

The shock loading of polycrystalline beryllium: residual microstructure and the effects of microstructure on internal spallation

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The residual microstructure of polycrystalline beryllium in both a rolled-reduced and hot-pressed form has been characterized by transmission electron microscopy following shock deformation at pressures up to 9.2 kbar, and the observation of residual shock softening was attributed to the activated-glide response of sub-grain boundaries to the shock wave passage. A marked difference in the shock-induced spallation in the roll-reduced material as compared with the hot-pressed material has been attributed to a difference in the density of grain-boundary ledge structure which induces larger spall cracks with a greater frequency when the density and size of the ledges is large.

1. Introduction

To examine some of the dynamic properties of beryllium, Stevens and Pope [1] have conducted a study of wave propagation in single crystal beryllium by shock-loading to selectively activate various slip systems [2], followed by a study of wave propagation and spallation in shock-loaded polycrystalline beryllium [1]. These researchers have noted that there are open questions related to the deviation in the behaviour of beryllium when subjected to shock-loading. In most metals, the compressive wave generated by shock-loading separates into a low-amplitude, high velocity elastic precursor wave followed by a lower velocity plastic wave. In beryllium, both of these waves deviate substantially from the idealized case. In their study of polycrystalline beryllium, an "as-pressed" material and a variety of "textured" materials were used to examine the effects of heat-treatment on this unusual dynamic property.

When a solid plate is subjected to explosive loading against one surface, the interference from the incident and the reflected wave from the oppo-

site surface will cause a tensile stress to be built up a short distance from the opposite surface. If this tensile stress exceeds the strength of the material, a thin layer on the opposite surface may separate from the plate. This rupture is referred to as a spall [3]. Stevens and Pope noticed a difference in the spall damage inflicted on the "as-pressed" material with respect to the "textured" material, but the microstructural feature responsible for this was not determined.

The present study is a continuation of Stevens and Pope's work in which an effort has been made to (1) characterize the microstructural feature responsible for the difference in spall damage inflicted on materials prepared by different heat-treatments, (2) examine the effects of heat-treatments involving the rolling of hot-pressed beryllium on the microstructural features, and (3) examine the effects of shock-loading at pressures below 10 kbar on the microstructure of both "as-pressed" and "textured" (roll-reduced) powder metallurgical beryllium. Therefore, it was concluded at the outset that the results of this study

should increase the understanding of the microstructural reaction of beryllium to rapid loading and help explain the unusual observations made in the previous studies as noted above.

2. Experimental procedure

The polycrystalline beryllium used in this study was supplied by Stevens and Pope of Sandia Laboratories, Albuquerque [1]. The samples were fabricated from a single billet of Kawecki-Berylco hot-pressed HP-10 beryllium with a purity of approximately 99.0%. Table I lists the quantity of impurities present.

During this study, two series of samples were used: an as-pressed set and a textured set unidirectionally rolled to a reduction of 13.5:1. The rolling was conducted at 775°C with approximately a 10% reduction per pass, and the samples were subsequently annealed at 705°C for ½ h.

Stevens and Pope determined that there was a concentration of basal planes parallel to the rolling direction, which they quantified using X-ray diffraction basal plane pole figures. Their results indicated that the concentration of basal planes in the roll-reduced samples was eight times as great as the random concentration of basal planes in the as-pressed material. This texturing caused by rolling has been mentioned elsewhere in the literature [4].

In addition, a commercially available 10 mil* beryllium sheet hot-pressed and rolled to thickness was used to establish the procedures. This material will be referred to as "as-received" beryllium in this study.

Both sets of samples supplied by Stevens and Pope were subjected to planar impact using a technique described by Karnes [5]. Discs 3 mm thick and 50 mm diameter were impacted uniformly by a 1 mm thick, 50 mm diameter fused silica flyer-plate disc. By recording the impact velocity, the approximate impact stress was determined. Each set of samples consisted of an annealed specimen and two shock-loaded specimens. The shock-loaded specimens in the as-pressed set were impacted at stress levels of approximately 7.2 and 9.2 kbar, while the shock-loaded specimens in the roll-reduced set were impacted at stress levels of approximately 6.6 and 9.2 kbar.

TABLE I Beryllium analysis (wt %)

BeO	C	Fe	Al	Mg	Si	Other	Be
0.700	0.048	0.120	0.043	0.004	0.032	0.040	Balance

*1 mil = 0.0254 cm.

