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METALLURGY AND CERAMICS

PRELIMINARY ATTEMPTS TO PRODUCE RANDOMLY ORIENTED WROUGHT BERYLLIUM AND THEIR RELATION TO TEXTURE DEVELOPMENT

By
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July 6, 1960

Nuclear Metals, Inc.
Concord, Massachusetts

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Nuclear Metals, Inc.
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Technical Director

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ABSTRACT

Several experiments were performed utilizing beryllium single crystals and polycrystalline material in an effort to produce randomly oriented wrought beryllium and to relate the deformation modes to texture development during plastic deformation.

It was determined that $(10\bar{1}2)$ type twinning appears to be one of the most predominant modes of deformation at temperatures as high as 1066°C (1950°F). This deformation mode is therefore instrumental in texture development. The prior texture of beryllium appears to affect the development of preferred orientations, especially at low reductions. The mechanical properties of compression rolled sheet approach those of upset sheet. The degree of preferred orientations developed by rolling beryllium at 1000°C is greater than that obtained by rolling at 450°C and annealing between each pass at temperatures of about 800°C. To date, that material which has most closely approached randomly oriented wrought beryllium exhibits a ductility of approximately 1%.

I. INTRODUCTION

Because of the metal beryllium's outstanding combination of physical, mechanical and nuclear properties (see Table I), there is much interest in its potential applications. The primary problem associated with the use of currently available beryllium is that of brittleness.

Tuer and Kaufmann[1] have shown that the lack of extensive slip on the (0001) basal planes* is one of the primary reasons for beryllium's lack of ductility. In single crystals of beryllium, up to 700°C, extensive slip in tensile elongation occurs on the $(10\bar{1}0)$ planes in the $[11\bar{2}0]$ directions. The main fracture planes at room temperature in a randomly oriented sample are the (0001) basal planes. The $(11\bar{2}0)$ prism planes are the planes of secondary fracture.

The reason for beryllium's lack of ductility has not been entirely explained, but there are basically three schools of thought regarding this "brittleness problem":

(1) Ductility can best be developed by the manipulation of crystallographic textures. The assumption here is that the metal is inherently brittle.

(2) Purification of the metal is necessary since the brittleness is believed to be due to impurities in solid solution. Yans, Donaldson and Kaufmann[2] have shown that some of the transition elements, especially iron, in solid solution have a definite detrimental effect on ductility, but the effects do not appear to be of the order of magnitude necessary to explain the very low ductility exhibited by beryllium. Oxygen is therefore suspected to be the most probable cause of solid solution embrittlement.

(3) The recently discovered β phase beryllium[3], (body-centered cubic structure, transition temperature approximately 1240-1270°C), which is potentially ductile, may be retained to room temperature.

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* Beryllium has a hexagonal close-packed crystal structure and $(c/a) = 1.568$.

Although the most economical and logical approach to the problem would be to prove or disprove the last two theories before starting work on the first, there is enough immediate interest in beryllium's potential to justify the simultaneous investigation and development of all three theories. This program has been primarily concerned with the first theory, i.e., the development of ductility by the manipulation of crystallographic textures in beryllium that is currently available.

A. Anisotropy of Beryllium with Special Emphasis on Sheet Products

When beryllium is mechanically worked, severe preferred orientation or crystallographic anisotropy is usually developed. Further because of the large degree of slip exhibited on the $(10\bar{1}0)$ planes and virtually negligible slip exhibited by the (0001) planes, this crystallographic anisotropy is translated into a severe anisotropy or directionality of mechanical properties, especially ductility. The type and degree of anisotropy (both crystallographic and mechanical) exhibited by beryllium sheet made by various fabrication techniques are discussed below.

1. Extruded and Transverse Rolled Sheet and/or Bi-directionally Rolled Sheet

By orienting the basal planes parallel to the plane of the sheet, Klein, Macres, Woodard and Greenspan [(4)] have obtained elongations in uniaxial tension of up to 40% in beryllium sheet rolled above $700^{\circ}C$. In this case, the main operative fracture planes are presumed to be the $(11\bar{2}0)$ planes. This "ductile" sheet was made by extruding powdered beryllium into a flat of rectangular cross-section and then rolling the flat transverse to the extrusion direction. Beryllium sheet with a very similar cyrstallographic texture can also be made by bi-directionally rolling a beryllium ingot. The crystallographic orientation of this type of sheet is described by means of pole figures in Fig. 1, and the same texture is illustrated schematically in Fig. 2a. Due to the high population of basal planes nearly parallel to the plane of the sheet*, this type of material has an almost complete lack of ductility perpendicular to the plane of the sheet, or in the "third dimension"[5]. Ductility in the thickness direction, or third dimensional ductility, is needed if the sheet is to be stressed in a complex fashion as in a structural assembly. If the sheet is to be strained in two directions simultaneously, or bi-axially, it must accommodate some strain in the third direction to preserve constant volume. In almost every practical case, sheet used in structural assemblies will be subjected to complex or bi-axial strain; some degree of third dimensional ductility is therefore an absolute necessity.

Further, the extruded- and transverse-rolled sheet and/or bi-directionally rolled sheet exhibits severe crack propagation on the highly oriented $(11\bar{2}0)$ planes[5]. This results in very anisotropic fracture characteristics and catastrophic failure. The third dimensional ductility of this type of material is approximately 0.1%. During the bi-directional rolling process, low rolling reductions can be used to reduce the degree of preferred orientation and thereby increase the third dimensional ductility[6], but when this is done the rolling process loses its economic attractiveness.

2. <u>Upset Sheet</u>

Beryllium sheet made by the upsetting process, i.e. by hot forging of a beryllium cylinder into a pancake or circular disc, has some degree of third dimensional ductility (2%) but slightly lower ductility in the plane of the sheet[5]. The ductility in the plane of the sheet in this type of material is normally around 10%, but has been reported to be as high as 20%. The increase in third dimensional ductility and decrease in the ductility parallel to the plane of the sheet is attributable to the relatively low basal plane population parallel to the

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* Beryllium exhibits almost no slip in a direction transverse to the (0001) planes.

plane of the sheet. The crystallographic texture of this type of sheet is portrayed by means of pole figures in Fig. 3, and shown schematically in Fig. 2b. As might be expected from the lack of preferred orientation in the prism planes with respect to any direction in the plane of the sheet, the $(11\bar{2}0)$ crack propagation problem is virtually eliminated and the material behaves isotropically in the plane of the sheet. But, the difference in ductility exhibited between the plane of the sheet and the thickness or third direction proves that some degree of anisotropy still exists. The material can be readily machined using normal techniques, however, and forming can be done to some extent.

In upset sheet, even though the degree of mechanical and crystallographic anisotropy relative to the cross-rolled or extruded and transverse rolled sheet is considerably reduced, the problem of directional mechanical properties still exists, and imposes difficulties on the aircraft designer, sheet metal fabricator, and nuclear engineer. Further, the process is not economically applicable to thin sheet production.

3. Randomly Oriented Beryllium

In the beryllium currently available, the anisotrophy of mechanical properties can be completely eliminated by producing a material with no crystallographic texture or preferred orientation. By definition, beryllium sheet with no preferred orientation must have a completely random crystallographic texture, as illustrated schematically in Fig. 2c.

Beryllium powder which has been merely hot compacted to 100% theoretical density, with no other hot work or plastic deformation, has a texture closely approaching random but does not exhibit much ductility (approximately 1 - 1-1/2%). A possible explanation of this phenomenon, other than the lack of preferred orientation, is that the bond between the powder particles or grains is not as perfect as that usually encountered in other metals and may be an area in which microcracks nucleate. In this particular case, an analogy may be drawn between beryllium and uranium powders [7]. Uranium powder may also be hot compacted to 100% theoretical density without plastic deformation and will exhibit almost zero plastic elongation in this state. This situation exists until the uranium compacts are mechanically wrought, at which point they become ductile.

From these considerations, it therefore becomes desirable not only to produce beryllium sheet with a random texture but also to hot work this material to such an extent that intimate contact between the powder particles or grains is achieved. The resulting material would then have completely isotropic mechanical properties, and hopefully, elongations in excess of those exhibited by hot pressed powder.

It should be noted that this randomly oriented wrought material should also have a fine grain size (20 - 40 microns) since past investigators have shown that the ductility varies inversely with grain size.

One of the first mechanisms that would normally be considered to achieve randomly oriented wrought beryllium would be the usually random grain nucleation which occurs when a metal is cycled through a phase transformation. The difficulty involved with this method is that the beryllium beta phase transformation occurs only 15 - 50^{o}C below the melting point, and when beryllium is heated to these temperatures severe grain growth occurs. The grain size has never been extensively refined by quenching from these temperatures. Further, normal mechanical working operations cannot be performed on specimens cycled through the phase transformation in order to reduce the grain size, because preferred orientations or textures will be developed.

B. The General Philosophy of the Investigation

With these facts in mind, and assuming that neither the retention of beta beryllium to room temperature nor the production of ductile beryllium by purification is in the immediate future, several experiments were performed to define the most promising approach toward producing randomly oriented wrought beryllium, with special emphasis on sheet material. The investigation had several aspects, including: the effects of prior preferred orientation on texture development; deformation and recovery characteristics of beryllium single crystals and polycrystalline material; and the effects of several fabrication variables on textures.

II. EXPERIMENTS, RESULTS AND DISCUSSIONS

In all experiments except those involving single crystals, the starting material was -200 mesh beryllium powder (Brush Beryllium Corp. Specification No. NP-100A). The specifications for this powder are given in Table II.

A. The Effects of Prior Preferred Orientation or Texture Memory on Extrusion Textures

If a previously worked specimen with a definite, preferred orientation could be subjected to a fabrication operation which would destroy its prior texture without developing a new one, a specimen with a completely random hot worked texture should result. To investigate this theory, discs of upset beryllium sheet (Fig. 4a) were extruded into rod. Normally, in upset sheet the basal planes are oriented primarily parallel to the plane of the sheet, and in an extruded rod, parallel to the extrusion direction in a fiber texture. In this experiment, it was hoped that during the extrusion of the upset discs a kind of transition texture would be achieved that was neither that of upset sheet nor extruded rod. Hopefully, this texture would be random. The process and its implications are illustrated schematically in Fig. 4.

Four extrusion billets were prepared by machining an upset sheet (reduction in area of 6:1 at 1850°F) into discs (0.920" dia. and 0.100 - 0.200" thick) which were then assembled into a billet clad in cold rolled (1020) steel. With the aid of conical dies, these billets were extruded at 1950°F to reductions in area of 2:1, 4:1, 6:1, and 8:1, respectively. All billets were evacuated and sealed at 1950°F before extrusion. All discs bonded to each other except those from the 2:1 reduction extrusion which rendered that rod mechanically untestable. Another attempt was made to obtain bonded discs with a 2:1 extrusion reduction by pressure welding the discs before extrusion in a vacuum at a temperature of 1650°F with an applied pressure of approximately 10,000 psi. This pressure welded assembly, subsequently extruded at a reduction of 2:1, still did not yield metallurgically bonded discs. The discs from the extrusions at higher reductions were metallurgically bonded, however, and the resulting rods were evaluated by means of the tensile test.* The ductility of the rod extruded at a reduction of 2:1, both parallel and perpendicular to the extrusion direction, was determined by means of bend tests in which Type A7-SR4 strain gauges were mounted on the tensile side of the bend specimen perpendicular to the bend axis. The tensile and bend test data obtained from these extrusions are shown in Tables III and IV, respectively.

A texture analysis of the upset and extruded rods is shown in Fig. 5. The (0001) or basal plane texture was evaluated by means of x-ray diffraction Norton rod scans [8]. The Norton rods were made by machining cylinders out of the extruded rods. The axes of these cyclinders were perpendicular to the extrusion direction and intersected the center of the extruded rod. The random level was calculated by the Harris technique[9] and is accurate within $\pm$ 5%. A (0001) trace for upset sheet is also shown in Fig. 5. This trace was also obtained by the Norton rod technique and the axis of the rod used was in the plane of the upset sheet. The random value used in this case was obtained experimentally since the Harris conditions of symmetry are not fulfilled.

As seen in Fig. 5, any reduction in excess of approximately 2:1 under the given extrusion conditions produces a severe, extrusion type texture, i.e. (0001) planes parallel to the extrusion direction. An extrusion reduction of 2:1 performed on beryllium sheet upset at a reduction of 6:1 at 1850°F produces cyclic intensity variations from 0.30 random to 1.75 random. It should be noted that the intensities obtained from hot pressed beryllium powder exhibit non-cyclic variations ranging from 0.75 to 1.45 random.

