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Effect of Purification on Basal Cleavage in Beryllium Single Crystals

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by

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The deformation of impure beryllium crystals by basal glide at room temperature invariably terminates by basal cleavage after a few percent strain. It is generally accepted that fracture of this type is caused by the splitting of low-angle boundaries, or bend planes, by obstacles that restrict the motion of the bend planes in the deforming crystal. The details of such a process have been developed by Stroh, who showed that the high tensile stress normal to the basal plane in the region where the bend plane is split results in the propagation of a basal cleavage crack, provided there is no available deformation mode having a shear component normal to the basal plane.

It has been postulated that purification of beryllium might lead to increased basal ductility by the removal of barriers to bend plane motion and possibly by the activation of non-basal deformation systems. Large increases in the amount of basal glide that can be sustained prior to fracture have been observed in crystals purified by zone-refining. In each case, eventual fracture was by sharp basal cleavage, suggesting that the split-bend plane model still applies to the fracture of beryllium of reasonably high purity.

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In the present work, calculations are made to show to what extent basal ductility can be increased by purification while the split bend-plane fracture process remains applicable. Experimentally observed ductilities are generally somewhat lower than those predicted by calculation because of premature failure due to defects introduced during specimen preparation and testing.

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Effect of Purification on Basal Cleavage in Beryllium Single Crystals

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Several investigators (1-4) have studied the deformation characteristics of beryllium single crystals of commercial purity, and have related the strong anisotropy of plastic deformation in single crystals to the problem of brittleness in polycrystalline beryllium. For most orientations, the principal deformation mode is basal slip, which, at temperatures between 300°K and 900°K begins at a critical resolved shear stress of approximately 1500 gms/mm^2, and terminates after 3-6% glide strain by cleavage on the basal plane in the neighborhood of $(11\bar{2}0)$ bend planes that are formed during the deformation.

This type of fracture, which is also observed in zinc at low temperatures, has been interpreted by Stroh (5) to be caused by the splitting of bend planes by obstacles to their motion. In the Stroh model, a $(11\bar{2}0)$ dislocation wall, or bend plane, moving along the slip direction under the action of the applied stress, encounters obstacles which cause part of the bend plane to be held up, the other part continuing to move. Stress concentrations are built up in the region where the bend plane is split, with a high tensile component normal to the basal plane, resulting in a microcrack at the end of the split wall. Because no deformation mode having a shear component at an angle to the basal plane is available to relieve the stress at the crack tip, the crack can propagate under a sufficiently high applied stress.

The Stroh criterion for crack propagation is given by

$$\frac{(\tau_s - \tau_o) D\sigma_n}{\text{Cos } \lambda} = \frac{\gamma}{\pi B} \ell n \frac{\theta}{B\tau_s} = k' \qquad [1]$$

where τ_s = resolved shear stress on the basal plane at fracture

τ_o = critical shear stress for basal glide

σ_n = resolved normal stress on the basal plane at fracture

D = minor diameter of the crystal at fracture

λ = angle between the tension axis and the slip direction at fracture

B = a complex function of elastic constants. $\frac{2}{B}$ is approximately equal to a mean elastic extensional modulus (6).

θ = misorientation across bend plane.

The small dependence of the logarithmic factor on θ and τ_s justify treating the right-hand side of [1] as a material constant. Crack propagation is thus pictured to take place when the value of the left-hand side of [1] reaches a critical constant value.

By assuming the existence of a linear stress-strain curve, Green and Sawkill (6) postulated that, on the basis of the above model, the shear strain prior to fracture is given by

$$a = \frac{\cos \lambda_o \cot \lambda_o}{D_o h \, \tau_o \tau_s} \cdot k' \qquad [2]$$

where $h = \frac{1}{a} \frac{\tau_s - \tau_o}{\tau_o}$.

D_o = initial diameter of crystal

λ_o = initial angle between tension axis and slip direction.

This expression describes the shear strain for small amounts of strain only, since changes in λ and D with deformation are not accounted for. Since large strains are not observed in commercial purity beryllium, [2] is in fairly good agreement with experimental results. Further support for the operational validity of the Stroh model is derived from the metallographic observation that basal microcracks are generally associated with bend planes in the manner suggested by Stroh (1).

The particular sequence of events that must take place for Stroh cracks to propagate suggests several possible effects of purification on basal cleavage in beryllium. It is evident from [2] that a reduction in critical shear stress by purification should result in larger amounts of strain to fracture. Similarly, a reduction in work-hardening rate should promote ductility. Physically, this would occur by a reduction in the barriers to the motion of bend planes. Finally, if it were possible for new slip systems to be activated by purification, a mechanism for the relief of stresses at the tip of a crack should be realized, and basal cleavage might no longer occur.

In crystals purified by floating-zone-refining, Herman and Spangler (7) have observed basal glide strains as high as 220%. The observed increases in ductility with purification appeared to be qualitatively consistent with predictions based on the Stroh fracture criterion, in that both yield stress and work hardening rate decreased with increasing purity. The eventual termination of deformation, however, was by basal cleavage at all purity levels studied, indicating that satisfaction of the conditions for propagation of Stroh cracks was merely displaced to higher strains by purification, rather than being suppressed completely.

