C00 - 2296 - 19

Diffusional Creep of Multicomponent Systems

Progress Report

J.C.M. Li

University of Rochester
Rochester, N.Y. 14627

February 1, 1978 - January 31, 1979

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

Prepared for the U.S. Department of Energy
under contract EY-76-02-2296*001

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED *EB*

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Abstract

The achievements this year can be summarized as follows:

1. Successful completion of photoelastic studies of three dimensional stress field caused by a cylindrical punch. A report is attached as Appendix I.
2. Successful completion of pencil slip studies in β -Tin single crystals. A report is attached as Appendix II.
3. Submission of the manuscript "Impression Creep of β -Tin Single Crystals" to Materials Science and Engineering. It has since been accepted for publication.
4. Equipment set up and preparation for experimental investigation of laser-excited diffusion and creep in solids. No positive results yet. Some negative results are described.

Photoelastic Studies of Three Dimension Stress
Field Caused by a Cylindrical Punch

This project is completed and a report is attached as Appendix I.

The highlights are:

1. The stress field (maximum shear stress) inside a cylinder caused by a coaxially loaded cylindrical punch is obtained by a stress-freezing technique.
2. At long distances from the punch (more than 3x punch radius) the observed stress field agrees very well with theoretical results using either the uniform displacement boundary condition or the uniform stress boundary condition.
3. At short distances from the punch (less than 3x punch radius), the observed stress field lies in between the two boundary conditions.
4. This result supports the use of mixed stress distributions under the punch in the previous finite element calculation of impressing creep velocities.

Pencil Slip in β -Tin Single Crystals

This project is also almost completed and a report is attached as Appendix II. Some highlights are:

1. The stress dependence of impressing velocity obeys a power law with a stress exponent of 4 for thick crystals, small stresses, or a rigid crystal support to prevent the bulge from forming.
2. For thin crystals, the impressing velocity of the punch is larger when a hole is provided in the crystal support to allow the bulge to form than when the hole is absent.
3. The exiting velocity of the bulge is about 40% of the entering velocity of the punch.
4. The slip lines on the surface can be explained by a cross slip mechanism.

Impressing Creep of β -Tin Single Crystals

A manuscript of this title has been submitted to Materials Science and Engineering and has been since accepted for publication.

This project was described in the last progress report.

Laser Activated Creep in Solids

A. Equipment set up

- a. The tunable laser system capable of providing laser radiation in wavelengths ranging from near ultraviolet (2500Å) to near infrared (2.6 μm) has been set up. The system included a Chromatix CMX-4 flash lamp pumped dye laser unit, an optical parametric oscillator, an automatic scanning unit, a helium-neon laser for optical alignment and a disc thermopile power detector. They are shown in picture 1.
- b. Since the laser pulse energy is very sensitive to the purity of the dye solution, normally a separate dye solution pumping module is needed for each dye. However, it would cost an additional total of nine thousand dollars. To avoid such expense an all stainless steel dye pumping module, which is easy for cleaning, was built along with a filter station which included twelve sets of microfilters and urethane tubings. They are shown in picture 2.
- c. An apparatus built for laser-activated creep studies is shown in picture 3. A small rectangular sample bar is compressed under a dead load. The compression is recorded by a LVDT as a function of time. The lower part of the apparatus is enclosed in a plastic box which serves as an environmental chamber for the sample. Dry nitrogen is sent into the chamber to keep moisture away. Quartz window is installed on the front wall of the chamber for the laser beam to enter the chamber. The laser beam strike the sample at the middle portion of the bar.
- d. An apparatus built for laser-activated impression creep testing is shown in picture 4. The punch is loaded horizontally on the surface of the sample. The load is transmitted through the level arm onto the

punch. The displacement of the punch is recorded as the deflection of the level arm. Laser light can be shone directly onto the surface of the sample.

- e. An apparatus for detecting laser activated hardness change in germanium is shown in picture 5. In this design, the indenter is attached directly to the core of the LVDT. The weight of the core and the indenter itself is used as the load applied on the sample. The entire arrangement is mounted on a rigid aluminum base which can rotate 360° in a vertical plane. The load on the indenter is varied by changing the angle of inclination of the loading axis. The laser beam can be shone directly at the spot of indentation. Any decrease in hardness or increase in the indentation will be sensed by the LVDT.
- f. An apparatus for coloring alkali halide crystals is shown in picture 6. It is a modified version of the apparatus originally designed by C.Z. Van Doorn.[†] A stainless steel wick is hanging near the cold zone inside the hot pipe to provide a place for the alkali metal vapor to condense. This arrangement is to avoid condensation of the vapor in the cold zone so that the metal vapor pressure can be maintained.

