

Characterization of beryllium foil produced by hot rolling

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Beryllium foil is important for a number of aerospace applications including honeycomb structures and metal-matrix composites. In this study, a method of producing beryllium foil directly from powder or flake is demonstrated. A variety of foils were produced in the thickness range 90–300 μm , free from defects such as pinholes and excessive surface roughness, and exhibiting sufficient formability for honeycomb manufacture. Foil produced directly from powder or flake exhibits crystallographic texture, microstructure, and formability equivalent to foil produced from more massive precursors.

1. Introduction

Future aerospace systems, such as hypersonic vehicles and advanced space exploration craft, will depend strongly on the availability of novel high-temperature structural materials having mechanical properties which far exceed those of current materials. In many cases, density rather than strength will be the dominant factor limiting the applications of current developmental structural materials. Among all of the structural metals and alloys, beryllium exhibits the highest specific modulus and strength. When integrated with advanced materials construction techniques such as honeycomb structures or metal-matrix composites, the favourable stiffness-to-weight ratio of beryllium can be further enhanced. Fabrication of honeycomb beryllium structures and continuous fibre-reinforced metal-matrix composites, however, depends on the availability of beryllium foil. Unfortunately, due to difficulties associated with manufacturing, beryllium foil is prohibitively expensive.

The difficulty in producing wrought beryllium articles arises from the fact that it exhibits a hexagonal close-packed (h c p) crystal structure. The h c p materials commonly exhibit anisotropic flow behaviour at low temperatures. Studies of beryllium single crystals have demonstrated that plastic slip occurs primarily on basal planes (0001) and on prism planes $\{10\bar{1}0\}$ [1–3]. For both basal and prismatic slip, the direction for slip is $\langle 11\bar{2}0 \rangle$. For polycrystalline materials in general, the Taylor–Von Mises criterion for extended ductility requires the operation of at least five independent slip systems [4]. Slip on the $\{10\bar{1}0\}$ $\langle 11\bar{2}0 \rangle$ and $\{0001\}$ $\langle 11\bar{2}0 \rangle$ systems provides for only four independent slip systems. Non-basal or “c + a” slip on pyramidal planes of the type $\{11\bar{2}2\}$ $\langle 11\bar{2}3 \rangle$ has been observed for beryllium single crystals but the

critical resolved shear stress required for pyramidal slip is two to three orders of magnitude greater than that required for basal slip at room temperature [2, 5]. Because non-basal (pyramidal) slip is restricted, polycrystalline beryllium cannot be deformed to any great extent at ambient temperatures.

Restricted ductility in polycrystalline beryllium has also been attributed to a low critical stress for basal plane cleavage [2, 6]. Impediments to dislocation motion, such as grain boundaries, impurities, or oxide inclusions, lead to dislocation pile-ups and increasing resistance to plastic flow. Ultimately, the critical resolved shear stress for plastic flow exceeds the critical stress for basal plane cleavage and microcracking results. Such microcracking has been observed in beryllium bicrystal experiments even for cases where the Taylor–Von Mises criterion is completely satisfied by the orientation of the crystals and the applied stress [7].

The mechanical properties of polycrystalline beryllium sheet are affected strongly by texture and impurity content. Typically, beryllium sheet is processed to exhibit a pronounced rolling texture involving the alignment of basal planes parallel to the plane of the sheet [8]. Such a texture allows for prismatic slip along $\langle 11\bar{2}0 \rangle$ directions within the plane of the sheet but no appreciable plastic flow in the through-thickness direction. Thus, strongly textured beryllium sheet can exhibit room-temperature tensile ductility in excess of 20%, yet exhibit little ductility in bending because bending deformation requires slip in the through-thickness direction as well as the in-plane direction. Purity is an important factor for polycrystalline beryllium because impurities raise the critical resolved shear stress for slip above the critical stress for basal plane cleavage. For example, it has

been demonstrated that the tensile ductility of polycrystalline sheet is reduced by dilute alloy additions of iron, chromium and nickel [9].

