Hot-Pressing of Electrolytic Grade CR Beryllium

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A brief discussion of the importance of hot-pressing to the beryllium industry is presented along with data pertaining to the powder characteristics and properties of CR electrolytic powder. The powder-manufacturing process and an elaborate quality-control procedure for inspecting the powder are described. The hot-pressing cycle is outlined, including blending, loading, pressing, and stripping. Related equipment is described, including the dies used to contain the beryllium. Properties of the hot-pressed block and the quality-control procedure for determining integrity of sections used from the hot-pressed block are discussed.

INTRODUCTION
Since 1950, beryllium has taken its place as one of the more promising metals to be integrated in the program of increased scientific technology, namely, in nuclear and space applications. Beryllium has a high elastic modulus and low density, which results in a rigidity/density ratio higher than those of other metals. In addition, it has good strength, high melting point, high corrosion resistance, and heat conductivity, as well as a low neutron-absorption cross section. The most significant undesirable characteristics are its toxicity, brittleness, and difficulty associated with its fabrication.

Most metals utilize a casting process followed by conversion to wrought product. At this time, techniques have not been developed to permit beryllium to be conventionally processed in such a manner. Casting results in a billet with extremely large grain structure, which, unfortunately, results in lowered mechanical strength. Besides the disadvantage of large grain structure, impurities, such as aluminum and iron, tend to segregate at the core of cast billets. This causes hot shortness and centerline cracking which renders the beryllium useless for further processing [1-2].

Since casting of beryllium was found to be impractical from an engineering standpoint, metallurgists in the field next resorted to a sintering process. This process, although more successful, generally resulted in compacts with low density. Low density in sintered beryllium products has generally been attributed to the inability to break down the beryllium oxide film which is concentrated about the individual beryllium particles. Although a fine structure is, therefore, attained by sintering, properties generally are low and ductility nil, due to more than 3% porosity in the structure [1-2]. British investigators have recently published reports claiming full densification for sintered compacts as the result of using an activated beryllium [3-6]. A second problem which accrues from the sintering process is the shrinkage effect commonly noted for such a process. Shrinkage in beryllium results...
in cracking, since the crystal structure of beryllium is not favorable to the relief of stresses generated from this effect.

The metallurgist is, therefore, limited to a process of hot-pressing in order to obtain a beryllium ingot capable of meeting the engineering properties of the metal required for it to be useful. Hot-pressing, in general, minimizes the disadvantages of the sintering process, and, as in the sintering process, grain size can be accurately controlled by the grinding conditions and the classification of the beryllium powder to be hot-pressed. Likewise, because pressure is an important variable in hot-pressing, the characteristic beryllium oxide film about the individual particles is ruptured and, hence, diffusion and consolidation can readily occur. Also, the use of pressure prohibits shrinkage and forces the consolidated product to conform to the geometry of the die cavity. Fortunately, equipment of suitable size is available in the beryllium industry to accommodate the fabrication of large size components currently in use today. Hot-pressing has, therefore, been established as the primary method for the fabrication of beryllium metal products. \[1-2\].

Until recently, most commercial hot-pressed products which had been fabricated in this country were made from the magnesium reduction of beryllium fluoride. The electrolytic process for the production of beryllium flake was first developed by the German firm, Degussa, and in 1945 this process was adopted by the French firm, Pechiney, who further developed the process to one of commercial importance. In general, the Pechiney process converts beryl ore to beryllium oxide, which is briquetted with carbon. The briquettes are then chlorinated, and the crude chloride is then purified. Purified chloride is then converted to beryllium flake by electrolysis. The method of electrolysis had not been used extensively in this country partly because of the prior commercial establishment of the fluoride process. Because of agreements reached between Pechiney and General Astrometals Corporation, electro­lytically produced beryllium has become available on the American market. By means of electrolysis, an extremely high-purity beryllium can be produced. This beryllium is generally lower in iron, carbon, aluminum, chromium, and other metallic contaminants. In addition, scandium can be held to 10 ppb or less in the electrolytic process. This latter purity could be of importance to the nuclear industry because expended reactor components could be cooled radioactively in lesser time periods and, hence, a lower beryllium requirement would ensue.

As with the magnesium fluoride process, the flake is contaminated with halogens. The flake, therefore, is melted, at which time the halogens are released and chlorine content is related to be less than 10 ppm. Powder is then produced from the cast billet.

Some reference has been made in the literature to the effect that high-purity beryllium produced by the electrolytic process is somewhat more difficult to hot-press than lower purity grades of beryllium \[3-6\]. The purpose of this paper is, therefore, to provide a better understanding of electrolytically produced powder and products resulting from the consolidation of this powder. It will be shown that high-purity electrolytic powder can be successfully fabricated and can be readily used as commercial product.

**PROCESS**

It is not intended to dwell upon the electrolytic process *per se*. It is enough to state that, using the process, an extremely high-purity beryllium can be made for the commercial market. The flake resulting from the process, as stated before, is pelletized and charged to a melting furnace (Fig. 1). Here the flake is melted in order to