B. Experiments performed and some preliminary results.

- a. Conventional compression creep testing under laser radiation were performed on a naphthalene polycrystalline rod by using the apparatus shown in picture 3. Samples of about 2 mm diameter and 7 mm long were tested in compression. A laser beam of about 3 mm diameter was shone at the middle portion of the sample rod during creep. The compression

[†] C.Z. Van Doorn, Rev. Sci. Inst. Vol. 32, No. 6 755, 1961.

of the sample was recorded continuously during the test period.

Naphthalene is an ideal material for observing laser activated creep in solids. The activation energy for self-diffusion is 42.7 K cal/mole (J.N. Sherwood and D.J. White, Phil. Mag. 15, 745 (1967)). The half of which corresponding to the vacancy motion energy has excitation wavelength of $1.34 \mu\text{m}$ which is in the frequency range of our laser system. However, scanning of the wavelength from $.8 \mu\text{m}$ to $2.6 \mu\text{m}$ did not show any increase in creep rate.

- b. Conventional compression creep and impression creep tests under laser radiation were performed on germanium single crystals. The activation energy for self-diffusion in Ge (68.5 K cal/mole: H. Letaw Jr., W.M. Portnoy and L. Slifkin, Phy. Rev. Vol. 102, 3, 636 (1956)) is different from the activation energy for creep (38.1 K cal/mole: S. Schafer, Phy. Stat. Sol. 19, 297 (1967)). The half of these values corresponding to the wavelength of $.83 \mu\text{m}$ and $1.50 \mu\text{m}$. They are both in the frequency range of our laser system. However, frequency scan of the laser beam from $.75 \mu\text{m}$ to $2.6 \mu\text{m}$ did not show any effect on the creep rate.

Since germanium is not transparent to radiation for wave lengths longer than $1.8 \mu\text{m}$, it was thought that the laser light could not penetrate the bulk material and the effect on the surface may produce changes in creep rate too small to be detectable. A more appropriate test is obviously the impression creep test where the deformation is only limited to the surface layer. Impression creep test were done on Ge single crystal surface by using the apparatus shown in picture 5 under the intense laser radiation. Unfortunately, still no effect on the impressing velocity was found.

c. Laser activated surface hardness test on Ge single crystal. It was reported that the surface hardness of the germanium single crystal decreases when a light radiation of wavelength between 2.0 and 4.0 μm , or shorter than .4 μm is shone on the surface during the test. (G.C. Kuczyuski and R.F. Hochman, Phy. Rev. Vol. 108, 4, 946 (1957)). Attempting to repeat the test with a sharp conic indenter (see picture 5) by using monochromatic laser radiation was not successful. One of the reasons may be the limited wavelength range that the laser system can provide. For the long wavelength it can only provide radiation up to 2.6 μm , while in the short wavelength side the present system can only provide down to .45 μm . Therefore the very limited wavelength in the radiation may be used to explain the failure of the test.

Impression Creep in Bismuth and Bismuth Antimony Alloy

Both bismuth and bismuth antimony alloy single crystals are difficult to grow. Since mechanical twinning occurs very easily in bismuth single crystals, special technique has to be used to avoid twinning during crystal growth. Attempts to grow large twin free bismuth single crystals using the zone melting facility built for growing β -Tin single crystal in the previous study were not successful.

Due to the extreme easiness of mechanical twinning it may not be feasible to obtain meaningful results in impression creep testing on single crystals of specific crystal orientations. This project may not be continued.

Impression Toughness

Some work has been done in this area. But no conclusive results can be reported at present. By driving an alumina (Al_2O_3) single crystal

rod into an aluminum block using the Instron, load displacement curves similar to the stress-strain curve in the conventional test were obtained. Detail studies of the effect of punching velocity on the load displacement curves are currently underway. These tests will actually open up a new area of dynamic hardness measurements.

Microelasticity Measurement

The work in this area is still in the stage of instrumentation. Testing apparatus is expected to be completed in the near future and both experimental and theoretical work on anisotropic crystals will begin.

PICTURE 1

PICTURE 2

PICTURE 3

PICTURE 4

46 1320

PICTURE 5

46 1320

K&E 10 X 10 TO 1/2 INCH 7 X 10 INCHES
KEUFFEL & ESSER CO. MADE IN U.S.A.

