Elasticity and Creep of Pb Single Crystals

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The rigidity modulus and Young's modulus of lead single crystals have been measured by static methods as functions of crystal orientation. The elastic parameters so obtained are in agreement with the previous values determined dynamically by Goens and Weerts. The bending-torsion effect is large for certain orientations, and its effect upon the measurement of rigidity modulus by torsion has been studied. The elastic limit for lead under longitudinal stress occurred when the resolved shear stress on the most favorably placed octahedral plane was about 2×10^6 dynes per cm^2. Observations have been made of the transient creep under longitudinal stress. The creep strain velocity at any instant was found to be proportional to the difference between the final strain reached under that load and the strain present at the instant.

GOENS and Weerts[1] have measured the adiabatic elastic constants of lead from torsional and flexural oscillations of single crystal rods. The elastic region under steady stress has been studied by Deming[2] who measured Young's modulus by the bending beam method.[3] He found definite elastic behavior with a linear relation between stress and strain up to a resolved shear stress of about 2×10^6 dynes/cm^2, beyond which creep and permanent set (after removing the load) appeared. The values of Young's modulus for crystals with orientation functions near zero agreed fairly well with those of Goens and Weerts, but for other orientations the agreement was poor.[4]

In the following, the elasticity of lead under steady stress has been studied by determining the rigidity modulus for various crystal orientations with a torsion apparatus, and the Young's modulus by observing the increase in length under applied longitudinal stress. Some observations of creep have also been made with the latter method.

The lead was from the same lot used by Deming.[5] The preliminary castings were made in an iron tube[6] in the shape of cylinders about 15 cm long and 0.9 cm in diameter. After being sharpened to a point at one end, they were coated with Alundum cement, dried, rolled in moistened asbestos paper, and slipped into a brass tube. They were formed into single crystals by the Bridgman method of lowering through a furnace. The rate of growth was about 2 cm/hr. and the gradient was about 10°C/cm. The cement-asbestos paper coating was removed from the final crystal by soaking in water. This procedure was believed to be less likely to produce strains than other methods such as growing in a glass tube, etc. The orientation of each crystal was determined after etching[7] by the reflection of light from crystal planes by the procedure described by Webb.[3] The reflections were mainly from the {111} and {100} planes, although fainter reflections were also observed from the {110} and {113} planes.

RIGIDITY MODULUS

The elastic properties of a cubic crystal such as lead may be expressed in terms of three main parameters, s_{11}, s_{12} and s_{44}, when referred to the crystallographic axes. One may also use a set of axes, x', y', and z', not coinciding with the

[1] E. Goens and J. Weerts, Physik. Zeits. **37**, 321 (1936).
[2] A. Deming, M.S. thesis, State University of Iowa 1939, unpublished.
[3] Wayne Webb, Phys. Rev. **55**, 297 (1939), footnote 26.
[4] This is now believed to be due to the reciprocal bending-torsion effect, the influence of which could not be effectively estimated with the apparatus used by Deming.
[5] We are indebted to Professor H. F. Moore of the University of Illinois for the material. It has about 0.005 percent impurity, according to an analysis made by Baker, Betty, and Moore (Trans. Am. Inst. Min. and Met. Eng. **128**, 111 (1938), Inst. of Metals Div.).
[6] A spectrographic analysis showed no impurities were introduced.
[7] Two etch solutions were used; a stronger to remove oxide (for ½ hr.) and a weaker (for several hr.). The two solutions are composed of nitric acid, acetic acid and water in the proportions 1 : 1 : 2 and 2 : 3 : 20. This procedure was suggested by Deming and is a modification of the one used by B. B. M.

