United States Patent Office 2,912,731 Patented Nov. 17, 1959

2,912,731

METHOD FOR CASTING GROUP IV METALS

Joseph B. Brennan, Cleveland, Ohio; Helen E. Brennan, executrix of the estate of said Joseph B. Brennan, deceased

No Drawing. Application August 2, 1956 Serial No. 601,628

2 Claims. (Cl. 22—212)

This invention relates to a method for preparing castings from metals of group IV of the periodic system of elements, such as hafnium, zirconium, thorium, titanium and their alloys. These highly refractory metals have found important uses in devices which operate at high temperatures, that is, temperatures above the service range of ordinary structural metals, such as steel. Group IV metals are highly reactive at elevated temperatures and readily combine with oxygen, water vapor and other materials, which results in embrittlement of the metal. Embrittled parts are undesirable in that they are difficult to machine and form with tools. So far as I am aware, no satisfactory casting procedure has been previously developed. It has been proposed to place titanium in a degassed crucible made from graphite or ceramic material and to melt the metal under vacuum. I have found, however, that this procedure is not adaptable to casting because of contamination from pick up of oxygen, hydrogen or carbon or other elements, apparently due to the prolonged period of contact with the mold surface containing such contaminants during the melting period.

The primary object of this invention is to provide a practical process for casting highly reactive metals, including hafnium, zirconium, titanium, thorium and their alloys, to produce castings free of embrittlement and contaminants picked up from the mold.

In accordance with my invention, the casting mold is preheated to at least a red heat, say 1070° F. to 2800° F., in vacuo of at least 25 microns to drive off any occluded gases, including oxygen, water vapor and other impurities, on or below the surface of the mold. The molten metal is then introduced into the preheated mold at a rapid rate, followed by immediate cooling of the mold and the casting therein. The cooling should take place quickly enough to form a solid skin on the surface of the casting in less than a minute. The casting and the cooling steps as well as the preheating step must be carried out under vacuum. Preferably, the cooling step is carried on concurrently as the metal is being cast, from the bottom up. It was surprising to find that group IV metals cast in accordance with this procedure were not embrittled since it is generally conceded that the rate of any chemical reaction increases with the temperature. Apparently, the step of preheating the mold in vacuo so effectively removes undesirable reactants from the surface of the mold cavity that the embrittling reaction is substantially eliminated due to lack of active contaminants. If minute quantities of undesirable reactants are present in the mold cavity, the combination between the metal and the reactant apparently is not extensive, probably because the reaction time is held to an absolute minimum by reason of immediate cooling.

Another surprising result of the method of the invention is that the casting may be easily removed from the mold. No undesirable adhesion occurs.

Molds suitable for use in the casting process of this invention must be made from a material which is essentially non-reactive at the melting temperature of the material being cast, at least during the time between casting and skin solidification. Highly refractory, relatively inert ceramic materials, such as quartz, zirconia, zirconia stabilized with magnesium oxide or pure dense graphite, have been found to meet these requirements. The latter two materials have been found to be best. The mold is placed in a vacuum chamber maintained at a vacuum of less than 25 microns. The preheating step may be conveniently effected by radio frequency electrical energy. The mold must be heated to at least a red heat, approximately 1070° F., and preferably higher, but not in excess of the melting point of the particular material being cast. Most group IV metals will melt in the range of 3200° to 3400° F. The preferable preheating temperature of the mold is around 2450° F. In addition to driving off occluded gases and contaminants on or below the surface of the mold cavity, the preheating step lessens the tendency to fracture, due to thermal shock, exhibited by the zirconia and magnesium oxide mold, for example. It is highly desirable that the contacting surface of the mold be finished to at least 70 microns surface smoothness (1 micron=.000001 meter limit of projection apparent on a surface).

The refractory metal, as for example titanium in rod or briquetted sponge or powder form, is melted and degassed in a suitable furnace and very rapidly poured into the mold which has been preheated as described above. A suitable pouring rate is at least one ounce per second. The titanium or titanium alloy or other metal has been previously degassed in vacuo at less than 25 microns. The melting and pouring steps are preferably carried out within a vacuum of less than 25 microns. Means for cooling the mold is also provided within the vacuum chamber so that the mold is cooled immediately progressively from the bottom up. In a preferred form of the invention, cooling begins immediately after the metal strikes the mold so that contact between the molten metal and the mold surface is made while the temperature thereof is in excess of 1070° F. to 2600° F., but immediately thereafter the temperature is reduced. The temperature of an outside portion of the casting in contact with the mold should be reduced by cooling from its melting point to below 2600° F. within one second of the initial contact of such portion therewith. In accordance with this procedure, clean sharp castings have been produced which are free of embrittlement.

