will be hung on the other end of the level arm with several possible arm ratios: 1:1, 1:1.2, 1:1.4, 1:1.6, 1:1.8 and 1:2.

The penetration of the thin rod into the specimen will be detected by a linear displacement transducer, a linear variable differential transformer (LVDT) type 24DCDT-500 from HP. The stroke range is ± 0.5 in, the scale factor is 23.9 VDC/in. ($\sim 1.06 \times 10^{-3}$ mm/mv). It will be energized by a low impedance 24 VDC power supply. The output of the LVDT will be connected to a strip chart recorder.

A calibration unit and holder for LVDT were constructed with four pieces of Al alloy 6061-T6 plates (7 1/2" x 2" x 1/2"). They are lined up parallel with respect to each other through two stainless steel rods, the bottom and top pieces serve to keep the unit parallel and rigid. The LVDT unit was fastened on the second piece from the top. This piece is movable. Two super stainless steel ball bushings inside the adapters were used to insure smooth and vertical movement. The height of this plate or the position of the LVDT unit can be adjusted through a threaded rod beneath it and through the third piece from the top, which is locked in place. When the threaded rod turns, it pushes or pulls the LVDT unit up and down. The amount of movement can be read from the disk attached to the bottom of the threaded rod. The disk was divided into 100 graduations, the threaded rod has 20 turns in an inch, hence each graduation is equivalent to 1/2000" vertical displacement.

Most items described above can be seen from picture on the top (Fig. 10), the unit for the LVDT was enlarged in the bottom picture (Fig. 11). The equipment building is near completion and the measurements will be made soon.

$$\overline{\Delta F} \left(= \frac{4\pi(1-\nu)\Delta F}{\mu b_x b_z} \right)$$

FIG. 1

ΔF vs X , WHERE X IS THE POSITION OF S_2 ; $Z_1 = 0.0, Z_2 = 1.0, h = 1.0, \nu = 0.3$
 $S_1: X_1 = 0.0, X_2 = 1.0$
 $-1.0 \leq X \leq 2.1$

FIG. 2

~~FIG. 4~~

ΔF vs z_2 , WHERE z_2 IS THE HEIGHT OF S_2

$z_1 = 0.0$

$S_1: X_1 = 0.0, X_2 = 1.0$

$h = 1.0$

$v = 0.3$

FIG. 3

DISLOCATION AS A VACANCY SOURCE I.

$$K = C_{\square}$$

$$\Omega_{xx} = v_{\square} (b_x/b_e)^2$$

$$\Omega_{yy} = v_{\square} (b_y/b_e)^2$$

$$\Omega_{zz} = 0$$

$$\Omega_{yz} = v_{\square} (b_y b_z / b_e^2)$$

$$\Omega_{xz} = v_{\square} (b_x b_z / b_e^2)$$

$$\Omega_{xy} = 2 v_{\square} (b_x b_y / b_e^2)$$

$$RT \ln(C_{\square} / C_{\square}^0) = \sum \Omega_{ij} \sigma_{ij}$$

DISLOCATION AS A VACANCY SOURCE II.

$$\Omega_{xx} = v_{\square} b_x [b_x - \nu_x (b \cdot \underline{b})] / b_e^2$$

$$\Omega_{yy} = v_{\square} b_y [b_y - \nu_y (b \cdot \underline{b})] / b_e^2$$

$$\Omega_{zz} = v_{\square} b_z [b_z - \nu_z (b \cdot \underline{b})] / b_e^2$$

$$\Omega_{yz} = v_{\square} [2b_y b_z - (b \cdot \underline{b})(\nu_y b_z + \nu_z b_y)] / b_e^2$$

$$\Omega_{xz} = v_{\square} [2b_x b_z - (b \cdot \underline{b})(\nu_x b_z + \nu_z b_x)] / b_e^2$$

$$\Omega_{xy} = v_{\square} [2b_x b_y - (b \cdot \underline{b})(\nu_x b_y + \nu_y b_x)] / b_e^2$$

$$RT \ln(C_{\square} / C_{\square}^0) = \sum \Omega_{ij} \sigma_{ij}$$

FIG. 4

IRON NITRIDE IN IRON

$$K = C_{\text{N}}$$

	A(100)	B(010)	C(001)
$\Omega_{xx}/V_{\text{Fe}}$	-.8	.07	.07
$\Omega_{yy}/V_{\text{Fe}}$	.07	-.8	.07
$\Omega_{zz}/V_{\text{Fe}}$	.07	.07	-.8

$$\text{A: } RT \ln(C_{\text{N}}/C_{\text{N}}^0) = -.8V_{\text{Fe}}\sigma_{xx} + .07V_{\text{Fe}}(\sigma_{yy} + \sigma_{zz})$$

$$\text{B: } RT \ln(C_{\text{N}}/C_{\text{N}}^0) = -.8V_{\text{Fe}}\sigma_{yy} + .07V_{\text{Fe}}(\sigma_{xx} + \sigma_{zz})$$

$$\text{C: } RT \ln(C_{\text{N}}/C_{\text{N}}^0) = -.8V_{\text{Fe}}\sigma_{zz} + .07V_{\text{Fe}}(\sigma_{xx} + \sigma_{yy})$$

MULTI-COMPONENT CREEP

$$K_{\text{A}} = C_{\square} C_{\text{A}}^{\text{S}} / C_{\text{A}}$$

$$\Omega_{yy} = V_{\text{A}}^{\text{S}} + (V_{\square} - V_{\text{A}})/3$$

$$\Omega_{xx} = \Omega_{zz} = (V_{\square} - V_{\text{A}})/3$$

$$\Omega_{xy} = \Omega_{yz} = \Omega_{xz} = 0$$

$$RT \ln(K_{\text{A}}/K_{\text{A}}^0) = V_{\text{A}}^{\text{S}}\sigma_{yy} + (V_{\square} - V_{\text{A}})\sigma_{\text{h}}$$

$$RT \ln(K_{\text{B}}/K_{\text{B}}^0) = V_{\text{B}}^{\text{S}}\sigma_{yy} + (V_{\square} - V_{\text{B}})\sigma_{\text{h}}$$

FIG. 5

Fig. 6

Fig. 7

Fig. 8

FIGURE 49

A ln-ln plot of steady state penetration velocity verses⁴ applied stress. The slope shows that the stress exponent value is 4.1 .

FIGS. 10 and 11