Although the upset material extruded at a reduction of 2:1 does not display any high degree of orientation, that preferred orientation

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* Prior to all mechanical testing, approximately 0.004 - 0.006 was removed from the specimens by etching in an aqueous solution of 10% H_2SO_4.

which it does possess appears to be quite discrete. This can be seen from the three intensity peaks appearing at zero degrees and $\pm 60^{o}$ to the extrusion direction. These peaks might suggest a higher degree of preferred orientation on a micro scale. Nevertheless, it can be said that the upset beryllium specimen extruded at a 2:1 reduction closely approaches randomly oriented wrought beryllium, at least on a macro scale. The primary difficulty is that this material's ductility does not exceed that observed in hot-pressed beryllium powder either parallel or transverse to the extrusion direction.

B. Some Deformation and Recovery Characteristics of Beryllium Single Crystals and Polycrystalline Material

Since the primary purpose of this investigation concerns texture-developing mechanisms and their kinetics, it was felt that a more basic understanding of the above phenomena would be instrumental in the production of randomly oriented wrought beryllium. In particular, the determination of the most predominant mode(s) of deformation in polycrystalline beryllium during the rolling process, and the effects of recovery and recrystallization on the orientations developed, would aid in predicting resulting rolling textures.

Several experiments were therefore performed on beryllium single crystals, and the existing literature was reconsidered. These experiments and their implications on the literature are presented below.

1. The Rolling and Annealing of Single Crystals

Several beryllium single crystals were prepared, in the manner outlined by Tuer and Kaufmann(1). The starting material for these crystals was lump beryllium, from which commercial beryllium powder is made by attritioning; its chemical composition is therefore within the limits presented in Table II. These crystals were clad in a thick mild steel (1020) jacket, rolled at 1000 - 1070^{o}C and then air cooled to room temperature. The orientation of these single crystals with respect to the rolling direction and plane is shown in Fig. 6, which also lists the total reduction in area used on each. A rolling temperature of

1000 - 1070°C (1832 - 1958°F) was chosen in an effort to duplicate the fabrication conditions used by previous investigators who have accumulated considerable data concerning Be textures.

a. As-rolled Crystals

After rolling, the steel clad was removed from the crystals by chemical etching, and the specimens were prepared, by mechanical polishing, for optical metallography and x-ray diffraction.

Figure 7 is a photograph of the (0001) face of specimen No. 1, where the twins present are of the $(10\bar{1}2)$ type. The twin indices were determined by optical metallography and x-ray diffraction techniques. As indicated by optical metallography, the traces of the twins on the (0001) face of the crystal were parallel to the $(10\bar{1}0)$ planes and on the $(10\bar{1}0)$ face parallel to the $(10\bar{1}2)$ planes. By means of x-ray diffraction, the angular misorientation between the (0001) planes in the matrix of the crystal and the twins was $85^{\circ} \pm 1^{\circ}$, which is in good agreement with the results of Tuer and Kaufmann (approximately 83°) [1]. In most metals, it is considered extremely unusual to observe twinning at a temperature (1000 - 1070°C) so close to the melting point of the metal (1283°C). Kaufmann and Tuer had observed the formation of twins in beryllium at temperatures as high as 800 - 850°C. It should be noted, however, that the orientation of Specimen No. 1 during rolling was ideal for this type of twinning.

The edges of the twin platelets are wavy, a configuration often associated with inclusions. The fact that the inclusions present in the beryllium single crystals do have an effect on the shape of the twins is shown in Fig. 8, which is an (0001) face of Specimen No. 1, prepared by electropolishing to emphasize the presence of inclusions.

The ends of the twins are often square or at an angle corresponding to a $(10\bar{1}0)$ trace. This effect may possibly be explained by the fact that the $(10\bar{1}0)$ plane is an active slip plane and stress relief by slip may therefore occur at the end of the twin, since it is $\sim 90^{\circ}$ to the $(10\bar{1}0)$ plane, thereby yielding an angular shaped end. Some twins also appear to have rounded and saber-shape ends.

In addition, on the sides of the twins there appear many small, thin twins (see Fig. 9), which seem to indicate that the process of twinning proceeds principally by means of the "starting up" of thin twins which are then absorbed by the major one. The thickening of the small individual twins appears to bring about a union of small twins forming one large twin. This information corroborates the results of Garber, Genden, Kogan, and Lazarev[10]. These small twins are not cracks or polishing effects, because they are optically anisotropic and remain so even after electropolishing.

No slip lines could be detected in any of the specimens, since the rolling temperature was considerably in excess of that needed for stress relief.

On the basal plane face of Specimen No. 1 many grains were found to exist at or very near the intersection of two or more twins. This indicates that the strains associated with twinning are reasonably high and twin intersections are preferential sites for the formation of recrystallization nuclei. Similar observations have been made with uranium. For example, Lloyd[11] was not able to obtain any recrystallized grains in uranium single crystals that had been deformed only by slip. Further, LaCombe[12] found grains, forming in uranium at (112) type twin intersections, of the same orientation as the (112) twins.

Some of the recrystallized grains occurring at twin intersections in the beryllium single crystals are shown in Fig. 10. It should be noted that, in most cases, the nucleated grain assumes the orientation of one of the intersecting twins; i.e., in polarized light, the nucleated grains appear to be of the same color as one of the intersecting twins regardless of the polarization angle. Also, when a grain nucleates at twin intersections, the grain appears to grow parallel to the twin which does not have the same orientation of the grain. This is also to be expected, since a higher energy is associated with a high angle boundary than with a low one. It should be noted that in the surface layers of the single crystals in contact with the iron clad, many grains were present with orientations that did not appear to be related to any of the twin orientations and were more or less randomly oriented. This was only a surface effect; when a few thousandths of an inch were polished off the surface of the specimen these grains were no longer visible.

In Samples Nos. 2 and 3, due to the different orientation with respect to Sample No. 1 during the rolling process, slightly different deformation modes were operative. For example, in Specimen No. 3, Laue back-reflection photographs indicated that only $(10\bar{1}0)$ slip had occurred. No twins were visible by means of optical metallography. The results obtained from Specimen No. 2 were more complex; $(10\bar{1}2)$ twins were evident by optical metallography as well as a second type of twin. The size and quantity of the second type was too small to allow exact determination of its orientation by x-ray diffraction techniques. On the basis of the crystallographic planes which the twin traces intersected, the second type of twin was of the $(11\bar{2}x)$ type and, as closely as the angular relationships could be determined, the last Miller index was 1. It should be noted that Garber et al. [10] reported only twinning systems of the $(10\bar{1}1)$, $(10\bar{1}2)$ and $(10\bar{1}3)$ type. Because of the difficulty in determining angular relationships of a twin trace in a deformed crystal, by optical metallography, further work should be done to determine accurately the existence of this second type of twinning, preferably by x-ray diffraction techniques.

Laue back-reflection techniques appeared to indicate that in Specimen No. 2 the two types of twinning previously discussed were also accompanied by $(10\bar{1}1)$ slip in the $[11\bar{2}3]$ direction. Garber et al. [10] also noted $(10\bar{1}1)$ slip but in the $[01\bar{1}0]$ direction. Since the type and direction of applied stress used by the above authors was different from that used in the present investigation, it is quite possible that the results from both investigations are not contradictory.

Tuer and Kaufmann have indicated that there is a "critical stress" needed for $(10\bar{1}2)$ twinning in beryllium, and this stress increases with increasing temperature. Since the critical shear stresses needed for slip decrease with increasing temperature, this would tend to indicate that at high temperatures the probability of twin formation drastically decreased. This is in contradiction with the results obtained by Garber et al[10], who indicate that the "critical stress" needed for $(10\bar{1}2)$ type twinning is insensitive to temperature changes (see Fig. 11). Since twinning was found to occur quite extensively at temperatures as high as 1070°C even in the presence of deformation by slip, as in Specimen No. 2, Garber's results appear to be more consistent with the current observations; that is, the "critical stress" needed for twinning, relative to that needed for slip, does not increase drastically with increasing temperature. Perhaps, as stated by Tuer and Kaufmann, the data they obtained can possibly by explained by the fact that Bell and Cahn[13] have reported no unique "critical stress" at room temperature at which twinning will occur on the $(10\bar{1}2)$ plane in zinc. The latter authors indicate that the stress at which twinning does occur was found to be a function of the mechanical perfection of the crystal; when small twin nuclei were not present initially, the stress for twinning was appreciably increased.

b. Annealing of the Rolled Single Crystals

After rolling, and metallographic examination, Specimen No. 1 was annealed in a vacuum at 750°C for two hours followed by air cooling. Metallographic examination of this heat-treated single crystal revealed that several unusual recovery and annealing mechanisms were operative, as follows:

1. Some of the original twins were absorbed by the matrix of the single crystal in what appears to be a diffusion-type process.

2. The recrystallized grains previously at the twin intersections sometimes were and sometimes were not absorbed by the single crystal matrix.

The first phenomenon, that is, the absorption of twins by the matrix material, is shown in Figs. 12 and 13. From these figures it can be seen that the twins are being absorbed by the matrix, and it should be noted that islands of the original matrix orientation begin to appear in the central portions of the twins. Further, in Fig. 12

the small grain at the intersecting twins has been partially absorbed by the matrix. Lloyd[11] observed very similar effects occurring in uranium single crystals. That is, the absorption of twins by the matrix occurred upon annealing. But, in the same specimens, Lloyd also observed the growth of twins.

The second phenomenon, the absorption of the recrystallized grains at the twin intersections, is illustrated in Fig. 12. However, these recrystallized grains are not always absorbed by the matrix during annealing, as evidenced by Fig. 14.

Some of the absorbed twins are slightly elevated above the matrix after mechanical polishing (see Figs. 13 and 14), indicating that the absorbed twin has a higher hardness than the original matrix material. The reason(s) for this increased hardness in the previously twinned region must be (1) incomplete stress relief in the absorbed twin, and/or (2) a composition gradient between the matrix and the absorbed twin.

2. The Case for Twinning as a Major Deformation Mode

Since extensive twinning occurred in the single crystals rolled at 1000 - 1070°C, it is reasonable to believe that twinning is a major mode of deformation at these temperatures and also at lower temperatures, as will be pointed out later in the text.

Careful consideration of the literature on preferred orientation in beryllium tends to confirm the above statement. For example, Greenspan[14] has done extensive work on determining the textures formed in beryllium sheet made by the process of rolling an extruded beryllium flat in a direction transverse to the extrusion direction. The change in the basal plane texture of this type of sheet as a function of the reduction ratio is shown in Fig. 15. It is obvious from these pole figures that the transition in texture from that of an extruded flat to that of the extruded transverse rolled sheet is not a gradual one, that is, the intensity peaks on the north and south poles in the case of the pole figure of the extruded flat (a) do not move gradually to a position 10°

or 20^{o} from the equator which is the texture of the rolled sheet (b) and (c). As can be seen from Fig. 15b (3:1 reduction), the grains appear to move discretely and, in this case, are either in the prior orientation of the extruded flat or in the new orientation indigenous to the transverse rolled sheet. The discrete movement of the grains can most readily be explained by a twinning mechanism. Also, the approximately 80 - 90^{o} rotation of the basal poles from the extruded flat to the transverse rolled sheet is consistent with $(10\bar{1}2)$ type twinning. One might speculate that in general the twinning mechanism is the primary mode of deformation for the case in question, but the general spread in the pole figures would appear to indicate that deformation by slip is also occurring, especially in the later stages of deformation. For example, the intensity peak in Fig. 15b occurs at about 20^{o} from the equator and, with higher reductions, moves to approximately 10^{o}, probably due to slip.

It is also obvious from the work of Greenspan that very similar effects are observed in the texture development of extruded and transverse rolled sheet regardless of rolling temperature. This would tend to indicate that twinning is a prevalent deformation mode at temperatures as low as $870^{o}C$ (which is in this case the lower temperature limit of Greenspan's work). Further, since it was pointed out in Fig. 7 that the "critical cleavage stress" for twinning does not change drastically with decreasing temperature, and since it is known from the work of Tuer and Kaufmann[1] that the critical shear stress for slip increases with decreasing temperature, it seems reasonable to assume that the twinning mechanism becomes even more predominant over slip at lower fabricating temperatures.