Samples were removed from the impacted discs by sectioning with a spark-cutting apparatus. The samples varied in thickness from 0.2 to 0.5 mm. Further preparation depended on the intended examination involved: samples used for X-ray analysis were lightly electropolished before insertion into a holder; samples used for microhardness testing were mechanically fine-ground through 600 grit with silicon carbide paper prior to electropolishing to insure a flat surface; and samples used to produce the thin foils used for the transmission electron microscopy were electropolished to a thickness which would permit transmission of the electron beam.

All electropolishing was accomplished by modifying Walter's and Fuller's electropolishing procedure found in the literature [6]. These modifications involved the use of the window technique with a subsequent change in the cleaning procedure and a different selection of voltage and current to render this technique applicable to a constant-current power supply. The modified procedure employed a solution of 70 ml 85% phosphoric acid, 35 ml glycerine, 5 ml freshly prepared Cr₂O₃, 50% H₂O solution. The solution temperature was maintained between 65 and 75°C. A Sorenson DCR 150-10A power supply was employed. Surface polishing was accomplished at a current setting of 1.4 A while electropolished thin sections were obtained at a current setting of 0.8 A.

Samples to be electropolished were clamped in tweezers, then the tweezers and the sample edges were coated with Microstop. A beaker containing the electropolishing solution was heated with gentle stirring; a nichrome strip hung in the beaker acted as the cathode. The smooth surface required for the X-ray analysis and the microhardness testing was created with a submersion of approximately 1 min in the electrolyte. The electropolishing needed to thin samples for the transmission electron microscopy was accomplished by turning the sample 180° every minute to produce a foil from the centre of the sample. The time required to thin a foil varied from approximately 3 to 20 min depending on the thickness and on the processing and deformation history of the sample.

After electropolishing, the sample was rinsed in

hot, de-ionized water to remove the remaining solution. This step was repeated to insure adequate washing, then the sample was freed from the tweezers with acetone. This was followed by two acetone rinses to ensure complete removal of the Microstop. The final cleaning step was a double rinse in ethyl alcohol to remove as much contamination as possible from the foil. The above cleaning procedure was only necessary for the samples thinned to TEM foils; the surface smoothing operations required for X-ray analysis and microhardness testing did not demand a sample absolutely free from contamination.

Microhardness testing was accomplished using a Vickers M12a Tester operated with a 100 g load. After the surface smoothing operation described above, the sample was positioned on the load table, an area was selected, and the hardness was determined. No less than 20 individual determinations were performed per sample. The results were then averaged and plotted in the form of histograms. A graph plotting the change in hardness with respect to the change in shock pressure was also prepared.

All transmission electron microscopy was performed on a Hitachi-Perkin Elmer H.U. 200F electron microscope equipped with a goniometer-tilt stage and operated at 200 kV accelerating potential. After the foil was cleaned, an area was trimmed off and mounted in the specimen holder. The mounted specimen was placed immediately in the high vacuum chamber in an effort to reduce oxidation on the foil. The foil was scanned, and representative areas were photographed. A com-

parison of the microstructure with respect to the texturing and deformation was then made.

3. Experimental results

The diffractograms obtained during the X-ray analysis demonstrated only the degree of texturing. The intensity of the reported peaks changed appreciably from the as-pressed material to the textured material, and the intensities changed only slightly with a variation in the deformation history.

The microhardness examination supported the concept of the work softening of beryllium subjected to high strain-rates as observed in Fig. 1. It can be seen in Fig. 1 that the small grained, textured material is harder than the large grained, as-pressed material. This increase in hardness is a consequence of not only the grain size, but also more specifically the dislocation characteristics of the materials [7], a point that will be discussed in detail in a later section of this paper. The anisotropy noted in this examination will also be dealt with more thoroughly in the Discussion.

Another experimental result that is evident from Fig. 1 is the general softening of the material as shock pressures are increased. This phenomenon has been reported in the literature [8, 9] for beryllium and other hcp metals for conventional loading. This result will also be discussed at length in the Discussion.

One of the objectives of this study has been to characterize the effects of shock loading on the microstructure of beryllium with the aid of a transmission electron microscope. An unexpected result was obtained: the influence of the preparation history is apparently much greater than the influence of the low-pressure shock loading. This statement is supported by examination and comparison of Fig. 2a and b. The micrograph of the textured, annealed material shown in Fig. 2a demonstrates that the material contains dislocation walls (A), dislocation networks (B), grain-boundary ledges (C), dislocation pinning and/or generation at the grain boundaries (D), and possible pinning of dislocations at inclusions (E). These features are identified by Bonfield [7] as characteristics of the microstructure of beryllium subjected to high strain-rates.