PICTURE 6

Appendix I

Photoelastic Studies of Three Dimensional Stress Field Caused by a Cylindrical Punch

S.N.G. Chu and J.C.M. Li

Abstract

A stress-freezing technique is used to study the stress distribution inside a cylindrical body produced by a coaxially loaded cylindrical punch. The flat end of the punch is pressed against the top flat surface of a vertical cylinder. After the stress is frozen in, a thin vertical diametric slice is prepared for photoelastic observation. The stress distribution is compared with theoretical results calculated for a semi-infinite body using the following two boundary conditions: uniform displacement of the punch and uniform pressure under the punch. The agreement is good at long distances (3 times the punch radius) with either boundary condition. At short distances, the observed stress distribution is in between but close to the uniform displacement boundary condition. These results support the use of mixed stress distributions under the punch in a previous finite element calculation of impressing creep velocities.

Introduction

The problem of determining the stress field in a semi-infinite elastic solid produced by a rigid body of prescribed shape pressed against the free surface is known as Boussinesq problem [1]. Exact solutions for punches of simple shapes, such as spherical, conical and cylindrical, have been found by various methods. A summary of the results is given in Goodier et. al's review article [2]. However, the same problem in a finite elastic body has not been solved exactly. A finite element computer analysis of the elastic stress field produced by a cylindrical punch of flat end loaded coaxially on the flat end surface of a large cylindrical elastic body [3] showed that the result agrees very well with that of a semi-infinite elastic body. This is so because the radius of the cylindrical body to be pressed is much larger compared to the radius of the punch (30 times).

Two simple boundary conditions have been analyzed for the Boussinesq problem with a cylindrical punch of flat end. One is uniform displacement under the punch corresponding to a rigid punch material. The other is a uniform stress under the punch corresponding to a soft (fluid like) punch material. The situation for an actual punch material must lie somewhere in between the two extremum conditions. An example is our previous study on the finite element analysis of impression creep. In order to obtain a uniform velocity profile under the punch, the boundary condition used was somewhere between the two extrema.

By the stress-freezing technique, the stress distribution around the punch is measured in this study and compared with exact solutions for semi-infinite solids obtained by Sneddon et. al [4,5] and by Terazawa [6].

Experimental Procedure

A stress freezing technique was employed in this study. The three dimensional stress field produced by the punch was frozen inside an epoxy resin block and subsequently sliced into thin sections for photoelastic observation. The material used for stress freezing was PLM-4B epoxy resin obtained from Photoelastic Inc. The as received material was a precasted cylinder of 5" in diameter by 12" long. A stainless steel cylindrical punch of 5 mm diameter was used. A box furnace with loading arrangements was built to contain both the cylinder and the punch. The cylindrical punch was loaded coaxially on the top surface of the vertical epoxy cylinder inside the furnace. The load can be applied from outside the furnace.

A four-point-bending calibration sample bar was cut from the same material. This preloaded sample bar was hung inside the furnace and went through the same heating and cooling cycle as the punched cylinder.

The heating rate was about $.48^{\circ}\text{C}$ per hour until it reached 122°C . The load was then applied and the temperature was raised rapidly to around 125°C . The sample was soaked for about 5 hours so that the fringes could be fully developed. The furnace was then cooled down very slowly at a rate of $.16^{\circ}\text{C}$ per hour to about 80°C and then faster at $.33^{\circ}\text{C}$ per hour to room temperature.

A diametrial section parallel to the cylinder axis was cut out by a sharp bend saw. The thickness of the section was about $3/8$ inch. The thickness of the section was further reduced down symmetrically on both sides on a milling machine until the decrease in the number of fringes were proportional to the decrease in thickness of the section. The final thickness was less than 1.5 mm. The surface of the section was polished transparent and the isochromatics was taken with a photoelastic model 061 diffuse light

polariscope.

Cautions were exercised to avoid heating during the sawing, milling and polishing processes.

In addition to the punch with a straight edge, another punch with a rounded edge was used together with a thin sheet of teflon placed between the punch and the sample. According to St. Venant's principle, this new punch will produce the same stress field as the old one except perhaps at locations near the punch.

Theoretical Stress Distributions

As mentioned in the introduction, the stress distribution in a semi-infinite body due to a cylindrical punch of a flat end was solved by Sneddon, et. al [4,5] for the uniform displacement boundary condition and by Terazawa [6] for the uniform stress condition. The consistency of the two solutions was investigated by solving Terazawa's problem using Sneddon, et. al's method (Hankel transformation). Indeed it was found that the new method was simpler and gave identical results.

