

SLIPPING OF BERYLLIUM SINGLE CRYSTALS AT LOW TEMPERATURES. III*

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Especially pure (99.98%) beryllium single crystals were deformed at 20° and 77°K. Slipping along the basal plane (0001) has been detected at 20° as well as at 77°K. Two deformation stages are discerned: an initial stage, when the displacement occurs in a thin layer adjacent to the slip band, and a later stage when the deformation is localized at the slip band. In regions between the bands, twisting has been detected which increases with the deformation. The twist of the blocks in the early stages may be ascribed to the effect of residual strains. In the later stages, when the rotation reaches 3°, one is forced to assume that twinning takes place during slip. The large magnitude of the relative displacement in the second stage may be explained by violation of continuity with subsequent healing of the contacts.

WE have shown elsewhere^{1,2} that single crystals of commercial beryllium, 99.7% pure, have a plasticity, particularly basal slip along (0001) plane, not only at room temperature but also at lower temperatures. We have found as the results of these investigations that the first microcracks (along the planes of the prisms and second-order pyramids) are formed as a consequence of non-uniform shear along individual basal slip bands. At low temperature, the non-uniformity of the shear increases, leading to an increase in brittleness. The non-uniformity in shear can be assumed to be due to impurities and that the shear would be more uniform in pure beryllium.

In this project we have investigated basal slip of single crystals of especially pure beryllium (99.98%) at low temperatures (20 and 77°K), at which brittleness of this metal manifests itself most.

1. SPECIMENS AND INVESTIGATION PROCEDURE

Single crystals were grown from the melt by slowly cooling pre-purified beryllium in vacuo.† The method of preparation of single-crystal specimens was described in detail in earlier papers.^{1,2}

The samples were oriented in such a way that the basal plane (0001) made an angle of 45° to the direction of the deforming forces, while the direction of the shear in the case of basal slip — the first-order diagonal [1120] was parallel to one

of the side faces of the specimen (Fig. 1). The specimens were deformed by uniaxial compression in a machine provided with a special fixture for low-temperature testing.^{3,4} The speed of deformation was constant at 0.03 mm/sec. The compression was carried out in sequence to $\delta = 0.1, 0.3, 0.7, 0.9, 1.7,$ and 6%. The sample was subjected after each step to x-ray, micro-interference, and electron-microscopic investigation by methods described in references 1, 2, and 5. In addition, it was possible to determine the overall picture of the slip at various stages of deformation from the displacement of scribe marks scratched beforehand on one of the lateral faces of the specimen, coinciding with the (1100) shear plane. These data were compared with the profile of the face of the specimen, perpendicular to shear plane (face ah, Fig. 1). To draw the profile of this face, use was made of an electron-microscopic photograph with a lacquer replica contrasted with chromium.

Usually the angle and the direction of contrast-

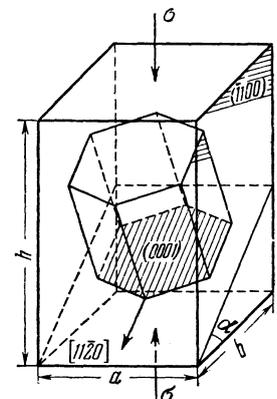


FIG. 1. Crystallographic orientation of the slip elements ($\alpha = 45^\circ$).

*For parts I and II see references 1 and 2.

†The pre-purified beryllium was graciously furnished to us by V. E. Ivanov and V. M. Amonenko.

ing are determined from the shadows produced by the magnesium-oxide crystals that settle on the replica that during the combustion of a magnesium ribbon.⁶ In this case, however, an exact determination of the length of the shadow is difficult because of the arbitrary placement of the cubic crystals of the replica. To increase the accuracy of the drawn profile, spherical particles were deposited on the replica. These particles were obtained from a 1% solution of coloxylin in amyl acetate by atomizing the solution in air.*