Fig. 2b, a micrograph of the as-pressed, annealed material, demonstrates that the hot-pressed structure is significantly different from the textured structure. There are few dislocations in the grain, yet the boundary is composed of a dense

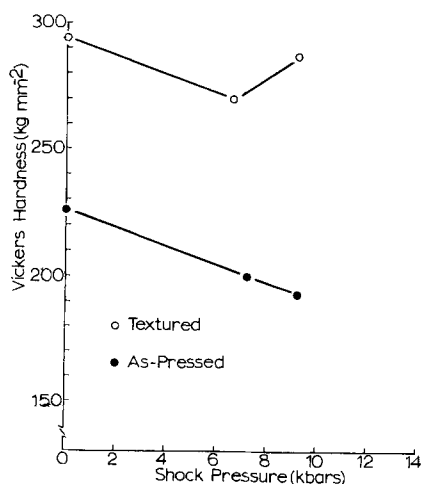


Figure 1 Residual hardness of shock-loaded beryllium.

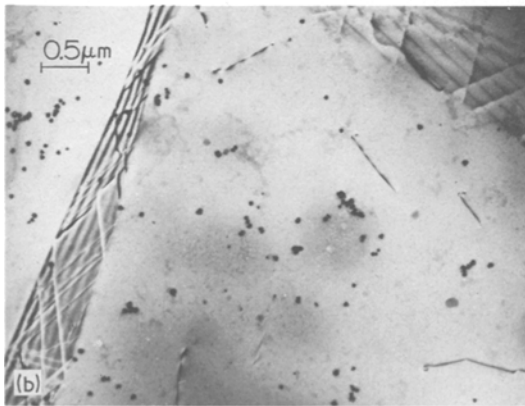
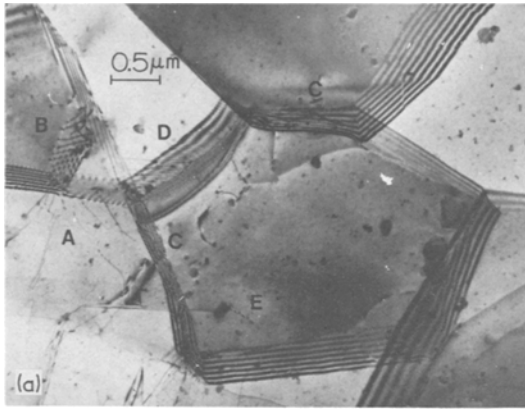


Figure 2 Annealed, as-fabricated beryllium microstructure. (a) Textured, annealed beryllium, (b) as-pressed, annealed beryllium.

ledge structure. The difference in the grain-boundary ledge structure is the most striking difference between the two forms of polycrystalline beryllium examined during this study, and this feature is thought to be responsible for the difference in spall characteristics encountered between the textured and the as-pressed materials.

Further inspection of Fig. 2 reveals a large difference in the grain sizes of these two materials. It was established that the grain size of the as-pressed material averages $30\ \mu\text{m}$, which is in agreement with the results obtained by Stevens and Pope [1]; however, it was established that the grain size of the textured material averages approximately $4\ \mu\text{m}$. This value is in agreement with a grain-size determination of hot-pressed rolled beryllium found in the literature [4]. The deviation between the grain size of approximately $18\ \mu\text{m}$ reported by Stevens and Pope and the results obtained in this study is possibly due to the failure of optical microscopy to reveal low-angle boundaries that are seen in the transmission elec-

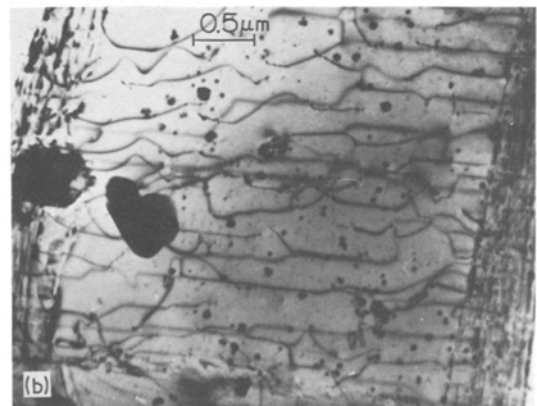
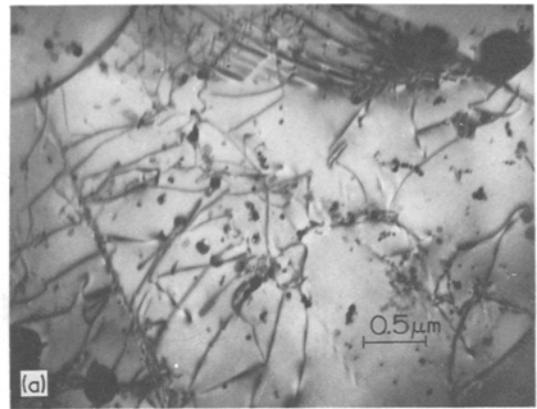


Figure 3 Textured, annealed, shock-loaded beryllium. (a) Shock-loaded at 6.6 kbar, (b) shock-loaded at 9.2 kbar showing dislocation emission from grain boundary.

tron microscope.