A quantitative treatment of Herman and Spangler's data is difficult, first, because the relative purities of their crystals were not measured, and second, because, as they point out, the observed ductilities are infulenced not only by purification, but also by factors such as accidental surface damage and bending constraints in testing. Such factors, which are always present to some extent, tend to cause premature failure, resulting in underestimates of ductility.

In the present work, an attempt is made to obtain a quantitative estimate of the effect of purification on basal ductility in the absence of adverse effects imposed by testing conditions, and to compare this estimate with experimentally determined ductilities.

ESTIMATE OF THE PURITY EFFECT

An estimate of basal glide strain prior to fracture as a function of purification can be obtained from the Stroh fracture criterion, Eq. [1], if changes in surface energy and elastic constants with purity are neglected.

The geometry of deformation on a single glide system requires that

$$\sigma_n = \frac{\tau_s}{\mathrm{Cot}\lambda} \qquad [3a]$$

and

$$D = D_o \left(\frac{\text{Sin } \lambda}{\text{Sin } \lambda_o} \right)^{1/2} \qquad [3b]$$

for the case $\lambda = \chi$, where χ = angle between tension axis and slip plane. Thus, one may write for [1],

$$\tau_s - \tau_o = \frac{k}{\tau_s} \; \frac{\text{Cot } \lambda \;\; \text{Cos } \lambda}{(\text{Sin } \lambda)^{1/2}} \, , \qquad [4]$$

where

$$k = \frac{k' (\text{Sin } \lambda_o)^{1/2}}{D_o}$$

The geometrical factor in the above expression, $\frac{\text{Cot } \lambda \;\; \text{Cos } \lambda}{(\text{Sin } \lambda)^{1/2}}$, is a function of the glide strain at fracture and the initial orientation of the crystal. The relationship is shown in Fig. 1 for the case $\lambda_o = 45^o$. For strains up to 350%, this relationship can be approximated by a straight line,

$$\frac{\text{Cot}\,\lambda\ \text{Cos}\,\lambda}{(\text{Sin}\,\lambda)^{1/2}} \simeq 0.840 + 2.35\,a. \qquad [5]$$

If linear hardening is assumed,

$$\tau_s = \tau_o + n\,a \qquad [6]$$

where

$$n = \frac{\tau_s - \tau_o}{a}$$

Combining [4] , [5], and [6] leads to a quadratic expression for glide strain in terms of yield stress and work hardening rate:

$$a = \frac{1}{2n^2}\left\{ 2.35\,k - \tau_o n + 2.35\,k\left[\left(\frac{\tau_o n}{2.35k}\right)^2 - \frac{2\tau_o n}{2.35k} + 0.608\,\frac{n^2}{k} + 1\right]^{1/2}\right\} \qquad [7]$$

Evaluation of this relationship requires the experimental determination of τ_o and n as functions of purity.

EXPERIMENTAL PROCEDURE

Purification was performed by floating zone-refining in an apparatus similar to that employed by Herman and Spangler (7). In order to obtain crystals covering a wide range of purity, both the purity of the starting material and the number of refining passes were varied. The starting materials employed were vacuum-melted and extruded Pechiney CR grade flake, and vacuum-distilled beryllium prepared from Pechiney CR flake (8). This material was also vacuum-melted and extruded prior to zone-refining.

Both 1/4 - inch and 1 1/4 - inch diameter crystals were prepared. The smaller crystals were seeded to obtain a basal plane inclination of 45^o to the rod axis. The large crystals were not seeded, since they generally grew with the basal plane parallel to the rod axis, so specimens of any basal orientation could be obtained by sectioning. Advantages of using 1 1/4 - inch diameter crystals were that several specimens of various purities could be obtained from a single zone-refined bar, and that such specimens would exhibit no axial concentration gradient.

In addition to the zone-refined crystals, a crystal of commercial purity was cut from a large-grained ingot prepared by vacuum melting of Brush QMV powder. Electric spark-discharge machining was employed to shape specimens for tensile testing. Cylindrical specimens having 1/2 - inch gauge lengths and 0.100 - inch gauge diameters with conical gripping surfaces were obtained both from 1/4 - inch diameter and 1 1/4 - inch diameter zone-refined bars. Specimens from the 1/4 - inch diameter bars were taken longitudinally with respect to the bar axis. One quarter inch diameter tensile blanks were spark-discharge trepanned from the 1 1/4 - inch diameter bars, as shown schematically in Fig. 2. These blanks were then shaped in an identical manner to that of the 1/4 - inch diameter zone-refined bars.

Final shaping of specimens was performed by electrochemical etching. This procedure was employed to remove .004 - .005 inches that might contain damage from spark machining.

The degree of purification obtained by zone-refining was measured by chemical analysis and by the ratio of electrical resistance at 4.2°K to that at 298°K. Resistance measurements were made on tensile blanks prior to final shaping. A dc method was employed.

