Reprinted from JOURNAL OF APPLIED PHYSICS, Vol. 34, No. 8 2254–2257, August 1963

Pressure Dependence of the Creep of Lead*

K. L. DEVRIES,† G. S. BAKER,‡ AND P. GIBBS§
University of Utah, Salt Lake City, Utah
(Received 19 September 1962)

Apparatus is described for maintaining hydrostatic environment, 1 in. in diameter by 4 in. long, with 8 to 12 electrical leads, up to 20-kbar pressure and various temperatures. Bending creep of 99.999+% lead is reported with an activation volume of about 21×10^{-24} cm^3, essentially independent of temperature between 0° and 57°C. Evidence for recrystallization is given.

INTRODUCTION

THE effect of pressure on properties of materials has been extensively reviewed.[1–3] Theories of creep have also been reviewed elsewhere.[4–6] Those relating creep to self-diffusion involve dislocation climb,[7,8] vacancy creep,[9,10] and motion of "joggy" dislocations[11] among other mechanisms. Bridgman[12] has investigated the effect of pressure on Young's modulus, the shear modulus, work hardening, elastic limit, ultimate strength, etc., for a large number of materials. Lazarus,[13] Daniels and Smith,[14] and Hughes *et al.*[15] have studied the effect of pressure on elastic constants of a number of solids. Christy[16] has reported the effect of pressure on creep in silver bromide. Butcher and Ruoff[17] have investigated the effect of pressure in 99.999% lead. The influence of pressure on self diffusion in sodium,[18] phosphorus,[19] zinc,[20] silver,[21] silver bromide,[22] and lead[23] has been found to approximate an equation of the type

$$D=D_0e^{-p\Delta V^\ddagger/KT},\tag{1}$$

where D is the diffusion coefficient, D_0 the value of D at zero pressure, p the pressure, $\Delta V^\ddagger$ the activation volume, T the temperature, and K Boltzmann's constant. Nachtrieb, Resing, and Rice[23] found a temperature-independent activation volume of 0.71 to 0.87 atomic volumes for self-diffusion in 99.999% lead between 253° to 301°C.

If creep were vacancy controlled, the activation volume for creep would be expected to be equal to that for self-diffusion, and hence given by

$$\Delta V^\ddagger=[KT/(p_2-p_1)]\ln(\dot{\epsilon}_1/\dot{\epsilon}_2),\tag{2}$$

where $\dot{\epsilon}_1$ is the deformation rate at pressure p_1. Butcher and Ruoff[23] reported the activation volume for creep in lead to be 0.80 atomic volumes at 70°C.

SAMPLE PREPARATION

Samples were prepared from 99.999+% lead.[24] Typical principal impurities were listed as Ag, Cu, Fe, and Bi, less than 1 ppm each. An ingot of the material was crushed in a press until it was approximately 0.375 cm thick. Beams were carefully cut from this with a hacksaw and carefully filed or sanded to various sizes 0.28 to 0.38 cm thick by 0.3 to 0.4 cm wide and 2.4 cm long, the sizes chosen to obtain about the same creep rate at different temperatures. Visual inspection of surfaces of the samples, after finishing revealed that the grain size varied from less than 0.1-mm to 1-mm diameter, with a mean size of approximately 0.5 mm. Sample 20 was melted and allowed to cool slowly, resulting in the center ⅔ of the sample's being one large grain.

---

* This work was supported by the U. S. Air Force Office of Scientific Research.

† The research is part of a dissertation submitted for Ph.D. in Physics at University of Utah.

‡ Present address: Aerojet General, Sacramento, California.

§ Address for 1963: Depto. De Fisica, Escola de Engenharia de Sao Carlos, Sao Carlos, Est. Sao Paulo, Brazil.

[1] C. A. Swenson, in *Solid State Physics*, edited by F. Seitz and D. Turnbull (Academic Press Inc., New York, 1960), Vol. 11, p. 41.

[2] K. L. DeVries, G. S. Baker, and P. Gibbs, *A Survey of High Pressure Effects of Solids*, WADC Technical Report 59-341 (1960).