Because of the desire for optimum rolling texture and material purity, and the limited room-temperature ductility of polycrystalline beryllium, the manufacture of beryllium foil is a significant materials processing challenge. Foil is commonly defined as wide metal strip having a thickness of 150 μm or less. As the thickness of the foil approaches the dimensions of microstructural features of the metal, the effect of microstructural irregularities becomes quite important. Large inclusions, coarse precipitates, or brittle grain boundaries can potentially span the entire foil thickness. Similarly, excessive surface roughness can produce local thickness variations which significantly reduce the net section strength of the foil. Local irregularities have a particularly strong influence on the foil ductility because they serve as sources for tensile instability and failure at nominal section stresses which are below the yield point.

A variety of foil production methods have been developed for those cph materials such as beryllium- and titanium-based materials which cannot be readily cold worked. Important alternatives to cold rolling include elevated temperature pack rolling [10–12], electron beam evaporation and vacuum deposition on to a static substrate [13], electron beam evaporation and vacuum deposition on to a moving strip [14, 15], sputter deposition [16, 17], hot rolling under vacuum [18, 19], machining of cast materials [20], vacuum melt spinning [21, 22], or direct cold-roll compaction of powders followed by sintering [23, 24]. Elevated temperature pack rolling has been the most widely used means of producing large quantities of foil, although sputter deposition is an important technique for producing thin beryllium structures of limited area. For the thinnest foils, chemical milling is used subsequent to pack rolling to eliminate surface discoloration and achieve the final thickness reduction [25].

It has been the purpose of the present research programme to produce beryllium foil by the pack rolling method, and to assess the importance of material purity, processing temperature, and reduction amount on the properties of the foil.

2. Experimental procedure

2.1. Materials preparation

The starting materials for this study were as follows: cross-rolled beryllium sheet prepared from hot-pressed powder block - this is a standard grade of beryllium sheet and exhibits a "pancake" grain morphology, and initially, the grains are 40 μm diameter and 5 μm thick; beryllium flake prepared electrolytically from halide salts; and powder prepared by crush grinding and having a particle size in the range of 10–50 μm . Chemical analyses of the foil precursor materials are presented in Table I. The three materials shown exhibit a range of purity and are typical of commonly available beryllium products. The electrolytic flake exhibited the greatest overall purity, although the residual chloride content was rather high.

TABLE I Impurity content of foil precursors (wt p.p.m.)

Impurity	Sheet	Flake	Powder
BeO	14100	3100	6000
Fe	1210	140	500
Al	530	155	200
Si	270	20	200
Cl	—	760	—

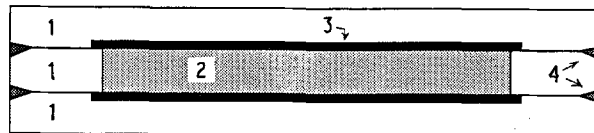


Figure 1 Schematic diagram of laminated assembly used to process foil for this study. 1, Protective encapsulation layers; 2, encapsulated beryllium; 3, release agent; 4, welds.

The processing method developed for this study was designed to encapsulate the beryllium into an oxygen-free environment and is based upon the building up of a laminate geometry. The laminate consisted of an outer ply of protective metal, a thin layer of an inert release agent, the reactive metal itself, an additional layer of the release agent, and a top ply of protective metal. A schematic diagram of this laminate geometry is shown in Fig. 1. The stacking sequence shown may be repeated to form a laminate containing several plies of beryllium. A complete description of the pack rolling method has been presented elsewhere [12].

The beryllium was inserted into a metal frame as shown in Fig. 1. The cross-rolled sheet was simply cut into a square and assembled into an appropriately sized metal frame. The electrolytic flake was vacuum hot pressed to full density at 675 $^{\circ}\text{C}$ under a pressure of 140 MPa. This fully dense section was then inserted into an appropriately sized frame. The beryllium powder was cold pressed into a frame using a single-action die of appropriate geometry. The powder was compacted under a pressure of 304 MPa and had a green density of 1.28 g cm^{-3} (70% full density). For each of the beryllium precursor materials, the metal frame containing the beryllium was mated with cover plates and sealed by welding at the edges of the assembled plates.