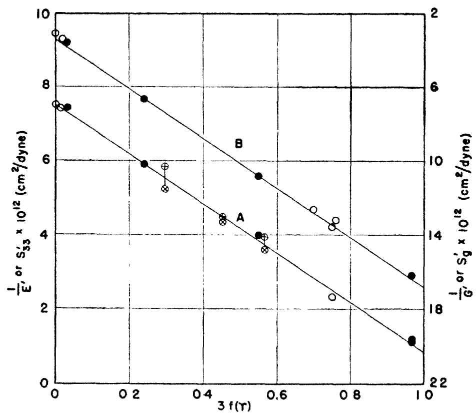

FIG. 1. Curve A, reciprocal of rigidity modulus; Curve B, reciprocal of Young's modulus, as functions of orientation. Lines and solid circles (●) are from Goens and Weerts. Other points are the writers' values. On Curve A, the symbols have the following significance: ○, no bending-torsion correction; ⊕, from "maximum slope" run; ⊗, from "minimum slope" run and correction factor ϕ.

crystallographic axes, and a set of "primed" parameters, s'_{ij}, which are, of course, functions of the main parameters and of the relative orientation of the two sets of axes. In general, a crystal either under axial torque or with an applied bending moment both twists and bends. With an appropriate choice of axes, the following equations[8] hold for a cylindrical crystal of length l and radius r:

$$\pi r^4\theta/2l = s'_g N - \bar{s}'_{34} M, \tag{1}$$

$$1/\rho = (2/\pi r^4)(\bar{s}'_{34} N - 2s'_{33} M), \tag{2}$$

in which θ is the angle of twist, N is the axial torque, ρ is the radius of curvature, and M is the component of the applied bending moment in the plane in which bending occurs. The parameters s'_g, s'_{33}, and $\bar{s}'_{34}$ are expressed in terms of the main parameters by the following:

$$s'_g = s_{44} + 4sf(\gamma), \tag{3}$$

$$s'_{33} = s_{11} - 2sf(\gamma), \tag{4}$$

$$\bar{s}'^2_{34} = 4s^2[(\gamma_1^6+\gamma_2^6+\gamma_3^6) - (\gamma_1^4+\gamma_2^4+\gamma_3^4)^2], \tag{5}$$

where

$$s = s_{11} - s_{12} - \tfrac{1}{2}s_{44}, \tag{6}$$

$$f(\gamma) = \gamma_1^2\gamma_2^2 + \gamma_1^2\gamma_3^2 + \gamma_2^2\gamma_3^2, \tag{7}$$

and the γ's are the direction cosines of the length of the crystal specimen with respect to the crystallographic axes. In the following, we shall define G' $(=1/s'_g)$ as the "free" rigidity modulus [N only; $M=0$ in Eq. (1)], and G as the modulus which is measured when bending is prevented [$1/\rho = 0$ in Eq. (2)]. If the crystal is free to bend, the expression for G' reduces to $G' = 2lN/\pi r^4\theta$, the usual "isotropic" formula. If the crystal is prevented from bending the reciprocal of the free modulus is given by[9] $1/G' = (1/G)\phi$ where $\phi = 1/[1-(\bar{s}'^2_{34}/2s'_{33}s'_g)]$ and G is given now by the "isotropic" formula. The correction factor ϕ is 1.00 for orientations in which the length of the specimen is in the [100] ($f(\gamma)=0$), [110] ($f(\gamma)=0.25$), and [111] ($f(\gamma)=0.33$) directions. It has a maximum value of about 1.25 for lead.

The torsion apparatus was of the usual type having for the measurement of the twist, θ, an optical system with a minimum observable angle of about one second of arc. The fixed end of the crystal was set in Wood's metal, and the movable end was held in jaws on a torsion head which was free to rotate in ball bearings. An approximately pure torque, clockwise or counter-clockwise, was applied to the torsion head by a suitable arrangement of four springs.

Three crystals for which the bending-torsion coupling was absent were grown and measured. The stress-strain[10] plots for these crystals were all straight and the slopes consistent, within the experimental error of about one percent. The points indicated by open circles (○), curve A, Fig.

TABLE I. Summary of data.

Crystal No.	$f(\gamma)$	ϕ	$\frac{1}{G'}\times 10^{12}$	$\frac{1}{E'}\times 10^{12}$	Stress at elastic limit§ Longitudinal	Resolved shear
1	0.000	1.00	6.91	(9.71)* 9.47	6.8	1.4
2	0.004[6]	1.02	7.11			
3	0.006			(9.61)* 9.31	4.1	1.8
4	0.099	1.21	11.50† 10.27‡			
5	0.151	1.19	13.3† 13.0‡			
6	0.189	1.12	14.8† 14.1‡			
7	0.233			4.70		
8	0.250	1.00	17.35	4.22		
9	0.254			4.41	4.3	1.8

* Before annealing.
† Computed from maximum slope.
‡ Computed from minimum slope and correction factor, ϕ.
§ In 10^6 dynes/cm^2.