The stress distribution for the two boundary conditions are:

1. Uniform displacement boundary condition

$$U_z = \begin{cases} \epsilon & z = 0 \quad \& \quad r \leq a \\ 0 & z = 0 \quad \& \quad r > a \end{cases}$$

where U_z is the displacement in z direction and a is the radius of the punch.

$$\sigma_r = -\frac{\sigma_A}{2} [J_1^0 - \zeta J_2^0 - (1-2\nu) \frac{J_0^1}{\rho} + \frac{\zeta}{\rho} J_1^1]$$

$$\sigma_z = -\frac{\sigma_A}{2} [J_1^0 + \zeta J_2^0]$$

$$\sigma_\theta = -\frac{\sigma_A}{2} [2\nu J_1^0 + (1-2\nu) \frac{J_0^1}{\rho} - \frac{\zeta}{\rho} J_1^1]$$

$$\tau_{rz} = -\frac{\sigma_A}{2} \zeta J_2^1$$

where $\zeta = z/a$, $\rho = r/a$, $\sigma_A = \frac{L}{\pi a^2} = \frac{2E}{1-\nu^2} \frac{\epsilon}{\pi a}$ and

$$J_m^n = \int_0^\infty \rho^{m-1} \sin \rho e^{-\zeta \rho} J_n(\rho \rho) d\rho$$

where L is the applied load, E is Young's modulus and ν is the poisson ratio.

The integral J_1^0 can be generated from the Lipschitz integral

$$\int_0^\infty e^{-ax} J_0(bx) dx = 1/\sqrt{a^2 + b^2} \quad (a>0)$$

by substituting $\zeta + i$ for a and ρ for b . J_0^1 and J_1^1 can be generated from the integral

$$\int_0^\infty e^{-ax} J_1(bx) dx = (\sqrt{a^2 + b^2} - a)/(b\sqrt{a^2 + b^2}) \quad a>0$$

by the same method. Then

$$J_2^0 = -\frac{\partial J_1^0}{\partial \zeta} \quad \text{and} \quad J_2^1 = -\frac{\partial J_1^1}{\partial \zeta}$$

The results are:

$$J_1^0 = \frac{2\zeta}{(\sqrt{(A+B)^2 - 4}) AB}$$

$$J_0^1 = \frac{1}{\rho} \left(1 - \frac{2\zeta}{\sqrt{(A+B)^2 - 4}} \right)$$

$$J_1^1 = \frac{2}{\sqrt{(A+B)^2 - 4} (A+B)} \left(\frac{1+\rho}{A} - \frac{1-\rho}{B} \right)$$

$$J_2^0 = \frac{2}{(\sqrt{(A+B)^2 - 4}) AB} \left\{ \frac{4\zeta^2}{((A+B)^2 - 4) AB} + \frac{\zeta^2}{A^2 B^2} (A^2 + B^2 + AB) - 1 \right\}$$

where

$$A = \sqrt{\zeta^2 + (1+\rho)^2} \quad \text{and} \quad B = \sqrt{\zeta^2 + (1-\rho)^2}$$

These expressions can be shown to be equivalent to those obtained by Sneddon [4].

The contours of local maximum shear stress:

$$\tau_{\max} = \sqrt{\frac{(\sigma_r - \sigma_z)^2}{4} + \tau_{rz}^2}$$

is plotted in Fig. 2 for $\nu = .5$ which is the Poisson ratio for the epoxy resin at the temperature used in this study for stress freezing. It is seen that the stress has a singularity at the edge of the punch.

2. Uniform stress boundary condition

$$\sigma_z = \begin{cases} \sigma_A = L/a^2 & z = 0 \text{ and } r \leq a \\ 0 & z = 0 \text{ and } r > a \end{cases}$$

$$\sigma_r = -\sigma_A a \left[I_0^0 - z I_1^0 - (1-2\nu) \frac{I_1^1}{r} + \frac{z}{r} I_0^1 \right]$$

$$\sigma_z = -\sigma_A a \left[I_0^0 + z I_{-1}^0 \right]$$

$$\sigma_\theta = -\sigma_A a \left[2 I_0^0 + (1-2\nu) \frac{I_1^1}{r} - \frac{z}{r} I_0^1 \right]$$

$$T_{rz} = -\sigma_A a z I_{-1}^1$$

where

$$I_m^n = \int_0^\infty p^{-m} J_1(ap) J_n(rp) e^{-zp} dp$$

The integrals I_m^n were tabulated by Sneddon et. al [7]. The maximum shear stress were calculated using the tabulated values of the integrals. Graphical interpolations were used to obtain coordinates for the constant maximum shear contour. The contour of local maximum shear stress is shown in Fig. 3. Unlike Fig. 2, no singularity exists at the edge of the punch. The maximum shear stress is at some distance below the punch.

