

COMPRESSION OF BERYLLIUM SINGLE CRYSTALS ALONG THE HEXAGONAL AXIS IN THE TEMPERATURE RANGE 4.2-900°K

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The present paper reports the results of an investigation of the mechanical properties of beryllium single crystals in which the basal plane is at right angles to the compression direction. In the temperature range 4.2-900°K beryllium single crystals of this orientation exhibited unusually high strength: at the temperature of liquid helium the maximum compressive strength reached a value of $\sigma_b = 532 \text{ kg/mm}^2$. It was found that at high temperatures (500-900°K) beryllium single crystals possessed some plasticity due to slip along the second-order pyramidal planes; at low temperatures plastic deformation was not observed and practically all the deformation before fracture was elastic.

Introduction

Plastic deformation disrupts the continuity of a crystal, and even at the earliest stages of plastic flow a partial fracture occurs [1, 2]. Therefore, one would expect that crystals oriented so that the principal types of plastic deformation are avoided should exhibit higher strength. From this point of view hexagonal crystals are of special interest, since they have a limited number of crystallographic slip and twinning elements, particularly at low temperatures [3-5].

In the case of uniaxial compression of hcp metal single crystals at right angles to the basal plane (0001), the orientation and the stress state are such that all the usual types of plastic deformation are suppressed (Fig. 1). Twinning along the plane $\{10\bar{1}2\}$ cannot occur, because volume changes on formation of twins are related to an expansion along a direction at right angles to the basal plane (0001). Shear in the basal plane (0001) and first-order prismatic plane $\{10\bar{1}0\}$ cannot occur because the tangential stresses in these planes are zero. Thus the only possible states are elastic compression or slip along other crystallographic planes (for example, pyramidal planes).

Lee and Brick [6] investigated the plastic properties of beryllium single crystals (98.8% purity) of various orientations in the temperature range 20-500°C, and showed that single crystals in which the basal plane (0001) is at right angles to the compressive forces exhibited high compressive strengths. The strength σ_b varied from 199.5 kg/mm² at 20°C to 150.5-158.9 kg/mm² at 300-500°C. These authors showed that at room temperature the beryllium single crystals underwent almost pure elastic deformation; no traces of twins

or slip lines were observed. Fracture occurred instantaneously on reaching the critical stress; the sample disintegrated into small particles. Lee and Brick were able to establish the indices of the planes along which fracture occurred by means of the Laue back-reflection method. These planes were the basal plane (0001) and three second-order prismatic planes $\{11\bar{2}0\}$. They noted also that asterism in the Laue diagrams developed only in two out of the three $[11\bar{2}0]$ zones. However, they gave no information on the nature of the deformation of beryllium single crystals of this orientation at the higher temperatures of 300 and 500°C.

Tuer and Kaufman [7] also carried out similar tests on beryllium single crystals of 98.89% purity. The compressive strength at room temperature was 140-168 kg/mm², which is approximately 25% lower than the value of σ_b given by Lee and Brick [6]. Tuer and Kaufman observed considerable plasticity at temperatures of 700-1000°C, but they did not describe the nature of the deformation at high temperatures.

In an earlier short note [8] the present authors described preliminary experiments on the low-temperature compression of 99.9% pure beryllium single crystals and of calcite single crystals in which the principal elements of plastic deformation were also suppressed by selecting a particular orientation.

These beryllium and calcite crystals exhibited exceptionally high values of the compressive strength: for example, fracture of a beryllium single crystal in liquid nitrogen occurred at a stress of 410 kg/mm², and for calcite at room temperature σ_b was 23 kg/mm². In beryllium single crystals of this orientation brittle fracture at low temperatures was replaced by some plas-

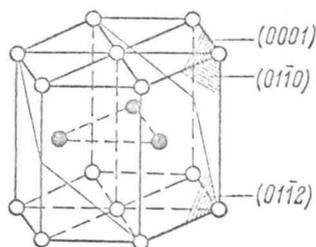


Fig. 1. Hexagonal close-packed structure of beryllium: $c/a = 1.568$. With this orientation of a single crystal the slip along (0001) and $\{10\bar{1}0\}$ and the twinning along $\{10\bar{1}2\}$ are avoided.

ticity at high temperatures. It was of interest to find the temperature dependence of the mechanical properties of high-purity beryllium single crystals, exhibiting high strength at low temperatures, and the nature of the deformation and fracture at high temperatures. For this purpose a study was made of beryllium single crystals oriented so that the basal plane (0001) made an angle of 90° with the axis of the deforming forces, in the temperature range 4.2 - 900°K .