[8] E. Goens, Ann. d. Physik **16**, 693 (1933).

[9] This may also be written in the equivalent form $1/G' = 1/G$ plus a "bending-torsion" correction. See W. A. Good, Phys. Rev. **60**, 605 (1941).

[10] More accurately—angle of twist *vs.* axial torque.

1, are the averages of a number of runs on these crystals and are in good agreement with the data of Goens and Weerts.[1]

For the crystals which had an appreciable bending-torsion correction ($\phi > 1$), the situation is more complicated. The ball bearings were not perfectly tight, but had a play estimated to be about 10^{-3} cm, which is actually about sufficient to allow free bending. Thus one might expect the stress-strain curve to have a slope corresponding to the free modulus (G'). If, however, the bearing reaction produced a partial restraint, the slope should correspond to something greater than G', i.e. nearer G. In the extreme case where the torsion head was initially definitely in contact with one side of the bearing torque in one sense would produce no bending while under the opposite torque very nearly free bending would occur. The stress-strain curve should then be two straight lines, one with a slope, for one sense of torque, corresponding to G, and the other to G'. These two cases are illustrated by curves C and D of Fig. 2, which are successive runs on crystal No. 5. In the first, only a pure torque was used, and the line is straight, with a slope fairly close to that corresponding to free bending. In curve D, a weight was hung on the torsion head sufficient to press it definitely against one side of the bearing. Since a constant bending moment (contrasted to a bending moment which is proportional to the torque) gives a line with a slope corresponding to G', a double slope line was expected and was actually obtained. The difference in the slopes of the two lines (14 percent) corresponds fairly well to the computed difference of 19 percent ($\phi = 1.19$) between G and G'. This experiment was repeated with another crystal with similar results. The same experiment with a crystal having no bending-torsion coupling gave a null result.

Of all the runs on the crystals having a bending-torsion coupling ($\phi > 1.00$), the results were about as follows: Either single straight lines were observed, with slopes corresponding fairly well to G', or they were of the double slope type. Examples of the latter are curves E and F, Fig. 2. From the various slopes observed for any one of these crystals (5 to 20 runs), the extreme values were picked out. The maximum was assumed to be that for free bending and G' was computed from it. The minimum value was assumed to give G and from it another value of G' ($1/G' = (1/G)\phi$) was computed. These two values are plotted in Fig. 1 with separate symbols and are given in Table I. The average of these two values should be and is reasonably close to the value of G'.

The diversity of slopes of the stress-strain relations for crystals with a large correction factor as compared with the constancy of slopes for

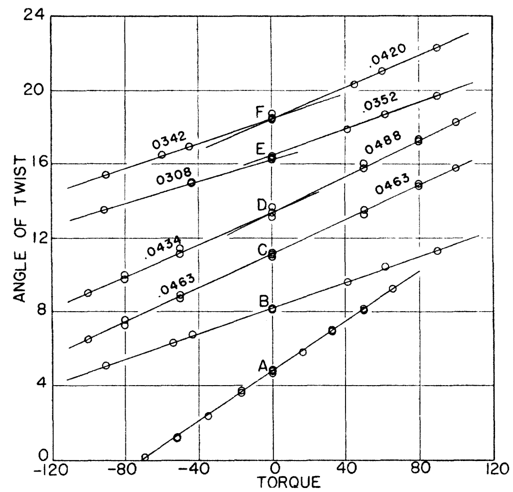

FIG. 2. Angle of twist against axial torque. Curve A is for a brass rod. Curves B to F are runs on lead single crystals. B and C are typical examples of straight line plots, while E and F are typical examples of runs of the double slope type. The slopes are shown by the numbers near the curves. The unit of torque is 1960 dyne cm and the unit of twist is 5.6×10^{-5} radian.

crystals with no (or very small) bending-torsion correction is shown in Fig. 3.

