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DMIC

FABRICATION AND STRUCTURAL APPLICATIONS

Beryllium was chosen for three items on the TACSAT I Satellite incorporating a nutation damped stabilization device designed several years ago by Hughes Aircraft.(1) Because of beryllium's inherent stiffness, Hughes made beryllium the choice for the bearing and power transfer assembly, the UHF helix antenna tubes, and the bicone antenna support. Beryllium also had potential usage in the antenna mast structure, but since this was a primary structure, beryllium was not to be chosen for this application until additional studies proved its worthiness. Several problems that cropped up during fabrication of these parts are presented along with the sources and the solutions. On the basis of the success of the TACSAT components and the studies involving the antenna mast, Hughes is producing a satellite that contains an adapter cone, spinning arms, despun platform, and housing for the beryllium and power transfer assembly, all manufactured from beryllium.

Attempts are being made at Battelle to produce 0.005-in.-diameter beryllium wire by hydrostatic extrusion drawing.(2) Hydrostatic pressures are applied to the unreduced stock, while controlled drawing stresses are simultaneously applied at a predetermined drawing speed. Process conditions and sequence (such as lubricant for the wire, drawing temperature, and pressurizing fluid) are being determined with a goal of producing sufficient beryllium wire for evaluation by potential users of the wire. The two best lubricants tried so far are Vydax and TFE. A temperature of 250 F has been selected, with qualifications, as the most desirable after studying drawing at temperatures between 80 and 350 F. Short coils up to 10 feet in length have been drawn at 60 percent reduction consistently at this temperature using the lubricants mentioned above. The researchers indicated that coils up to 800 feet in length would be attempted. Tensile properties of hydrostatic-drawn beryllium wire were measured, but inconsistent data were obtained. This may have been due partially to defective starting material.

In another program recently completed by Brush Beryllium Company, attempts were made to produce high-strength 5-mil beryllium-alloy wire with ultimate strengths of 300 ksi or more.(3) Thermally reduced powder and electrolytic flake were classified into various fractions, modified with additions of iron, cobalt, and copper, and then hot pressed and machined into 1-inch-diameter billets for extrusion. These were warm extruded at 800 F and wire drawn at the same temperature. The effect of impurities, grain size, and composition on work hardening and strengthening were investigated. The variations in wire drawing force, fracture mode, and crystal orientation due to working also were studied. Electrorefined beryllium gave optimum combinations of mechanical properties in fine drawn wire. Stress-strain characteristics in the drawn wire were found to be independent of oxide level, hot-pressed grain size, and iron and cobalt chemistry. This is due to oxide and impurity segregation in grain boundaries and subsequent striation parallel to the drawing direction. The best engineering properties obtained on 5-mil wire were 227 ksi ultimate strength, 170 ksi yield strength, and 2.1 percent elongation. On the basis of literature sources, a 1-mil drawn wire would potentially achieve strengths near 250 ksi. Variations in mechanical properties due to chemical milling and shelf life were also examined. While 20- and 10-mil wire were unaffected by shelf life, some 5-mil wire batches showed degraded properties, possibly from surface corrosion or aging.

A DMIC report on deformation processes involved in the preparation of beryllium wrought products was recently published describing a variety of wrought beryllium products that are available, how these products are made, some typical properties with emphasis on texturing effects, and important problem areas.(4) The authors note that Government sponsorship of research and application programs on wrought beryllium products has been significantly curtailed since 1968, and emphasis has been placed more on basic research. Reports coming out after 1968 have been mostly final reports ending long-term projects that started in 1965 and 1966. Little support has been given toward improving the processes that could lower costs of beryllium wrought products in the future.

Along these same lines, a cost-performance study was conducted to show how tradeoffs among such performance factors as structural weight, manufacturing difficulties, reliability, and safety can be exploited.(5) Several materials besides beryllium were rated according to how they compared with aluminum. These were titanium, boron-epoxy, and carbon-epoxy fiber-reinforced resin composites, and boron-aluminum-reinforced metallic composite. Tradeoff parameters on these materials were compared in an integrally stiffened skin construction as well as in a honeycomb construction. All the materials showed weight-performance superiority over aluminum, and beryllium was rated slightly better than aluminum. Costs for most of these materials were substantially higher than those for the baseline aluminum-stiffened skin; beryllium of both construction types ranked last according to cost performance. The authors point to reductions in fabrication costs for beryllium as a particularly important factor that will help increase its potential for future structural applications.