Drops of the solution, suspended in air, contract into spheres on the order of 0.1μ in diameter after the evaporation of the amyl acetate. Under suitably chosen conditions one or two spheres settle on the replica within the field of view of the electron microscope. By measuring the radius of the sphere R and the length of the shadow H it is possible to determine the contrasting angle from the following formula

$$\alpha_1 = 2 \tan^{-1} (R/H) \quad (1)$$

2. RESULTS AND DISCUSSION

Unlike technical beryllium, in which slip is observed only at 77°K and above,[†] basal slip in pure beryllium occurs even at 20°K. It manifests itself in the form of thin straight-line tracks on the lateral faces of the specimen. The appearance of the first band is preceded by a stage of block formation, which is observed from the fragmentation of the Laue dots and deformation without formation of slip tracks. However, in pure beryllium this process is weakly pronounced. For example, it was noted with the aid of an electron-microscope that in pure beryllium the first bands appear at 77°K after 0.3% compression, while in technical beryllium the bands arise only after 3% compression. Furthermore, in pure beryllium the yield point is more than 10 times lower. The lower resistance of single-crystal pure beryllium to shear combines with the weak development of the block formation in the initial stage of deformation. The table lists the mechanical characteristics of beryllium crystals of various purity at 20 and 77°K.

Purification of beryllium leads to a reduction of deformation in block formation and to an increase in the overall plasticity. The slip tracks of pure beryllium at low temperatures are similar in external appearance to the slip tracks of technical

*The procedure of determining the angle of contrasting with the aid of coloxylin small spheres was developed by I. M. Fishman, and will be described in detail elsewhere.

†At 77°K only weak basal slip occurs in technical beryllium.

Test temperature (degrees K)	Purity of beryllium (percentage by weight)	Deformation prior to appearance of bands (percent)	Yield	Total residual compression (percent)	Ultimate strength
20	99.7	No slip bands observed.		2.4	48.0
	99.98	2.0	14.0	8.8	37.0
77	99.7	3.0	42.0	6.0	52.0
	99.98	0.3	3.4	22.0	34.0

beryllium at 200°C and above. This is explained by the fact that the deformation by block formation, which precedes the basal slip, is sufficiently small in either case. The prismatic slip which occurs in technical beryllium at 400°C is observed in pure beryllium at 77°K. As the compression stress increases, the number of slip bands and the displacement in each band also increase. Lines scratched beforehand on a polished lateral face of the specimen bend and shift during the compression process.

3. INVESTIGATION OF THE PROFILE OF THE SPECIMEN SURFACE

Figure 2a shows an electron-microscope photograph of a portion of the face parallel to the $(\bar{1}100)$ shear plane of the specimen, deformed 0.3% (at 77°K) by a compression stress of 3.4 kg/mm^2 . The black lines at 45° to the vertical axis of the specimen are the tracks of basal slip bands. The scribe marks intersect the slip track. At the initial stage of the deformation, the shear is not observed directly on the slip band. As the distance from the track of the band increases, the shear increases and reaches a maximum at a distance of 0.4μ . The remaining portion of the crystal shifts as a whole and therefore the scratch remains straight. Thus, the curved portion of the scribe mark near the slip plane shows a region in which the shear deformation, usually ascribed to the slip band, is concentrated. Figure 2b shows schematically details of an electron-microscope photograph (Fig. 2), in which the reproduction does not show the scribe marks with sufficient contrast). The residual relative shear in this region is

$$\gamma = S_n/b_0 = 0.63,$$

where b_0 is the thickness of the shear region, and S_n the displacement of the scribe mark in the direction of the shear. The straight sections of the scribe marks on both sides of each slippage band are no longer parallel after the deformation. This could be explained by the influence of the remain-

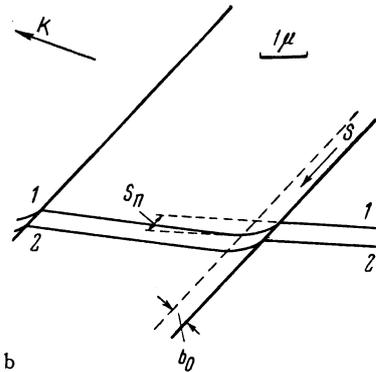
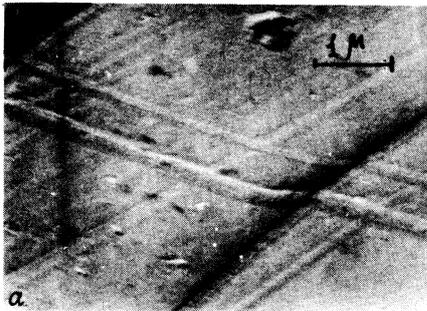


FIG. 2. a) cracks of the basal slip bands on the shear plane (1100) (See Fig. 1). Temperature 77°K. Deformation 0.3%. The curved lines near the slip bands are the scratch marks. b) arrangement of the details of the photograph. Dotted line illustrates the region of the localization of the shear, 1-1 and 2-2 are the scribe marks.