YOUNG'S MODULUS

The apparatus used for determining Young's modulus[11] and observing creep was developed particularly for measuring very small extensions of metal single crystals under longitudinal stress. It consists of an optical lever system attached to the specimen, a change in length of which causes the image of a scale to move past the cross hair in a low power microscope. The magnification of the device is about eight hundred. If one considers the smallest definite observable change in scale reading as 0.01 mm (one-tenth of the smallest scale division), the corresponding least observ-

[11] This is E' Eq. (4) and is $1/s'_{33}$.

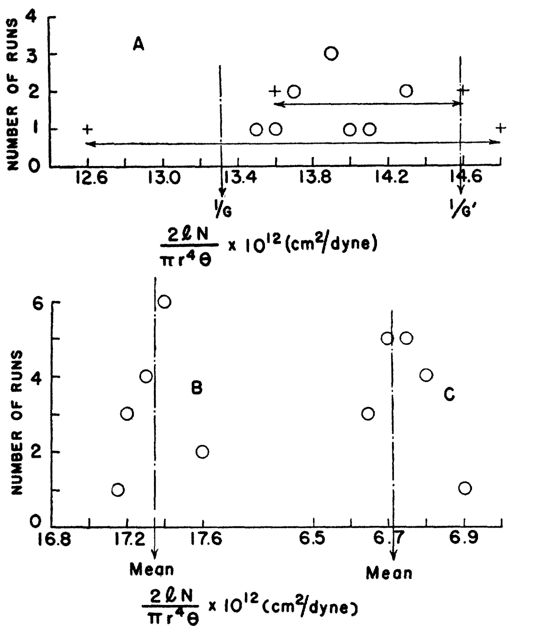

FIG. 3. The number of runs giving values of $2lN/\pi r^4\theta$ to the closest value plotted against these values. A is for a crystal (No. 6, $\phi=1.12$) which has a large bending-torsion coupling and B and C are for crystals having zero and small coupling (No. 8 and No. 2), respectively. A value of $2lN/\pi r^4\theta$ obtained from a straight run is plotted as a circle (○) while the two values obtained from a double slope are plotted as plus signs connected by the arrow ↔.

able increment of strain on a specimen 7 cm long is 1.8×10^{-7}. A fairly accurate stress-strain curve can therefore be run with the upper limit of allowable strain as low as 4×10^{-6} above the strain caused by the initial, or zero, load. By the use of a photoelectric multiplying device very similar to, and suggested by, one used by McKeehan and Cioffi,[12] the accuracy of observation is increased and the lower limit of observable strain reduced. With it, in fact, a 1-cm scale deflection of a galvanometer corresponds to a strain increment of about 2×10^{-8} (on a 7 cm length), and a determination of Young's modulus can be made with a total strain not exceeding about 10^{-6} beyond the initial value. The initial load was made small enough to produce in a crystal of 0.65 cm² cross section a stress of 0.6×10^6 dynes[13] per cm².

Young's modulus was determined by the photoelectric method for five crystals with orientation functions near zero and near 0.25. The results are plotted as open circles (○), Curve B, Fig. 1, and are given in Table I. They agree well with the adiabatic[14] determination of Goens and Weerts. The load-extension curves for both increasing and decreasing loads were straight lines, with no observable hysteresis or permanent set. Two of the crystals (see Table I) gave somewhat high values for $1/E'$ when first run but yielded values in agreement with Goens and Weerts after an anneal of three hours at 200°C, followed by slow cooling. Crystals which gave the expected value on the initial determination were not annealed.[15]

ELASTIC LIMIT AND PLASTICITY

Three of the crystals were taken into the plastic region. This was done mainly by the visual observation method. The load was increased by steps of about 275 grams. In all cases the load which caused the first deviation from a linear relation between load and extension was accompanied by easily observable creep and by subsequent permanent set when the load was removed. This was taken to be the elastic limit. Elastic limits so determined are given in Table I both as longitudinal stress (column 6) and as