A recent issue of KBI "Keynotes" illustrated some of the applications of beryllium in Apollo 12.[6] Components, such as a beryllium inertial-guidance platform, an optical unit assembly, an alignment optical telescope housing, a seismometer cylinder, and a number of other lunar-based scientific experiments with structural components and heat-dissipating members of beryllium, are described and illustrated.

The results of a Navy program aimed at producing beryllium sheet having improved ductility was reported by Franklin Institute.[7] Through use of low-temperature (200 to 500 C or 390 to 930 F) rolling procedures coupled with intermediate and post-rolling annealing procedures, better bend ductilities were achieved at 100 C (212 F) with beryllium sheet than with a reference powder and ingot beryllium sheet tested in the same manner and at the same width to thickness ratio of 6:1. A bend ductility as high as 5-1/2 percent has been achieved together with a reported ultimate tensile strength of 124 ksi on samples prepared by "programmed rolling", starting with a temperature of 750 C (1380 F) and ending at 450 C (840 F) followed by an anneal for 15 minutes at 625 C (1155 F). Similar results have also been achieved by rolling to an 85 percent reduction at 300 C (570 F) and subsequently annealing at 625 C. These optimum combinations of bend strength and ductility are achieved for beryllium sheet in the partially recrystallized condition. High bend ductilities were also achieved in some cases for material in the fully recrystallized condition. The reasons for enhanced ductility under these conditions of thermomechanical processing are not well understood. Texture is suspected of being an important variable which will be investigated in a future program.

EXTRACTIVE METALLURGY

Basic extraction-refining-conversion processes for beryllium are summarized in a recently published paper by Kawecki Berylco.[8] Flow diagrams which illustrate the different steps in commercial processes are included. Price projections for increasing volumes of production are illustrated for several mill products. A brief commentary gives the current and anticipated availability of beryllium and new products, and the kind of technological advances that can be expected during the next few years.

Because of potential economic advantages, the Bureau of Mines investigated the direct electrowinning of beryllium from BeO with several anode materials using a molten-salt electrolyte containing 56LiF-37BeO.[9] Porous carbon, carbon pipe, graphite pipe, and graphite tubing were tested to determine which anode material gave the highest current density with the lowest cell voltage. The porous carbon anode operated in the electrolyte at 780 F for several months before noticeable corrosion occurred because of reaction with oxygen liberated from BeO as CO or CO_2. The authors were able to collect the electrodeposited metal as powder, or as small spheres by coalescing at 1300 C (2370 F). The metal was about 99 percent pure. The primary impurities were aluminum, calcium, and iron. Porous carbon anodes achieved the best current-carrying capacities at up to 16 amp/dm^2 at a cell voltage of 3.3 volts. Cathode current efficiency was 50 to 70 percent. When cell voltages on other anode materials were raised above 3 volts, the anodes polarized, causing the current density to decrease rapidly to 1 amp/dm^2. Even though cell voltage was then raised to 5 volts, the anodes remained passivated.

PHYSICAL AND MECHANICAL METALLURGY

A discussion of factors affecting dimensional stability in beryllium mirrors and methods which have been investigated to minimize instabilities has recently been presented in a DMIC Memorandum by Maringer.[10] Residual stresses in beryllium introduced by (1) machining, (2) anisotropic thermal expansion in beryllium crystals through crystallographic texturing, and (3) electroless nickel plates are among the common sources of service instability. While these stresses are not always as great as the microyield strength (stress required to cause a plastic strain of 10^{-6} in./in.), creep has nevertheless been observed when stresses smaller than the microyield strength are applied. To avoid these instabilities, a reduction in the residual stresses is required by stress-relieving heat treatments, by minimizing preferred orientation through powder-reattritioning techniques, isostatic compaction, or pressureless sintering, and by increasing the microyield strength through alloying, grain refinement, or other techniques.

