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INVESTIGATION OF PLASTIC DEFORMATION OF BERYLLIUM SINGLE CRYSTALS SUBJECTED TO COMPRESSION

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An x-ray press-camera for studying plastic deformation of single-crystal and polycrystalline samples in the stressed state is described. The plastic deformation of beryllium single crystals of reagent grade purity was studied in this apparatus for three different orientations. The x-ray photographs obtained during various stages of deformation are associated with various portions of the deformation curves. On the basis of this investigation, certain conclusions have been reached concerning the mechanism of plastic deformation in beryllium.

As a rule, the elements of plasticity of single crystals are studied after deformation of the samples by special testing machines. Although one can, by this means, arrive at information concerning the type of plastic deformation the crystals have experienced (be it slipping, twinning, kinks, etc.), neither metallographic nor x-ray investigations performed on previously deformed samples yield sufficiently complete information about the mechanism or about the kinetics of these processes. Clearly a better way to identify the elements of plastic deformation or to study the kinetics of deformation or the failure mechanism of single crystals and also the only way to measure the magnitude of elastic deformation of the crystals by direct x-ray techniques is to x-ray the samples during the mechanical testing process.

In this paper we describe an x-ray investigation of the plastic deformation of single crystals of beryllium of various orientations. The selection of beryllium single crystals for this study was based on the specific plasticity of this material as well as on the search for new elements of plasticity in high-purity beryllium samples. In this paper we shall describe only the investigation of the kinetics of plastic deformation of single crystals of Be of reagent grade purity (99%) with well-known elements of plasticity: slip along the (0001) basal planes, prisms of the first kind $(10\bar{1}0)$, and twinning along the planes of pyramids of the first kind $(10\bar{1}2)$.

A special x-ray camera [1] was constructed for this study of plastic deformation of these samples. It consisted of a compact mechanical press with automatic loading, equipped with a mechanism for simultaneously photographing the deformed samples "by transmission" and "reflection". The x-ray photographs could be made either at a fixed load or directly during the process of loading at a slow rate. Not only does this camera make it possible to x-ray the sample while it is deforming, but it also makes it possible to elucidate the shape of the deformation curve.

The x-raying of samples during the compression process was performed on a Type URS-70K1 apparatus with unfiltered iron x radiation. The specimens were made from single crystals of beryllium obtained by slow crystallization from the melt contained in a beryllium oxide crucible. Specimens in the form of rectangular parallelepipeds of dimensions 2×2×4 mm were prepared in the usual way: electric spark cutting, polishing, chemical etching, annealing, and electrical polishing. Only samples with a relatively perfect structure were used in this study, i.e., we did not use samples containing large grains.

The deformation of the samples was studied in the following way. The crystal was loaded step-by-step according to a prescribed program. Dynamometric and strain gauge sensors were used to evaluate the load and the deformation. Then the sample was irradiated. Cycles of loading and photographing the sample were continued until failure occurred. Some of the x-ray photographs obtained in this way are shown in Fig. 1a-c.

Samples of three different orientations (I-III) were selected for the studies of plastic deformation; these are shown schematically in Fig. 2.

Figure 2 shows a schematic representation of deformation curves rather than actual experimental curves. This is because the small press-camera was not rigid enough and it did not permit one to measure the deformation of the samples with the desired accuracy. During the tests we were able to record only the loads with accuracy; the deformations were determined in arbitrary units only.

The samples with orientation II were subjected to a parallel study in a standard testing machine. We shall refer to this machine-generated deformation curve throughout this paper.

I. The basal plane (0001) perpendicular to the loading axis. The side faces of the samples were $(10\bar{1}0)$ and $(11\bar{2}0)$ planes.