The typical microstructure of the shock-loaded beryllium studied is revealed in Figs. 3 and 4. It should be kept in mind that the majority of the spots seen in these photographs are not structural features but are contamination due to the preparation technique. This contamination seemed to be minimized if both the Cr_2O_3 solution and the H_3PO_4 were freshly prepared.

It can be observed from Figs. 2 to 4 that the annealed, as-pressed beryllium demonstrates a low density of dislocations, but it also contains a dense ledge structure in the grain boundaries. It should be noted that some areas containing a greater density of dislocations were seen indicating a state of heterogeneous strain exists. The existence of heterogeneous shear has been attributed by Bonfield [7] to the relative ease of slip on the basal plane coupled with the limited slip on other planes. Shock loading increases the density of dislocations which creates areas of extensive interactions, but the dense ledge structure remains the

single, most unique characteristic of this material. During this study, no hexagonal networks were seen in the as-pressed material. The textured, annealed material demonstrated features identified by Bonfield [7] as characteristic of beryllium that has been subjected to high strain-rates: dislocation walls, hexagonal dislocation networks, other complex arrays, and particle-dislocation interactions. These features were apparently enlarged in the shocked material, and the density of dislocations

consequently increased.

While the arrangements of dislocations in the textured and shock-loaded and as-pressed and shock-loaded samples were similar as observed from Figs. 3 and 4, there appeared to be a marked difference in the density of grain-boundary ledges in the as-pressed material which gave rise to a greater number of grain-boundary related dislocations emitted after shock loading. On comparing the unshocked textured and as-pressed specimen structures, it was concluded that although the grain size was smaller in the textured material, the grain-boundary ledge density was higher in the as-pressed material.

Fig. 5 illustrates several examples of ledge structure in the as-pressed beryllium and several interesting features of ledges. In Fig. 5a dislocations are observed to be extending into the matrix from what appear to be grain-boundary ledge sources. In Fig. 5b, dislocation loops generated by dislocations from grain-boundary ledges are observed. While such loop formation has been postulated to occur from grain-boundary ledges, there have not been any previous observations in support of the postulate [10].

4. Discussion

Examination of the data obtained during the microhardness testing reveals a wide spread in the values derived in any one sample. This is a result of the limited slip systems available for flow in beryllium at room temperature. As pointed out by

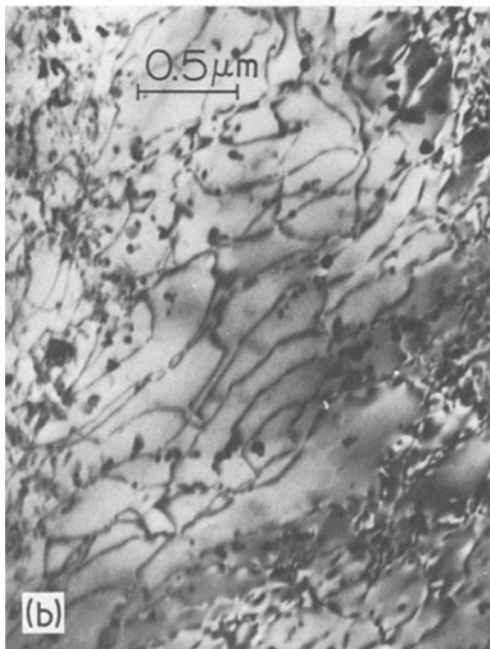
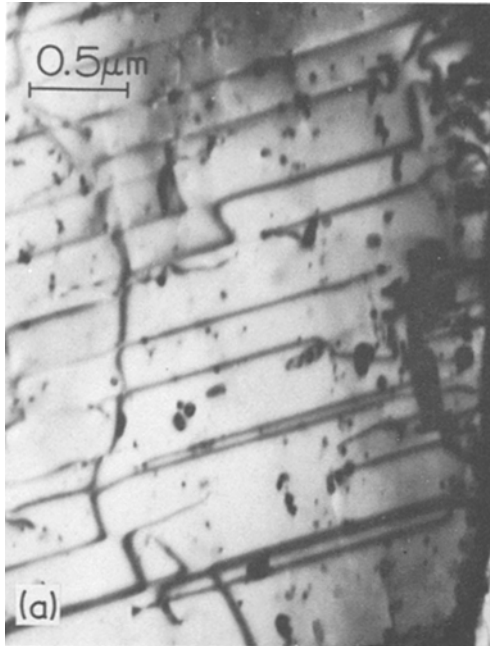
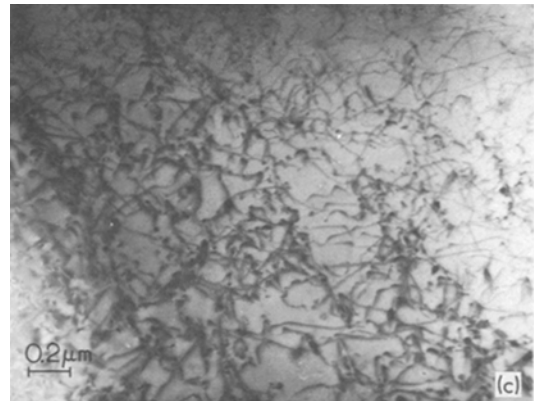