Tensile testing was performed on a Tinius-Olsen Elect-o-Matic Tensile Testing Machine at a strain rate of 6.7×10^{-4}/sec. Crosshead travel, corrected for machine deflection, was used as a measure of specimen elongation. Grips were of the self aligning variety, with a conical section for seating the specimen. All measurements were performed at room temperature.

RESULTS AND DISCUSSION

Degree of Impurity Removal by Zone-Refining

Photometric determinations (9) of specific impurities as a function of starting material, number of zone-refining passes and position in the zone-refined bar are presented in Table I. Values of $S\left(= \dfrac{R4.2^{\circ}K.}{R298^{\circ}K.}\right)$ are given in Fig. 3.

Although the chemical analyses give quantitative information on specific impurities, techniques are available for only a few elements in the ppm range. Therefore, analytical results are not complete enough to be useful in the correlation of mechanical behavior with purity. On the other hand, the resistance ratio can be used to describe the relative over-all purity of any given

Table I. Impurity Distribution in Zone-Refined Bars

Material	Amount of Impurity (ppm) Fe	Cr	Mn	Al	Si	Ni	Cu
Vacuum-melted Pechiney Flake							
Before zone-refining	225	10	20	75	<20	115	55
Three passes, first portion to freeze	60	<1	<5	8	<20	194	96
Five passes, first portion to freeze	50	<5	<5	20	<20	220	
Five passes, last portion to freeze	1290	110	230	960	40	30	10
Distilled Beryllium							
Before zone-refining	2-3	1-2	10-15	15	<20	1-2	<10
Six passes, first portion to freeze	3	<1	1	<5		12	17
Six passes, last portion to freeze	13	2	11	18	<20	1	<4

sample. It is quite sensitive to purity changes, and, as will be seen, provides a convenient parameter for describing the impurity dependence of mechanical behavior. Within the limitations of its insensitivity to the presence of insoluble impurities and its small dependence on differences in structure, resistance ratio is therefore a useful tool for purposes of the present discussion.

Mechanical Properties

Resolved shear stress-resolved shear strain curves of crystals of various purities are presented in Fig. 4. The crystal designations employed are given in Table II. The data, in general, are in agreement with those of Herman and Spangler (7), in that there is a trend toward increased basal ductility with increasing crystal purity. The apparent reversal of this trend for the highest purity crystals L - 4 and L - 5 are probably a result of premature failure. Also, all crystals eventually failed by basal cleavage, indicating that the Stroh mechanism remains applicable even in the highest purity crystals.

Two additional features of the data of Fig. 4 make possible a comparison of experimentally determined ductilities with theoretical estimates based on Eq. [7]:

1.) There is a continuous decrease in the critical resolved shear stress for basal glide, τ_o, with decreasing soluble impurity content as measured by resistance ratio. This relationship is shown in Fig. 5.

2.) The stress-strain curves are approximately linear, justifying the assumption of Eq. [6]. In addition, the linear work hardening rate, n, decreases in a continuous manner with decreasing impurity content, as shown in Fig. 6.

Table II. Description of Single Crystals Employed in Tensile Tests

Specimen	Zone-Refined Bar	Distance From Start End (cm)	S
0-1	Brush Ingot, Not Refined	————	0.40
S-1	3-pass distilled, 1/4 inch	1.0	0.0045
S-2	3-pass distilled, 1/4 inch	3.0	0.015
L-1	5-pass Pechiney, 1 1/4 inch	0.5	0.026
L-2	5-pass Pechiney, 1 1/4 inch	3.0	0.036
L-3	5-pass Pechiney, 1 1/4 inch	4.3	0.045
L-4	6-pass distilled, 1 1/4 inch	1.3	0.00094
L-5	6-pass distilled, 1 1/4 inch	4.4	0.0019

Assuming that the curves of Figs. 5 and 6 represent adequately the dependence on purity of τ_o and n, it is possible to solve Eq. [7] for the glide strain to be expected at any given level of purity. In making this calculation, the following values were assumed: $\gamma = 600$ ergs/cm^2, $B = 10^{-12}$cm^2/dyne, $\theta = 5^o$, and $\tau_s = 10^8$dynes/cm^2. Then for a crystal 2.5 mm in diameter and oriented such that $\chi_o = \lambda_o = 45^o$, the constant $k = 4.3 \times 10^{15} \frac{\text{dynes}^2}{\text{cm}^4}$.

The results of the calculations are given by the solid curve of Fig. 7. The experimentally determined ductilities are in fairly good agreement with the calculated values up to purities corresponding to S = 0.05. At higher purities, premature failure results in much lower ductilities than would be anticipated.

For a purity level corresponding to $S = 10^{-3}$, Fig. 7 indicates that glide strains of the order of 300 % may be possible in the absence of adverse effects imposed by testing conditions. Such values are similar to those observed in typically ductile hexagonal close-packed crystals such as magnesium. It is evident, therefore, that quite extensive basal plane ductility in beryllium can be achieved by purification without the necessity of causing a change in the ductility-limiting mechanism.

SUMMARY

1. An estimate was made of basal plane ductility of beryllium single crystals as a function of purification, based on the assumption that the propagation of Stroh cracks is the operating fracture mechanism. In arriving at this estimate, it was assumed that strain hardening is linear, and that surface energy and elastic properties are unaffected by purification.