Both Figs. 2 and 3 resemble those of two dimensional isochromatics due to a strip punch [8].

Results and Discussion

The isochromatics of stress fringes under the punch in a rz plane are shown in Figs. 4 and 5. Fig. 4 is for the punch with the straight edge and Fig. 5 is for that with the rounded edge and a teflon sheet placed at the interface. It is seen that they are very similar to each other. To make quantitative comparisons, the (maximum) shear stress for each fringe can be calculated by

$$\tau_{\max} = CN/t$$

where t is the thickness of the section (1.29 mm for Fig. 4 and 1.41 mm for Fig. 5), N is the fringe order number and C is the stress optical coefficient. The fringe order number can be assigned by starting from the farthest fringe from the punch indentation with $N = 1$. The stress optical coefficient was obtained by using a four-point bending calibration bar as described before. Since the stress distribution in the bar is known, the value of C can be determined and found to be 41.2 kg/fringe/m for Fig. 4 and 40.3 kg/fringe/m for Fig. 5.

The calculated $\tau_{\max}/\sigma_A$ along the z -axis from the position of fringes taken as the middle of the dark region is compared with theoretical values for semi-infinite solid as shown in Fig. 6. The values calculated from both Figs. 4 and 5 are shown and it is seen that they agree with each other very well.

However, the stress distribution agrees with neither the uniform displacement boundary condition nor the uniform stress boundary condition. It lies somewhere between the two boundary conditions and closer to the uniform displacement condition. It is interesting to note that at 125°C the epoxy resin has a Young's modulus four order of magnitude lower than the stainless

steel punch and yet the stress distribution still deviates from that of uniform displacement boundary condition. This observation seems to support the use of a mixed stress distribution under the punch in our finite element calculations of impressing creep velocities. /

Conclusions

1. The stress freezing technique in the determination of three-dimensional stress distribution is found useful in the coaxial loading of a cylindrical punch of a flat end onto the flat surface of a large cylinder.

2. The stress distribution at long distances from the punch ($3 \times$ punch radius) is found to be the same as that calculated for a semi-infinite body using either of the following two boundary conditions: uniform displacement of the punch and uniform pressure under the punch. At short distances from the punch, the stress distribution is close to that of uniform displacement boundary condition and lies between the stress distributions of the two boundary conditions.

3. The stress distribution caused by a punch of rounded edge and a thin teflon sheet placed at the interface is the same as that caused by a punch of straight edge and no teflon sheet.

4. The present results support the use of mixed stress distributions under the punch in a previous finite element calculation of impressing creep velocities.

References

1. J. Boussinesq, Application des potentiels a l'etude de l'equilibre et du mouvement des solides elastiques, Gauthier-Villars, Paris, 1885.
2. J.N. Goodier and P.G. Hodge, Survey in Applied Mathematics: I. Elasticity and Plasticity, New York, 1958.
3. H.Y. Yu and J.C.M. Li, J. Mat. Sci. 12 (1977) 2214-2222.
4. I.N. Sneddon, Proc. Cambridge Phil. Soc., 42 (1946) 29.
5. J.W. Harding and I.N. Sneddon, Proc. Cambridge Phil. Soc., 41, (1945) 16.
6. K. Terazawa, J. Coll. Sci. Imp. Univ. Tokyo, 37 (1916) article 7.
7. G. Eason, B. Noble and I.N. Sneddon, Phil. Trans. A 247 (1955), 529.
8. A. Nadai, "Plasticity", McGraw-Hill, New York 1931 p. 248-249.

46 1320

K&E 10 X 10 TO 1/4 INCH 7 X 10 INCHES
KEUFFEL & ESSER CO. MADE IN U.S.A.

FIG. 1

Const. Displacement

0

14

2

46 1320

K-E 10 X 10 TO 1/2 INCH 7 X 10 INCHES
KEUFFEL & ESSER CO. MADE IN U.S.A.

1

.289

.267

.214

.187

2

.133

3

.080

.054

.027

Fig. 3

Fig 4

FIG. 5

FIG. 6

Appendix II

Pencil Slip in β -Tin Single Crystals

S.N.G. Chu and J.C.M. Li

Abstract

Pencil slip is observed when a punch is pressed over the (001) surface of a β -Tin single crystal. A bulge of the same size and shape as the punch appears at the opposite surface directly across the thickness of the crystal. The development of the bulge was affected by punching stress, punching time, and the ratio between the diameter of the punch and the thickness of the crystal. The effects of punching stress on the entering (impressing) velocity of the punch and on the exiting velocity of the bulge are reported. Dislocation mechanisms consistent with slip line observations are proposed.