Additional evidence for twinning as one of the major deformation modes in beryllium can be obtained from the information contained in Fig. 5. The initial starting material for the "memory" experiment whose results are shown in Fig. 5 was upset beryllium sheet. The upset sheet has basal pole intensity peaks at approximately plus and minus 25^{o} to the normal to the plane of the sheet which is also the extrusion direction (see Fig. 5). If $(10\bar{1}2)$ type twinning is the major deformation mode after extruding this upset sheet as explained in Section IIa of the text, one would expect approximately an 85^{o} displacement of the basal pole peaks observed in the upset sheet. That is, at the lower extrusion reductions $(10\bar{1}2)$ type twinning should yield basal pole peaks at approximately minus and plus 60^{o} from the extrusion direction. This in fact does occur. It should be noted that in the case of the 2:1 reduction of a basal pole peak is also exhibited at approximately 0^{o} to the extrusion direction, probably because all the grains in the previous upset sheet orientation have not yet twinned, due to the low extrusion reduction, and a complete extrusion texture has not yet been developed. (The major basal pole peaks in the upset sheet occurred at approximately 25^{o} to the normal to the plane of the sheet, but there is a general spread in basal pole population from 0^{o} to 30^{o} to the normal to the plane of the sheet.)

At higher extrusion reductions (4:1, 6:1 and 8:1), the basal pole peak (at 60^{o} to the extrusion direction) found in the 2:1 reduction moves to a location of approximately 80^{o} to the extrusion direction. This 20^{o} displacement (from 60^{o} to 80^{o}) is probably caused by slip.

To further corroborate the above hypothesis, a rolling experiment was performed on the extruded flats used by Greenspan [(14)]. One of these extruded beryllium flats was cut at an angle of 30^{o} to the extrusion direction, canned in mild steel, and rolled transverse to the extrusion direction at a temperature of 1038^{o}C (1900^{o}F) to a total reduction of 5:1.

A schematic representation of the texture of the as-extruded flat used by Greenspan [(14)], and the aforementioned angularly machined flat, is shown in Fig. 16a and 16a'. It should be noted that the primary difference between the two is that in the as-extruded flat the $(10\bar{1}0)$ planes are 90^{o} and 30^{o} to the extrusion direction, and in the angularly machined flat the $(10\bar{1}0)$ planes are 0^{o} and 30^{o} to the extrusion direction.

(0001) and $(10\bar{1}0)$ pole figures of both the as-extruded and the machined flat before and after rolling are shown in Fig. 17. The pole figures of the as-extruded flat before and after rolling were taken from Greenspan[14]. The pole figures of the machined flat before rolling were constructed from Figs. 17a and 17b. Greenspan used a rolling reduction of 6:1, while the machined flat was subjected to a reduction of 5:1. It is assumed that this small differential in rolling reductions affected only the degree and not the type of texture developed.

The important thing to note in this experiment is that in both cases the texture changes can be explained almost completely by $(10\bar{1}2)$ type twinning. Further, due to the large spread in orientation, especially in the case of the machined flat, one must come to the conclusion that slip, although not the predominant mechanism of deformation, certainly accompanies twinning at these temperatures. The type of slip that occurs cannot be determined from the present data.

C. The Effects of Some Rolling and Annealing Variables on Preferred Orientation in Beryllium Sheet

1. The Preferred Orientation and Mechanical Properties of Compression Rolled Sheet

To determine how closely the properties of compression rolled* sheet approach those of upset sheet, a disc of steel-clad, hot-pressed QMV beryllium powder approximately 4" in diameter and 1" thick was compression rolled at a rolling temperature of 1010°C (1850°F) to a reduction of 7-1/2:1. The rolling was done at the Allegheny Ludlum Steel Corporation in Brackenridge, Pennsylvania. Following the rolling process, the steel clad was removed from the sheet by chemical etching, and the sheet was vacuum annealed at 750°C (1382°F) for one hour and furnace cooled. The pole figures describing the resulting preferred orientations

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* In the compression rolling process[15], [5], deformation by pure compression is simulated by means of rolling. That is, small reductions are taken at each rolling pass and the rolling is done from a large number of directions.

are shown in Fig. 18. Essentially, the type of texture achieved in the center of the sheet is the same as that at the edge. The primary difference between them is that the basal plane population parallel to the plane of the sheet is slightly higher on the edge than it is in the center. The uniaxial tensile properties of this sheet are given in Table V.

Bend tests were performed on specimens of this sheet with varying width/thickness ratios. The resulting data (deflection at fracture vs width/thickness) are plotted in Fig. 19. Also included are similar data obtained for upset sheet, hot-pressed sheet and extruded, transverse-rolled sheet[5]. These data indicate that the ductility of this sheet in the thickness or "third dimensional" direction is the order of 0.5%[5].

2. The Effects of Intermediate Annealing on the Preferred Orientation of Compression Rolled Sheet

Eighteen discs of hot pressed [1066°C (1950°F), 200,000 psi], -200 mesh QMV beryllium powder were rolled to varying reductions by means of compression rolling at 450°C (842°F). The specimens were clad in mild steel prior to rolling. Nine of the specimens were annealed between each rolling pass at a temperature of 850°C for approximately 15 minutes and the remaining nine specimens were annealed at approximately 700°C between each pass. The complete rolling schedule for all 18 specimens is given in Table VI.

After removal of the mild steel clad by chemical etching, (0001) basal plane pole figures were made of each of the eighteen sheets. The maximum basal pole intensity observed on the pole figure was plotted at the respective rolling reduction (see Fig. 20). These data are shown in comparison with the maximum intensity developed during compression rolling or upsetting if no intermediate anneals are used and the rolling temperature is on the order of 1000°C. The latter data were obtained from Yans and Kaufmann[16]. It should be noted that the degree of preferred orientation in the sheet rolled at 450°C and subjected to intermediate anneals is lower than that obtained in the sheet produced by compression rolling or upsetting at 1000°C with no intermediate anneals. There does not appear to be any difference in preferred orientation between the sheet annealed at 850°C and the sheet annealed at 700°C. Metallographic specimens were taken from a few sheets subjected to intermediate anneals at 850°C and 700°C respectively. It was difficult to tell from the metallographic specimens whether or not recrystallization had occurred in any of the specimens, not only because of the low magnifications usable with polarized light, but also the relatively "dirty" microstructure of the powder beryllium product. The presence of mechanical twins was not detected, indicating that the sheets were certainly stress relieved.

To explain this decrease in preferred orientation accompanying the intermediate annealed specimens, further quantitative work must be done in determining the recrystallization textures of polycrystalline beryllium. From the single crystal experiments involving annealing, one can only infer that the mechanism causing this effect may involve not only recrystallization but twin absorption. For example, the starting material in this case was randomly oriented hot pressed beryllium block deformed by rolling. This block may have been deformed primarily by twinning, and during the annealing process these twins may have been absorbed by the matrix, thus resulting in a net deformation not accompanied by an intensive development of preferred orientation. Although this hypothesis certainly appears conceivable, the effect of recrystallization nuclei and their subsequent growth must be considered. In summary, the mechanism by which this effect occurs is rather nebulous at the moment and further investigation is needed.

III. CONCLUSIONS

1. Twinning, especially type $(10\bar{1}2)$, appears to be one of the most predominant modes of deformation in beryllium, if not the most predominant, and has a large effect on texture development.

2. Twinning in beryllium occurs at temperatures as high as 1070°C (1958°F).

3. The prior texture of beryllium appears to affect the development of preferred orientations, especially at low reduction ratios.

4. The mechanical properties of compression rolled sheet (reduction ratio 7-1/2:1) approach those of upset sheet; its third dimensional ductility is approximately 0.5%.

5. The degree of preferred orientation developed by rolling beryllium at 1000°C is greater than that obtained by rolling at 450°C and annealing between each pass at temperatures of about 800°C.

6. To date, that material which has most closely approached randomly oriented wrought beryllium (upset 6:1; extruded 2:1) exhibits a ductility of approximately 1%.

IV. TABLES AND FIGURES

Table I

Some Physical, Mechanical and Nuclear Properties of Beryllium (at room temperature)

Property	Value
Density (lbs./cu. in.)	0.0658
Melting Point (°F; °C)	2340; 1283
Heat Capacity (C.G.S. units)	0.50
Thermal Conductivity (C.G.S. units)	0.35
Electrical Resistivity (% I.A.C.S.)	40
Modulus of Elasticity (p.s.i.)	40×10^6
Yield Strength (p.s.i.)	35-70,000
Tensile Strength (p.s.i.)	60-90,000
Thermal Neutron Absorption Cross Section (barns/atom)	0.0090
Thermal Neutron Scattering Cross Section (barns/atom)	7.0
Slowing Down Length (cm) (fission energy to thermal energy)	9.9

Table II

USAEC Nuclear Grade Chemical Specification
for -200 Mesh Beryllium Powder

Element	Weight %
Be (assay)	98.5*
BeO	1.2
Al	0.14
Ag	0.0005
B	0.0002
Cd	0.0002
Ca	0.02
C	0.12
Cr	0.03
Co	0.0005
Cu	0.015
Fe	0.16
Pb	0.002
Li	0.0003
Mg	0.06
Mn	0.015
Mo	0.002
Ni	0.04
N	0.05
Si	0.10
Zn	0.02

* Minimum specification. All other specifications maximum.

Table III

The Uni-axial Tensile Properties of Extruded Beryllium Rod
Made from Upset Beryllium Sheet (6:1; 1850°F).
The Tensile Direction Is Parallel to the Extrusion Direction

Property*	Extrusion Reduction					
				8:1**		
	2:1	4:1	6:1	a	b	c
Yield strength (0.05%)	unable to test because of poor bonding	50,600	55,870	--	--	32,500
Engineering tensile strength (p.s.i.)		61,750	64,600	78,900	73,750	64,200
True tensile strength (p.s.i.)		63,970	67,700	78,900	73,750	67,050
% reduction in area		3.50	4.60	0	0	4.16

Note: Textures of these rods are shown in Fig. 5.

* The values presented are averages obtained from 2 or 3 specimens.

** Because of the wide variation in results among the 3 samples tested from this reduction ratio, all the data are presented.

Table IV

Uni-Axial Ductility of the Most Random Extruded Upset Sheet

As Determined by Means of Bend Testing*

The extrusion reduction is 2:1.

Item	% Elongation	
	Parallel to extrusion direction	Perpendicular to extrusion direction
Total strain gage reading (% elongation)	1.08	0.84
Elastic portion of stress strain curve	~ - .10	~ - .10
Total plastic elongation	0.98	0.74

* Strain gage placed on tension side of 1/2" long bend specimens 1/8" wide and 0.040" thick.

Table V

The Average Uni-Axial Tensile Properties of Beryllium Sheet

Made by Compression Rolling of Hot Pressed Block

Rolling Temperature: 1010°C (1850°F)
Reduction in Area: 7.5:1
Annealed at 750°C (1382°F) for one hour and furnace cooled

Property	Value
Modulus of elasticity (p.s.i.)	38.5×10^6
Yield strength (0.05% offset)	50,250
Engineering tensile strength (p.s.i.)	77,700
True tensile strength (p.s.i.)	89,900
% reduction in area	17

Table VI

Rolling and Annealing Schedule Used on the Eighteen Specimens Subjected to Compression Rolling and Intermediate Anneals

The Rolling Temperature was 450°. Specimens annealed for 15 minutes at temperature for last pass.