2. Tensile tests on crystals purified by zone refining provided the necessary information on the variation in critical resolved shear stress and work hardening rate as a function of purification in order to quantitatively predict the basal glide strain prior to fracture over a wide purity range.

3. Experimentally observed ductilities agreed fairly well with the above predictions over an appreciable portion of the purity range studied. At the highest purities, the observed ductilities were significantly lower than the predicted values. This is attributed to adverse effects imposed by testing conditions.

ACKNOWLEDGMENTS

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FIGURE CAPTIONS

Fig.	Caption
1	Strain dependence of geometrical factor $\frac{\text{Cot}\,\lambda\ \text{Cos}\,\lambda}{(\text{Sin}\,\lambda)^{1/2}}$
2	Location and orientation of tensile specimens trepanned from 1 1/4 - inch diameter zone-refined bars
3	Resistance ratio gradients in zone-refined bars
4	Resolved shear stress-resolved shear strain curves for beryllium crystals of different purities
5	Purity dependence of critical resolved shear stress for basal slip
6	Purity dependence of basal work hardening
7	Basal ductility of beryllium single crystals as a function of purity

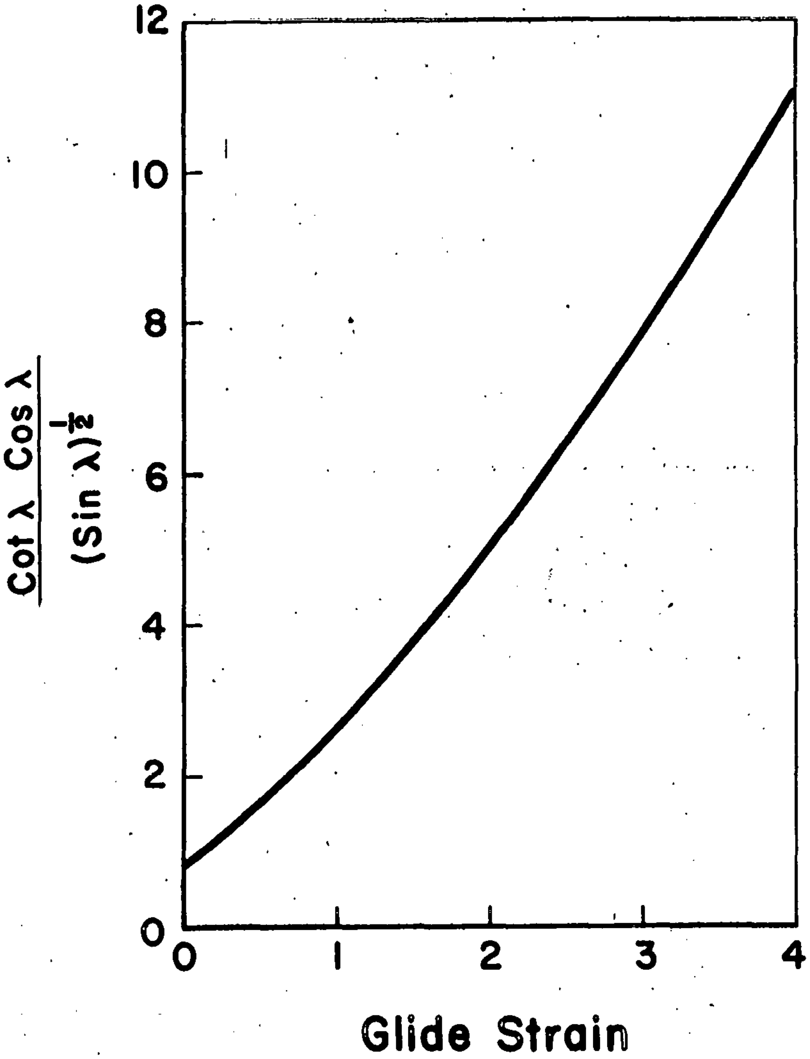

Fig. 1 Strain dependence of geometrical factor $\frac{\text{Cot } \lambda \ \text{Cos } \lambda}{(\text{Sin } \lambda)^{1/2}}$

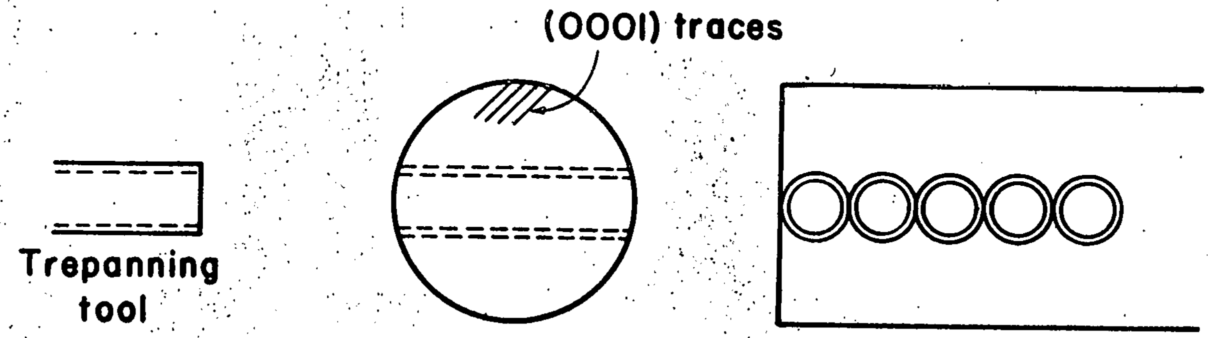

Fig. 2 Location and orientation of tensile specimens trepanned from 1 1/4 - inch diameter zone-refined bars