Introduction

Pencil slip or prismatic punching is a rare slip phenomenon. It occurs only in crystals with a unique crystallographic slip direction and several slip planes which can form a closed prism whose axis is parallel to the slip direction. When a concentrated stress, such as that due to a punch, is applied to the crystal surface in the slip direction, the material will glide along the prism planes as if a prism or a cylinder of material is pushed out of the solid.

The phenomenon was first discovered in 1910 by Taricco [1,2] in a PbS single crystal. When a blunt indenter was tapped sharply on the (001) cleavage face, a square pyramid rose on the opposite (00 $\bar{1}$) face with the bases of the pyramid parallel to [001] and [010] directions. Mügge [2,3] in 1914 observed the same phenomenon in tetragonal phosgenite and showed that the mechanisms for both PbS and phosgenite were caused by crystallographic glide. Similar phenomena were observed in b.c.c. iron [4], mercury [5] and silver chloride [6] by showing that they sometimes have corrugated slip surfaces made up of strips of slip planes bounded by a common slip direction. On a macroscopic scale the apparent slip surface may not be a crystallographic plane. The slip surface is determined by maximum resolved shear stresses. Taylor in 1926 [4] was the first to model the slip by a bundle of rods slipped along their length. Later Nye [6] in 1949 called it pencil slip. In the same year (1949) Smakula and Klein [7] demonstrates that the strain produced by a blunt punch on (100), (110) or (111) faces of a b.c.c. thallium halide single crystal can be transmitted along the cubic directions to the opposite cubic faces where it appears as truncated square pyramids with edges parallel to the

$\langle 110 \rangle$ directions. Seitz [8] in 1950 named this phenomenon prismatic punching and proposed prismatic dislocations of $\langle 100 \rangle$ Burgers vectors gliding along the prism formed by $\{011\}$ slip planes as the mechanism for transmitting the strain.

Although in f.c.c. metals, the (111) and $(\bar{1}\bar{1}1)$ planes form a closed prism with axis parallel to the $a/2 [0\bar{1}1]$ Burgers vector, no pencil slip or prismatic punching has ever been observed. Nabarro [9] attributed that to the possible dissociation of the $a/2 [1\bar{1}0]$ Burgers vector into two permissible $a/\sqrt{2} [\bar{1}01]$ and $a/\sqrt{2} [1\bar{1}0]$ Burgers vectors during deformation. Unless both of them slips by exactly the same amount, the general direction of the slip will not be along the prism axis.

In this report, complete pencil slip caused by a cylindrical punch is observed for the first time in a metal single crystal. β -Tin has a body center tetragonal structure with a c/a ratio of .5457. Pencil slip is found to occur in $[001]$ direction surrounded by (110) and $(\bar{1}\bar{1}0)$ planes. The effects of punch size and shape, punching stress and crystal thickness on the shape and velocity of the cylinder (or pencil) slipping out of the opposite surface are studied. A possible slip mechanism is proposed for material transport underneath the punch.

Experimental Techniques

Single crystal ingot of β -tin was grown by vacuum zone melting. β -Tin granules of three nines pure were zone refined prior to crystal growth. The single crystal ingot was believed to be better than four nines pure. Crystal orientations were determined by x-ray back-reflection method. Slabs parallel to (001) plane were acid sawed from the single crystal ingot. The surfaces were further polished flat on an acid planer. Two sample thicknesses, 4.3 mm and 11.5 mm, were used.

To demonstrate the pencil slip in β -tin, two different kinds of punches were used. One was cylindrical in shape and the other was a punch of the letter Y. Quantitative measurement of entering velocity of the punch (impressing velocity) and the exiting velocity of the bulge at the other side of the crystal were made as a function of punch stress and the size of the punch. These experiments were done on an impression creep apparatus described elsewhere [10] with a modification in the crystal support inside the furnace to allow the material to slip out of the bottom surface of the crystal slab and at the same time to record the height of the bulge as a function of time. After the test the bulge was polished off on emery paper so that the crystal can be used again by punching at another location. All the tests, except the one with the letter punch, were done in air at a temperature of $29.7 \pm 0.4^\circ\text{C}$. The test for the letter punch was done at room temperature in air by placing the single crystal slab on a thin sheet of rubber or a thick paper towel and tapping gently with the letter punch on the top surface of the crystal.