Suitable apparatus for casting titanium and similar group IV metals in accordance with this invention are shown and described in my copending applications Serial No. 406,809 filed January 24, 1954, now Patent No. 2,858,586, granted Nov. 4, 1958, Serial No. 539,406 filed October 10, 1955, and Serial No. 586,627 filed May 27, 1956. In such apparatus the mold is advanced through an evacuated embracing sleeve, one zone of which is cooled by circulating fluid therethrough to accomplish cooling from the bottom up as the mold advances through said zone.

If desired, the mold and metal melt may be centrifuged in vacuo during filling in accordance with my copending applications Serial No. 406,809 filed January 24, 1954, Serial No. 545,261 filed November 7, 1955, and Serial No. 586,627 filed May 27, 1956. Centrifuging the preheated mold during filling permits completely filling the mold before a skin solidifies adjacent the wall of the mold cavity. This in turn permits fast melting and fast casting in a high thermal conductivity mold at a low temperature because the mold is filled before the skin solidifies in any area.

Example

One end of a ⅝" diameter rod of titanium alloy was

suspended in a 15 kw., 450,000 cycle tapered frequency coil surrounding such rod end and the rod end was melted as it was advanced within the coil. The molten metal was collected in a graphite pouring crucible preheated to 3300° F. by 15 kw. of 2,000 cycle energy under 25 microns of vacuum. The melting step was carried out under 10 microns of vacuum. Within 5 seconds the titanium melt in the crucible was poured into a graphite mold disposed therebelow and preheated to a temperature of 2600° F. in 25 microns of vacuo. The casting apparatus was designed to permit cooling the mold rapidly from the bottom up. The filling of the mold took about one second, and coolant was applied to the mold instantly. A skin formed on the casting adjacent to the cooled walls of the cavity in less than 40 seconds. During the melt of the rod the vacuum momentarily increased to almost 200 microns but in less than 50 seconds it was down to 25 microns and remained below this value during casting and cooling. The casting was readily removable from the mold and had a shiny, smooth bright surface.

The power was shut off on the rod melting coil immediately when the crucible therebelow had sufficient melt therein, usually in about 15–25 seconds for the above specified melt of about 5 ounces.

The mold below the pouring crucible or ladle may be moved out of position when filled and replaced and the cycle repeated in a succeeding mold.

The entire cycle is preferably automatically energized and timed with standard controls so that each step including melting, pouring, casting and cooling is accurately controlled.

This application is a continuation-in-part of my copending application Serial No. 344,237 filed March 23, 1953, now abandoned. Many widely differing embodiments of the invention will occur to those skilled in the art and it is not my intention to limit the invention to the specific details illustrated or described since various changes can be made without departing from the spirit and scope thereof.

I claim as my invention:

1. A method for casting a highly refractory group IV metal comprising preheating a mold to a temperature of between 1070° F. and the melting point of the metal to be cast, said mold being made from a refractory material stable at temperatures above the melting point of said metal, feeding said metal from a source outside said mold rapidly in molten form into said preheated mold while the surface of said mold is at said preheating temperature, and immediately cooling the mold to form a skin on the casting within less than 1 minute after the molten metal contacts the mold surface, said preheating, feeding and cooling steps being carried out under a vacuum.

2. A method for casting a highly refractory group IV metal comprising preheating a mold to a temperature of between 1070° F. and the melting point of the metal to be cast, said mold being made from a refractory material stable at temperatures above the melting point of said metal, feeding said metal at a rate of not less than 1 ounce per second from a source outside said mold in molten form into said preheated mold while the surface of said mold is at said preheating temperature, and immediately cooling the mold to form a skin on the casting within less than 1 minute after the molten metal contacts the mold surface, said preheating, feeding and cooling steps being carried out under a vacuum.

References Cited in the file of this patent

UNITED STATES PATENTS

1,251,951	Ashdown	Jan. 1, 1918
1,776,053	Voightlander	Sept. 16, 1930
1,930,408	Bailey	Oct. 10, 1933
1,946,450	Bailey	Feb. 6, 1934
1,966,615	Croning	July 17, 1934
2,361,382	Camin	Oct. 31, 1944
2,548,897	Kroll	Apr. 17, 1951
2,564,337	Maddex	Aug. 14, 1951
2,684,297	Urban	July 20, 1954
2,709,842	Findlay	June 7, 1955
2,713,183	Winkler	July 19, 1955

FOREIGN PATENTS

22,181	Great Britain	of 1895
266,305	Great Britain	July 21, 1927
698,303	Great Britain	Oct. 14, 1953

OTHER REFERENCES

The Iron Age, "Titanium," vol. 70, pub. Oct. 16, 1952, pp. 105–113.