PRESSURE DEPENDENCE OF HIGH TEMPERATURE CREEP IN SINGLE

CRYSTALS OF INDIUM

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ABSTRACT

The activation volume for the high temperature steady-state creep of high purity single crystal indium in the pressure range of 0.8-5.5 kbars has been obtained using the change of slope method. The activation volume obtained is $12.0 \pm 0.9 \mathrm{cm}^3/\mathrm{mole}$ which corresponds to 0.76 atomic volume.

INTRODUCTION

At temperatures greater than one half of the melting point, creep in indium is a diffusion controlled process,[1] being limited by stress directed, non-conservative motion of the dislocations in the lattice. The high temperature creep rate over a relatively wide range of stresses can be expressed as[2]

$$\dot{\epsilon} = \frac{A \, a \, \sigma^{m+1} \, D}{G^m} \qquad (1)$$

where $1 < m < 5$, A is a pressure independent constant, a is the lattice parameter, σ is the applied stress, and G is the

shear modulus.

The diffusion coefficient, D, is given by,

$$D = D_0 e^{-(Q_d + PV_d)/kT} \tag{2}$$

where Q_d and V_d are respectively the activation energy and activation volume for self diffusion, P is the hydrostatic pressure and D_0 is the pre-exponential frequency factor.

In a steady-state creep experiment the activation volume may be obtained by the change of slope method where the pressure is periodically cycled between two different pressure levels, P_1 and P_2, while keeping the temperature and deviatoric stress constant, and the steady-state creep rates corresponding to P_1 and P_2 are measured. Using this method V_d is given by

$$V_d = -\frac{kT}{P_2 - P_1} \ln \frac{\dot{\epsilon}_2 G_2^m D_{0_1} a_1}{\dot{\epsilon}_1 G_1^m D_{0_2} a_2} \tag{3}$$

or

$$V_d = V_c - \frac{kT}{P_2 - P_1} \ln \frac{G_2^m D_{0_1} a_1}{G_1^m D_{0_2} a_2} \tag{4}$$

where

$$V_c = -\frac{kT}{P_2 - P_1} \ln \frac{\dot{\epsilon}_2}{\dot{\epsilon}_1} \tag{5}$$

is the apparent activation volume from creep measurements.

In cubic metals D_0 shows little variation with pressure and the magnitude of the second term on the right hand side of Eq. (4) is about 2-5 per cent of V_d.[3]

Experimentally measured values of V_d are generally smaller than the atomic volume, Ω, being of the order of 2/3 to 3/4 Ω in close packed lattices and 1/3 to 1/2 Ω in the more open metals. The only metal in which V_d has been found larger than Ω is aluminum where from creep studies[4] V_d equals 1.3 Ω. A theoretical basis for this result has been provided by the study of Blandin and Deplante[5] on the electronic structure of defects. Blandin and Deplante found that Friedel oscillations in the electronic density about a vacancy defect should cause the vacancy volume in the trivalent metal, aluminum, to be slightly larger than the atomic volume. In view of this result measurements of the activation volume in trivalent indium were made to ascertain whether large activation volumes are a general feature of trivalent metals.

EXPERIMENTAL PROCEDURE

Single crystal specimens were grown by Monocrystals, Inc., from 99.999% pure indium obtained from Cominco Products Inc. The crystals were in the form of 3/16 in. diameter rods with a gauge length of 1.25 in. and were randomly orientated. The ends of the crystals were beaded to facilitate gripping.

The specimens were tested at temperatures of 67-115°C and at pressures up to 5.5 kbars. The pressure vessel used for the creep tests is shown in Fig. 1. The pressure vessel consisted of two vessels, one containing the specimen and the other the loading weight. The upper vessel was enclosed by a furnace regulated by a proportional controller and the temperature was maintained to within ±0.1°C. Dow Corning 200 silicon oil was used for the pressurizing media at the lower pressures and for tests above 4 kbars white gas was used.