Figure 4 As-pressed, annealed, shock-loaded beryllium. (a) Shock-loaded at 7.2 kbar showing long dislocations presumably emitted from a grain boundary as in Fig. 3b, (b) same specimen as (a) showing more uniform distribution of dislocations following 7.2 kbar shock loading, (c) shock-loaded at 9.2 kbar showing complex tangles of dislocations.



London *et al.* [11] only two slip systems normally operate for these conditions: basal slip (0001) $[11\bar{2}0]$ and prism slip $\{10\bar{1}0\}$ $[11\bar{2}0]$. Pyramidal slip only occurs at elevated temperature or at very high stresses. Thus, if an indentation were made on a grain in which one of these slip systems was available, a lower hardness would result with respect to an indentation made on a grain in which no easy slip system was available.

Another factor forwarded to explain these results is the generation of thermal microstresses in beryllium and other hcp metals upon cooling. These microstresses are created by the anisotropy of these materials with respect to their thermo-mechanical properties; i.e. the differences between the thermal coefficient of expansion in relation to difference in the expansion or contraction along the various crystallographic planes and directions. Microstrains are generated during heating and cooling because the individual grains mutually restrict any anisotropic dimensional changes. Armstrong and Borch [12] state that in beryllium these microstrains are a result of compressive stress normal to the basal plane and isotropic tensile stress in the basal plane, and that these stresses can potentially generate strains comparable to conventionally measured bulk yield and fracture stress values. Consequently, these authors feel that this anisotropic behaviour can generate stresses capable of influencing the deformation behaviour of beryllium, especially if the thermal history of the material involves temperatures above 700°C .

Stevens and Pope [1] have explained the variation in the elastic precursor wave propagation with differing texture by reasoning that as the basal planes are aligned due to the rolling reduction, the microstrains generated by the anisotropic thermal properties are reduced. Certainly this factor is in part responsible for the deviation of hardness values noted above.

The characteristic features of the dislocations in the textured material were hexagonal dislocation networks, dislocation walls, and particle-dislocation interactions. These features are related to beryllium that has been subjected to deformation accompanied by a high strain-rate. Walls, networks and particle-dislocation interactions contribute to local work-hardening [7]; therefore, they are responsible for fixation of glide dislocations in the limited slip systems. Comparison of the annealed, textured material that possesses features which stop the limited glide with the annealed, as-pressed