Pass No.	Thickness Initial (in.)	Thickness Final	700°C Annealing Temperature Approx. Time in Furnace (min.)	700°C Annealing Temperature Approx. Time to Cool to 450°C (min.)	850°C Annealing Temperature Approx. Time in Furnace (min.)	850°C Annealing Temperature Approx. Time to Cool to 450°C (min.)	Number of Specimens
1	.800	.750	25	18	30	15	9
2	.750	.706	60	15	28	32	9
3	.706	.662	29	15	26	77	9
4	.662	.618	31	15	29	15	9
5	.618	.574	31	17	27	14	9
6	.574	.530	30	15	27	15	9
7	.530	.498	22	30	35	19	8
8	.498	.466	27	13	20	32	8
9	.466	.433	22	12	30	14	8
10	.433	.400	27	12	27	15	8
11	.400	.380	23	12	24	46	7
12	.380	.360	23	11	25	12	7
13	.360	.340	26	10	23	30	7
14	.340	.320	25	25	21	11	7
15	.320	.307	25	95	23	13	6
16	.307	.294	25	23	25	11	6
17	.294	.281	25	12	31	10	6
18	.281	.266	28	90	30	10	6
19	.266	.257	27	10	23	10	5
20	.257	.248	25	10	22	10	5
21	.248	.239	23	38	25	15	5
22	.239	.228	27	10	24	49	5
23	.228	.221	25	10	22	16	4
24	.221	.214	23	10	27	10	4
25	.214	.207	23	10	25	10	4
26	.207	.200	23	10	24	10	4
27	.200	.194	23	18	25	85	3
28	.194	.188	30	15	22	18	3
29	.188	.182	25	95	27	13	3
30	.182	.177	28	10	25	10	3
31	.177	.173	26	10	24	13	2
32	.173	.169	23	85	23	103	2
33	.169	.164	23	10	27	10	2
34	.164	.160	22	10	23	10	2
35	.160	.156	22	10	20	10	1
36	.156	.152	22	10	19	11	1
37	.152	.148	22	09	21	09	1
38	.148	.145	21	10	17	10	1

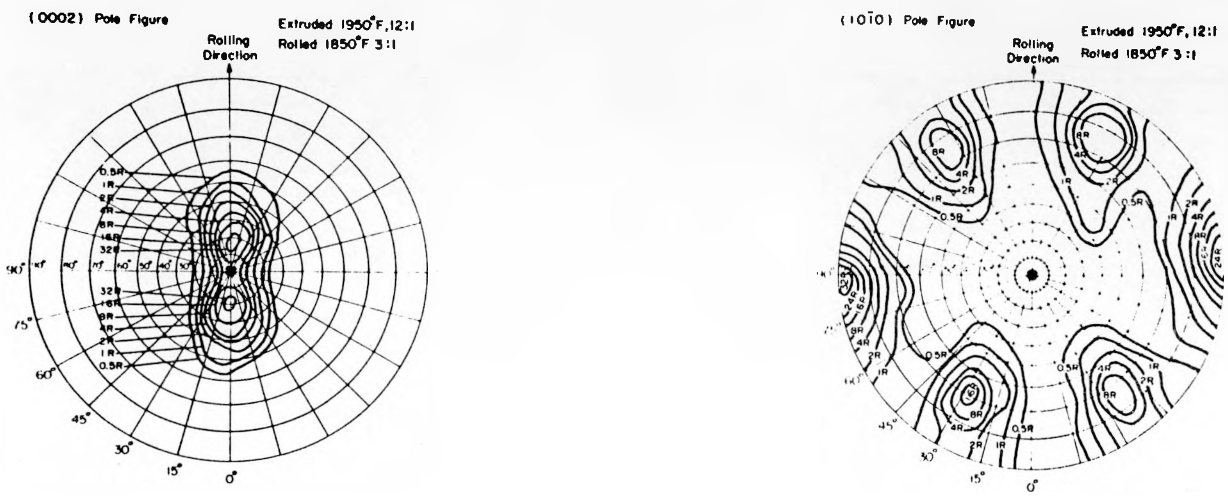


Fig. 1. Basal and prism plane pole figures for extruded and transverse rolled beryllium sheet.[5] RF-6149(a) and RF-6148(a).

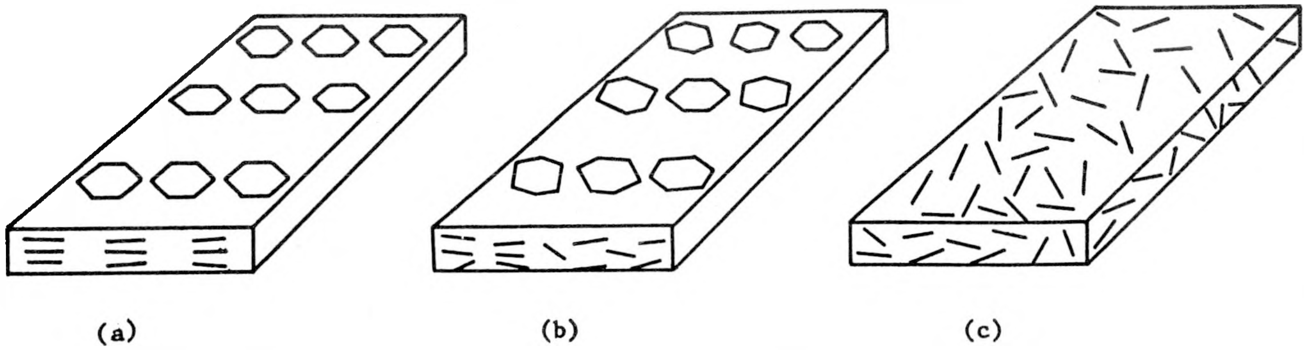


Fig. 2 (a, b and c)*. Pictorial representation of the textures obtained by: (a) extrusion and transverse rolling and/or bi-directional rolling; (b) upsetting; (c) a hypothetical fabrication technique yielding randomly oriented sheet.

Note that in 2a there is 0% elongation transverse to the plane of the sheet accompanied by severe $(11\bar{2}0)$ crack propagation in the textured prism planes. In 2b there is approximately 2% elongation transverse to the plane of the sheet. This is also accompanied by isotropic crack behavior. In 2c all properties are completely non-directional because of the random texture. Drawing No. RA-1402.

* The lines shown on front faces of a and b and all faces of c are basal plane traces.

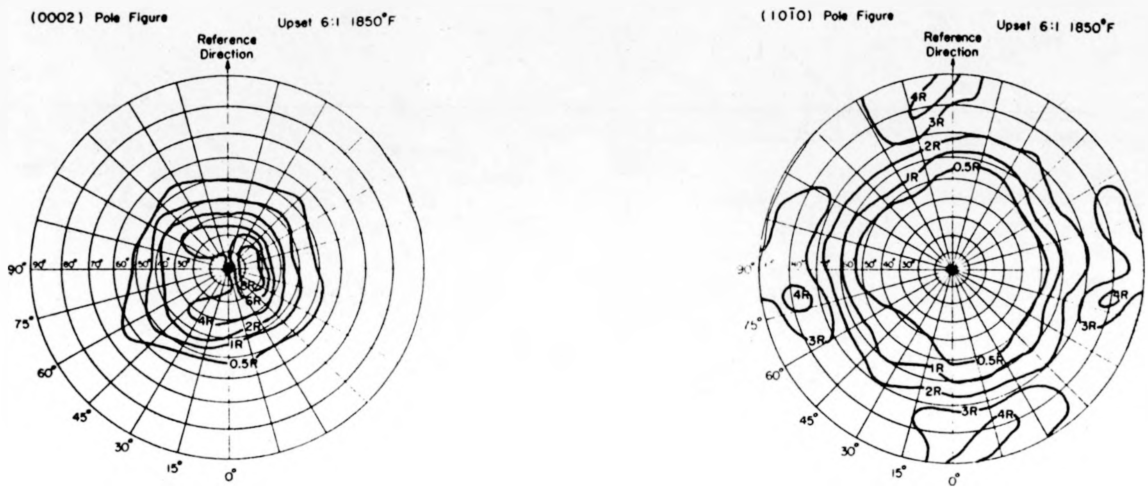
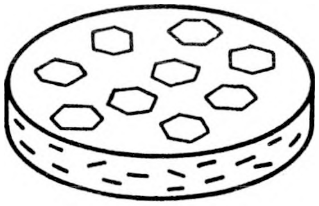
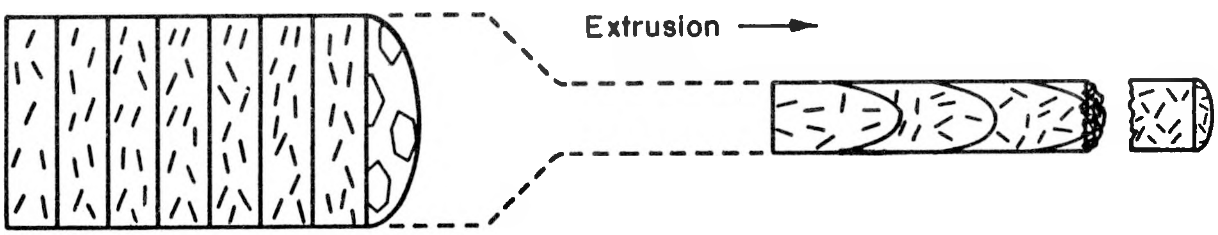


Fig. 3. Basal and prism plane pole figures for upset sheet. RF-6149(b) and RF-6148(b).



(a)
Schematic Illustration of the texture of an upset disc starting from hot pressed powder. The edges of the disc contain (0001) basal plane traces.

(b)
Schematic illustration of the texture of an extruded rod starting from hot pressed powder. Lines shown are (0001) basal plane traces.



(c)
Cross section of an extrusion billet made up of upset discs.

(d)
Cross section of billet shown in (c) after extrusion. Parabolic lines indicate the new positions of the previously flat interfaces between the upset discs. Note the "random" texture. Lines shown are (0001) basal plane traces.

Fig. 4. Utilization of the "memory" effect to achieve a random texture. (a) and (b) texture of upset sheet and extruded rod respectively not subjected to any prior work. (c) and (d) - the utilization of a prior texture to effect a change in the texture of an extruded rod. Drawing No. RA-1283

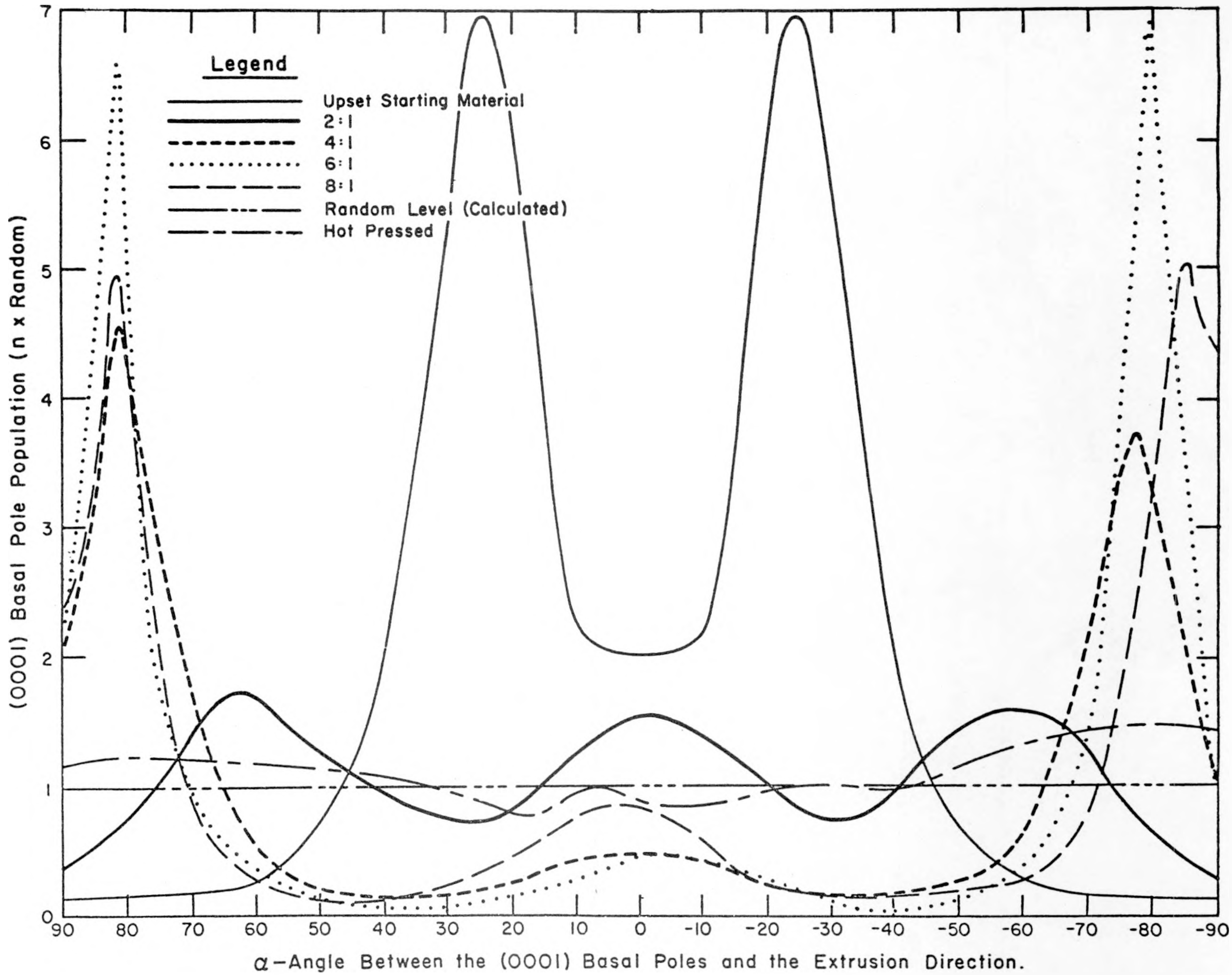
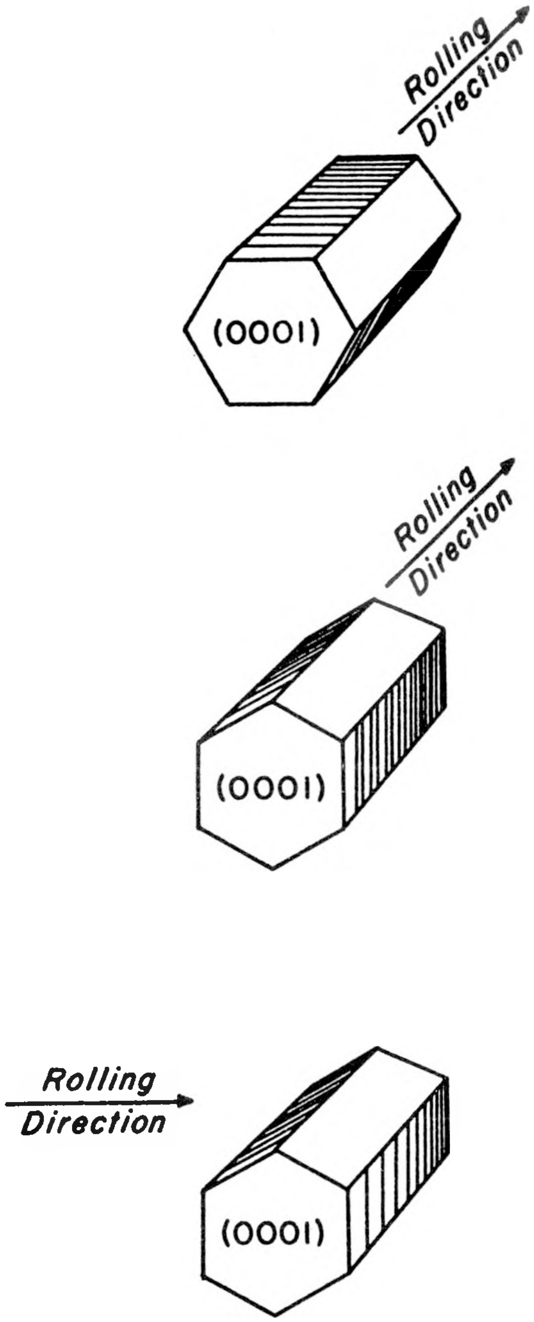


