HIGH-PURITY SHAPE CASTING WITH AN ELECTRON-BEAM FURNACE

C. W. Dean
R. E. McDonald
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METALS AND CERAMICS DIVISION

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OCTOBER 1967

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C. W. Dean and R. E. McDonald

ABSTRACT

An electron-beam melting furnace has been utilized to produce laboratory quantities of high-purity metals for many Oak Ridge National Laboratory research programs. The furnace hearth originally produced pancake-shaped ingots. Although high-purity samples could be produced in this shape, they contained cold shuts, porosity, and a coarse-grain structure. Because of the shape and other undesirable properties, rod and wire were difficult to produce from such castings.

To overcome these difficulties, we developed a casting technique for use with the electron-beam furnace that permitted a molten charge of high-purity metal to be drop-cast into a cylindrical or rectangular chill mold. Subsequent fabrication was easier and less expensive because of the more desirable ingot shapes. In addition, a higher material yield was realized. This drop-casting technique and melting equipment have been used to cast fabricable shapes of Ti, Zr, Nb, V, Ta, W, Mo, and their alloys.

INTRODUCTION

The range of research programs at Oak Ridge National Laboratory requires a wide variety of metals and alloys of extremely high purity. Because certain impurities can be vaporized during the melting operation and because there is no contamination from molds or electrodes, the electron-beam furnace is useful for producing high-purity material. With the exception of alloys containing desirable constituents of high vapor pressure, the quality of the melted product has been generally superior to equivalent material processed by other means. In the melting of most refractory metals, high-purity melt products are produced on a routine basis at ORNL with a very low interstitial impurity content. By remelting several times before final casting is attempted, homogeneity is achieved on the high melting point, difficult-to-alloy metals. However, many of these alloys are difficult to fabricate when cast as pancakes.
This report covers the development of a drop-casting technique that greatly improved the fabricability of the electron-beam melted alloys.

Electron-beam melted metals and alloys are used in a variety of ORNL research and development programs. High-purity refractory-metal wire and rod are used extensively in physical property research such as electrical resistivity measurements. Sheet and rods are being used as creep specimens for alloy evaluation and mechanical properties testing. In addition, electron-beam melted refractory metals and alloys are currently being used for end plugs in reactor fuel cans and capsules, as hardware in liquid-metal corrosion loops, and as fixture material in high-temperature, high-vacuum research.

In research and development work of this nature, "miniature" melts are necessary because of the time and material costs involved in making larger melts. The configuration of drop-castings makes them more desirable than pancakes or buttons for development and planning purposes because a drop-casting may be scaled up more realistically to a production-size ingot to be made by industry.

**EXPERIMENTAL SECTION**

**Equipment Design**

The electron-beam furnace employed in this study is an original NRC Equipment Corporation design that has undergone considerable modification by ORNL. Although originally designed with one electron gun and one hearth, this furnace is now equipped with two electron guns and double hearths. Figure 1 shows the configuration of the furnace before modification, and Fig. 2 shows the furnace after modification. The double-gun and -hearth configuration allows two melts to be made without opening the furnace, thereby doubling the furnace throughput capacity.

The cylindrical furnace shell is constructed of 1/2-in.-thick stainless steel plate with inside dimensions of 23 in. in diameter and 60 in. in length. A water-cooled copper shield is used to protect the stainless steel shell from radiant heat and metal splatter. This shield can be readily removed from the furnace for cleaning when necessary.
Fig. 1. Electron-Beam Furnace. Original configuration.
Fig. 2. Electron-Beam Furnace. Modified configuration.
The furnace evacuation system, consisting of two 10-in. diffusion pumps, a 5 hp-30 cfm roughing pump, and two 1 hp-30 liter/min backing pumps, is capable of reducing the internal pressure to $1 \times 10^{-6}$ torr in approximately 20 min. Unless high outgassing rates are encountered during metal purification, this evacuation system is capable of maintaining a pressure of $5 \times 10^{-6}$ torr throughout the melting cycle. This high pumping capacity permits most of the volatile impurities to be removed during melting. Provision is made for backfilling the furnace chamber with a purified inert atmosphere when desired, in order to expedite the cooling of ingots.

A measure of the purification from interstitial impurity elements is shown by the chemical analysis in Table 1 of various refractory metals and alloys, before and after electron-beam melting. Figures 3 and 4 show the microstructures of electron-beam melted molybdenum and vanadium. Both these microstructures give a further indication of the internal cleanliness of metals after electron-beam melting.