ing elastic stresses which have opposite signs on both sides of each band.⁷ In this case the elastic relative shear is

$$\gamma_1 = \tan \varphi_1 - \tan (\varphi_1 - \alpha_1),$$

where φ_1 is the angle between the normal (N-N) to the slip band and the initial direction of the scribe mark (B-B), and α_2 is the angle of rotation of the scribe mark, which equals approximately half the angle between the directions of the straight segments of the scribe mark (B₁-B₁) on both sides of the band after deformation (see Fig. 3). From Fig. 2a we obtain $\alpha_2 \approx 1^\circ$ when $\varphi_1 = 35^\circ$, which yields $\gamma_1 = 0.026$. This value of α_1 corresponds approximately to the yield point.

Further deformation leads to localization of the shear on the slip band. The scribe marks break and the tracks of the slip band shift a considerable distance from each other. Figures 4a and 4b show an electron-microscope photograph and a diagram of a portion of the lateral face of the specimen, deformed by 6% at 77°K.* The compression was 8.5 kg/mm² before unloading. The

*The specimen was repolished after 1.7% compression and fresh scribe marks were scratched on the side surface. Thus the shift of the marks on Fig. 4 corresponds to a deformation of 4.3% of a specimen previously deformed by 1.7%.

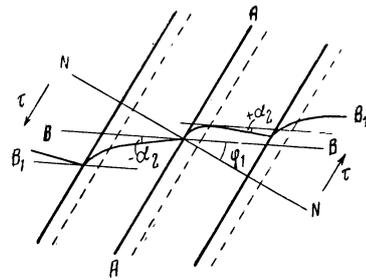


FIG. 3. Illustrating the calculation of the residual elastic stress. A-A) tracks of slip bands on the (1100) shear plane; N-N) normal to the slip plane; B-B) initial position of scribe mark; B₁-B₁) position of scribe mark after a small plastic deformation ($\delta = 0.3\%$); τ) direction of shear.

shear along the slip plane reached a considerable value (2.5μ). Distortions near the slip plane manifest themselves more sharply. The photograph shows clearly the complicated relief of this region.

A striking fact is that the sections of the scribe marks adjacent to the band are less curved in Fig. 4 than in Fig. 2. This is apparently due to localization of the shear in the band. The relative rotation of the straight sections of the marks indicates an increase in the residual stress.* The residual relative shear at this stage of basal slip is difficult to estimate, since the thickness of the deformed layer is very small. It is found thus that, in sufficiently pure beryllium, a shear that is concentrated in the slip band occurs at 77°K. In commercial beryllium, a similar shear is observed only at temperatures exceeding 400°C.²

From the data obtained it follows that in beryllium the initial stage of the basal slip is localized in the region adjacent to the slip band and covers a layer several hundreds or thousands atomic distances thick. Starting with a certain stress, the slip is concentrated in the slip band and is characterized by a large relative shear.

The slip should result in a change in the profile of the lateral face of the specimen (face ah of Fig. 1). Rosenhain⁹ has observed in a single crystal of iron a sawtooth contour, corresponding to

*Large local distortions occur in certain sections of the face bh (see Fig. 1), leading to a considerable increase in the angle of rotation of the scribe marks, α_2 (see Fig. 3). Figure 5 shows the corresponding electron-microscope photograph. In this case $\alpha_2 \approx 3^\circ$, corresponding to $\gamma_1 \approx 0.067$. So large a value cannot be attributed to the influence of the residual stress alone. The rotation of the marks may reflect in addition to the residual stress, also the rotation of the crystal through twinning in slip, similar to that described by Brilliantov and Obreimov.⁸ It is important to note that in our case we deal with slip along the basal plane of a hexagonal crystal, slip considered by most authors as purely translational. In reference 8, however, the substance investigated was rock salt, in which the slip may be more complicated.