The strain rates were measured using an external linear differential transformer system which is shown in Fig. 2. In this system the soft iron transformer core is connected to the load, and thus the specimen, by a length of piano wire rod and a brass spacer. The core is contained within the 1/16 in. bore of a 5/16 in. diameter length of non-magnetic stainless steel pressure tubing which is sealed off at the bottom and connected into the pressure vessel using a standard high pressure fitting. The linear variable differential transformer is situated around the pressure tubing allowing detection of the core movement, and thus the strain, to be made through the tubing wall. Calibration of the system and measurement of total strain was accomplished using the micrometer assembly shown in Fig. 3. A micrometer drive motor for displacing the transformer and a recorder actuated

switching circuit were employed to attain maximum sensitivity. Further details of this system are described elsewhere.[6]

RESULTS AND DISCUSSION

A typical creep curve for the high temperature creep of indium is shown in Fig. 4. Measurements were taken in the quasi-linear or steady-state region, which occurred between strains of 1.5% and 6 or 7% for the specimens studied. Fig. 5 shows part of a creep curve obtained from a pressure cycling test. The apparent activation volumes obtained from pressure cycling are shown in Fig. 6 for a typical run. The apparent activation volumes for eleven single crystal indium specimens are shown in Table I and the individual volumes for specimen 5 are given in Table II. The average value for the eleven specimens is $12.0 \text{cm}^3/\text{mole}$ with a standard deviation of $\pm 0.9 \text{cm}^3/\text{mole}$. This correponds to $.76\Omega$ ($\Omega = 15.7 \text{cm}^3/\text{mole}$).

Corrections to the apparent activation volume were not made since the pressure derivatives of G and D_0 have not been determined. In general G should exhibit a positive pressure derivative and in non-cubic metals D_0 has been found to increase with pressure[7] so the magnitude of the correction term may be somewhat minimized. For the pressures used the activation volume does not appear to be pressure dependent; however, knowledge of the pressure dependence of G and D_0 is

needed to substantiate this.

Since the value of the activation volume for creep in indium is less than the atomic volume and of the same relative magnitude as for the close packed metals (Table III) it appears that a large activation volume is unique to aluminum. At present the activation volume in indium has not been measured by diffusion studies, nor have any calculations of the vacancy formation volume been made thus preventing any comparisons from being made.

ACKNOWLEDGMENTS

REFERENCES

1. R. E. Frenkel, O. D. Sherby, and J. E. Dorn, Acta Met.
    3, 470 (1955).

2. P. G. McCormick and A. L. Ruoff, "Creep Under High Pres-
    sures," Mechanical Behavior of Materials Under Pressure,
    edited by D. L. Pugh, Elsevier Publishing Co., to be
    published.

3. A. L. Ruoff, "Activation Volumes for Creep," Physics of
    Solids at High Pressures, Edited by C. T. Tomizuka and
    R. M. Emrick, Academic Press, (1965).

4. B. M. Butcher, H. Hutto, and A. L. Ruoff, Appl. Phys.
    Letters 7, 34 (1965).

5. A. Balandin and J. L. Deplante, "Size Effects and Chemical
    Interactions," Metallic Solid Solutions Edited by
    J. Friedel and A. Guinier, W. A. Benjamin, Inc., (1963).

6. B. M. Butcher, R. H. Cornish, and A. L. Ruoff, A.S.M.E.
    Publ. 64-WA/PT-30.

7. N. H. Nachtrieb and C. Coston, "Self Diffusion in Tin at
    High Pressure," Physics of Solids at High Pressures,
    Edited by C. T. Tomizuka and R. M. Emrick, Academic
    Press, (1965).