Once encapsulated, the beryllium may be processed by any number of thermomechanical processing routes. Because the final properties of rolled beryllium sheet and foil are primarily governed by the texture developed during rolling, the experiments of this study were designed to vary the texture. This was achieved by varying the processing temperature, amount of rolling reduction, and by employing cross rolling. Hot rolling was performed isothermally. The desired reductions were obtained in 15–20 rolling passes with reheating for 1–2 min between passes. A summary of the rolling experiments conducted for this study are presented in Table II.

Subsequent to elevated temperature deformation, the beryllium foil was removed from the protective jacket. This was readily accomplished by simply slitting the edges off of the strip with a large press shear

TABLE II Foil produced for this study

Beryllium source	Rolling temperature (°C)	Cross rolled	Final thickness (μm)	Reduction ratio
Sheet	900	No	100	12:1
Sheet	750	No	90	13:1
Sheet	900	Yes	130	9:1
Sheet	750	Yes	90	13:1
Flake	900	Yes	300	17:1
Flake	750	Yes	290	17:1
Powder	900	No	175	20:1
Powder	750	No	210	19:1

and peeling the layers apart. For each of the materials produced, metallographic analysis was conducted to determine the effect of processing variations on grain size and grain size anisotropy. Photomicrographs were taken on unetched specimens under polarized light. Evaluation of mechanical properties included bend ductility determination. Formability in bending is an important attribute if the beryllium foil is to be used for honeycomb fabrication.

X-ray analysis was conducted on each specimen to determine the degree of crystallographic texture. The textures of the foils were determined by pole-figure analysis. Measurement of the pole densities was performed with a four-axis single-crystal Picker diffractometer utilizing filtered $\text{CuK}\alpha$ radiation and a 0.5 mm incident beam diameter. X-rays were produced with a constant potential power supply at 36 kV and 18 mA. All measurements were in the Schultz reflection geometry [26] over the range of 90° – 20° χ (0° – 70° from the foil surface normal), and over the full 360° sample rotation about its normal. Data points were spaced by 5° on both axes to yield 1009 observations per foil. The (0002) reflection was used in all foils at a 2θ of 52.5° . Sufficient dwell time per point was used to reduce 1σ statistical counting errors to 5% or less. Foil samples were supported in a goniometer head on a thick silicon single-crystal wafer backing. The silicon wafer was cut at such an angle that no diffracted intensity could be observed at any of the angular positions used during the pole figure scan. This wafer served to block any transmitted X-rays from contributing to spurious background diffraction from the goniometer head itself.

Raw observations were corrected for geometrical aberrations and finite thickness effects by comparison with thin uniform powder samples of beryllium with closely similar absorption [27]. However, these corrections were found to be significant only at χ angles smaller than 40° , as expected for the Schultz method. Random pole densities were estimated from the powder measurements and by averaging each set of pole figure stepped-scan measurements. These two procedures agreed within 10%. Background intensity was estimated by measurements without a beryllium sample in place, and with a beryllium sample in place at a 2θ value far from the allowed reflection. The former gives an indication of air scatter, the latter air scatter plus bremsstrahlung scattering accepted by the detector.

3. Results and discussion

3.1. Microstructures

Representative microstructures from five of the processed foils are shown in Figs 2–6. Shown in Figs 2 and 3 are foils processed at 900 and 750 °C, respectively, using powder-source sheet as a precursor material. The foils shown were processed by cross rolling; thus, the microstructures in the longitudinal and transverse directions did not differ appreciably. Processing at 900 °C has resulted in recrystallization and excessive grain growth. In many regions of the foil, the entire thickness of the foil is spanned by just one or two grains. Evidently, the BeO particles observed in this material did little to restrict the degree of grain growth. Processing at 750 °C resulted in a finer microstructure, Fig. 3, because the processing temperature was too low to allow recrystallization to occur.