Fig. 5. The effect of extrusion reduction on the (0001) basal plane orientation of previously upset (6:1, 1850°F) beryllium sheet. Norton Rod Techniques used. Drawing No. RA-1282.

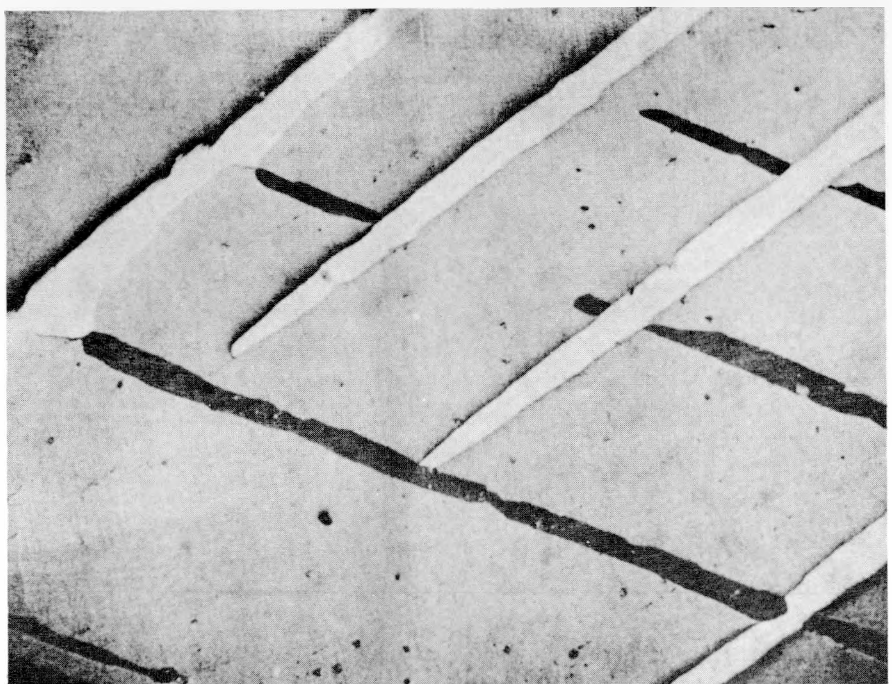


Specimen No. 1
Reduction in area = 4% ± 1/2%

Specimen No. 2
Reduction in area = 13% ± 2%

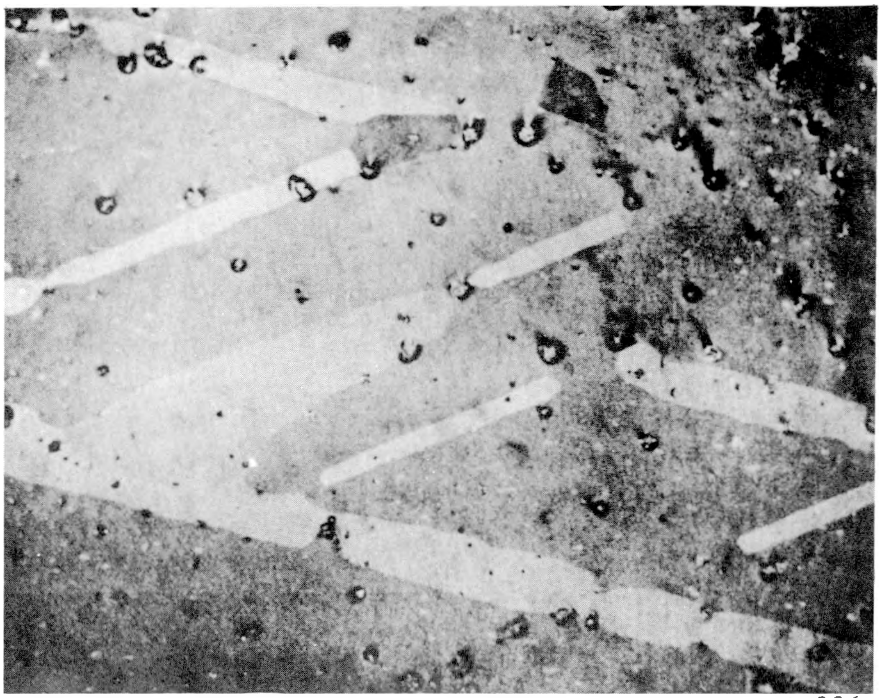
Specimen No. 3
Reduction in area = 20% ± 2.5%

Fig. 6. The orientation during rolling of the three crystals used. In all cases, the rolling plane is perpendicular to the (0001) plane, i.e., horizontal. The arrows indicate the rolling direction. Fabrication temperature, 1000 - 1070°C (1832 - 1958°F).
Drawing RA-1403



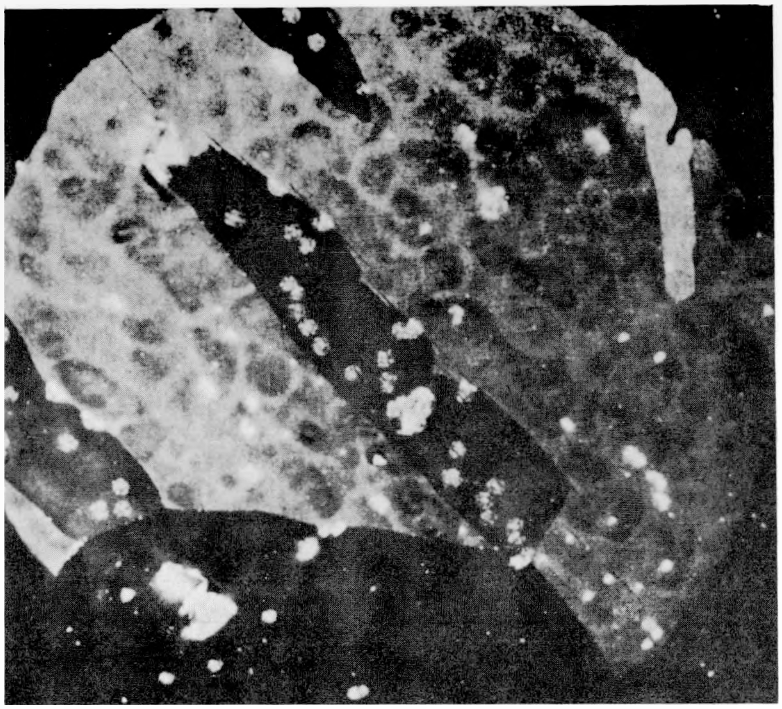
B-306-j

Fig. 7. The (0001) face of single crystal No. 1. $(10\bar{1}2)$ type twins are shown. Rolled at 1000-1070°C to a reduction of approximately 4% and then quickly air cooled. See Fig. 6 for the rolling orientation. 50X, polarized light, as-polished surface.



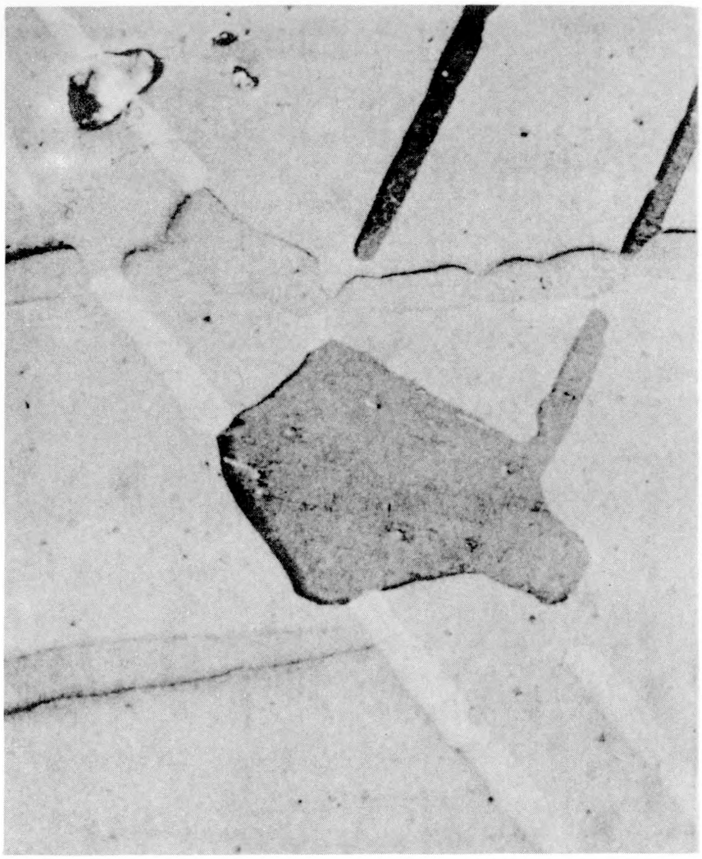
B-306-d

Fig. 8. The $(000\underline{1})$ face of single crystal No. 1. Type $(10\bar{1}2)$ twins are shown. Note the manner in which the inclusions affect the twin surfaces. 50X, polarized light, electropolished surface.