1. Material, Preparation of Samples, and Experimental Procedure

Beryllium single crystals were grown from the melt in a vacuum chamber using beryllium oxide crucibles [9]. Beryllium distillate of 99.97% purity* was used as the initial material. The purity of the single crystals grown was 99.9%. The orientation of the single crystals was determined by the direct Laue diffraction method. An oriented single crystal was cut into samples of rectangular shape of electric-spark machining [10]. Subsequent treatment of the single-crystal samples of beryllium, consisting of etching, grinding and polishing, was similar to the method of preparation of beryllium single crystals described earlier [9]. The orientation of the crystals was such that the basal plane (0001) was parallel to the end surfaces of the sample and one of the lateral (side) surfaces coincided with the second-order prismatic plane $\{11\bar{2}0\}$. Laue diffractograms of the samples indicated that the initial structure of the beryllium single crystals was highly perfect (Fig. 2).

The crystals were deformed in a special press [11] at temperatures of 4.2 , 77 , 300 , 500 , 700 and 900°K . The compression tests at high temperatures were carried out in an atmosphere of helium in order to prevent oxidation of the samples. The rate of deformation was 0.013% per sec. To avoid penetration of the sample into the compression surfaces of the press and consequent error in the determination of the sample deformation, Pobedit spacers were used (Pobedit is a tungsten-cobalt-carbon-titanium alloy). Two mutually perpendicular side surfaces of the samples were polished before the metallographic investigations. Indexing of the actual slip and fracture planes was accomplished by plotting the stereo-

graphic projection from a back-reflection Laue diagram and comparing the results with the microanalysis data.

2. Results

The nature of the behavior of the beryllium single crystals under load was different in different temperature ranges:

a) The samples tested at 4.2 - 300°K exhibited brittle fracture and the deformation preceding fracture was predominantly elastic;

b) The samples compressed at 500 - 900°K showed some plastic deformation before fracture.

This difference appeared clearly in the compression curves (Fig. 3). First of all we notice the high values of the yield point and compressive strength, the two quantities differing little from one another in the temperature region 4.2 - 300°K . The total deformation δ_{tot} of these samples was small and mainly elastic.

At the temperature of liquid helium the compressive strength σ_b reached a maximum value of 532 kg/mm^2 and the elastic deformation preceding fracture amounted to $\delta_e = 1.1\%$ out of total deformation $\delta_{\text{tot}} = 1.7\%$ (Table 1). With increase of the test temperature the maximum compressive strength decreased, falling to 210 kg/mm^2 at 300°K ; the elastic part of the deformation before fracture also decreased, while the plastic deformation δ_p increased (at $T = 300^\circ\text{K}$, $\delta_p = 1.0\%$). At 300 - 900°K the compression curves of the samples had more pronounced yield points and regions of plastic flow with slight hardening. The usual temperature dependence of the yield point was observed and, what is most important, some plasticity appeared: the relative plastic compression δ_p increased with increase of temperature and reached 9.2% at $T = 900^\circ\text{K}$.

Figure 4 shows the temperature dependence of the yield point σ_s and the compressive strength σ_b of the tested samples. In spite of the different nature of the fracture of the single crystals at low and high temper-

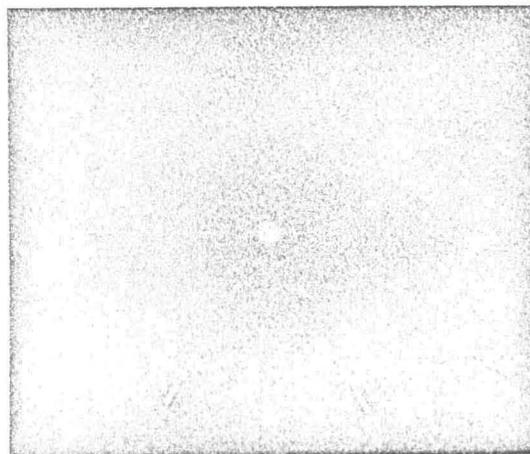


Fig. 2. Laue transmission diagram of a beryllium single-crystal in the initial state.