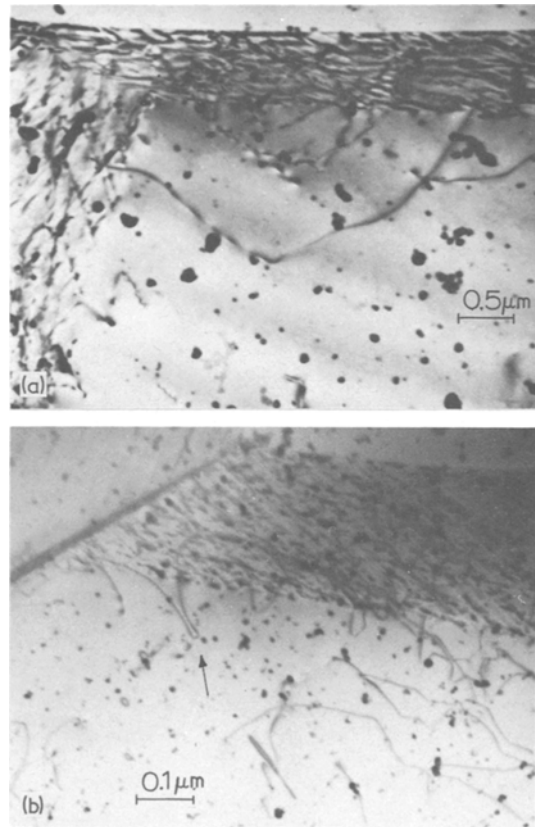


Figure 5 Grain-boundary ledge structures in the as-pressed beryllium. (a) As-pressed, annealed Be showing dislocations attached to ledges in the grain boundary, (b) dislocation loops associated with ledges (arrow) in a grain boundary following shock loading to 9.2 kbar.

material that is relatively free of dislocations and features acting as barriers to dislocation glide, explains the large deviation between the average hardness of these two materials. Of course, the difference in the grain size will accentuate the hardness deviation; the larger-grained, as-pressed material inherently possesses a longer path for free dislocation glide than the smaller-grained, textured material.

The general softening of the beryllium with increasing shock pressure that can be seen in Fig. 1 has been reported in the literature [8, 9] for beryllium and other hexagonal close-packed metals. The occurrence of work-softening during shock-loading is reasonable, for Edwards *et al.* [9] state that strain softening is enhanced by a high strain-rate. Deighton and Perkins [8] propose a model to explain this phenomenon, the basis of which is the stress-activated glide of sub-boundaries. They state that softening is a consequence of deformation and not of stress; therefore, work-softening is poss-

ibly due to the disintegration of the dislocation cell structure by a combination of low angle sub-boundaries to form higher angle sub-boundaries. This process leads to fewer sub-boundaries with a higher angle of misorientation, and these boundaries possibly become fixed when the dislocations in the boundaries become so closely packed that they lose their identity. Of course, when the sub-boundaries become fixed, the work-softening process is reversed. The results of this study support this proposal. It will be recalled that as the shock pressure increased in the textured material in which the identification of sub-boundaries was possible, the sub-boundary features appeared to increase in size. In the textured material with the greatest abundance of sub-boundaries in the annealed condition, it is seen that the softening trend is reversed between shock pressures of 7.2 and 9.2 kbar. Deighton and Perkins' proposal suggests that this is due to a fixation of the sub-boundaries which then act as conventional barriers to dislocation glide.

The features such as hexagonal dislocation networks, dislocation walls and particle-dislocation interactions introduced in the textured material because of the high strain-rate encountered during the rolling operation were one of the obvious microstructural differences between the textured and the as-pressed materials. These differences have been used to explain the large deviation between the average hardness of these two materials. However, these features do not clearly establish a correlation between the microstructural differences and the observed difference in the spall crack characteristics between the textured and as-pressed material.

In their work on the wave propagation and spallation characteristics of polycrystalline beryllium [4], Stevens and Pope noted that although the spall strength does not vary significantly between the textured and the as-pressed materials, the distribution and spall characteristics do vary with respect to a difference in texture. They state that during "metallurgical examination the spall damage in the as-pressed material is seen to be transgranular cleavage cracks, somewhat randomly oriented in direction and distributed over the centre half region of the $\frac{1}{8}$ in. thick sample. In contrast, the spall damage in the texture material is characterized by fewer cracks, which are more continuous and parallel to the plane of the plate". They attributed this difference in spall damage to

the preferred orientation of basal planes parallel to the rolling direction in the textured material which created a more continuous path along which cleavage could progress. As a result of the present study, a microstructural feature can be forwarded as the cause for the observed difference in the spall damage between the textured and the as-pressed beryllium.

The single, most striking microstructural difference between the as-pressed and the textured materials was the occurrence of a dense ledge structure in the grain boundaries of the as-pressed material when compared with the textured material. Ledges possess an effective Burgers vector, but in several ways these Burgers vectors are different from the Burgers vectors of lattice dislocations: [10, 13] the Burgers vector of a ledge is larger than the Burgers vector of a dislocation, and the magnitude of the Burgers vector of a ledge is sensitive to the misorientation of the adjacent grains that share the boundary. The density of the ledge structure will increase with an increase in the angle of misorientation between the two grains. Murr [13] states that grain-boundary ledges are responsible for emission of dislocations during the first stage of yielding; emission of glide dislocations from ledges have been directly observed previously.