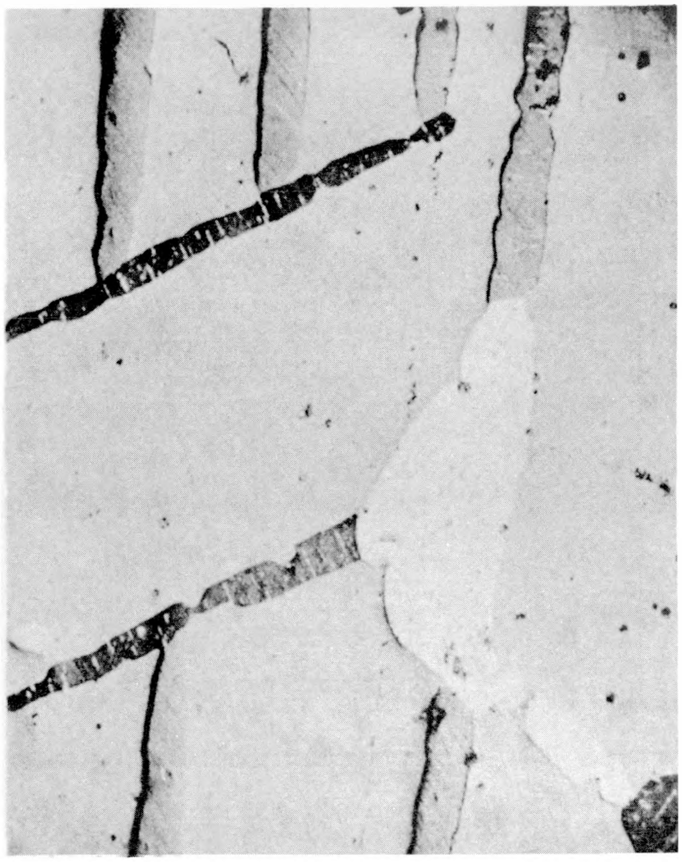


B-306-e

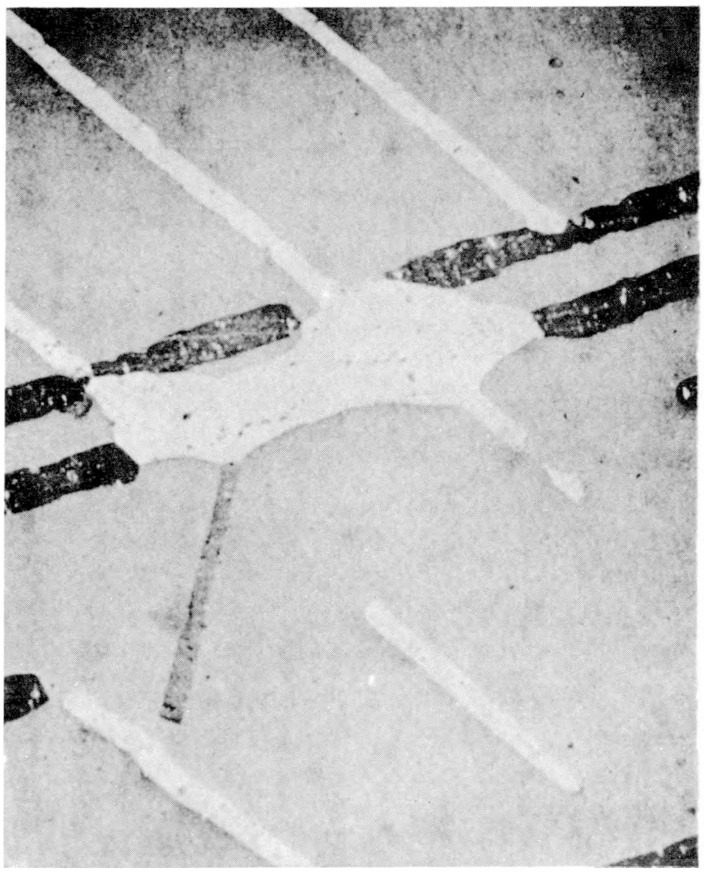
Fig. 9. The $(10\bar{1}0)$ face of single crystal No. 1. $(10\bar{1}2)$ type twins are shown. Note the small incipient twins surrounding the major twin. Some of these small twins have been absorbed. 150X, polarized light, electropolished surface.



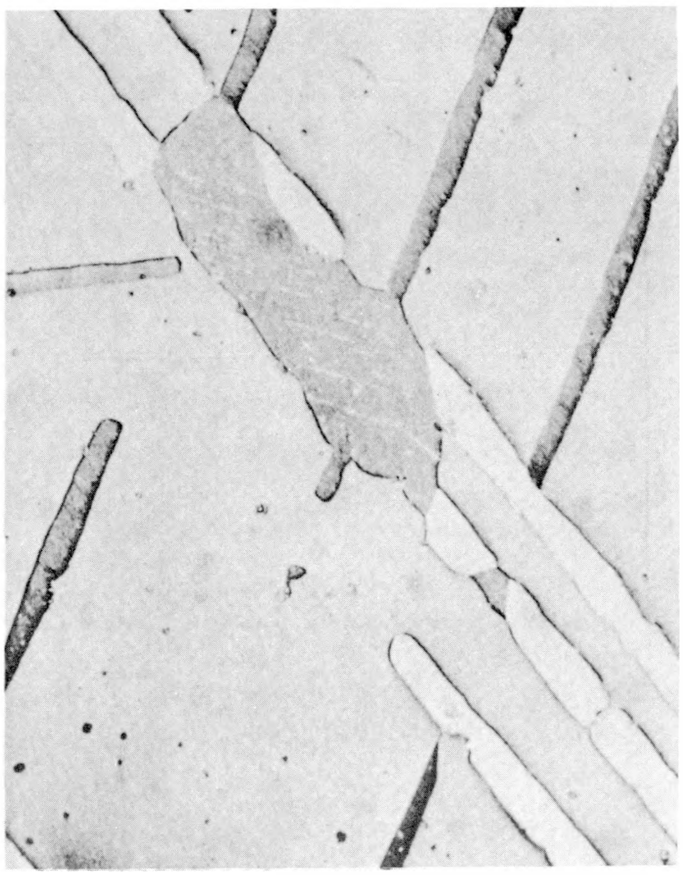
Photomicrograph B-306-F1



Photomicrograph B-306-h



Photomicrograph B-306-a



Photomicrograph B-306-i

g. 10. The (0001) face of single crystal No. 1. Note the formation of grains at twin intersections. 50X, polarized light, as-polished surface.

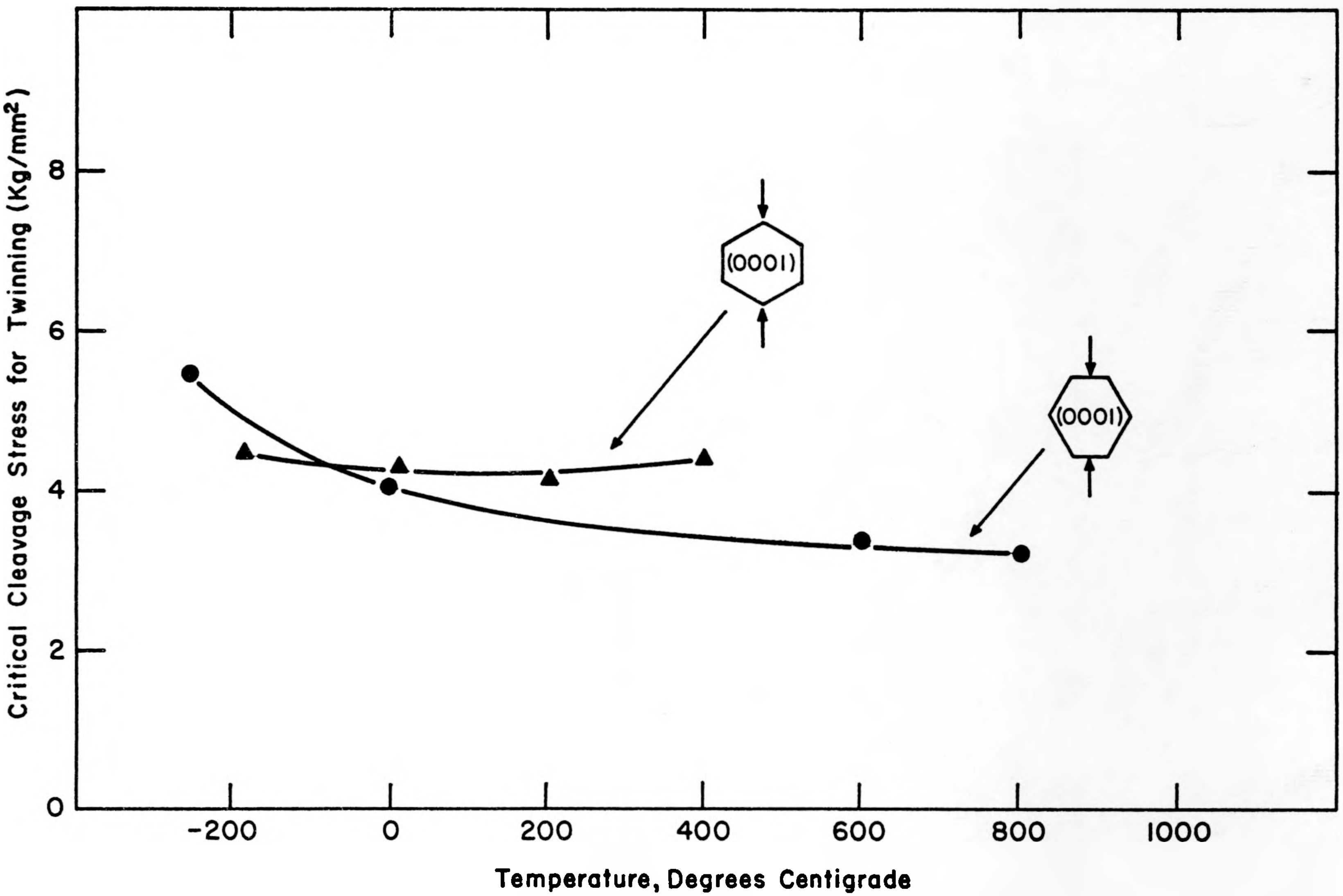
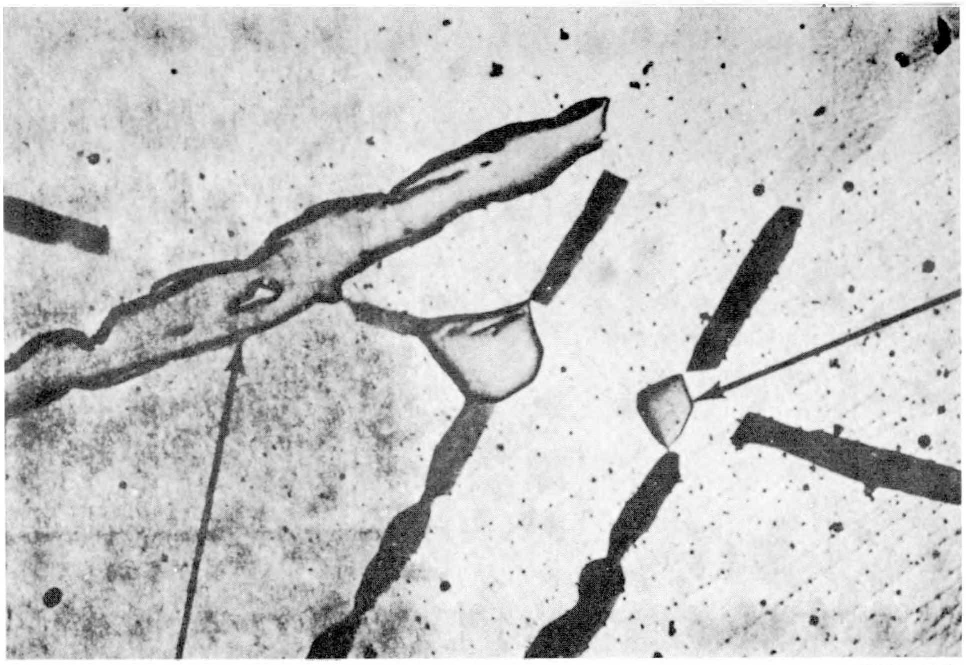


Fig. 11. The temperature dependency of the critical cleavage stress for twinning of beryllium on the $(10\bar{1}2)$ plane. From Garber, Genden, Kogan and Lazarev (Ref. No. 10). Drawing RA 1404.



Partially absorbed small grain at intersecting twins

TF5-31

(10$\bar{1}$2) twin being absorbed by the matrix

Note island in twin of original matrix orientation.

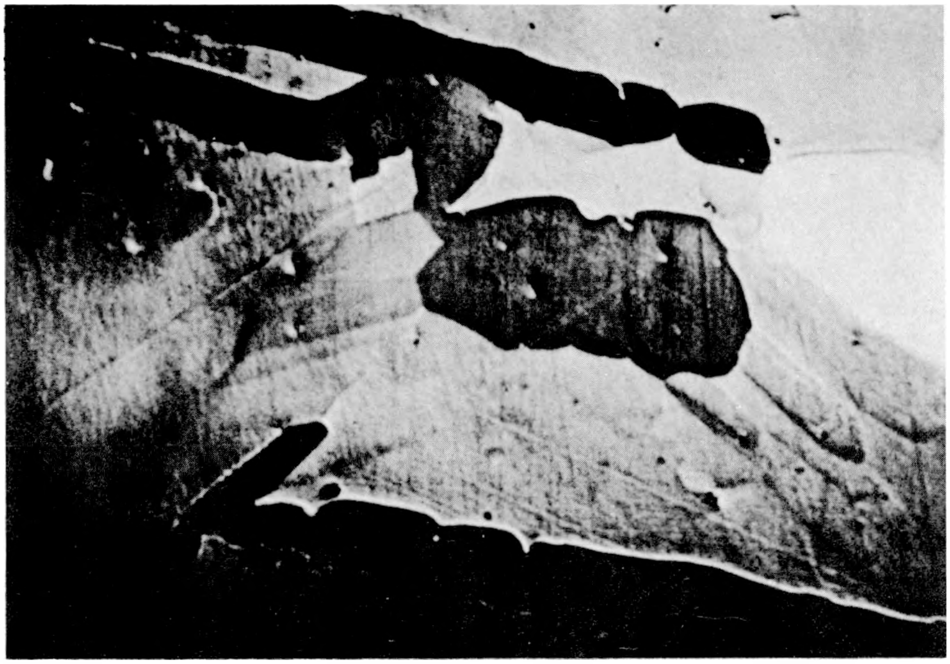
Fig. 12 - The (10$\bar{1}$0) face of specimen No. 1 after heat treatment at 750°C for 2 hr in vacuum. Note that the twins are beginning to be absorbed by the matrix and assume its orientation. The edges of the twins are irregular and islands of the matrix orientation are forming within the twin itself. Also note that some of the small grains formed at twin intersections are beginning to be absorbed. 60X, polarized light, mechanically polished surface.