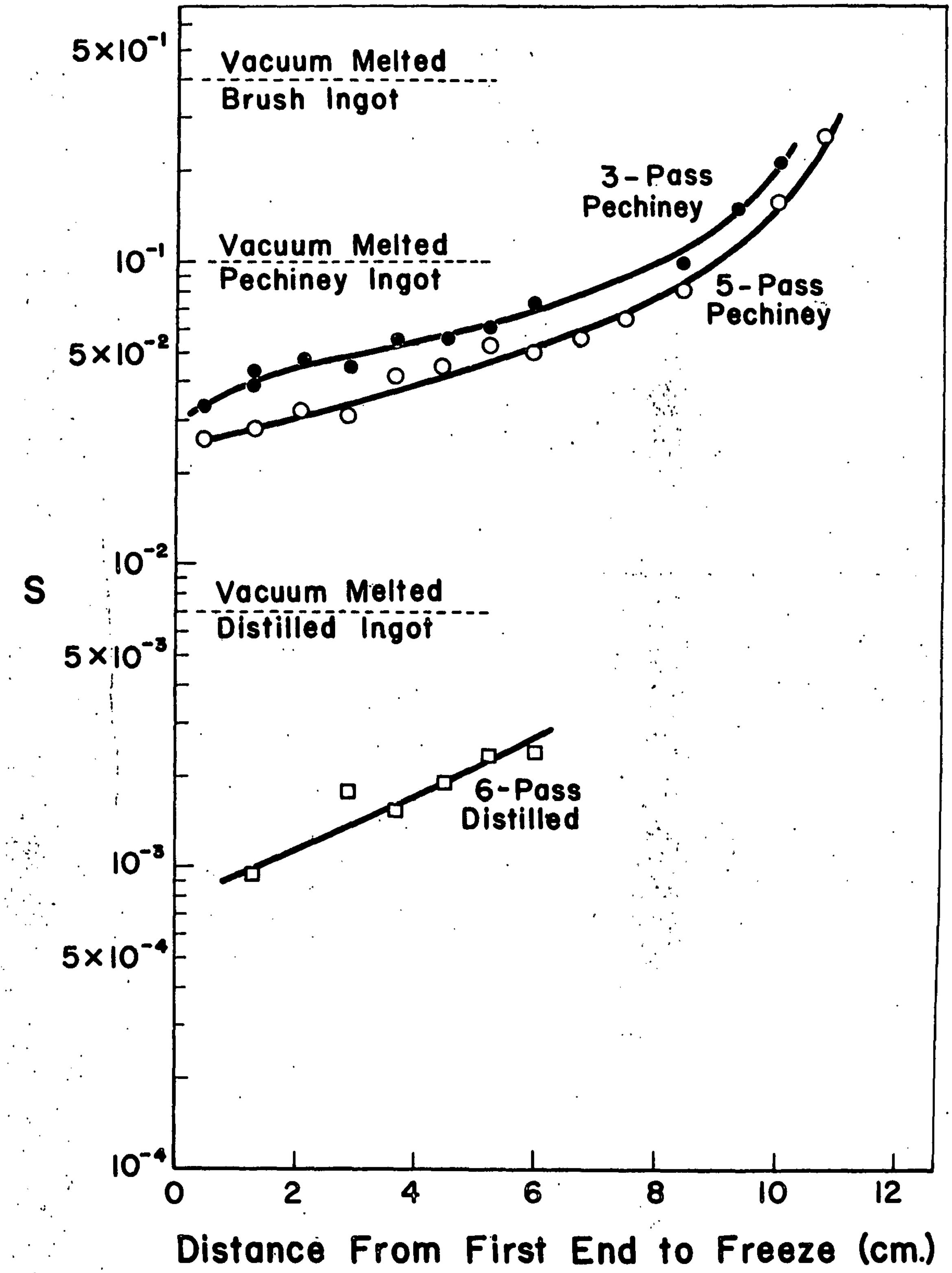

Fig. 3 Resistance ratio gradients in zone-refined bars

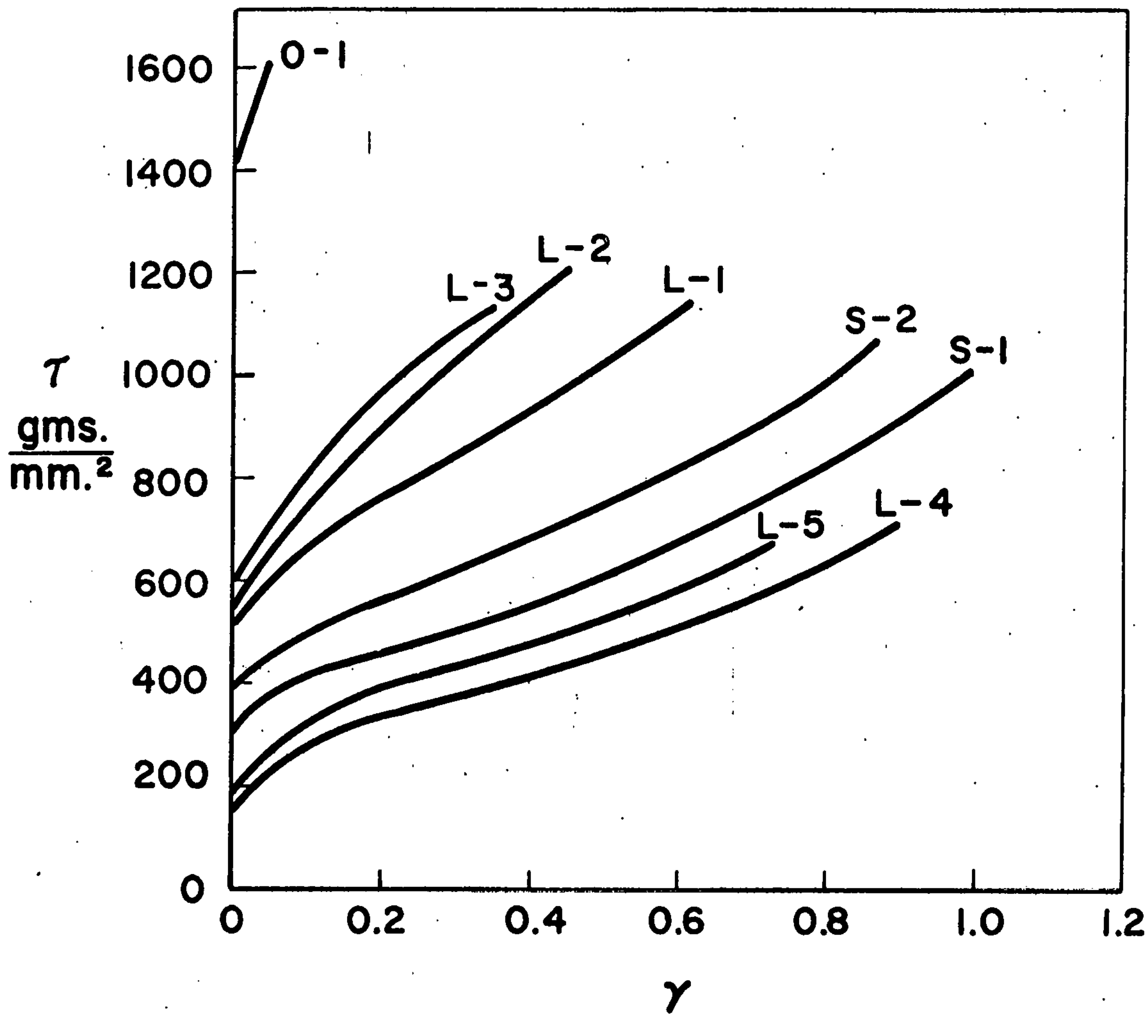

Fig. 4 Resolved shear stress-resolved shear strain curves for beryllium crystals of different purities