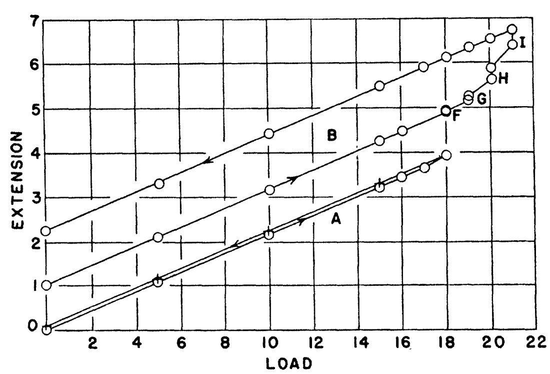

FIG. 4. Load against extension for crystal No. 1. Each unit of load produces a stress of 0.415×10^6 dynes/cm². The zero load is 0.67 dyne/cm². Each unit of extension corresponds to a longitudinal strain of 18×10^{-6}. Curve B was taken directly after curve A and the ascending branch coincides with the descending branch of curve A, but is offset to avoid confusion. The first elastic limit observed for this crystal was at 14 on the load scale.

[12] L. W. McKeehan and P. P. Cioffi, Phys. Rev. **28**, 146 (1926).

[13] For the orientation, $f(\gamma)=0$, with the smallest modulus this is an initial strain of about 5×10^{-6}.

[14] The theoretical difference between the reciprocals of the adiabatic and isothermal modulus is 0.016×10^{-12} cm²/dyne and is too small to show in the graph.

[15] Crystal No. 9 was also high, but this was a crystal with a somewhat irregular cross section. It had also had considerable previous use.

resolved shear stress (column 7). The resolved shear stress was obtained as follows. The glide plane is known to be the most favorably situated of the octahedral planes and the component of longitudinal stress parallel to this plane was obtained and then resolved into the nearest glide direction [110]. This resolved shear stress, for any orientation, comes out fairly close to four-tenths of the longitudinal stress, except for the case where glide may take place on two equally favorably situated glide planes. In this case the resolved shear stress is half as great as for resolution on a single plane. Crystals Nos. 3 and 9 seemed to satisfy the former condition, while crystal No. 1 was so nearly oriented along a cube edge that the assumption of double glide appeared correct. Computing the resolved shear stress on this assumption we obtain the values in the table. If single glide for No. 1 is assumed, a value twice as high is obtained.

It must be realized that any specification of elastic limit, the presence of permanent set, presence or absence of hysteresis, etc., are inherently indefinite and conditioned by the particular observational method used. Thus, the writers feel safe in saying that the load-extension curve is linear and the same for increasing and decreasing loads up to a fairly well-defined point, corresponding to a resolved shear stress of not more than 2×10^6 dynes per cm^2. It is implied, however, that this is true within the limitations of the apparatus, namely, the ability to determine a change in strain of about 2×10^{-7} which may occur in, say, five or six minutes. The same change occurring in five or six hours is not considered observable.[16] Elastic curves were obtained whether the runs were made in ten minutes or in one hour, but a run which took a week could conceivably be different.

A decrease in the load after the specimen had been taken slightly beyond the elastic limit produced a load-extension curve parallel to the increasing load curve, but offset by the amount of the plastic deformation. Curve A, Fig. 4 is an example of this behavior. Curve B, taken directly after A, shows the effect of again increasing the load. The first, very small creep was observed at F, the maximum load of the previous curve. At G, H, and I, the lower point indicates the reading taken as soon as possible after reaching[17] the indicated load. The higher point indicates the extension reached after several minutes when the creep had practically stopped. The curve from I down is parallel to the increasing load

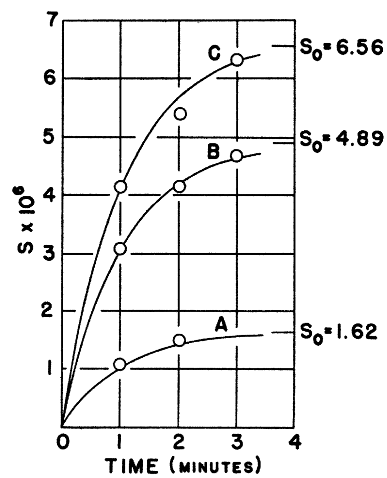

FIG. 5. Creep as function of time. Ordinate is longitudinal strain, S; abscissa, time in minutes.

curve. In general, after a specimen was left loaded until the creep seemed to stop, its subsequent behavior was elastic almost up to this point with no certain change in modulus (not more than 1 percent). The elastic limit of the crystal in Fig. 4 has been raised about 50 percent by other runs of this sort. No self-annealing at room temperature was observed, i.e., after the elastic limit was pushed up to a certain value, it stayed there.