Completely ab-
sorbed twin

Partially ab-
sorbed twin

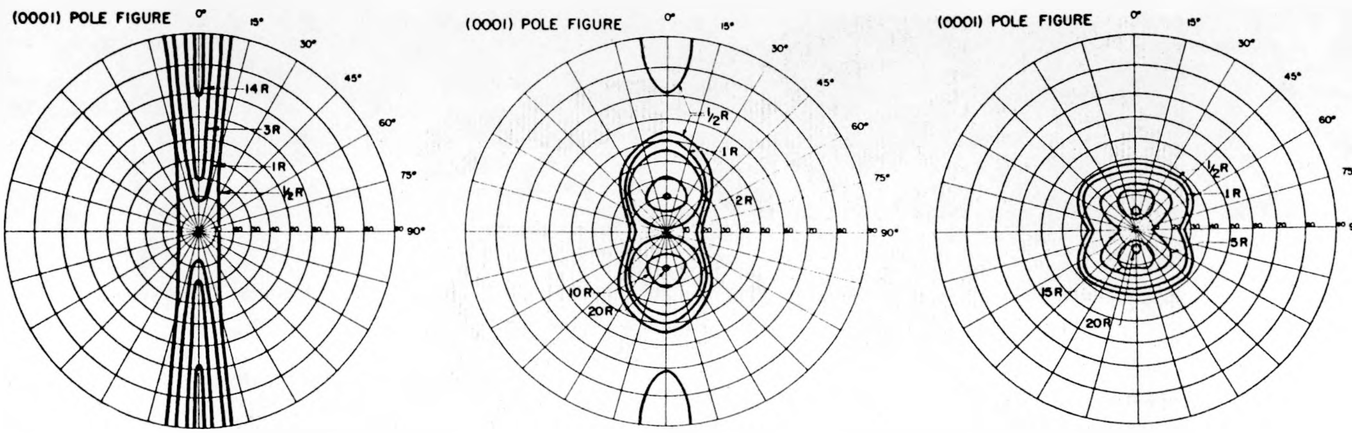
TF1-18

Fig. 13 - The $(10\bar{1}0)$ face of specimen No. 1 after heat treatment at 750°C for 2 hr in vacuum. Note that the completely absorbed twin is slightly elevated above the matrix material, indicating a variation in hardness between the matrix and the absorbed twin. 60X, oblique polarized light, mechanically polished surface.



TF1-16

Fig. 14. The (0001) face of specimen No. 1 after heat treatment. Note that small recrystallized grains still remain, but the twins that previously surrounded them have been absorbed by the matrix. 60X oblique polarized light, mechanically polished surface.



(a)
as extruded

(b)
rolled 3:1

(c)
rolled 6:1

Fig. 15. (0001) pole figures for (a) a beryllium flat extruded at 1038°C with a reduction of 18:1; extrusion direction, 90°. (b) the same extruded flat shown in (a) but after being reduced by rolling at 1038°C at a reduction of 3:1. (c) the same as (b) but with a reduction of 6:1. The rolling direction in (b) and (c) is 0°. (From Greenspan, Ref. 14). RF 1923, RF 1927, RF 1929.

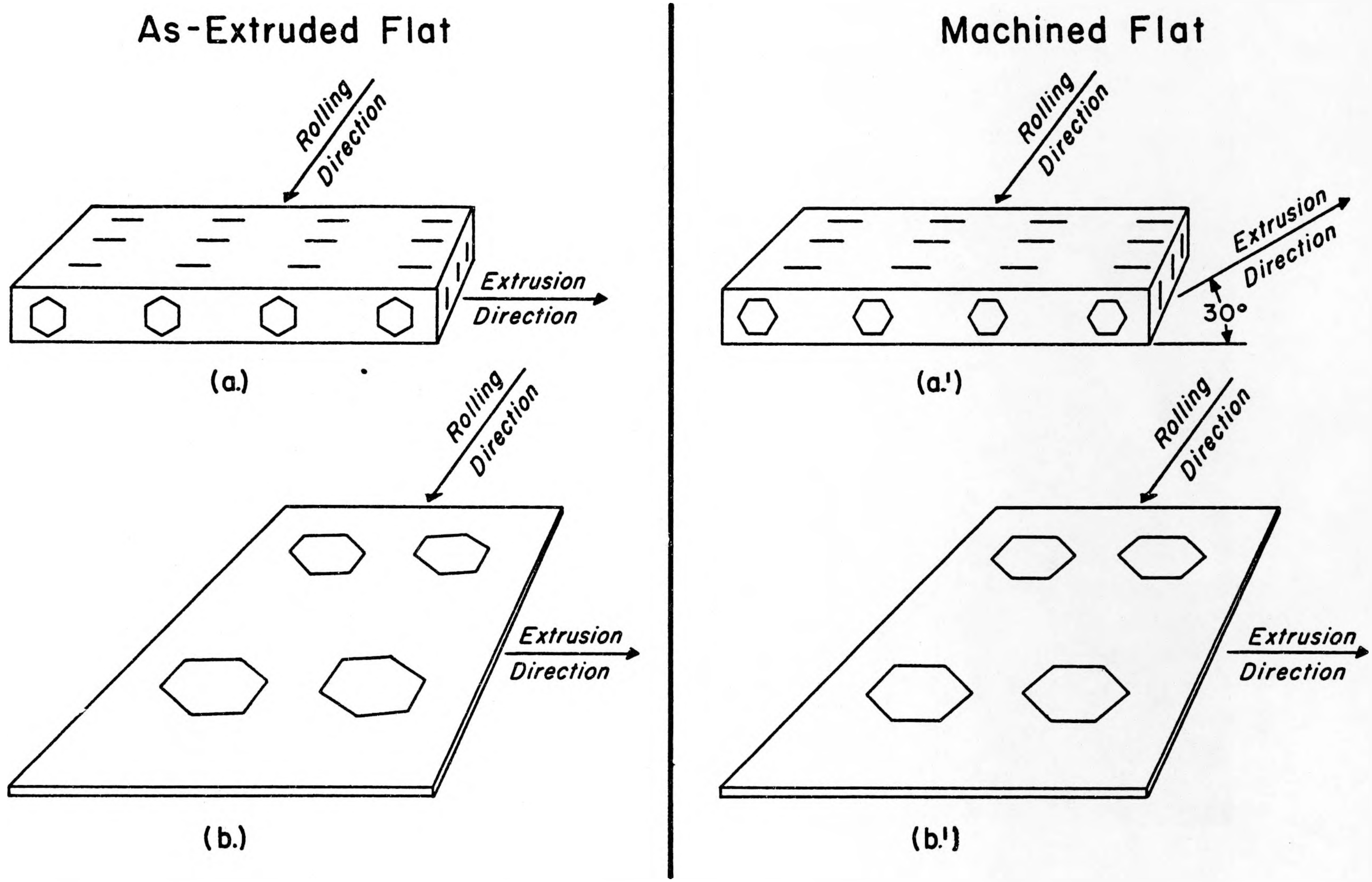
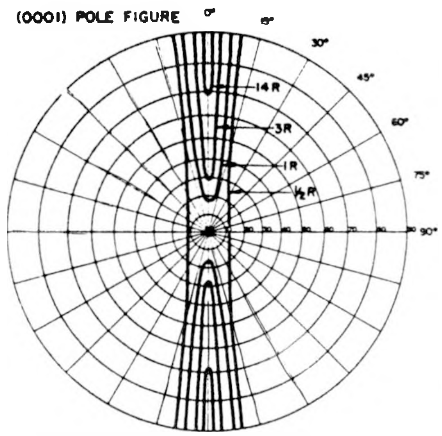
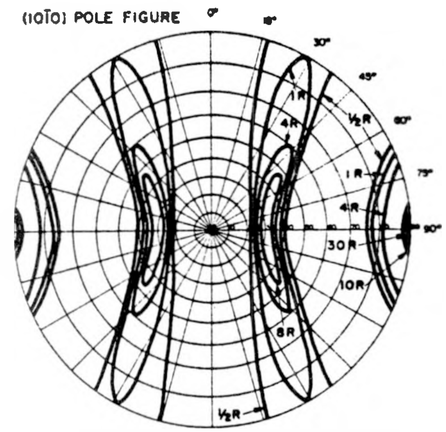


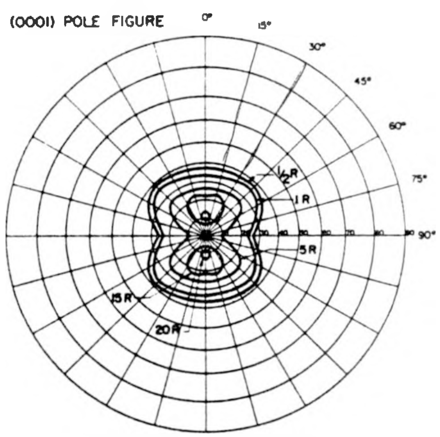
Fig. 16. Schematic representation of the textures resulting in (a) the as-extruded flat, and (a') the angularly machined flat before and after rolling. All lines except those forming hexagons are traces of the (0001) basal planes. Drawing RA 1405



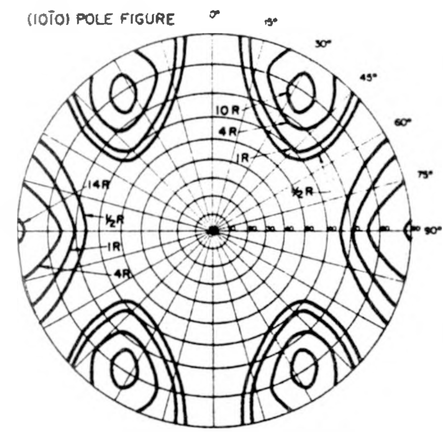
(a) (0001) pole figure of an as-extruded flat, 1038°C 18:1. RF 1923



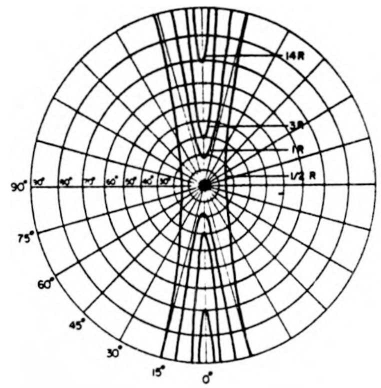
(b) (10$\bar{1}$0) pole figure of an as-extruded flat, 1038°C 18:1. RF 1924



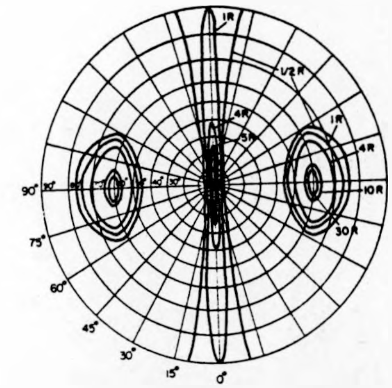
(c) (0001) pole figure after rolling an as-extruded flat, 1038°C, 6:1. RF 1929



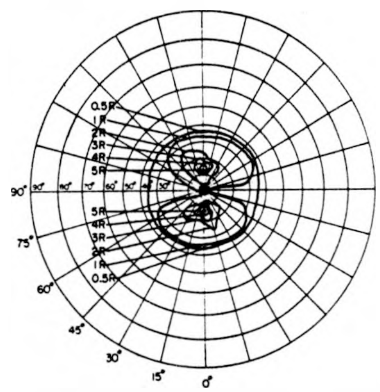
(d) (10$\bar{1}$0) pole figure after rolling an as-extruded flat, 1038°C, 6:1. RF 1930