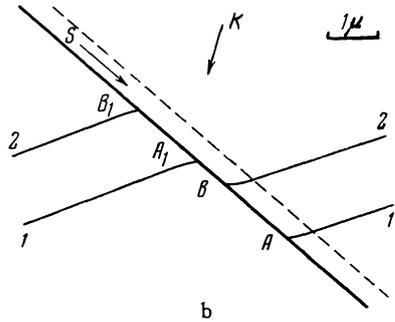
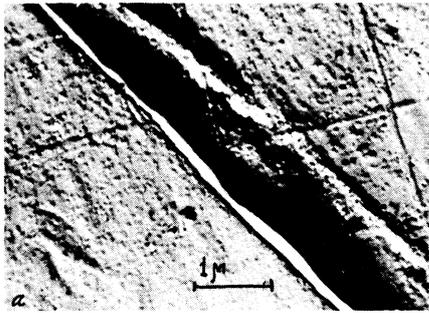


FIG. 4 (A, B). Tracks of the basal slip bands at 77°K. Deformation 6%. The shear is localized on the slip plane and amounts to 2.5μ . $AA_1 \approx BB_1$.

“pure” slip. Greenland,¹⁰ however, found in a mercury single crystal a profile that evidences non-uniform shear of various layers of the crystal. In beryllium one can observe two types of profiles, a smooth one at the start of the deformation and a rough (sawtooth) one at a certain stage of deformation. Micro-interference investigations confirm this statement (Fig. 6). In a specimen compressed by 0.3% at 77°K, the interference bands curve smoothly along the slip track, and the absolute shear displacement amounts to, on the average, one interference fringe (0.22μ) (Fig. 6a). In a specimen compressed by 6% at 77°K, the interference fringes break up on the slip tracks, the abso-

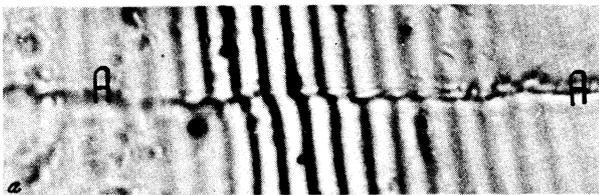


FIG. 6. Interference fringes on the ah face (Fig. 1). Temperature 77°K. A - A) track of slip band, a) deformation 0.3%, b) deformation 6% ($\times 1000$).

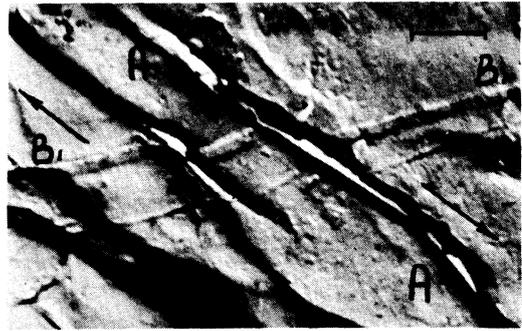


FIG. 5. Illustrating mechanical twinning in basal slip. A - A) tracks of the basal slip band; $B_1 - B_1$) position of the scribe mark after plastic deformation ($\delta = 6\%$).

lute shear displacement amounts to 9 or 10 interference fringes ($2 - 2.25 \mu$) (Fig. 6b).

It was shown earlier² that one of the causes of brittle fracture of single-crystal commercial beryllium is non-uniform shear along the basal slip band. The uneven shear manifests itself in the different displacement of the scribe marks that cross one slip band. In the case of pure beryllium no unevenness in shear is observed even if the shear is in the slip band and amounts to 3 to 5μ . The elimination of impurities from the beryllium contributes to the greater uniformity of the shear in basal slip, eliminates the appearance of microcracks along the planes of the second-order prisms and pyramids at various stages of deformation, and consequently raises the plasticity of the crystal.

The plasticity of purified beryllium single crystals manifests itself also in the fact that at 77°K, along with single slip bands, stacks consisting of many closely-spaced slip bands are produced. The appearance of such stacks is observed even at 0.7% deformation. The stack is 10 to 15μ thick, and the shear displacement is 0.5 to 0.8μ . Figure 7 shows electron-microscope photographs of a stack of basal slip bands in a specimen deformed 0.7% at 77°K. An electron-microscope investigation of the



FIG. 7. Group of basal slip bands on the ah face (Fig. 1) at 77°K and 0.7% deformation.