Each gun chamber has a separate 4-in. short-series diffusion pump that evacuates the chamber to $5 \times 10^{-7}$ torr. Two locally fabricated focusing coils, spaced to give maximum spot intensity, and a yoke-type coil for beam deflection are in use on each gun column. These coils permit the operator to control the "spot size" and deflection of the electron beam.

### Table 1. Chemical Analysis of Interstitial Impurities Before and After Electron-Beam Melting

<table>
<thead>
<tr>
<th>Material</th>
<th>Before, ppm</th>
<th>After, ppm</th>
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<tbody>
<tr>
<td></td>
<td>C</td>
<td>O</td>
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<tr>
<td>Titanium</td>
<td>200</td>
<td>1090</td>
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<tr>
<td>Molybdenum</td>
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<td>710</td>
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<tr>
<td>Vanadium</td>
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<td>1260</td>
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<td>Zirconium</td>
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<td>Tantalum</td>
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<tr>
<td>Tungsten</td>
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<tr>
<td>Nb-1 wt % Zr</td>
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<td>71</td>
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<tr>
<td>Ta-10 wt % W</td>
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<td>29</td>
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<tr>
<td>W-26 at. % Re</td>
<td>&lt;20</td>
<td>59</td>
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Fig. 3. Microstructure of Electron-Beam Melted Molybdenum
Etchant: 50 ml NH₄OH and 50 ml H₂O₂.

Fig. 4. Microstructure of Electron-Beam Melted Vanadium. Etchant:
50 ml H₂O, 25 ml NH₄OH, 10 ml H₂SO₄, and 10 ml HF.
Being rated at 60 kw (20,000 v and 3 amp), the furnace has adequate power for melting most refractory metals and alloys. Cathodes are machined from electron-beam melted T-111 (Ta-8 wt %, W-2 wt % Hf) alloy. They are approximately 1 in. in diameter and 1/8 in. thick and have a concave face, for initial focusing, that is polished to a very smooth finish.

To accelerate the cooling processes and minimize contamination, melts are made in a variety of water-cooled copper mold shapes, which are of four basic designs: pancake, button, and finger for initial consolidation and purification; and either rectangular or cylindrical drop-casting molds for the final casting. Figures 5 and 6 show some

Fig. 5. Electron-Beam Furnace Pancake Mold.
of the molds that are in use. The button mold for drop-casting is split in half for easy removal from the skull that remains after casting, and a 1/2-in.-to 1-in.-diam hole is bored through the bottom of the mold to permit the metal to flow rapidly into a drop-casting mold placed directly below. The drop-casting apparatus, as shown in Fig. 7, consists of a cylindrical copper mold that is 2 in. OD and 3 1/2 to 6 1/2 in. in length. This mold is split to facilitate removal of the ingot and is clamped together for casting. During the casting operation this mold is located directly beneath the specially modified button mold.

By having the button mold walls and the walls of the drop-casting mold "head" describe an angle of approximately 60° with respect to the horizontal direction, the molten metal flows very rapidly into the drop-casting mold.
Fig. 7. Schematic Drawing of Drop-Casting Apparatus.

Drop-Casting Procedure

The original furnace design and associated hearths permitted the melting of only pancake shapes approximately 4 in. in diameter by 3/4 in. thick. Such ingots were not readily amenable to fabrication because of a heterogeneous grain structure, which results from this type of casting arrangement. Demands for high-quality rod and wire products necessitated the development of an electron-beam melting technique capable of producing ingot shapes amenable to swaging and wire drawing operations.

The technique that we developed encompasses the following steps: (1) purification and consolidation of melting stock into a button configuration, and (2) remelting of the button in a specifically designed button mold that permits a rapid and continuous flow of the molten mass
into a copper chill mold of the desired shape. The charge is drop-cast through a hole in the bottom of the chill mold.

Drop-casting differs in many respects from what is commonly referred to as "drip-casting." In drip-casting the material is melted in a button hearth with a hole in the bottom in such a manner that the molten metal runs slowly into the vertical mold below. This produces an irregular surface and an inhomogeneous internal condition.

In order to make a drop-casting, scrap and/or chops stock is first melted in a conventional button mold. Figure 8 shows normal starting
stock for electron-beam melting. By turning the button from side to side and remelting several times, homogeneous alloying is achieved even for the most hard-to-melt, difficult-to-alloy refractory metals. When consolidation, purification, and homogeneity of the melt are assured, the button mold is removed and the drop-casting apparatus is installed.