II. The basal plane (0001) and direction of the basal slip $(11\bar{2}0)$ are oriented 45° from the loading axis.

III. The plane of a prism of the first kind $(10\bar{1}0)$ is oriented perpendicular to the load axis. The side

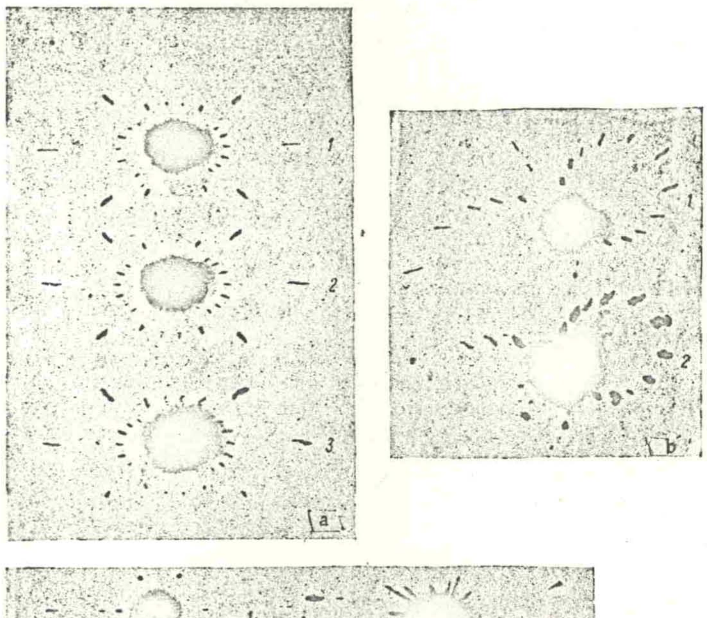

Fig. 1. X-ray photographs of initial (1) and deformed (2-6) samples with orientations I (a), II (b), and III (c).

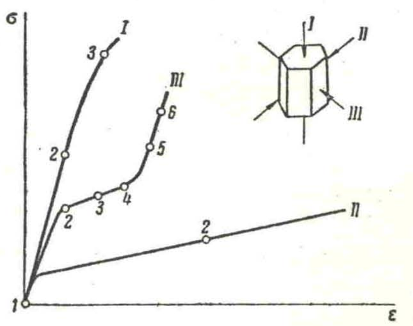

Fig. 2. Deformation curves (schematic) and the orientation scheme of the crystals in this investigation. 1-6) Points corresponding to x-ray photographs in Fig. 1a-c.

faces of the sample are the (11$\bar{2}$0) and (0001) planes.

The choice of the orientations indicated above is based on the following considerations.

In the case of type I orientation, the well-known elements of plastic deformation of beryllium do not occur; twinning along (10$\bar{1}$2) planes cannot occur since this would require dilation in a direction opposite to that of the applied load, and slipping along the (0001) and (10$\bar{1}$0) planes is eliminated because there is no component of the force in the slip direction [11$\bar{2}$0]. This orientation is convenient for observation and investigation of pyramidal slip, which, according to [2, 3], has been observed in the metal when its purity was 99.9% at a temperature of about 200°C. The deformation of single crystals of beryllium of such an orientation was studied in [2-7]. It was shown that in all cases when the crystals were compressed at room temperature only plastic deformation was observable and failure occurred at high stresses, which, according to data of various authors, varied in the range 140 [6] to 210 kg/mm^2 [2].

Orientation of type II was most convenient for slipping along the basal plane. This kind of deformation of beryllium has been studied in great detail [6, 8-10]. Here our main interest will be in comparing results involving the comparison of critical shear stress data from x-ray and microscopic data and also the investigation of the character of structural changes at high deformations.

The compression of beryllium single crystals in the III orientation has been studied in [4, 11]. It was shown that when the samples are compressed along directions perpendicular to the (10$\bar{1}$0) or (11$\bar{2}$0) planes, crystal twinning along (10$\bar{1}$2) planes occurs such that the crystal gradually passes over into a completely twinned situation. It is of interest to study the kinetics of this process of the subsequent deformation of the twinned crystal.

Sample with orientation I. The photographs were taken with the x-ray beam directed perpendicular to the (10$\bar{1}$0) or (11$\bar{2}$0) planes of the single crystal. Four samples of the same type were studied. The x-ray photograph of one of these crystals in the initial state is shown in (1) of Fig. 1a. For compressive stresses less than 60 kg/mm^2 no changes were detected in x-ray photographs. Beyond this point the reflections began to separate, which corresponded to a weak fragmentation and the formation of blocks. This process intensified with increasing compression: Fig. 1a (2 and 3) shows illustrative data for the x-ray process, taken at loads of 70 and 150 kg/mm^2. No plastic deformation effects were detected, even up to failure of the crystals. The deformation curve for this case is shown in Fig. 2 (curve I), corresponding to the elastic region. The reflection photographs exhibited a small shift in the position of the reflections, but because these shifts were small, it was not possible to make a quantitative evaluation of the magnitude of the elastic compression of the lattice.

When the crystals of this orientation were compressed in the standard testing machine, the measured values of deformation before failure amounted to 1.5%. For loads less than 60 kg/mm^2 the deformation curve was characterized by a completely elastic region while at higher loads some plasticity was detectable (0.3 to 0.7%); based on the data given above, it is clear that this plasticity is completely determined by the block-formation processes.

Failure of the crystals which we studied occurred at loads of 130, 155, 162, and 170.3 kg/mm^2. One sample failed as a result of static aging after being subjected to the load for 1.5 h, rather than failing during the process of application of successively higher loads. The x-ray photographs of samples prior to failure did not exhibit any peculiarities and were characterized by the growth of the block-formation process. When the samples failed, they crumbled into small particles, just as observed in [2, 4].