Experimental Results

1. Demonstration of Pencil Slip

Figure 1 illustrates a typical pencil slip in β -tin single crystal along $[001]$ direction caused by a flat end cylindrical punch loaded perpendicular to the (001) surface. The diameter of the punch was .53 mm and the thickness of the crystal was 4.3 mm. The upper picture shows the hole created by the cylindrical punch on the top (001) surface and the lower picture shows the cylinder (bulge) slipped out of the bottom $(00\bar{1})$ surface of the crystal slab. Some general features of the bulged out cylinder are:

- a. The cross-sectional area of the cylinder increases slightly towards the base of the cylinder.
- b. Except near the end of the cylinder where the cross section has the same shape and size as the punch, the cross section gradually deviates from circular shape to a square pyramid with the edges of the base parallel to $\langle 110 \rangle$ directions. This is shown more clearly in Figure 2.
- c. Large slip steps appear on the cylinder surface.
- d. The height of the cylinder is always less than the depth of the hole.

To demonstrate pencil slip for a punch of more complicated geometry, Figure 3 shows the pencil slip due to a letter punch. The depth of the impression is less than .5 mm. A letter Y of exactly the same size as the impression appeared on the opposite $(00\bar{1})$ surface directly underneath the impression.

2. Punching stress dependence of the impressing velocity.

The punching stress dependence of the impressing velocity was determined for both with and without a hole in the crystal support. A hole larger than the punch and directly under it was to allow the material to bulge or slip out of the crystal. Without such a hole, the support was rigid enough to prevent any material from bulging out. The results are shown in Figure 4. The punch diameter was .77 mm and the sample thickness was 4.3 mm which gives the diameter to thickness ratio of .177. The punching stress dependence of the impressing velocity without the hole obeys a power law with a stress exponent of 4 which is consistent with the results of a previous study [10]. The impressing velocity with the hole is consistently higher than the one without the hole and also the former increases more rapidly with applied punching stress than does a power law. However, at low stresses, the slope of the curve approaches the value of 4. It means that, at very low stress, the effect of the hole disappears.

3. The effect of diameter to thickness ratio

To investigate the effect of the diameter to thickness ratio on the entering (impressing) velocity, another punch of .53 mm instead of .77 mm was used on the same sample (4.3 mm thick). In this case, the diameter to thickness ratio was .123 instead of .177 for the previous case. The effect of punching stress on impressing velocity is shown in Fig. 4 and it is seen that the effect of the hole in the crystal support disappeared for this diameter/thickness ratio. This happened despite the fact that there was a small bulge appeared at the bottom surface. Another experiment was carried out for a punch diameter of .608 mm and a sample

thickness ratio is .053. This is shown in Fig. 5. It is seen that a power law of a stress exponent of 4 appeared again as though the hole was not there in the crystal support.

4. The punching stress dependence of the bulging velocity

Figure 6 shows the effect of punching stress on the velocity of the exiting material at the bottom surface measured during the same test as the one with diameter to thickness ratio of .177 as shown in Fig. 4. Only the velocities in the high stress region were recorded. They exhibited the same stress dependence as the impressing velocity of the punch. However, the bulging velocity of the exiting material is only about 40% of the impressing velocity of the punch. In the low stress region, the exiting velocity was erratic and hence not recorded.

5. The topography of the bulge

The shape of the bulge usually varies with the applied punching stress. For very low stress, the bulge has the same shape as the punch. When the punching stress increases, the cross-sectional area of the bulge increases also and slip steps appear on the side surfaces. For high punching stress, the bulge becomes pyramidal in shape. Figure 2 shows such a graduate change of bulge shape when the punching stress was raised by small steps. Figure 7 shows an abrupt change of shape from cylinder to pyramid when the punch is subjected to a large increase in stress. Especially for the case of thin crystal with a large punch diameter such as the case for the diameter to thickness ratio of .177, not only the bulge grows into a pyramid, the indentation also grows into an inverted pyramid as shown in Figure 8. This behavior approaches a case of perfect pencil

slip so that other slip systems are no longer needed for continued deformations. This may be the reason for increased impressing velocity as well as the high stress dependence.

Discussion

One of the important findings in this study is that pencil slip in most cases does not play an important role in impression deformation. Except for the diameter to thickness ratio greater than .177, the material pushed by the punch into the bulk of the crystal is always more than the material slipped out of the bottom surface. Since material can not be accumulated too much directly underneath the punch, it has to be transported away by a certain dislocation mechanism. One of the possibilities is by cross slip. Another is the climb of prismatic loops. However at 30°C, dislocation climb is unlikely to be a dominant mechanism. This has been confirmed in the previous study [10]. If cross slip is operative, it may interfere with pencil slip. The thicker the crystal or the higher the stress, the less is the possibility for a prismatic loop to survive the journey to reach the bottom surface.