The creation of a ledge has been discussed by Das and Marcinkowski [14]. They state that the plastic deformation of a polycrystal creates a heterogeneous shear of a grain boundary caused by the passage of crystal glide dislocations from one grain to another. Since the directions of the Burgers vectors do not coincide in the two grains, the passage of dislocations through the grain boundary creates the mismatch called a deformation ledge. The character of a ledge is determined by the type of dislocations that create it. Deformation ledges are associated with a large strain field that must be relieved; the crystal will relieve this strain field either by initiation of further crystallographic slip or by nucleation of a microcrack.

In crystallographic slip, new loops are generated in regions adjacent to the ledges. The negative half of the loops are pushed into the crystal while the positive half of the loops are pulled toward the ledge. Strain relief can also be accomplished by the climb of grain-boundary dislocations in the grain boundaries. Whether plastic flow in the form of crystallographic slip or microcrack initiation will relieve the stress field of a ledge is determined by which process will cause the greatest decrease in

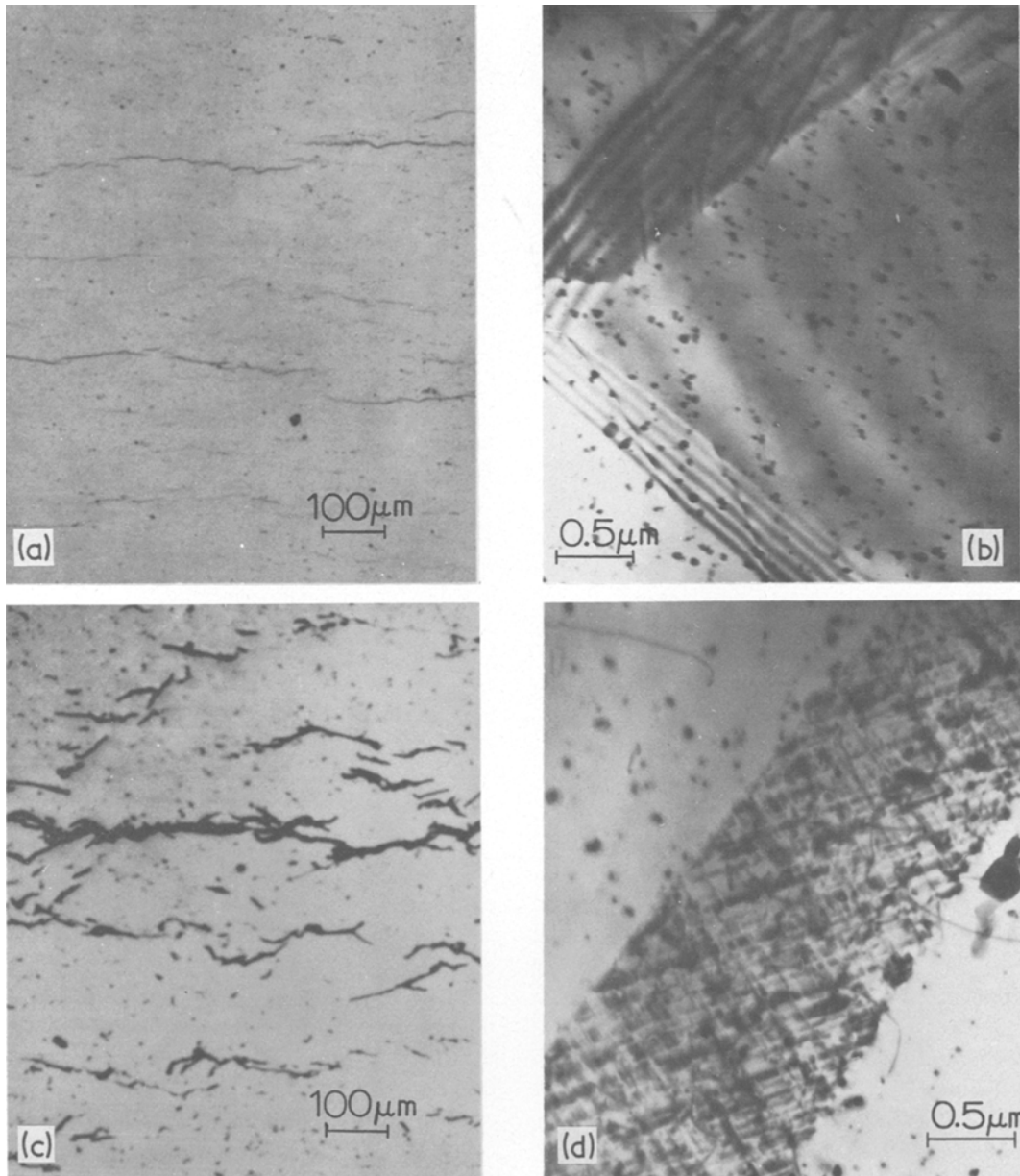


Figure 6 Comparison of spall crack morphologies in shock-loaded Be and typical, annealed, unshocked, grain-boundary ledge structure. (a) Optical micrograph of spall cracks in textured, annealed Be following shock loading to 9.2 kbar, (b) electron micrograph of grain-boundary ledges in textured, annealed Be, (c) optical micrograph of spall cracks in as-pressed, annealed Be following shock loading to 9.2 kbar, (d) electron micrograph of grain-boundary ledges in as-pressed, annealed Be. [(a) and (c) are courtesy A. L. Stevens, Sandia Labs].

free energy.

Crack initiation becomes favourable as the size of the ledge increases. The type of microcrack formed is determined by the type of ledge; different ledge characteristics will create different types of microcracks – intergranular or transgranular. The newly formed crack will grow until it reaches an equilibrium size that is determined by the mag-

nitude of the strain field being relieved. Thus, the larger the ledge, the longer the equilibrium length of the stable microcrack. These small, stable microcracks have the potential to grow larger upon application of an external stress.

Das and Marcinkowski expand their discussion of the formation of microcracks to include an equation to describe the relation between unlimi-

ted extension of the microcrack with an externally applied stress and the effective ledge length in a unit length of a boundary:

$$\sigma_{\text{crit}} = 2F_S/B$$

where σ_{crit} = stress at which the crack becomes unstable with respect to unlimited expansion, F_S = surface energy of the material, and B = the effective ledge length in a unit area of a grain boundary. Thus, as the density of ledges increases, the magnitude of the external stress required to extend the microcrack decreases.