The progress of creep with time at such points as G, H, and I, Fig. 4, is of some interest. Curves A, B, C, Fig. 5, depict this behavior. The strain existing at each of the lower points G, H, I in Fig. 4 is set equal to zero, with time equal to zero also, and the subsequent longitudinal strain ($S=\Delta l/l$) under the steady load is plotted as a function of time. The experimental points are

[16] The reason for this is that the apparatus is temperature sensitive, because of unequal expansion of crystal and measuring system. With the best compensation of this, good thermal insulation, etc., it is always probable that some small change taking place in several hours is due to temperature change. Long time observations were therefore precluded.

[17] The load was applied by gradually running mercury into a container hung from the lower end of the crystal. Adding the 275 gram increment took not more than twenty seconds.

indicated by circles and the curves are plotted from the equation:

$$S = S_0(1 - e^{-at}) \quad (8)$$

with a equal to unity and the values of S_0 shown on the curves. The constant a was found from suitable pairs of experimental points. The values actually found were: from points on A, 1.14; from B, 1.04, 0.87, 0.97; from C, 1.18, 0.70, 1.00. Another set of three similar curves, taken previously on this same crystal at somewhat lower loads, were also fitted quite well with a equal to unity. After several intervening runs, which pushed the elastic limit higher yet, a set of two curves taken beyond I, Fig. 4 (at 22.3 and 22.6 on the arbitrary load scale) was fitted by $S = 7.00(1 - e^{-0.36t})$ and $S = 7.29(1 - e^{-0.36t})$. It is to be noted that the a is definitely less. Although the fitting of such a curve to three points is not very sensitive to changes in a, a value of unity for a produced no fit at all.

The strain velocity, during creep, is given by

$$dS/dt = S_0 a e^{-at} = a(S_0 - S), \quad (9)$$

that is, the velocity is proportional to the creep (strain) still to be accomplished. If F, curve B, Fig. 4, is taken as the elastic limit, G, H, and I are 1, 2, and 3 load units, respectively, beyond this. The values of S_0 on curves A, B, and C, Fig. 5, are 1.98, 9.06, and 20.96×10^{-6} strain unit, respectively above the extrapolated elastic curve. These numbers are roughly in the ratio of 1 : 4 : 9, or proportional to the square of the load added above the elastic limit. Thus, if it is valid to extrapolate the strain velocity relation, Eq. (9), back to this point, such loads if applied suddenly would produce initial creep rates proportional to the square of the load above the then existing elastic limit. These initial strain velocities are, of course, equal to 1.98, 9.06, and 20.96×10^{-6} per minute. Chalmers[18] has found very similar results for tin, although he found a linear relation between strain velocity and stress in this region of "transient creep," and found that the previous history of the specimen had no effect.

The data on creep presented here are not very extensive but it seems that results of this type should throw useful light on the general problem of plasticity in metals and particularly the validity of Taylor's dislocation theory.[19] Thus the relations of Eqs. (8) and (9) may imply that under a given load there are a certain number of dislocations which are capable of moving (or disappearing) in such a way as to change the specimen length by a certain amount, and that the number of these which move (or disappear) per unit time is proportional to the number present at that time. A further increase in load then makes an additional number of dislocations effective, and so on throughout the region of transient creep. In this way one might, with more extensive data, arrive at quantitative results for density of dislocations, energy to move them, etc.

Finally, the writers do not believe that their data on elasticity warrant any change in the elastic parameters s_{11}, s_{12} and s_{44} from those recommended by Goens and Weerts. The isothermal parameters are, then, in cm²/dynes: $s_{11} = 9.28$, $s_{12} = -4.24$, $s_{44} = 6.94$, all $\times 10^{-12}$, and the compressibility, computed from these is 2.40×10^{-12} cm²/dyne.

[18] B. Chalmers, Proc. Roy. Soc. **156**, 427 (1936).

[19] See Frederick Seitz and T. A. Read, J. App. Phys. **12**, 470 (1941), particularly Figs. 43 and 44.