Foil processed from the hot-pressed electrolytic flake exhibited a similar correlation between processing temperature, degree of recrystallization, and final grain size. The material processed at 900 °C exhibited fairly coarse grains while the material processed at 750 °C was fine-grained. Also observed for the material processed from electrolytic flake, was evidence of prior particle boundaries. Boundaries of original flake particles were delineated by voids and inclusions which could be expected to reduce significantly the mechanical integrity of the foil. It is believed that these defects are related to the residual chloride

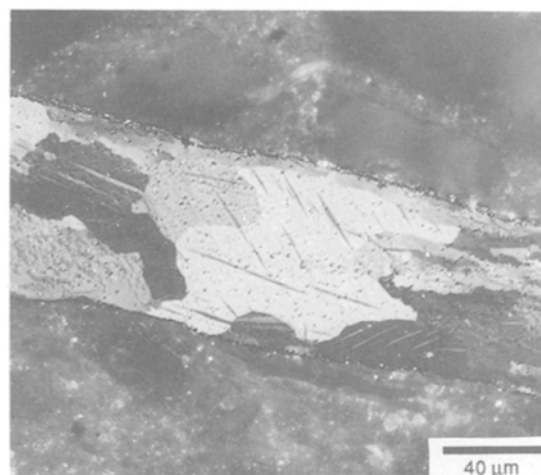


Figure 2 Foil processed from powder-source sheet at 900 °C, polarized light. Note large, recrystallized grain structure.

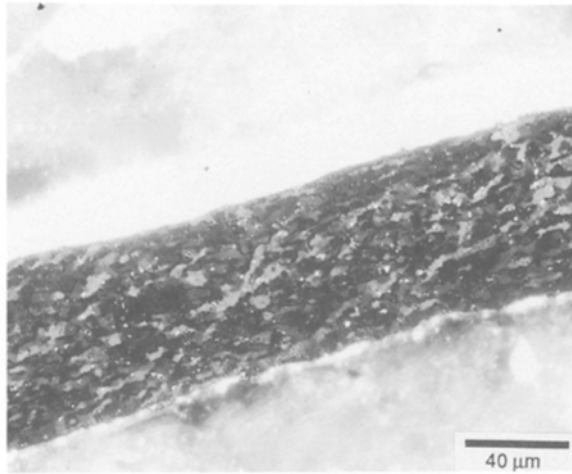


Figure 3 Foil processed from powder-source sheet at 750 °C, polarized light. Note fine, unrecrystallized grain structure.

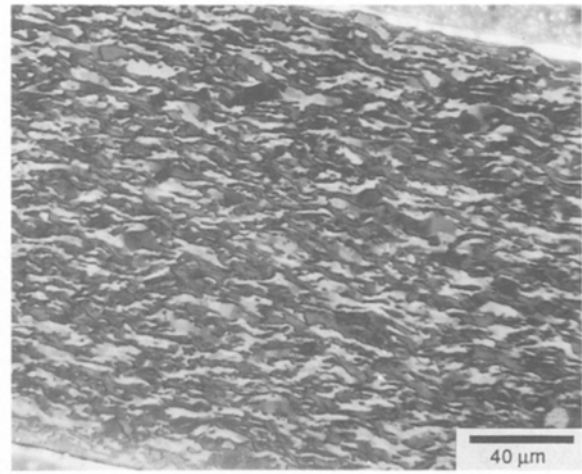


Figure 6 Foil processed from beryllium powder at 750 °C, polarized light. Fine, unrecrystallized grains have resulted.

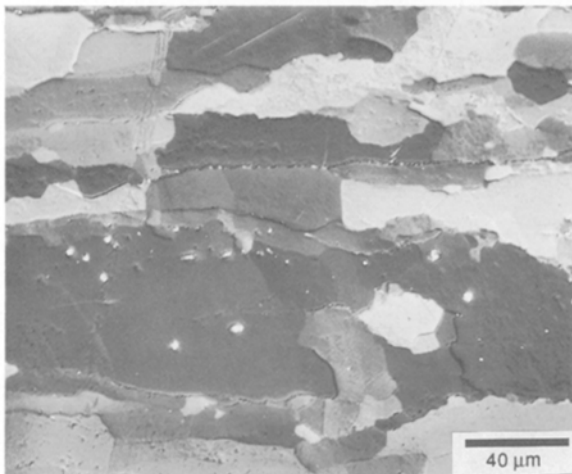


Figure 4 Foil processed from electrolytic beryllium flake at 900 °C, polarized light. Note large recrystallized grain structure and evidence of contamination at prior particle boundaries.