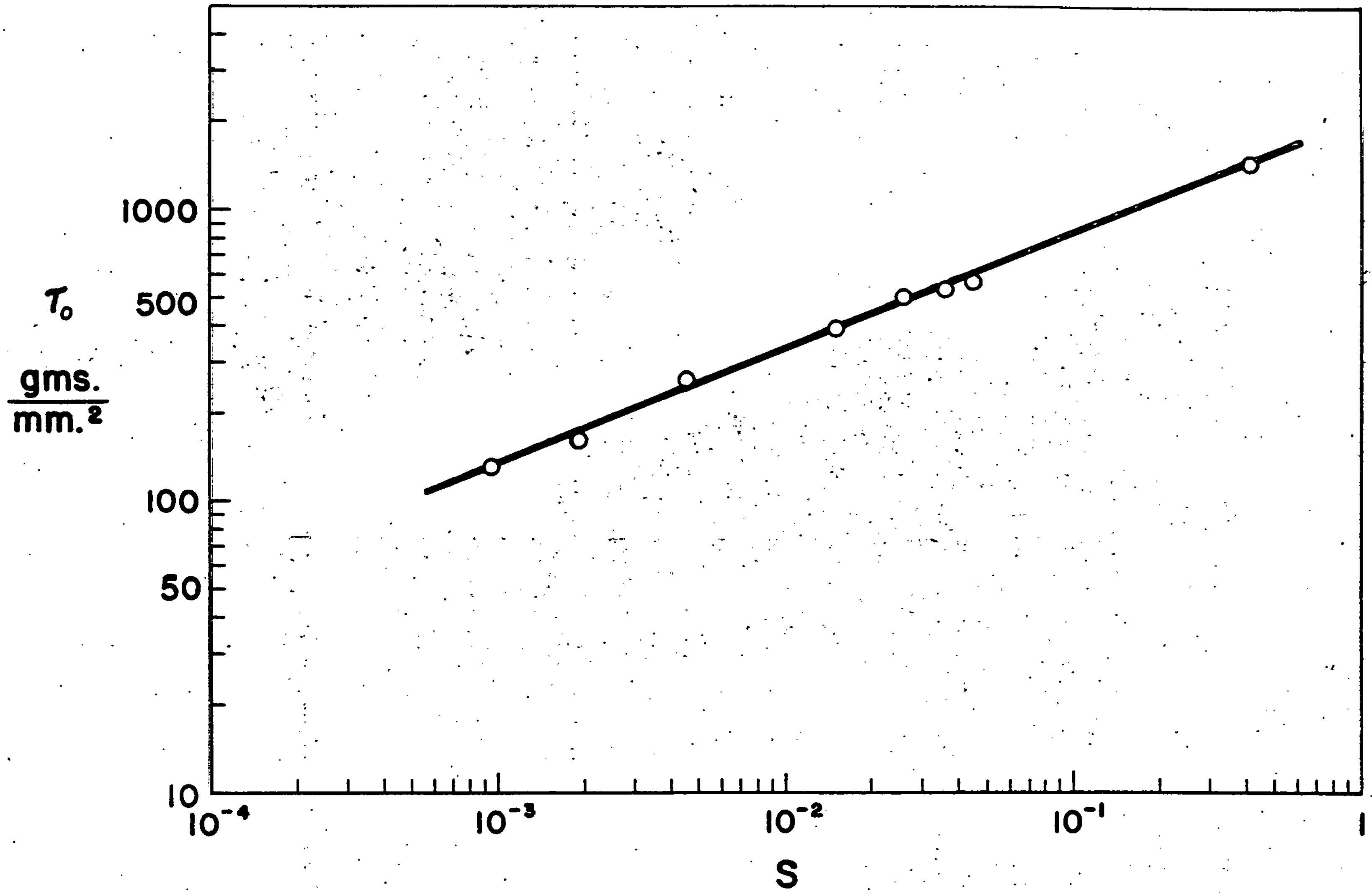

Fig. 5 Purity dependence of critical resolved shear stress for basal slip

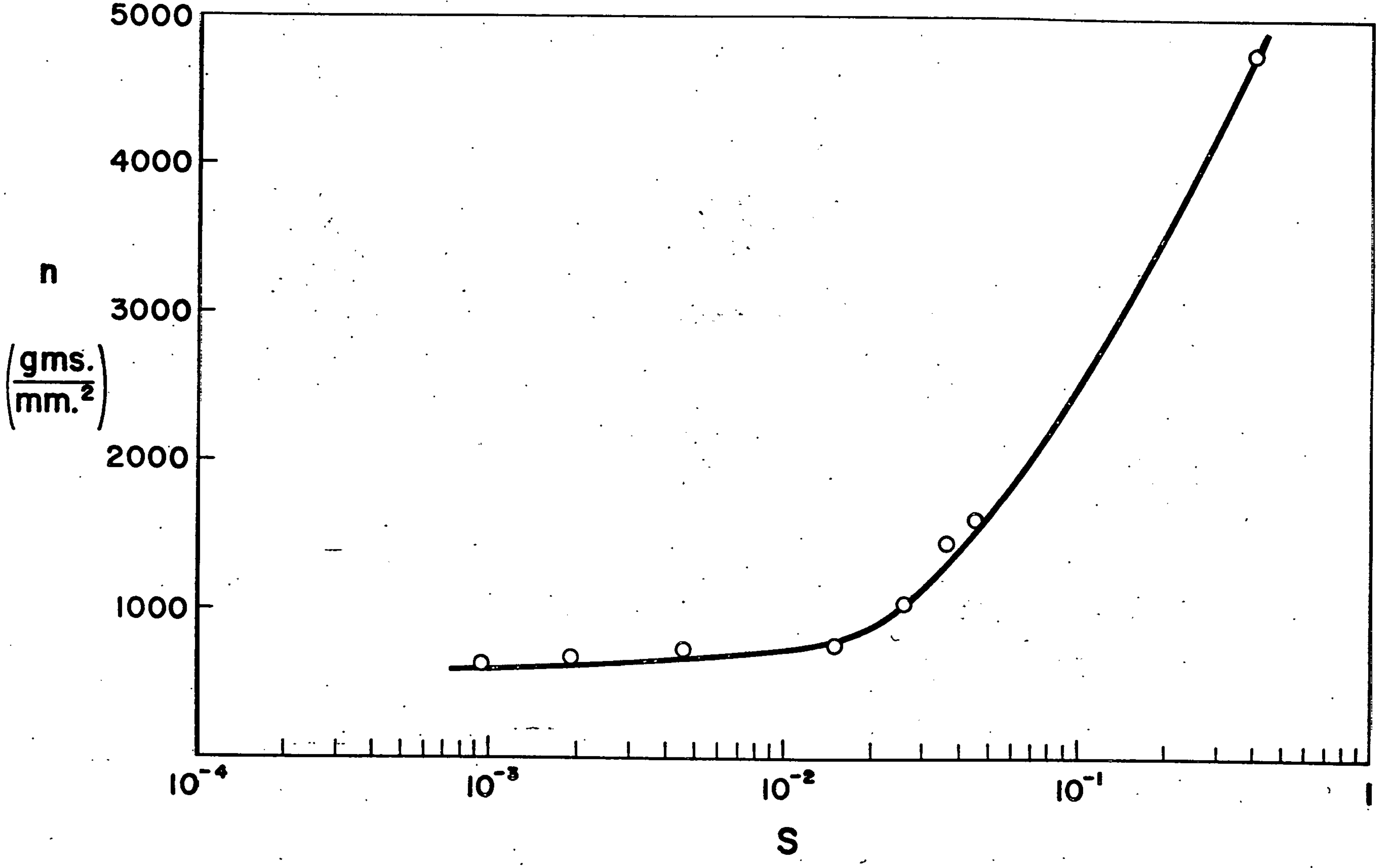