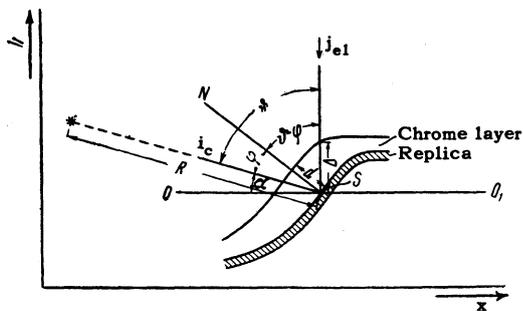


FIG. 8. Illustrating the determination of the profile. i_c – contrast direction, j_{e1} – direction of electron beam, $\alpha_1 = 15^\circ$ – angle between the contrast direction and the undeformed face. OO_1 – undeformed face, S – surface element of the replica, N – normal to the surface element of the replica, φ – angle between the normal to the surface element and the contrast direction.

structure of the stack was made with the aid of chrome-stained lacquer replicas. A photograph of Fig. 7 was used to draw a profile of the slip-band stack.

4. INVESTIGATION OF THE PROFILE BY PHOTOMETRY OF THE ELECTRON-MICROSCOPE PICTURE

Let an element S of the surface of the replica be arbitrarily oriented relative to the beam i_c of the contrasting substance (Fig. 8). The amount of substance condensing on the surface element is determined from the expression

$$q = dQ/dS = Q \cos \varphi / 4\pi R^2, \tag{2}$$

where Q is the total amount of evaporating substance, R is the distance from the evaporator to the surface element S of the replica (R is considerably greater than the linear dimensions of the replica), and φ is the angle between the direction of i_c and the normal to the surface element S .

If the replica is examined in an electron microscope, the electron beam passes through a stained layer of thickness

$$\Delta = \frac{d}{\cos(\vartheta - \varphi)} = \frac{Q \cos \varphi}{4\pi R^2 \rho \cos(\vartheta - \varphi)}, \tag{3}$$

where d is the thickness of the layer of stained substance in the normal direction to the surface element S of the replica, and R is the density of the stained substance.

The electron beam is partly absorbed in the layer Δ and is scattered (one can neglect the absorption in the lacquer replica compared with the absorption in the chromium). The intensity of the electron beam after passage through a layer Δ is

$$J_\Delta = J_0 e^{-\mu \Delta}, \tag{4}$$

where μ is the absorption coefficient. Since

$$dh/dx = \tan(\vartheta - \varphi), \tag{5}$$

(cf. Fig. 8), the ordinates of the profile h can be determined from

$$h = \frac{1}{\beta} \int_0^x f(x) dx, \tag{6}$$

where $\beta = \mu Q / 4\pi R^2 \rho$ is a constant, and

$$f(x) = \ln \frac{J_0}{J_\Delta} \cdot \frac{\sin(\vartheta - \varphi)}{\cos \varphi}, \tag{7}$$

which follows from (2), (3), and (4).

It is not necessary to determine β in order to calculate h , since $\vartheta = \varphi$ for a horizontally-placed element S of the replica (see Fig. 8), i.e., we have from (3) and (4)

$$\ln(J_0/J_{\Delta h}) = \beta \cos \vartheta. \tag{8}$$

Here $J_{\Delta h}$ is the intensity of the electron beam passing through the horizontal portion of the replica.

Inserting into (6) the value of β from (8), we get

$$h = \cot \vartheta \left[\int_0^x \left(\frac{\ln(J_0/J_\Delta)}{\ln(J_0/J_{\Delta h})} - 1 \right) dx \right]. \tag{9}$$

The density D of the photographic plate is proportional, within a definite exposure interval, to the intensity of the electron beam. One can therefore express the integrand $f(x)$ in (9) as follows:

$$f(x) = \frac{\ln(D_{\max}/D)}{\ln(D_{\max}/D_h)} - 1. \tag{10}$$

It must be noted that D_{\max} is the density produced in an electron-microscope plate by an electron beam that has experienced no scattering or absorption, i.e., that has passed through a section of the replica on which there is no contrasting substance. In our case such a spot is the shadow region produced by the coloxilin sphere, placed on the replica to determine the direction and angle of contrast. D_h is the density on the horizontal portion of the replica, while D is the density of the replica at the point with the given value of x . Thus, to determine the profile of the face it is enough to carry out a photometric measurement of the electron-microscope plate, find $f(x)$, and integrate (9). In other words, the area bounded by the curve at $f(x)$ for various values of x will yield all the points of the sought profile of the individual slip band.