The preceding discussion has attempted to identify the microstructural feature responsible for the difference in the spall crack formation of the as-pressed material with respect to the textured beryllium originally reported by Stevens and Pope. A dense deformation ledge structure exists in the as-pressed material while in comparison fewer ledges are seen in the grain boundaries of the textured material. Fig. 6 compares an optical micrograph of the spall damage introduced in the as-pressed material with an electron micrograph of the as-pressed, annealed material and an optical micrograph of the spall damage introduced in the textured material with an electron micrograph of the grain-boundary structure of the textured, annealed material. This comparison is in direct agreement with the above equation proposed by Das and Marcinkowski: for the same level of applied external stress, the crack formation in the as-pressed material was much more dense because the effective number of ledges in a unit area or length of boundary in this material was much greater than this value in the textured material. The critical crack propagation stress was lower than in the as-pressed material, thus accounting for the increase in the density of these cracks with respect to the density of cracks in the textured material. The ordering of the basal planes caused by the texturing probably accounts for the ordering of the spall cracks as suggested by Stevens and Pope.

5. Conclusions

In this study, the microstructural feature that is responsible for the difference in the spall damage inflicted on the as-pressed, polycrystalline beryllium with respect to textured, polycrystalline beryllium has been determined to be the deformation ledge structure that is dense in the as-pressed material while less frequent in the grain boundaries

of the textured material. The material with the higher crack density also possesses the higher ledge density, and this fact qualitatively supports the equation; $\sigma_{\text{crit}} = 2F_S/B$. It has been shown that the ledge structure is acting as a dislocation source in this material as proposed by various researchers.

The softening of beryllium with shock loading has been explained using a model based on the stress-activated glide of sub-boundaries with eventual fixation of these sub-boundaries as their angle of misorientation becomes greater. The as-pressed material is softer in the annealed condition with respect to the textured material both because of a less complicated dislocation structure with a lower density of dislocations, and because of a larger grain size.

It has been shown that the heat-treatment involving rolling reduction followed by annealing seems to affect the microstructure more than low-pressure, shock loading in the range from 0 to 9.2 kbar. The roll-reduced material possessed microstructural features identified with material that has undergone deformation accompanied by a high strain-rate, i.e. dislocation walls, hexagonal dislocation networks, complex dislocation arrays, and particle-dislocation interactions. In comparison, the as-pressed structure has a low dislocation density in the annealed state, but its grain boundaries generally possess a dense ledge structure. Shock loading increased the dislocation density, but this increase occurred with only an increase in the dislocation interactions and the size of dislocation features apparent. Features such as twins were not introduced as a consequence of the shock loading.

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