On the basis of these results, it is possible to draw the following picture of the deformation of beryllium crystals of basal orientation. The crystal lattice experiences only plastic deformation up to loads between 50 and 60 kg/mm^2. Afterwards, because of existing imperfections in the crystal lattice, a gradual redistribution of dislocations takes place with the formation of defective regions with small-angle boundaries. The source for motion of dislocations are variations in stress and, consequently, the presence of sinks for defects. Dislocation pileup, which occurs when the stress increases, is accompanied by the formation of the first microcracks and destruction of the sample. It is evident that the distribution of imperfections in the original crystals plays an important role in this process; therefore, even samples which appear to be duplicates of each other have different strength. This is the reason for the wide spread in experimental values of the strength as given by various authors.

Sample of orientation II. The deformation of samples of this orientation was studied simultaneously with the standard testing machine and with the x-ray camera. The shape of the deformation curves for crystals with orientations convenient for slipping along the basal plane are shown in Fig. 2 (curve II). The critical shearing stress was determined in three different ways: on the basis of the deformation curve from the break in the curve corresponding to the onset of slip; from the appearance of the first slip lines as seen during an examination of a deformed sample through a microscope, and by the appearance of asterism spots on the x-ray photographs. The value of $\tau_{0001} = 1.2$ kg/mm^2 calculated from the deformation curves was somewhat higher than the values obtained by the other two techniques ($\tau_{0001} = 1.05$ kg/mm^2).

The x-ray photographs of the crystals of this orientation in the initial state and after plastic deformation caused by basal slipping are shown in Fig. 1b. When the samples were compressed, as is evident from the x-ray data, after the small elastic region in the curves an intense basal slip is observed; the orientation of the crystal remains essentially unchanged. At higher deformations the displacement along the basal plane is accompanied by bending of the slip plane and the appearance of cracks. However, because of the specific characteristics of the compression tests, total failure of the sample does not occur at this point. The x-ray photograph Fig. 1b (2) characterizes the slip process with the formation of kinks; in this case the shape of the sample is already changing. X-ray studies prove that the elements of plastic deformation, except for slip along the basal plane, are absent in this case.

Samples of orientation III. X-ray investigations of the compressive process of samples in a direction normal to prisms of the first kind made possible a detailed investigation of the kinetics of twinning in beryllium. The shape of the deformation curve and of the different stages of compression are shown by the x-ray photographs in Fig. 2 (curve III)[1] and Fig. 1c, respectively.

Compression of crystals of orientation III allows two elements of plastic deformation to enter the picture: prismatic slip in the system (10$\bar{1}$0) [11$\bar{2}$0] and twinning along (10$\bar{1}$2) planes. Analysis of the x-ray photograph Fig. 1c and of the deformation curve shows that both forms of plasticity appear almost simultaneously at a stress of about 10 kg/mm^2; however, it is evident that the beginning of slip predominates. This is supported by the readily observed asterism in the Laue spot of Fig. 1c (2 and 3). As entirely new regions of the crystal are included in the twinning process, prismatic slip is reduced in importance because of the presence of a large number of stress concentrators. The first kink in curve III (Fig. 2) characterizes the onset of intense twinning. The process of twinning proceeds mainly in the region between the first and second kink in curve III. Different stages of this process are characterized by x-ray photographs of Fig. 1c (2-4). The first of them corresponds to a sample which has preserved its original orientation and which has a small number of twins. Twinning spans not only the undeformed parts of the crystal but also the parts undergoing shear. Weak twinning is also observed after the second kink in curve III; however, the x-ray photograph which corresponds to this portion of the deformation curve essentially characterizes a crystal with new orientation. It is interesting to note that the reorientation of the sample as a result of twinning occurs in such a way that after the crystal has undergone complete twinning, it has an orientation close to orientation I. Since twinning occurs without breaking the crystal, further deformation is possible. This process is characterized by the second branch of curve III (Fig. 2) and the x-ray photograph Fig. 1c (5 and 6). Although the normal to the basal plane in the twinned crystal is tilted by 6° from the axis of applied stress, slip along the basal plane is not observed even up to stresses which result in failure. Clearly, this is caused by hardening of the specimen resulting from the previous plastic deformation. Failure of reoriented crystals as well as of samples with orientation I, a (1) occurs at high stresses (120 to 140 kg/mm^2).

Thus in this paper we have studied the mechanism and kinetics of the deformation of single crystals of technical grade beryllium of various orien-

[1] Curve III is not smooth because twinning is accompanied by the appearance of discontinuities on the deformation curve.

tations. The resulting data,which in many cases could be compared to the results of investigations of beryllium single crystals performed by the standard techniques, showed the usefulness of this method for investigation of structural changes during the deformation process as applied to the plasticity of beryllium.

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