[3] P. W. Bridgman, *The Physics of High Pressure* (G. Bell and Sons, Ltd., London, 1949).

[4] *Creep and Recovery* (American Society for Metals, Cleveland, Ohio, 1957).

[5] E. Dorn, Editor, *Mechanical Behavior of Materials at Elevated Temperature* (McGraw-Hill Book Company, Inc., New York, 1961).

[6] H. G. Van Bueren, *Imperfections in Crystals* (North-Holland Publishing Company, Amsterdam, 1961).

[7] J. Weertman, J. Appl. Phys. **26**, 4213 (1955).

[8] J. Lothe, J. Appl. Phys. **31**, 1077 (1960).

[9] F. R. N. Nabarro, *Proceedings of the Conference on Strength of Solids* (The Physical Society, London, 1948), p. 75.

[10] C. J. Herring, J. Appl. Phys. **21**, 437 (1950).

[11] A. Seeger, Phil. Mag. **46**, 1169 (1955).

[12] P. W. Bridgman, *Studies in Large Plastic Flow and Fracture* (McGraw-Hill Book Company, Inc., New York, 1951).

[13] D. Lazarus, Phys. Rev. **76**, 545 (1949).

[14] W. B. Daniels and C. S. Smith, Phys. Rev. **111**, 713 (1958).

[15] D. S. Hughes, E. B. Blankenship, and R. Mins, J. Appl. Phys. **21**, 294 (1950). [See also Geophysics **16**, 577 (1951).]

[16] R. W. Christy, Acta Met. **2**, 284 (1954).

[17] B. M. Butcher and A. L. Ruoff, J. Appl. Phys. **32**, 2036 (1961).

[18] N. H. Nachtrieb, J. A. Weil, E. Catalano, and A. W. Lawson, J. Chem. Phys. **20**, 1189 (1952).

[19] N. H. Nachtrieb and A. W. Lawson, J. Chem. Phys. **23**, 1193 (1955).

[20] T. Liu and H. G. Drickamer, J. Chem. Phys. **22**, 312 (1954).

[21] C. T. Tomizuka, in *Progress in Very High Pressure Research*, edited by F. P. Bundy, W. R. Hibbard, and H. H. Strong (John Wiley & Sons, Inc., New York, 1961); (also private communication 12 February 1962).

[22] D. S. Tannhauser, J. Phys. Chem. Solids **5**, 224 (1958).

[23] N. H. Nachtrieb, H. A. Resing, and S. A. Rice, J. Chem. Phys. **31**, 135 (1959).

[24] Purchased from American Smelting and Refining Company, Central Research Laboratories, South Plainfield, New Jersey.

APPARATUS AND EXPERIMENTAL PROCEDURE

The composite high-pressure vessel used in these studies is shown schematically in Fig. 1. With piston and bottom plug in place, it provided a working space 1 in. in diameter by 4 to 5 in. long. It has been tested to 20 kbar, although the present creep measurements were only carried on up to 10 kbar. The piston was sealed with a Bridgman unsupported area seal, using polyethylene as the soft seal material. The bottom plug, shown in Fig. 2, introduced 8 (or sometimes 12) electrical leads into the pressure vessel. Figure 2 also illustrates the means by which the creep apparatus was mounted on the bottom plug for easy assembly in the pressure vessel. Pressure was determined from the force applied to the piston, calibrated with 2.1% gold-chrome[3] and also manganin[1,3] coils.