Fig. 6 Purity dependence of basal work hardening

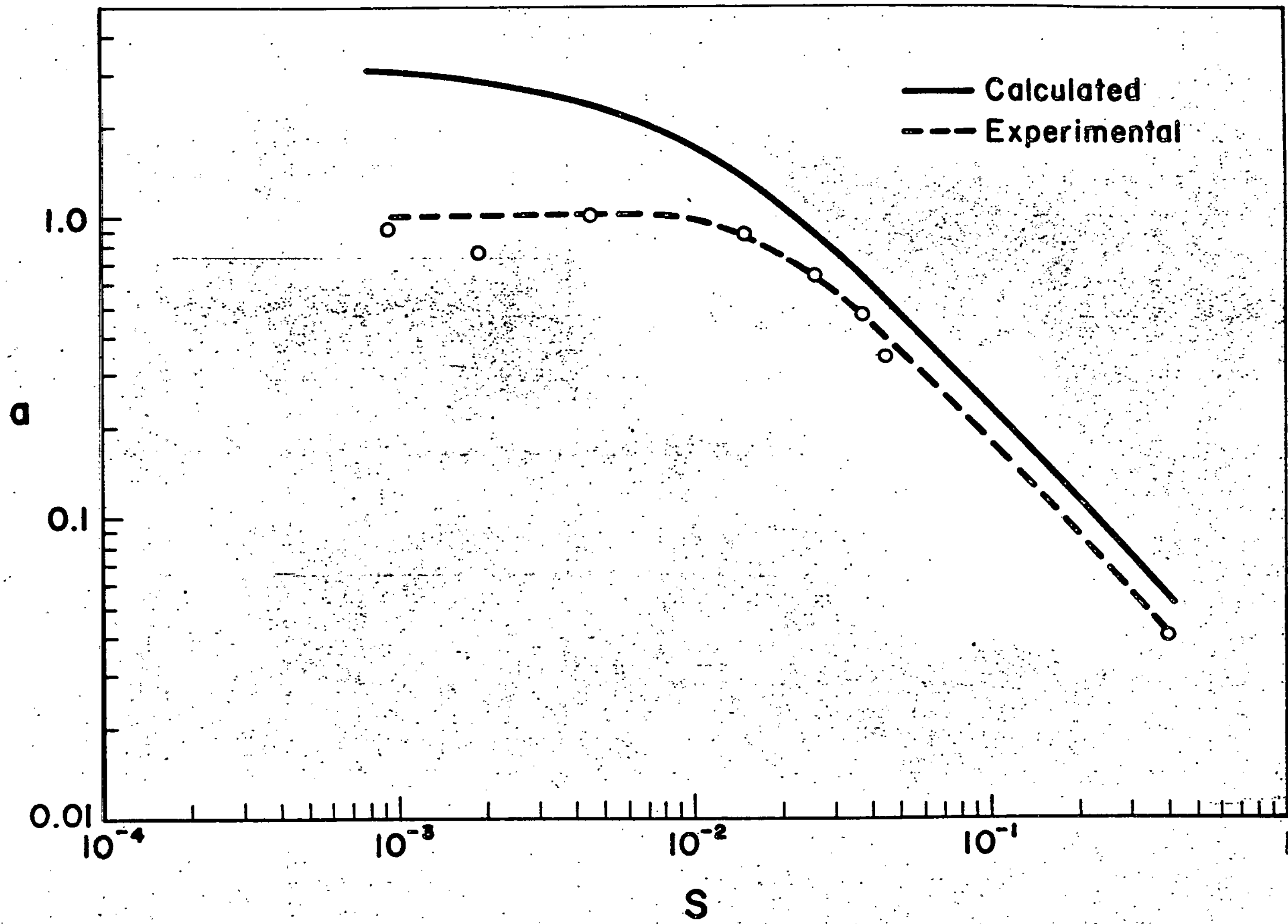

Fig. 7   Basal ductility of beryllium single crystals as a function of purity