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PROCESS FOR IMPREGNATING POROUS SOLIDS

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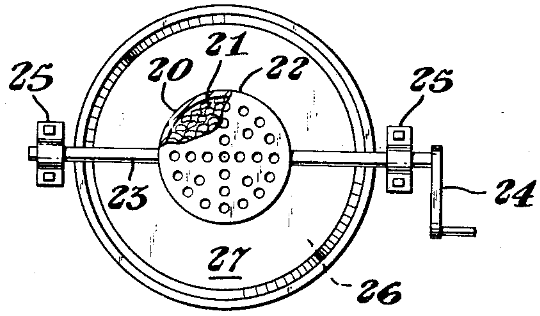

Fig. 3

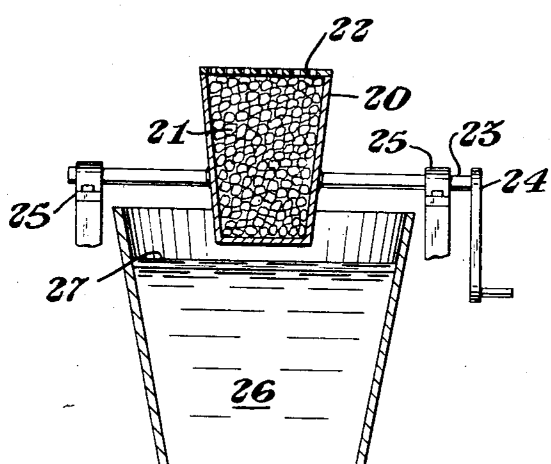

Fig. 1

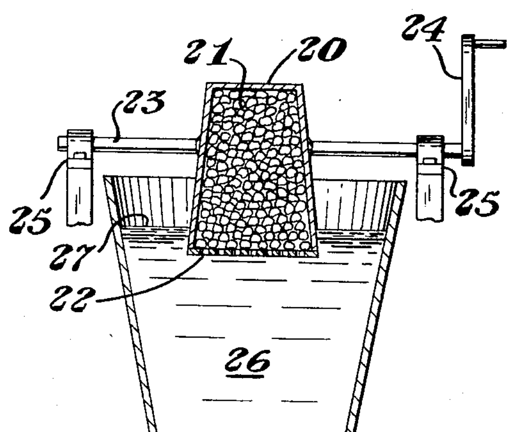

Fig. 2

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PROCESS FOR IMPREGNATING POROUS SOLIDS

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This invention relates to the impregnation of porous solids with metals and more particularly concerns a novel method of vacuum casting of metals into porous solids.

Casting of metals, particularly light metals such as magnesium, aluminum and alloys thereof, into void spaces has long presented problems. The commonly employed methods of gravity feed, pressure feed and vacuum feed all present great difficulty when attempts are made to fill spaces having small cross-sectional areas and irregular configuration. Even with the use of expensive equipment and controls, venting and the like, such methods are not suitable to impregnate the voids of porous solids. In recent years, a method has been devised for impregnating a porous material such as coke with a light metal such as magnesium by preheating the coke and plunging it into molten magnesium. However, this technique exposes a substantial portion of the surface of the molten magnesium to the air. The exposure of the molten magnesium to the atmosphere can produce burning and thereby requires the application of relatively large quantities of flux to eliminate or extinguish the burning. Also, exposure of the magnesium to the atmosphere and consequent burning produces MgO which increases the viscosity of the molten metal and adversely affects the impregnation of the coke. Likewise, the use of relatively large quantities of flux in the vicinity of coke causes contamination of the magnesium impregnated coke product causing it to be hygroscopic.

It is clear, therefore, that a need exists in the art for a simple and economical method for impregnating porous solids with metals which produces a more pure product and one which is not beset by the acknowledged problems of the art.

It is an object of this invention to provide a simple and economical method for impregnating porous solids with metals. A further object is to provide a novel process for impregnating porous solids with metals whereby a minimum of contamination from fluxes and metal oxides is obtained. These and other objects and advantages of the present process will become apparent from a reading of the following detailed description.