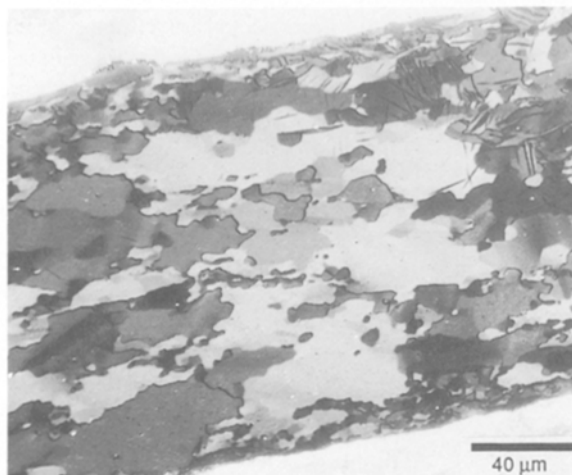


Figure 5 Foil processed from beryllium powder at 900 °C, polarized light. Note large recrystallized grain structure.

present on the surface of the flake resulting from the electrolytic process. An example of the microstructure of cross-rolled foil produced from electrolytic flake at 900 °C is shown in Fig. 4.

Of particular interest for this study is the foil produced directly from powder. For the two experiments conducted to produce foil directly from powder, foils were obtained which exhibited a high-quality surface finish and which were free of defects such as pinholes and edge cracks. The microstructures transverse to the rolling direction for foils produced at 900 and 750 °C are shown in Figs 5 and 6, respectively. As with the other experiments of this study, the material processed at 900 °C, Fig. 5, exhibited a coarse recrystallized grain structure while the foil produced at 750 °C, Fig. 6, exhibited a finer microstructure. The foil processed at 900 °C from powder did not coarsen to the same degree as the foils produced from powder-source sheet or electrolytic flake. Although the foil produced at 900 °C exhibited a coarse recrystallized grain structure, no regions of foil were found in which one or two grains spanned the entire foil thickness. Though not shown, the microstructural features in the longitudinal direction are somewhat elongated due to the unidirectional processing which was employed for the two powder-source foils.

The three foil materials shown in Figs 2, 4 and 5 exhibited a range of final grain sizes even though all were processed at equal temperatures (900 °C) and similar reduction amounts. The difference in grain size among the sheet-, flake-, and powder-processed material is related both to prior processing history and to material purity. For the foil processed from powder-source sheet, the 900 °C processing temperature coupled with stored energy of prior deformation processing provided a strong driving force for recrystallization and grain growth. Thus, the foil produced from the powder-source sheet exhibited the largest grain size. For the foils produced from flake and powder, there was no stored energy of prior deformation and thus, the driving force for recrystallization and grain growth was related to the effect of processing temperature alone. The grain sizes obtained in these two cases was thus smaller than that of the foil produced from powder-source sheet. The foil processed from electrolytic flake exhibited slightly larger grains than the foil processed from powder, although not as large

as the foil processed from powder-source sheet. This may be related to the higher purity of the electrolytic flake as compared to the powder. A higher purity material provides fewer obstacles to grain growth.

3.2. Texture

Several factors may influence texture development in metals, including the extent of deformation, the temperature of deformation, and the degree of deformation anisotropy. Each of these effects were also evident in the present study. The texture information determined in this study is presented below as $[0001]$ pole figures. Intensity contours are employed which represent integer multiples of the calculated random distribution. The symmetry of textures obtained was sufficient to allow for presentation of the data employing a half-section of the standard pole figure. The section is chosen such that the horizontal represents the rolling direction (RD), the vertical represents the cross rolling direction (CD), and the centre of the figure represents an axis perpendicular to the rolling plane.

Of the three foil precursor materials, only the powder-source sheet had an initial texture which differed from a completely random one. The initial texture of the powder-source sheet is shown in Fig. 7. The texture shown is typical of beryllium cross-rolled sheet and is the desired texture for maximum in-plane tensile ductility. The maximum degree of preferred orientation measured for this material corresponds to six times the random value. The initial texture of the hot-pressed electrolytic flake and the cold-compacted powder was assumed to be random.