The electron beam, broadly focused, traverses the button until the button surface is completely molten. The beam is now "pinpointed" or concentrated while continuing to traverse the molten surface. The button now melts to deeper penetrations until the entire charge is molten except for a thin layer at the bottom. An increase in furnace power rapidly melts the bottom of the button and the molten metal is drop-cast into the vertical chill mold.

RESULTS AND DISCUSSION

The rapid drop-casting action prevents "coining" and cold shuts and gives an internally sound ingot having good surfaces. Because of their differential cooling rate from top to bottom, pancakes and buttons usually have shrinkage cavities in them. In a drop-casting, the body of the ingot solidifies before and at a greater rate than does the feeder head. This solidification sequence results in the elimination of shrinkage cavities from the body of the ingot. The feeder head, which contains the shrinkage cavity, is necessarily removed for subsequent remelting.

The internal structure of pancakes and drop-castings differ in many respects. Pancake configurations generally exhibit a fine-grained structure at the bottom and a much coarser grain structure at the top. This difference in grain size is attributed to the vast differences in solidification and cooling rates exhibited within these regions. The microstructure of a molybdenum pancake, illustrated in Fig. 9 shows the definite line of demarcation that exists between the grain sizes. In addition, grain boundaries extend through the entire thickness of some pancakes, as illustrated in Fig. 10 for tungsten. Drop-cast ingots are internally homogeneous and exhibit a uniform grain size throughout, as shown in Fig. 11 for molybdenum.
Fig. 9. Electron-Beam Melted Molybdenum Pancake Showing Large Nonuniform Grains. 3x. Etchant: 50 ml NH₄OH and 50 ml H₂O₂.

Fig. 10. Electron-Beam Melted Tungsten Pancake Showing Grain Boundaries That Extend Completely Through the Pancake. 3x. Etchant: 50 ml NH₄OH and 50 ml H₂O₂.

Fig. 11. Electron-Beam Melted Molybdenum Drop-Casting Showing Equiaxed Grains of Uniform Size. 3x. Etchant: 50 ml NH₄OH and 50 ml H₂O₂.
Because of large nonuniform grains, many pancakes break apart when an attempt is made to fabricate them into a usable shape. An example of the merit of drop-casting is exhibited by vanadium, which cannot be fabricated as a pancake but may be fabricated readily as a drop-casting.

Not only do drop-castings have a more desirable grain structure for fabrication but they are also in a configuration that is much easier to fabricate. A cylindrical drop-casting is readily amenable to swaging and drawing operations, while sheet, plate, or bar are easily fabricated from ingots having rectangular cross sections. Fabrication of rod or wire from pancake configurations requires a prior and costly sectioning operation. Further difficulties are encountered because the sectional strips are rectangular and contain a broad range of large grains and therefore are not readily amenable to the formation of cylindrical configurations. For many refractory metals this sectioning operation is fruitless because of excessive breakage along the grain boundaries of large grains.

Fabricability improvements afforded by drop-casting are also accompanied by a considerable gain in material yield. An average yield for drop-cast ingots is approximately 80% in the ingot condition, but almost 100% yield is attained on fabrication. A drop-casting must be overcharged by approximately 20% to allow for the skull and feeder head. Figure 12 shows a skull and head that have been removed from a drop-cast ingot. To increase the efficiency of the process, the material from both skull and head may be used as high-purity feed material for arc furnaces or may be recycled in the electron-beam furnace. Pancakes attain almost 100% yield on melting, but on fabrication the average yield is considerably lower.

Ingots up to 3/4 in. in diameter and 5 1/2 in. long of V, Nb, Mo, Zr, Ti, and Nb-1% Zr are routinely drop-cast. Refractory alloys such as W-26 at. % Re, W-35 at. % Re-18 at. % Mo, and Ta-10% W have been successfully drop-cast in ingots 1/2 in. in diameter and 2 1/2 in. in length. Figures 13 and 14 show a number of these castings.
Fig. 12. Skull and Head from Drop-Casting.
Fig. 13. Cylindrical Drop-Castings.
Fig. 14. Rectangular Drop-Castings.
CONCLUSIONS

The quality of electron-beam melted drop-cast materials is superior to that produced by other methods. Interstitial impurity levels are generally reduced to satisfy most requirements. The low impurity levels combined with the favorable grain structure obtained by drop-casting improves alloy fabricability. In addition, the shapes of the drop-cast ingots are readily amenable to fabrication methods such as swaging, rolling, and wire drawing. Electron-beam techniques promise to continue to play a prominent role in the development and technology of metals.

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