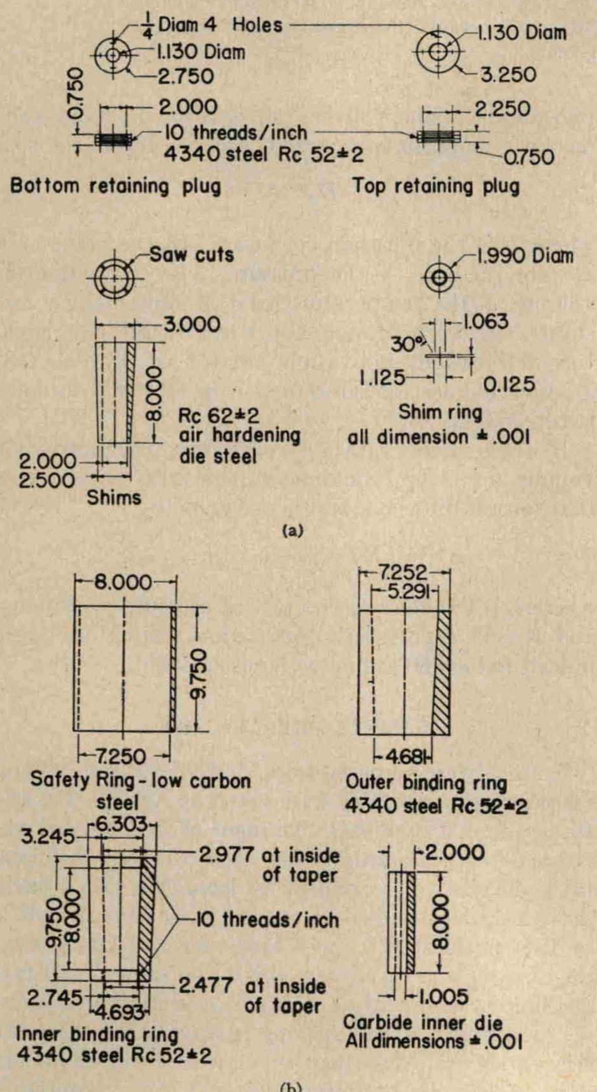

FIG. 1. (a) and (b), high-pressure vessel component parts shown in partial cross section. Safety ring, outer binding ring, inner binding ring, shims, and die are assembled successively in a hydraulic press. Shim rings are then introduced between both screw-in retaining rings and die.

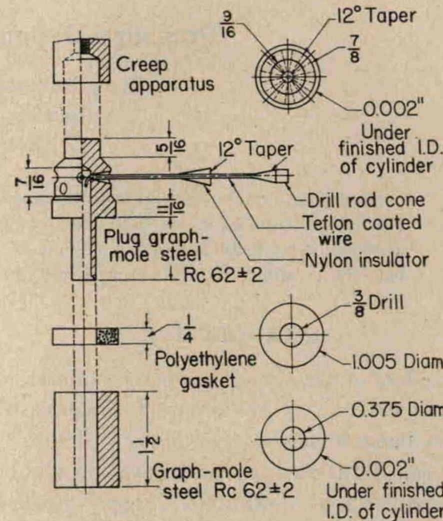

FIG. 2. High-pressure plug with 8 electrical leads (dimensions are in inches).

The temperature was controlled by a continually stirred water bath in which the pressure vessel was completely immersed. Heat was provided by a 1 kW immersable heater regulated by a mercury control and relay.[25] The temperature varied by less than $\pm\frac{1}{2}$°C as continually recorded on a Bristol Dynamaster dc potentiometer. In room-temperature studies, the ambient was controlled to ±2°C. Low-temperature studies were made with the bath packed in ice, with a maximum temperature variation of 2°C during creep.

Figure 3 shows schematically the creep stressing apparatus, which was surrounded by a stainless steel tube. The sample rested in slots in the tube which served as knife edges for 3-point loading. The other knife edge consisted of a wire loop attached rigidly to the spring which supplied the stressing force. Since a spring was used to load the sample, the load changed slightly with deformation. However, weak springs with

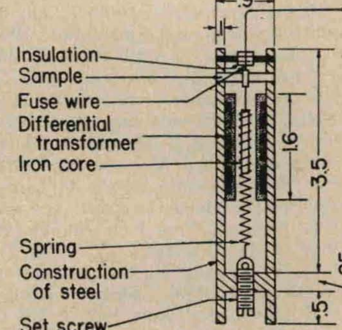

FIG. 3. Cross section of creep apparatus (dimensions are in inches).

[25] Type CF-708, Philadelphia Scientific Glass Company, Quakertown, Pennsylvania.