TABLE I

Summary of Activation Volumes for Creep in Indium

Specimens	T(°C)	P_1(kbars)	P_2(kbars)	Number of Slope Ratios	V_c (cm^3/mole)
1	67.2	0.41	2.90	30	10.8
2	95.0	0.83	3.59	25	14.2
3	100.5	0.83	3.59	29	11.4
4	101.3	0.83	3.59	15	11.9
5	101.3	0.83	3.59	24	11.8
6	98.6	0.83	3.59	28	12.6
7	111.0	0.83	3.59	27	11.4
8	105.5	2.14	4.00	15	11.5
9	97.5	4.21	5.17	32	12.2
10	111.3	3.18	5.16	40	12.5
11	115.8	4.14	5.52	18	11.6

TABLE II

Activation Volumes $(cm^3/mole)$ Obtained from Successive
Strain Rate Ratios for Specimen 5

13.1	12.6	11.1	11.9
11.2	12.1	11.6	12.4
11.9	11.9	11.8	11.4
11.6	11.7	12.3	11.2
12.6	11.6	12.2	10.3
12.4	12.5	12.2	10.1

TABLE III

Activation Volumes for High Temperature Creep in Metals

Material	Structure	V_d(cm^3/mole)	V_d/Ω
Na	bcc	9.8[a]	.41
K	bcc	25.0[b]	.53
P	cubic	30.1[c+]	.44
Pb	fcc	12.3[d] 13.9[e]	.76
Al	fcc	13.5[f]	1.3
In	fct	12.0[+]	.76
Zn	hcp	5.95[g+]	.65
Cd	hcp	8.2[g+]	.63
Sn	tet.	5.12[h+]	.32

a   A. L. Ruoff, unpublished results.

b   C. R. Kohler and A. L. Ruoff, J. Appl. Phys. 36, 244 (1965).

c   K. L. DeVries, P. Gibbs, H. Miles and H. S. Staten, J.
      Appl. Phys. 35, 536 (1964).

d   K. L.DeVries, G. S. Baker and P. Gibbs, J. Appl. Phys. 34,
      2254 (1963).

e   B. M. Butcher and A. L. Ruoff, J. Appl. Phys. 32, 2036 (1961).

f   See Reference 4.

g   K. L. DeVries and P. Gibbs, J. Appl. Phys. 34, 3119 (1963).

h   L. DeVries, G. S. Baker and P. Gibbs, J. Appl. Phys. 34,
      2258 (1963).

+   Apparent Value.

FIGURE CAPTIONS

Figure 1. Pressure vessel used for creep studies.

Figure 2. LVDT strain measuring system.

Figure 3. LVDT calibration assembly.

Figure 4. A typical indium creep curve.

Figure 5. Partial creep curve for pressure cycling test.

Figure 6. Plot of strain rate versus strain for specimen 6
showing individual activation volumes obtained.

Bridgeman seal

Furnace

Specimen grips
Specimen

Supporting tube

Tie rod

Holding frame

Mitre ring seal

Load

Mitre ring seal

Standard high pressure connection

Pressure tubing to measuring system

GRIPS

SPECIMEN

WEIGHTS

BOTTOM PISTON
(O-RING SEAL)

HIGH PRESSURE
TUBING

MUSIC WIRE
CONNECTING ROD

SOFT IRON
CORE

BRASS SPACER

LINEAR VARIABLE
DIFFERENTIAL
TRANSFORMER

HIGH PRESSURE TUBING

MICROMETER

TEFLON BEARING

TEFLON SPACER

LINEAR VARIABLE DIFFERENTIAL TRANSFORMER

COMPRESSION SPRING

$\dot{\epsilon}$ (sec^{-1})

$T = 372\ °K$
$\sigma = 1.8$ bars

.76 k bars

3.61 k bars
$V_c = 10.8\ cm^3/mole$
$V_c = 12.0\ cm^3/mole$

.79 k bars
3.55 k bars
$V_c = 11.9\ cm^3/mole$
$V_c = 12.0\ cm^3/mole$

.74 k bars
3.61 k bars
$V_c = 11.8\ cm^3/mole$
$V_c = 11.6\ cm^3/mole$

.81 k bars
3.61 k bars
$V_c = 12.6\ cm^3/mole$
$V_c = 13.0\ cm^3/mole$

.78 k bars
3.62 k bars
$V_c = 12.8\ cm^3/mole$
$V_c = 11.8\ cm^3/mole$

.81 k bars
3.59 k bars
$V_c = 12.8\ cm^3/mole$

.82 k bars
3.60 k bars
$V_c = 12.8\ cm^3/mole$

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