The requirement for design information in connection with the beryllium neutron reflectors used in the NERVA engine has prompted metallurgists at Westinghouse Astronuclear Laboratory to obtain fracture-toughness data on vacuum hot-pressed beryllium block in the as-pressed condition, after heat treatment and after irradiation at liquid nitrogen temperature.[11] Results obtained with WOL (wedge-open-loading) type samples are presented in Figure 1 which shows the effect of test temperature, sample orientation, strain rate, and heat treatment on the fracture toughness, K_{Ic}. The testing direction had a small but noticeable effect on the fracture toughness, with somewhat higher values being obtained when samples were loaded transverse to the pressing direction than when they were loaded in the pressing direction. The various loading rates had little effect on K_{Ic}.

Fracture-toughness values obtained from samples of the DCB (double-cantilever-beam) type gave somewhat different values for K_{Ic} (~9 ksi$\sqrt{\text{in.}}$ compared with 15 ksi$\sqrt{\text{in.}}$), but demonstrated the same temperature response. This specimen design also allows some measure of the crack-arresting ability and the K values required for crack reinitiation.

Russian investigators have made some interesting observations on basal-plane slip behavior as a function of temperature and purity level.[12] Using

$$\delta = \frac{\text{Resistance at 300 K}}{\text{Resistance at 4.2 K}}$$

as a measure of purity level, they have determined its effect on the critical resolved shear stress for

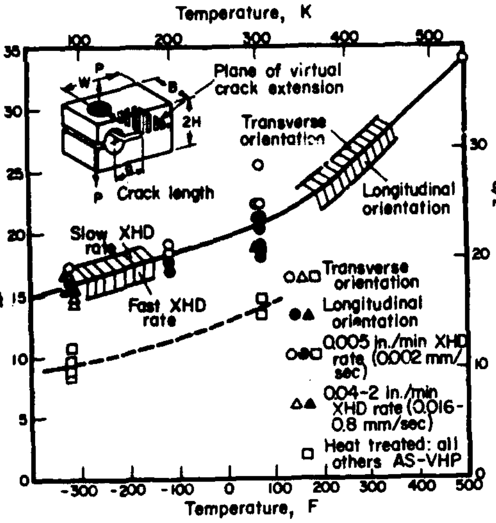

FIGURE 1. BERYLLIUM FRACTURE-TOUGHNESS TEST RESULTS OBTAINED WITH WOL SPECIMENS(11)

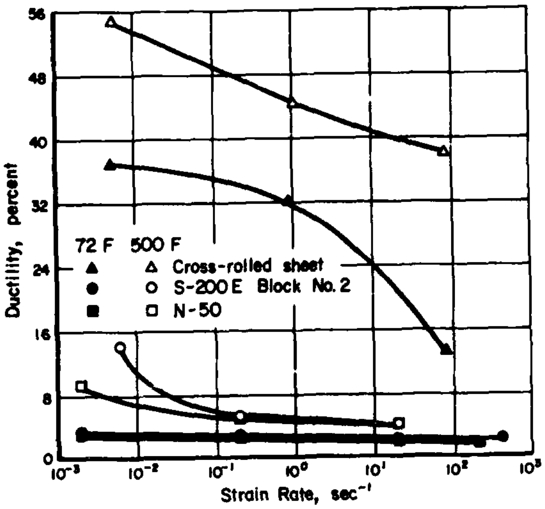

FIGURE 2. DECREASED DUCTILITY OF BERYLLIUM WITH INCREASED STRAIN RATE(13)