*The purity of beryllium is given without allowance for oxygen.

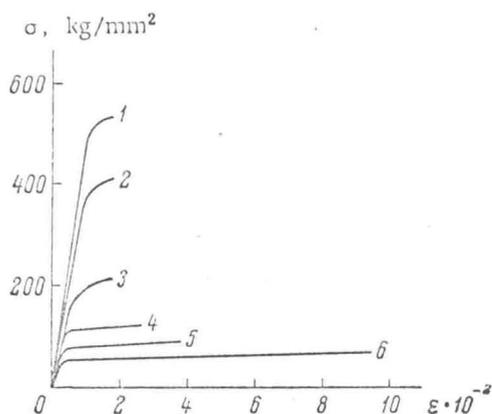


Fig. 3. Stress-strain curves for beryllium single crystals compressed along their hexagonal axes. Temperature ($^{\circ}\text{K}$): 1) 4.2; 2) 77; 3) 300; 4) 500; 5) 700; 6) 900.

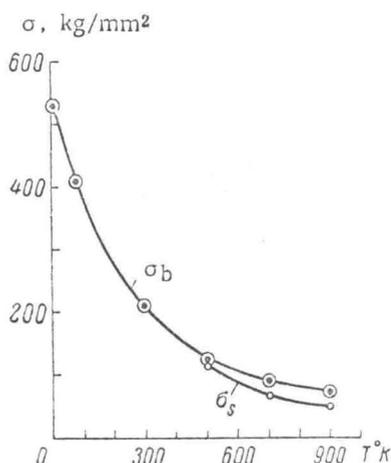


Fig. 4. Temperature dependence of the compressive strength σ_b and the yield point σ_s of beryllium single crystals on compression at right angles to the basal plane (0001).

atures, the $\sigma_b(T)$ curve is monotonic. The $\sigma_s(T)$ curve has physical meaning only from the temperature at which considerable shear appears along certain crystallographic planes. Therefore in Fig. 4 the $\sigma_s(T)$ curve joins the $\sigma_b(T)$ curve in the temperature range 300-500 $^{\circ}\text{K}$.

Since optical and electron-microscopic studies of the surfaces of the crystals deformed in steps showed no traces of slip or twinning, the slight plastic deformation preceding fracture at low temperatures is obviously due to processes of block formation occurring immediately before fracture.

High stresses, close to the compressive strength, are necessary to fragment the single-crystal structure (formation of blocks), as indicated by the following observations:

1) up to stresses close to the compressive strength the sample recovered fully its original dimensions after unloading;

2) on reaching the maximum (breaking) stress the crystals fractured instantaneously, disintegrating into very small particles.

The main dimensions of the particles obtained on fracture of beryllium single crystals increased with rising temperature from $\bar{d} = 1-2 \mu$ at $T = 4.2^{\circ}\text{K}$ to 100μ at 300°K .

Figure 5 shows photomicrographs of the particles formed on fracture of beryllium single crystals. We see that micron size particles formed by low-temperature fracture do not have definite crystallographic habit, in contrast to fracture blocks of the single crystals at room temperature. Irregular surfaces of the fragments formed in low-temperature fracture may be the result of a change in the ratio of the crystallographic axes c/a due to deep cooling. In such a case the linear expansion should be very strongly anisotropic.† Moreover, considering the process of crack formation as the accumulation of dislocations [13], we may conclude that the low mobility of dislocations at low temperature impedes crack growth; the fracture surfaces are conchoidal with a large number of crack nuclei which have hardly grown. This also leads to a rise of σ_b . We may assume that an increase of temperature not only lowers the limit of block formation but also makes the fracture surfaces more crystalline. However, in spite of the more pronounced crystalline nature of fracture no traces of deformation were found in the fracture planes. This is a further proof of the nonshear nature of the residual deformation found on fracture of beryllium single crystals in the temperature region 4.2-300 $^{\circ}\text{K}$. The assignment of indices to the fractured planes indicates that microcracks appear and propagate in the second-order prismatic planes $\{11\bar{2}0\}$, basal planes (0001) and second-order pyramidal planes $\{11\bar{2}1\}$.