Figure 9 shows the profile of a stack of slip

bands in pure beryllium. It is seen that the profile is smooth (as in the case of single bands during the earlier stage of deformation). The thickness b of an elementary band in the stack, in the case of a 0.7% deformation, is estimated to be 700 Å, while the absolute shear ($\Delta S = b - a$) on the band is approximately 200 Å. Consequently, the relative shear in the region adjacent to the slip band is $\gamma = \Delta S/b = 0.3$. In the case of a single band, $\gamma = 0.6$ at $\delta = 0.3\%$. For a uniform overall deformation in the stack, the shear at each band is somewhat smaller than in individual slip bands, this being compensated for by the closer placement of the bands in the stack.

5. INTERPRETATION OF THE PHENOMENA

It is interesting to compare the data obtained with the results of investigations on the fine structure of slip bands of the following plastic metals; mercury,¹⁰ zinc,¹¹ and aluminum.¹² In these metals the profile is believed by the authors to be smooth and the average shear per band is believed to be several thousands of angstroms. The relative shear per band is several units. Since these data pertain to the initial stage of deformation, one can conclude that pure beryllium exhibits typical plastic properties at low degrees of compression.

The observed phenomena can be interpreted in terms of the dislocation theory as follows. In the initial stage shearing occurs of an entire stack of glide planes, which leads to bending of the lines near the future slip bands, as can be seen from Fig. 2. The resolving power of the electron-microscope would permit observation of steps not less than 100 Å on the curved portion of the scribe line. Judging from Fig. 2a, no such steps appear, and consequently we can assume that the adjacent glide planes does not exceed 100 Å. This corresponds to a linear density of 10^6 per centimeter of the edge dislocations in a direction perpendicular to the glide planes. Assuming that the distance between the dislocations in each glide plane is of the same order, we obtain a figure of 10^{12} per cm^2 for the dislocation density in this zone.

As the relative shear reaches 0.6, corresponding, under these assumptions, to an emergence of 20 dislocations on the lateral surface of the crystal at each glide plane, the active planes become stronger, since some of the dislocation stick together. As the stresses increase further, this leads to localization of the deformation on one the glide planes. The region of localization of large shear displacements is usually called a slip band, as was done in the present paper.

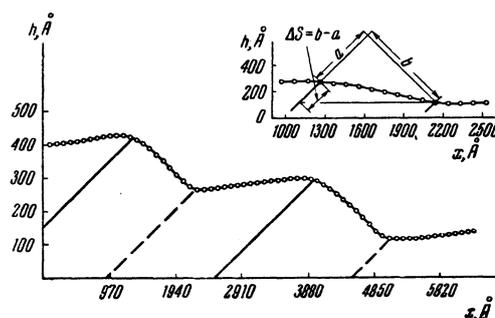


FIG. 9. Profile of a stack of slip bands in pure beryllium (cf. Fig. 7). b) thickness of elementary band in the pocket, equal to 700 Å, ΔS) absolute shear in band, equal to 200 Å. The relative shear is $\gamma = 0.3$.

The shear localized in a slip band at 77°K reaches several microns, which amounts to approximately 10^4 atomic distances. It is hard to believe that a regular motion of so large a number of dislocations is possible without a noticeable strengthening. It is therefore more probable that the localized shear is due to damage to the continuity with subsequent healing of the contacts.¹³

6. CONCLUSIONS

1. In crystals of 99.98% pure beryllium a clearly pronounced slip is observed along the (0001) basal plane, beginning with the temperature of liquid hydrogen (20°K).

2. The character of the slip differs substantially at different stages of deformation. At small degree of compression, there is no shear directly on the band, and the slip takes place in layers adjacent to the band. The remaining part of the crystal, between the two bands, moves as a whole. The residual stresses produce an elastic shear of opposite sign in the layers of the crystal adjacent to the band. At considerable degrees of compression, the slip is concentrated on the band and is characterized by a large relative shear. A characteristic feature of this stage is the development of a sawtooth profile in the crystal face, which, in the case of basal slip,⁸ can be considered as the result of twinning along planes with large indices which combines with the break in the continuity and a subsequent healing of the contacts over the slip surface.¹³

3. The non-uniformity of the shear is due to the presence of impurities; purification of the beryllium makes for more uniform shear along each slip band, leading in turn to an increase in the plasticity of the metal. At 77°K one observes a formation of stacks of basal slip bands.

A method was developed for plotting the face

profiles of deformed crystals, which makes it possible to determine the principal parameters of the fine structure of single and multiple slip bands.

The authors expressed their gratitude to I. M. Fishman for preparing and contrasting of the replicas and taking the electron-microscope photographs.

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