The textures developed as a result of the deformation conditions employed, previously outlined in Table II, generally fell into two categories: the ideally oriented "cross-rolled" texture, or the so-called "extrusion" texture. Within each of these classifications, the level of preferred orientation exhibited by the various foils differed only by degree.

The cross-rolled texture was developed for the four materials listed in Table II which were subjected to cross rolling. The best example of an ideal $[0001]$ texture obtained in the present study is shown in Fig. 8. The texture shown was obtained for the foil produced by cross rolling the powder-source sheet at a processing temperature of 750°C . The deformation

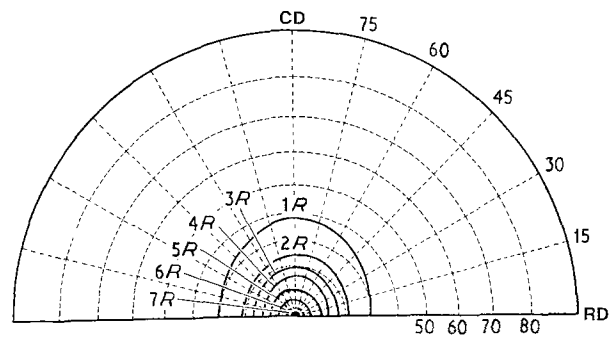


Figure 8 "Cross-rolled" texture of foil produced from powder-source sheet by cross-rolling at 750°C . The preferred orientation corresponds to seven times random.

employed in the processing of this foil increased the degree of preferred orientation from six times random, Fig. 7, to seven times random, Fig. 8. The foils produced from electrolytic flake at 750 and 900°C , as well as the foil produced from cross-rolled sheet at 900°C also exhibited the "cross-rolled" texture to varying degrees.

The extrusion texture was developed for those foils which were processed by unidirectional rolling deformation. An example of this texture is shown in Fig. 9 for the case of foil produced from cold-pressed beryllium powder at 750°C . The $[0001]$ basal plane poles are concentrated in a band about the axis of deformation. Such a texture is commonly observed for beryllium extrusions [8]. The extrusion texture was obtained for each of the four materials listed in Table II which were not subjected to cross rolling. The degree of preferred orientation was slightly affected by the processing temperature. For example, the maximum intensity observed for the foil shown in Fig. 9 was seven times the calculated random distribution. When the higher processing temperature of 900°C was used, however, the maximum preferred orientation corresponded to five times random. This is shown in the pole figure diagram of Fig. 10. Based on these results, it would be expected that lower processing temperatures than those used in this study would result in an even greater degree of preferred orientation than that observed here.

The texture determinations of this study demonstrate that preferred orientations can be obtained in foil produced from powder or flake, and can be enhanced in foil produced from previously deformed sheet. Ultimately, however, the limited amount of rolling reduction associated with the processing techniques of this study place a limit upon the degree of preferred orientation which can be obtained. Typically, beryllium sheet and foil is formed by extrusion of hot-pressed powder and subsequent rolling deformation perpendicular to the extrusion direction. The large deformation obtained with such a processing route can lead to a preferred orientation as high as 32 times random [8, 9]. Such a large degree of preferred orientation is probably not obtainable using the processing technique of this study. It remains to be demonstrated, however, if such a high degree of preferred orientation is necessary for foil applications.

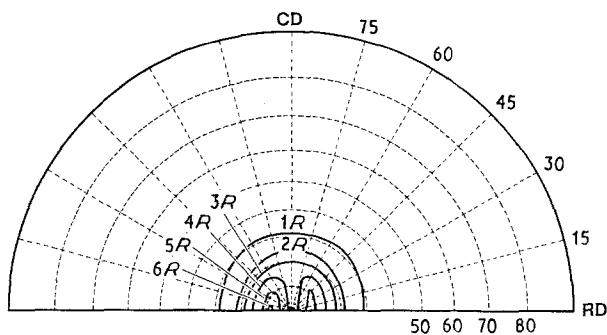


Figure 7 Initial texture of powder-source sheet used to make foil. The preferred orientation resulting from prior processing corresponds to six times random.