3. Discussion of Results

A. High Strength of Beryllium Single Crystals of Given Orientation

Comparison of the results given in Tables 1 and 2 shows that in a wide range of temperatures the value of the maximum fracture stress of beryllium single crystals of the given orientation ($\alpha = 90^{\circ}$) exceeds by a factor of 10 the value of the compressive strength σ_b of beryllium single crystals oriented favorably for basal slip ($\alpha = 45^{\circ}$). In this case the orientation dependence (anisotropy) of the compressive strength is more important, since the temperature dependence of the maximum fracture stress is approximately the same for different orientations.

The high value of the compressive strength of beryllium single crystals of the given orientation, particularly at low temperatures, is due to practically the whole of the prefracture deformation being elastic. There is no plastic deformation in the principal crystallographic planes and, therefore, the generation of fracture nuclei during loading is avoided.

† Information is available indicating a rise of c/a on lowering the temperature in the region 0-1000 $^{\circ}\text{C}$ [7].



Fig. 5. Photomicrographs of particles obtained on fracture of beryllium single crystals. a) $T = 4.2^\circ\text{K}$, $\sigma_b = 532 \text{ kg/mm}^2$, $\bar{d} = 1.0\text{-}2.0 \mu$; b) $T = 300^\circ\text{K}$, $\sigma_b = 210 \text{ kg/mm}^2$, $\bar{d} = 100 \mu$. ($\times 200$).

These experiments confirm once again the unfavorable influence of plastic deformation on the strength of materials. As in the case of whiskers, macroscopic single crystals are very strong under conditions ensuring large elastic deformation before fracture.

In single crystals of the given orientation the elastic deformation is spread throughout the crystal, and compression throughout the crystal is more uniform. The only possible mechanism for the observed small residual change of the crystal shape is the process of block formation which occurs at very high stresses immediately before fracture. This is indicated by the fracture of the whole crystal into particles of one to several microns in dimensions.

Although the compressive strength falls considerably at high temperatures (500-900°K), the value of σ_b nevertheless remains much higher than the compressive strength of beryllium single crystals of other orientations. It is interesting that at these high temperatures beryllium single crystals have a high yield point corresponding to deformation by slip along pyramidal planes.

B. Pyramidal and Basal Slip in Beryllium Single Crystals at High Temperatures

The characteristic feature of the deformation of beryllium single crystals of the given orientation in the temperature range 500-900°K is the instantaneous localized shear along second-order pyramidal planes on fracture, in contrast to the fragmentation into small particles at low temperatures. Figure 6 shows a photo-

TABLE 1. Plasticity and Strength Parameters of Beryllium Single Crystals on Compression along the Hexagonal Axis

	h , mm	f_0 , mm ²	T , °K	σ_b , kg/ mm ²	σ_{b0} , kg/ mm ²	δ_e , %	δ_p , %	δ_{tot} , %	$E \cdot 10^4$, kg/mm ²
Be-2 . . .	2.18	1.72	4.2	532	—	1.1	0.6	—	4.745
Be-5 . . .	2.11	1.65	77	410	—	0.9	0.9	—	4.55
Be-7 . . .	2.06	1.73	300	210	—	0.6	1.0	—	3.5
Be-11 . . .	4.56	7.42	500	124	112	0.4	4.3	27.3	3.1
Be-14 . . .	4.05	5.8	700	90	68.9	0.3	7.5	38.5	3.01
Be-18 . . .	4.05	5.16	900	72	50.4	0.3	9.2	45.0	2.39