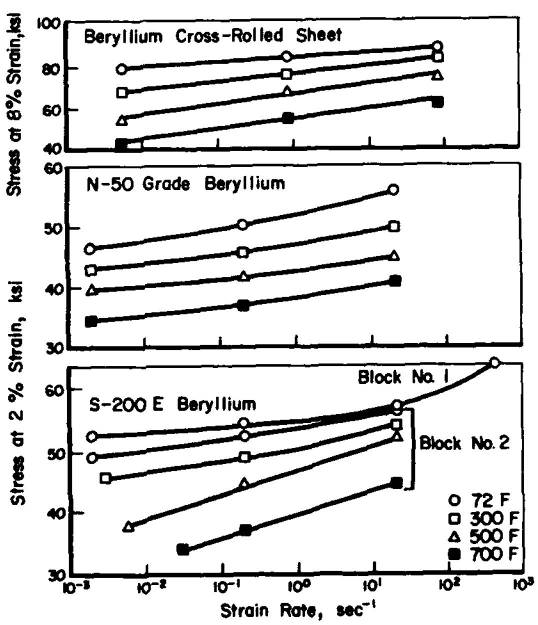

FIGURE 3. FLOW STRESS VERSUS STRAIN RATE FOR THREE BERYLLIUMS(13)

basal slip, the work-hardening rate, the slip distance, and the slip-band width at temperatures between 4.2 and 300 K. The basal-plane deformation characteristics were most sensitive to purity level at the lowest test temperatures (less than 20 K). Large changes in critical resolved shear stress occur in this range, with the more impure crystals showing little plasticity at or below 20 K. The work-hardening rate and slip-band width decreased with lower test temperature and increased impurity content, as would be expected from the increased work-hardening rates which were also observed under these conditions.

Investigators at GM's Materials and Structures Laboratory have determined the uniaxial tensile properties of various grades of commercial vacuum hot-pressed beryllium (N-50, S-200E, S-200D) and of S-200 cross-rolled sheet over a range of strain rates from 10^{-3} to 10^{3} and temperatures from 72 to 700 F.(13) Their results, shown in Figures 2 and 3, indicate reduced ductility and increased flow stress with increasing strain rate, especially at elevated temperature.

Two papers have recently been published describing an etch-pit technique which has proven to be useful in the analysis of fractured surfaces.(14,15) The technique, which makes use of a diluted 20 percent CrO_3, 14 percent H_3PO_4, 1 percent H_2SO_4 solution, forms etch pits whose geometric shape depends on the orientation of the etched surface. In addition to the three basic orientations (0001), ($10\bar{1}0$), and ($11\bar{2}0$), the Allison workers have determined the etch-pit configurations at 20, 40, 60, and 80 degrees from (0001) toward both ($11\bar{2}0$) and ($10\bar{1}0$) and from ($11\bar{2}0$) toward ($10\bar{1}0$), and have found them to be unique to their specific orientations.(15) Examples in the use of the technique for the analysis of the orientation of regions of fracture initiation and propagation are cited in both references.

At a recent beryllium conference, efforts were described that are now being applied by Brush Beryllium Company to control the impurity content and to apply heat treatments to hot-pressed beryllium block and to machining blocks produced from them.(16) This combination of impurity control and heat treatment has been found to produce materials with tailored properties.

The "microalloying" elements of most significance are iron and aluminum which may react with beryllium in combination to form $FeAlBe_4$, or the iron may react independently to form $FeBe_{11}$. Alternatively, the iron may exist in solid solution and the aluminum which has a very low solid solubility may exist as free aluminum usually found at the grain boundary. Brush Beryllium has a proprietary method of determining the amount of aluminum and iron distributed freely, or in the compounds.

Foos has related the mechanical properties of hot-pressed beryllium to the free-aluminum content and to the iron distributed in the matrix as $FeBe_{11}$. An example of one of these relations is shown in Figure 4 in which the ductility at 1050 F is shown to be sensitively dependent on the free-aluminum content.

The control of impurity content coupled with heat treatment has led to the development of beryllium with much improved ductility, especially at elevated temperatures. This material has been used in aircraft brake and rocket applications. This has also led to the development of a grade of beryllium (S-240) highly formable at 900 to 1000 F.