β -Tin has a body center tetragonal structure with a c/a ratio of .5457. The possible slip systems for [001] compression are (110) [001], (100) [001], (110) $[\bar{1}\bar{1}1]$ and (121) $[\bar{1}01]$ types of systems. They are shown in Figure 9. Among these possible slip systems, only [001] and $\langle 111 \rangle$ types of Burgers vectors were found operative along (110) and $(\bar{1}\bar{1}0)$ planes [12]. The [001] Burgers vector with (110) and $(\bar{1}\bar{1}0)$ slip planes form the unique system for pencil slip while the $\langle 111 \rangle$ Burgers vectors are the main system for cross slip. The combined [001] and $\langle 111 \rangle$ Burgers vectors provided a way for the prismatic loops to glide along the prisms formed by two pairs of the four (101) types of slip planes, thereby transport the material away from the highly compressed region underneath the punch. This is shown in Figure 10 for a square prismatic loop. Since the stress decreases rapidly with increasing distance from

the punch the whole loop may not be able to reach the surface. However, the portion near the punch may still emerge to the surface and produce a slip line around the impression parallel to $\langle 110 \rangle$ direction. This is consistent with the observation in the previous study [10].

Conclusions

1. Pencil slip is observed for the first time in β -tin single crystals in the $[001]$ direction along (110) and $(\bar{1}10)$ slip planes. When a punch is pressed over the (001) surface, a bulge of the same size and shape as the punch appears at the opposite surface directly across the thickness.
2. The stress dependence of the entering (impressing) velocity of the punch obeys a power law with a stress exponent of four for thick crystals, small punching stresses, or a rigid crystal support to prevent the bulge from forming.
3. For thin crystals or when the ratio of the punch diameter to sample thickness is larger than 0.1, the impressing velocity of the punch is larger when a hole is provided in the crystal support to allow the bulge to form than when the hole is absent. With the hole in the support the stress exponent is 9 which is larger than four. However, these behaviors disappear at low stresses.
4. The stress dependence of the exiting velocity of the bulge also obeys a power law with a stress exponent of 9. However, the exiting velocity is about 40% of the entering velocity of the punch.
5. A possible cross slip mechanism is proposed for the case of thick crystals, small punching stresses, or a rigid crystal support to prevent the bulge from forming. The extra material can be transported to the top surface by this mechanism.

References

1. M. Taricco, Atti. Accad. Nag. Lincei Rc. 19/2 508 (1910).
2. F.R.N. Nabarro, "Theory of Crystal Dislocation", p. 264, Oxford 1967.
3. O. Mügge, Neues Jb. Miner. 1914/1, 43.
4. G.I. Taylor and C.F. Flam, Proc. Roy. Soc., A112, 337 (1926).
5. K.M. Greenland, Proc. Roy. Soc. A163, 28 (1937).
6. J.F. Nye, Proc. Roy. Soc. A198, 191 (1949).
7. A. Smakula and M.W. Klien, J. Opt. Soc. Am. 39 445 (1949).
8. F. Seitz, Phys. Rev. 79, 723 (1950).
9. F.R.N. Nabarro, Phys. Rev. 79, 894 (1950).
10. S.N.G. Chu and J.C.M. Li, to be published in J. of Mat. Sci. and Eng.

FIG. 1

Fig. 2

F14.3

FIG. 4

Sample Thickness: 4.3 mm
 A $d: .77 \text{ mm}$ $d/\lambda = .177$ $T = 24.6 \pm 3^\circ \text{C}$
 B $d: .53 \text{ mm}$ $d/\lambda = .123$ $T = 24.8 \pm 3^\circ \text{C}$
 C $d: .41 \text{ mm}$ $d/\lambda = .092$ $T = 24.8 \pm 3^\circ \text{C}$

FIG 5

$d = 0.000 \text{ m}^2$
 $T = 11.5^\circ \text{ m}^2$
 $T = 298 \pm 3^\circ \text{C}$
 $d/l = 0.053$
 $\alpha = 4.0 \times 10^{-3}$
 $\omega = 0.0001$

$d = .77 \text{ mm}$
 $T = 4.5 \text{ s}$
 $\lambda_{\text{th}} = .177$
 $\tau = 27.6 \pm 3.2$

10^7
 10^8
 F14.6

FIG 7

46 1320

FIG. 8

Slip Systems in β -Tin Single Crystal
(BCT Structure $c/a = 0.5457$)

FIG. 9