TABLE 2. Principal Elements of Plasticity as a Function of Orientation

Orientation	T , °K	σ , kg/mm ²	σ_0 , kg/mm ²	Nature of plastic deformation
$P \perp (0001)$	77	410	—	Elastic compression; slip along (0001) and $\{10\bar{1}0\}$ and twinning along $\{10\bar{1}2\}$ avoided by orientation
	300	210	—	
$P < 45^\circ (0001)$	77	34	3.4	Slip along basal plane (0001)
	300	18	2.16	
$P \parallel (0001)$	77	103	5.3	Slip along $\{10\bar{1}0\}$ planes, twinning along $\{10\bar{1}2\}$; slip along (0001) avoided by orientation
	300	64	3.7	

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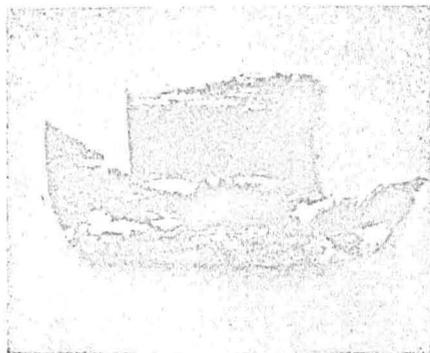


Fig. 6. Beryllium single crystals deformed at 500°K and macrosheared along the second-order pyramidal plane ($11\bar{2}4$). $\delta_p = 4.3\%$, $\delta_{tot} = 27.3\%$, $\sigma_b = 124 \text{ kg/mm}^2$, $\gamma = 60$. ($\times 10$).



Fig. 7. Slip traces in a beryllium single crystal deformed at $T = 700^\circ\text{K}$, $\delta_p = 5.7\%$, $\sigma_b = 78.4 \text{ kg/mm}^2$. The plane of the photograph coincides with the first-order prismatic plane ($10\bar{1}0$). The inclined slip traces correspond to shear along the second-order pyramidal planes ($1\bar{2}14$) and ($1\bar{2}12$); horizontal traces represent slip along the basal plane (0001). ($\times 340$).



Fig. 8. Fracture of beryllium single crystals at $T = 500^\circ\text{K}$, $\delta_{tot} = 27.3\%$, $\sigma_b = 124 \text{ kg/mm}^2$. a) Longitudinal bending of (0001) layers with formation of cracks in the basal plane; ($\times 340$). b) Principal elements of fracture: ($1\bar{2}14$), ($11\bar{2}0$), and (0001). ($\times 200$).

graph of a beryllium single crystal which was deformed at $T = 500^\circ\text{K}$ to fracture and which suffered a macro-shear of relative magnitude $\gamma = 60$ along a second-order pyramidal plane. By plotting the stereographic projection from the back-reflection Laue diffractogram it was found that the macroshear on fracture occurred along one of the $\{11\bar{2}4\}$ planes. It is characteristic that the macroshear accounted for almost 80% of the total deformation after fracture. At still higher temperatures the fracture of single crystals was also preceded by similar macroshear.

The microscopic picture of slip in beryllium single crystals of the given orientation is of great interest. Two types of slip traces were found on the polished surfaces of samples tested at 500, 700 and 900°K; these were inclined and horizontal traces. The inclined traces and some of the horizontal traces represented the emergence of pyramidal slip traces, mainly those due to slip along the second-order pyramidal planes

$\{11\bar{2}4\}$ in the direction $[11\bar{2}3]$ (Fig. 7). Shear along this plane predominated both in the initial stages of the plastic deformation and in the fracture of the single crystals, which usually occurred after macroshear along one of the $\{11\bar{2}4\}$ planes. In addition, slip was observed along the second-order pyramidal plane $\{11\bar{2}2\}$ and the first-order pyramidal planes $\{10\bar{1}1\}$. Other horizontal traces on both surfaces of the sample obviously belonged to a different slip system, since the density of the horizontal traces was greater than the density of pyramidal slip traces. In our opinion some of the horizontal slip traces are simply traces of basal slip. Although the contribution of basal slip to the total deformation of the beryllium single crystals of the given orientation was negligibly small, the existence of such slip for this orientation was nonetheless extremely interesting. The direction of shear along the basal plane coincides with one of the diagonals of the plane (0001). This was established by measuring the breaks in the inter-

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ference bands at intersections of the horizontal slip traces in two mutually perpendicular faces of the sample [12]. It should be noted that the direction of shear in basal slip in the case of our orientation is $[2\bar{1}10]$, in contrast to the usually observed direction $[11\bar{2}0]$ in beryllium single crystals oriented at $\alpha = 45^\circ$.