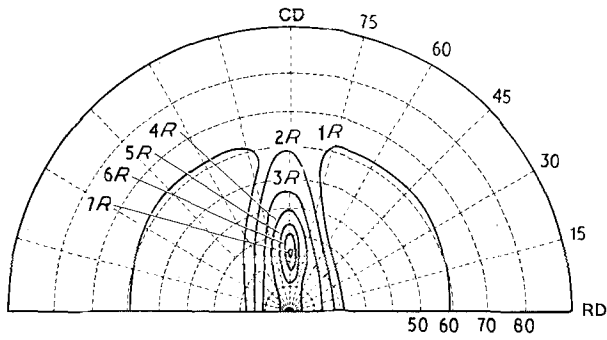


Figure 9 "Extrusion" texture of foil produced from beryllium powder by unidirectional rolling at 750°C. The preferred orientation corresponds to seven times random.

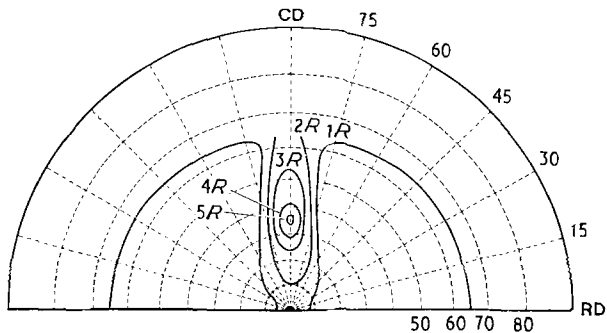


Figure 10 "Extrusion" texture of foil produced from beryllium powder by unidirectional rolling at 900°C. The higher processing temperature has resulted in a preferred orientation which corresponds to five times random.

3.3. Formability

Measurable room-temperature bending ductility has been obtained for some of the foil produced in this study. The majority of materials produced exhibit brittle behaviour, however. Shown in Fig. 11 is a foil with sufficient ductility to be plastically formed at room temperature into the shape shown. The permanent plastic deformation corresponds to an outer fibre strain of 1%. Such behaviour was exhibited by the foil which was processed from powder-source sheet at 900°C for both the unidirectionally processed material and the cross-rolled material. The ductility ob-

served is due in large part to the coarse grain structure of the material, Fig. 2. For large grains which span the entire foil thickness, plastic slip need not be accommodated at neighbouring grain boundaries. Thus, plastic deformation may occur in a manner analogous to single-crystal behaviour for the coarse-grained materials produced in this study. Room-temperature bend ductility was not observed for any of the foils produced at the lower processing temperature of 750°C. This is due to the finer grain size of these materials as well as the increased degree of crystallographic orientation associated with lower processing temperatures. As described in Section 1, bending deformation of polycrystalline textured sheet is hindered both by the need for plastic accommodation at grain boundaries (Taylor-Von Mises criterion) and by the diminished number of slip systems oriented for through-thickness slip deformation. Room-temperature bend ductility was further hindered for the materials produced from the hot-pressed electrolytic flake by contamination at prior particle boundaries. Such contamination resulted in intergranular failure at a net section stress well below that required for plastic slip.

Utilization of beryllium foil for honeycomb structures requires the forming of the foil into a corrugated section which is subsequently joined to other corrugated sections at nodal points to build up a cellular core structure [28]. Although some of the foils produced in this study exhibited room-temperature bend ductility, the degree of bending achieved was insufficient for that required for fabrication of honeycomb core. To achieve the necessary degree of formability, the current practice in the beryllium industry employs forming at elevated temperatures. At intermediate temperatures, polycrystalline beryllium exhibits both a decrease in flow stress and an increase in tensile ductility [29]. In order to evaluate the potential of the foils processed in the current study for formation into a corrugated structure, specimens of each of the foils of this study were warm pressed using a specially constructed die. Shown in Fig. 12 are several examples of foil which exhibited poor bend ductility at room temperature yet deform readily when warm-pressed at