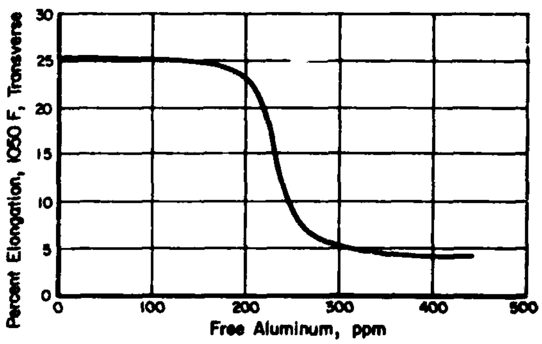

FIGURE 4. ELEVATED-TEMPERATURE DUCTILITY OF HOT-PRESSED BERYLLIUM AS A FUNCTION OF THE FREE-ALUMINUM CONTENT(16)

POWDER METALLURGY

In a brief note, investigators at SRI have described a method for producing isotropic consolidated beryllium.(17) These authors claim that a large part of the texturing is due to the aligning of the powder particles during the initial powder pouring and have described a procedure to avoid texturing. The procedure consists of hydrostatically pressing the powder, breaking it up into larger agglomerates, rehydrostatically pressing the powder, and then pressureless sintering.

Also at SRI, the calculated integrated X-ray intensities expected from various crystallographic planes were compared with values measured on randomly packed spherical powder.(18) Agreement between the measured and calculated intensities, as shown in Table 1, and the disagreement with data from the ASTM Card File Index have led these authors to conclude that the ASTM data do not reflect intensity values for a random sample.

These values of integrated intensity may be used as a semiquantitative guide for deducing the preferred orientation in a specific direction in the sample. However, exposure of a different surface is necessary for each direction in which the preferred orientation is required. Determination of a complete pole figure by this method, thus, would be tedious.

CORROSION

A recent translation from Russian literature summarizes the experimental research on the corrosion, oxidation, and protection of beryllium in various gaseous environments over a wide range of temperatures and pressures.(19) Effects of purity, moisture, surface treatments, flow rates, and gas pressure on corrosion rates are discussed on the basis of X-ray diffraction and optical- and electron-microscopic studies on film development and growth. Emphasis is placed on oxidation kinetics of beryllium and its alloys, beryllides, and other compounds. Possible effects on oxidation behavior of beryllium due to neutron radiation are indicated. A full chapter is devoted to the methods of protecting beryllium against corrosion and increasing wear resistance with surface coatings, and in particular, by anodizing and electrolytic coating. Vacuum deposited coatings also are discussed at length. A brief summary of flame-spraying techniques, diffusion coatings, melt-dip coating processes, enameling, and cladding by roll bonding to obtain corrosion-resistant surfaces is also given. The book is well documented with numerous references, most of them coming from domestic sources.

TABLE 1. CALCULATED AND OBSERVED INTENSITY VALUES OF ISOTROPIC BERYLLIUM(18)

Plane, hkil	d-Spacing A: Calculated(a)	d-Spacing A: Observed from Sintered Block	Intensity, I/I_o: Calculated(b)	Intensity, I/I_o: Observed from Sintered Block	Observed from Spherical Powder	Average of Observed Intensities	ASTM Observed Intensities
10$\bar{1}$0	1.980	1.98	25.7	31.8	30.6	31	20
0002	1.792	1.79	24.9	29.4	27.4	28	14
10$\bar{1}$1	1.733	1.73	100.0	100.0	100.0	100	100
10$\bar{1}$2	1.329	1.33	13.2	10.8	11.0	11	12
11$\bar{2}$0	1.143	1.14	15.7	15.4	18.0	16	12
10$\bar{1}$3	1.023	1.02	18.8	19.0	20.8	20	12
20$\bar{2}$0	0.9899	0.990	3.0	3.0	5.0	4	2
11$\bar{2}$2	0.9637	0.964	23.2	24.0	24.6	24	8
20$\bar{2}$1	0.9542	0.954	17.3	18.3	20.1	19	--
0004	0.8960	0.987	3.9	5.4	5.8	5	--
20$\bar{2}$2	0.8665	0.867	6.2	4.2	4.8	5	--
10$\bar{1}$4	0.8163	0.817	8.1	9.1	8.7	9	--

(a) The calculated spacings for beryllium were obtained using the crystal lattice parameters: a = 2.2859, c = 3.5843 A.

(b) A value of B = 0.5 A was used in the intensity calculations.

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R. W. Endebrock, Editor