We shall consider possible mechanisms for the appearance of basal slip in beryllium single crystals when tangential stresses in the basal plane (0001) are zero because of the selected orientation of the crystals relative to the compressive forces.

Since the stresses generated on deformation of the crystals are considerable, elastic bending of the lattice is possible and consequently tangential stresses may appear in the basal plane (0001) which in turn gives rise to shear deformation. An estimate of the magnitude of the elastic bending of the lattice sufficient for the appearance of tangential stresses greater than the critical shearing stresses \ddagger and producing basal slip, gives values of the bending angle less than 1° .

This is not the only possible way in which shear can appear in the basal plane of beryllium single crystals of the given orientation. We shall assume that dislocations moving in a pyramidal plane and causing shear in that plane are pinned at some obstacle; a stress concentration is produced around this obstacle. The compressive force at the obstacle will then be oriented at some angle to the basal plane [the angle between the planes (0001) and $(11\bar{2}4)$ amounts to $38^\circ 06'$]. Such orientation of the compressive forces with respect to the (0001) plane and the stress concentration in this plane may consequently give rise to tangential stresses which will cause shear. It should be noted that, in our case, shear in the basal plane destroys the continuity of the crystal. Fracture on shear in the basal plane (0001) occurs along the planes of the second-order prism $\{11\bar{2}0\}$ and the second-order pyramid $\{11\bar{2}4\}$, and, moreover, the (0001) layers are bent, forming cracks in the basal plane ** (Fig. 8).

A considerable divergence should be noted between the present results and those of foreign workers [6, 7]. As mentioned above, the compressive strength of beryllium single crystals along the hexagonal axis has, according to Lee and Brick [6], a weak temperature dependence in the range 20-500°C. Moreover, the maximum fracture stresses of beryllium single crystals at 500°C are higher than at 300°C.

In the present paper we have shown that the compressive strength decreases monotonically with increasing temperature in the range 4.2-900°K. The compressive strength σ_b decreases almost by a factor of 3 between 300 and 900°K, while Lee and Brick reported that the compressive strength changed by about 20% in the same range. This difference between the reported temperature dependences of the compressive strength can be explained by the different purities of the materials. The weak variation of the compressive strength with increase of temperature is related to strong hardening

caused by dispersion ageing in the process of high-temperature plastic deformation of the less pure beryllium.

Conclusions

1. Beryllium single crystals of 99.9% purity oriented and stressed in such a way as to avoid the possibility of plastic deformation in the principal crystallographic planes exhibit exceptionally high compressive strengths, especially at low temperatures.

2. At high temperatures (500-900°K) beryllium single crystals of the given orientation exhibit plasticity in the form of slip, mainly along the second-order pyramidal planes $\{11\bar{2}4\}$ along the direction $[11\bar{2}3]$. Pyramidal slip in beryllium single crystals has a high critical shear stress.

3. Basal slip is also observed, and it may be the result of either elastic bending of the lattice due to high critical stresses of the pyramidal shear, or the result of stress concentration at obstacles impeding the motion of dislocations in pyramidal planes.

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\ddagger The calculation was carried out on the assumption that the law of critical tangential stresses is valid.

**We may also assume that slip along the planes $\{11\bar{2}4\}$ is the result of penetration of a tetrahedron with the bounding planes (0001), $(1\bar{2}14)$, $(\bar{2}114)$ and $(11\bar{2}4)$ along the hexagonal axis c . The penetration of such a wedge obviously produced a longitudinal bending of the (0001) layers by forces acting from within along the direction $(11\bar{2}0)$.