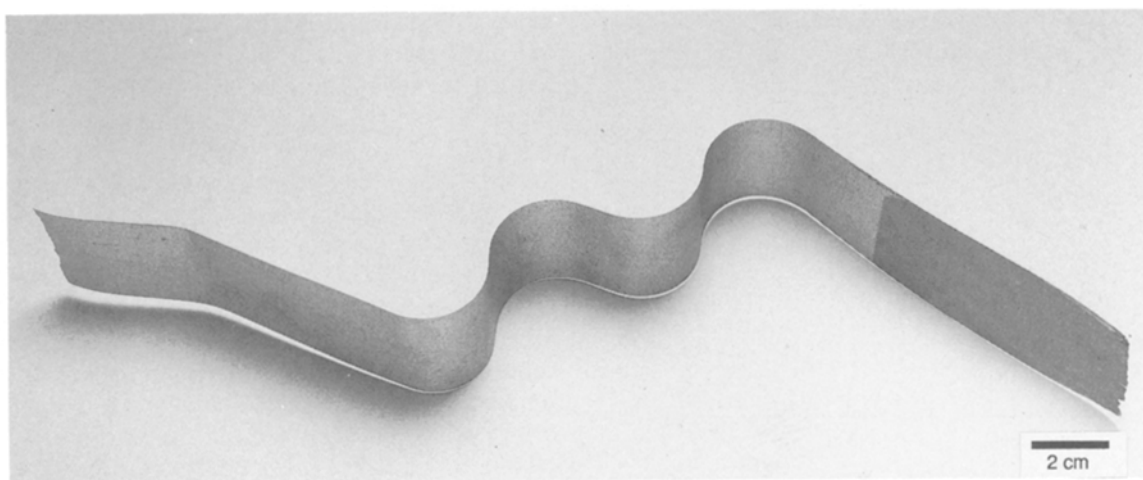


Figure 11 Foil exhibiting room-temperature bend ductility of 1%.

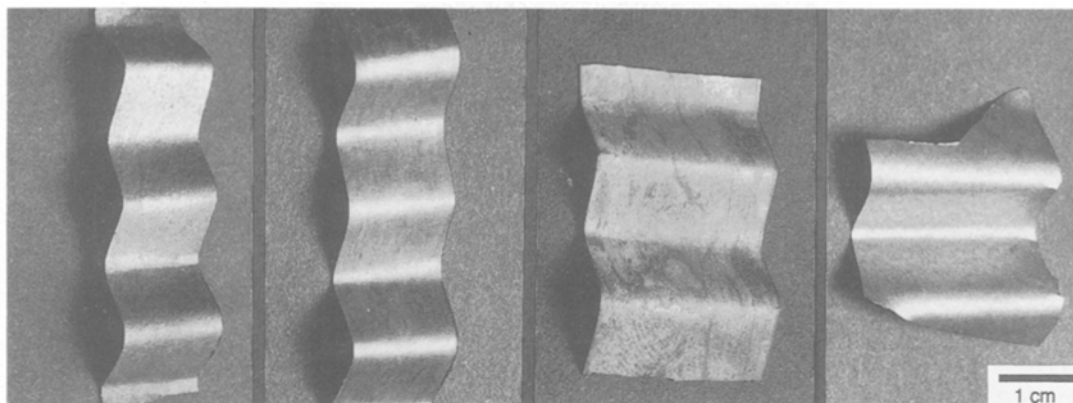


Figure 12 Examples of foil warm pressed into corrugated shape at 600 °C. Shown (left to right) are foil produced from powder at 900 °C, foil produced from powder at 750 °C, foil produced from flake at 900 °C, and foil produced from flake at 750 °C.

600 °C. It is noted that foils exhibiting both the extrusion texture and the cross-rolled texture exhibited formability. This result demonstrates that texture development may not be important as a criterion for evaluating the potential utility of beryllium foil for honeycomb application.

4. Conclusions

In the present study, a foil processing method has been demonstrated which by-passes much of the processing which is traditionally required to produce foil. The production of foil by the direct pack rolling of beryllium powder or beryllium flake, as demonstrated in the present study, may lead to a less costly means of producing beryllium articles. The foils produced exhibit oriented crystallographic textures up to seven times random for both the extrusion texture and the cross-rolled texture, good surface quality and, in some cases, 1% bend ductility at room temperature. Further, the foils are capable of being formed into corrugated shapes at elevated temperatures which may be later assembled into honeycomb core.

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