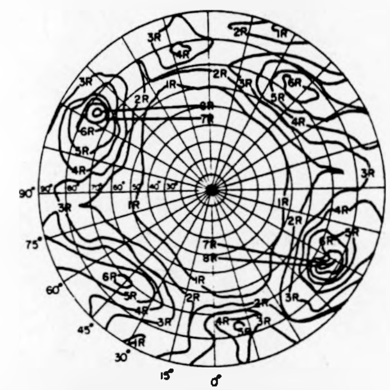
(a') (0001) pole figure of the angularly machined extruded flat, 1038°C, 18:1. RF7306



(b') ($10\overline{1}0$) pole figure of the angularly machined extruded flat, 1038°C, 18:1. RF 7309

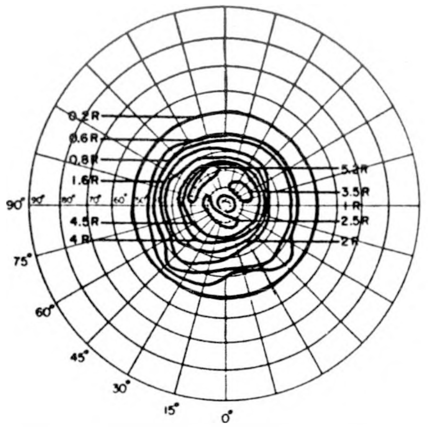


(c') (0001) pole figure of the angularly machined extruded flat, after rolling at 1038°C, 5:1. RF 7308

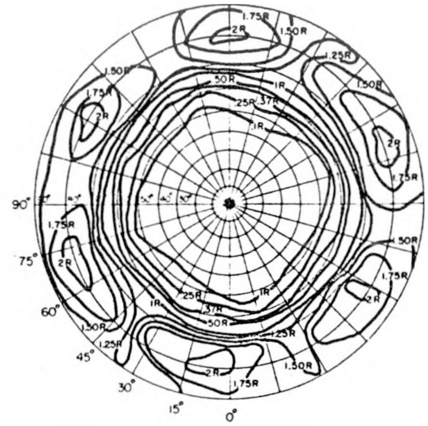


(d') ($10\overline{1}0$) pole figure of the angularly machined extruded flat, after rolling at 1038°C, 5:1. RF 7307

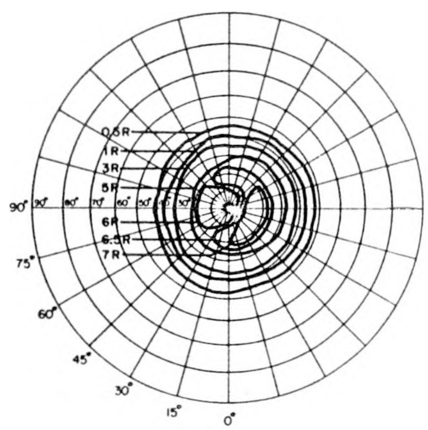
Fig. 17. Basal [(0001)] and prism [($10\overline{1}0$)] plane pole figures representing the textures obtained by rolling an as-extruded flat (a, b, c, d) and a angularly machined flat (a', b', c', d'). The rolling direction is 0° and the extrusion direction is 90°. In pole figures a, a', b and b' the direction parallel to the width dimension of the extruded flat is the vertical diameter of the pole figure. Figures a' and b' were constructed from a and b.



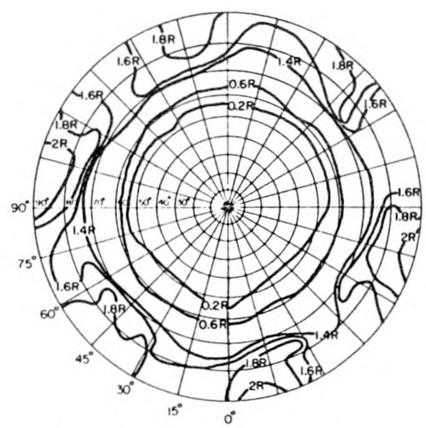
(a) (0001) pole figure.
RF 7303



(b) $(10\bar{1}0)$ pole figure.
RF 7304



(c) (0001) pole figure.
RF 7305



(d) $(10\bar{1}0)$ pole figure.
RF 7302

Fig. 18. (0001) and $(10\bar{1}0)$ textures of compression rolled, hot pressed beryllium block. (a) and (b) were taken from the center of the sheet, and (c) and (d) from a point close to the edge of the sheet. The rolling was done at 1010°C (1850°F), 7.5:1 reduction.

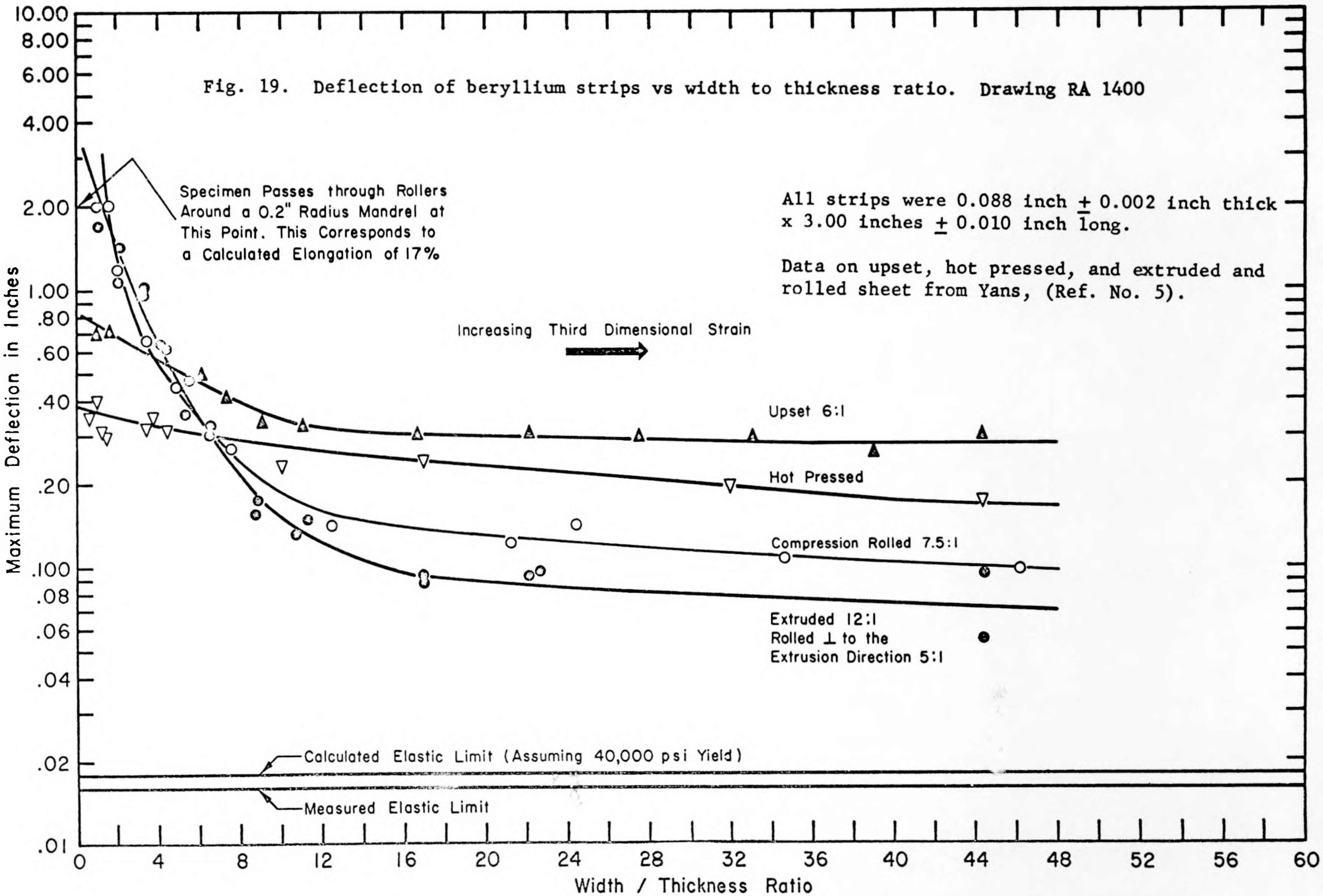


Fig. 19. Deflection of beryllium strips vs width to thickness ratio. Drawing RA 1400

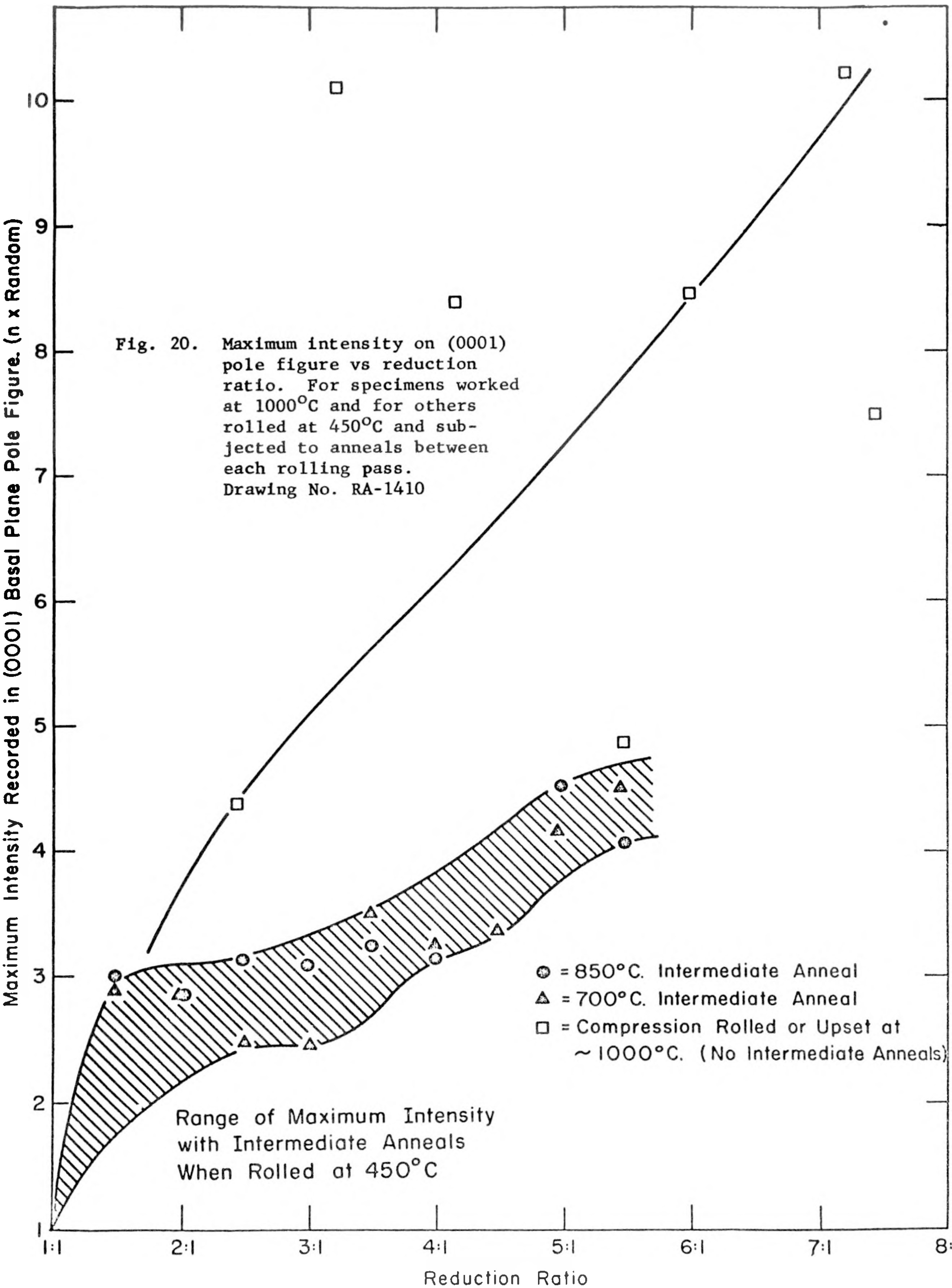


Fig. 20. Maximum intensity on (0001) pole figure vs reduction ratio. For specimens worked at 1000°C and for others rolled at 450°C and subjected to anneals between each rolling pass.
Drawing No. RA-1410

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