In carrying out the process of the present invention, a container is provided which has a single end open to the atmosphere and which is rotatable about a transverse axis between the open end and the opposing closed end. A quantity of porous solids of the desired particle size are added to the container and the open end is closed with a cover containing perforations which are smaller than the particles of porous solid. The covered container of porous solids is then positioned with the perforated end down and immersed in the molten metal a sufficient distance to assure that the molten metal will form a liquid seal thereby closing off the interior of the container from the atmosphere. One preferred method of accomplishing this is to position the container above the molten bath of a metal such that rotation of the container will bring the perforated cover below the surface of the molten metal. The container is then rotated to place the perforated end below the surface of the molten metal, and is allowed to remain in this position for a predetermined period of time. At the end of such period, the container is rotated to raise the perforated end above the surface of the molten metal. As the perforated end of the container moves upward, most of the molten metal which is not contained

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within the pores of the solid will drain out of the container through the perforations leaving the metal-impregnated solids within the container. These impregnated solids are then easily removed and the container may be recharged with additional porous solids for impregnation.

A better understanding of the present invention together with its attendant objects and advantages will be facilitated by reference to the accompanying drawings in which:

FIGURE 1 is a sectioned side elevation of the porous particle filled container positioned over the molten metal.

FIGURE 2 is a sectioned side elevation of the porous particle filled container inverted and in contact with the molten metal.

FIGURE 3 is a plan view of the covered container in upright position above the molten metal bath.

In the preferred embodiment illustrated in the drawings, container **20** is filled with porous solid particles **21** and covered with perforated cover **22**. Rod **23** is attached to opposing sides of container **20** at a point more than half way between the covered top **22** and the bottom of container **20**. Rod **23** is fitted with a rotation handle **24** and bears against bearing surfaces **25** to facilitate rotation of both rod **23** and container **20**. Molten metal **26** containing a thin covering of flux **27** and container **20** are positioned initially as in FIGURE 1 so that upon rotation of container **20** as shown in FIGURE 2, the perforated cover **22** will be below the surface of the molten metal **26** thereby sealing off the interior of the container **20** from the atmosphere.

Suitable porous solids for use in this invention include any normally solid particulate material having interconnected pores or void spaces and which is relatively dimensionally stable at the temperature of the molten metal which it must contact. Of particular utility are coke, sponge iron and compacts of various metals such as iron made by compressing irregularly shaped metal particles to form a cohered structure. Conveniently, a particle size of greater than about ¼ inch is employed.

The container used herein may be of substantially any geometry and made from any material having structural characteristics so as to be operable at the temperature of the molten metal which it will contact. Cylindrical or frustoconical containers made of iron or steel are readily available and relatively inexpensive so are usually preferred. To be useful herein, the container must have a single end open to the atmosphere. This end is fitted with a perforate cover which may be securely attached and which is removable. The perforations in the cover are smaller than the particle size of the porous particles but sufficiently large to permit easy and unrestricted flow of molten metal into and out of the vessel.

A means is likewise provided for rotating the vessel to a desired position. It is normally most convenient to load the vessel with porous solid while it is in the upright position, i.e., with the open end upward. After loading and covering, the vessel is inverted and immersed sufficiently below the surface of the molten metal to assure that all openings to the atmosphere are submerged below the surface of the molten metal. The molten metal thereby forms a liquid seal which prevents air from entering the vessel.

Metals suitable for use in the process of this invention to impregnate porous solids include magnesium, aluminum, sodium, potassium, lithium, strontium, barium, calcium, rare earth metals such as thorium or yttrium and mixtures and alloys thereof. These metals are employed in molten form at a temperature sufficient to provide the desired degree of fluidity.

To produce effective impregnation of the pores of the porous solid material, it is necessary that the pores be interconnected and that the atmosphere of such pores and of the interior space of the container be at least partially reactive with the molten metal which is to be introduced therein. For example, when magnesium is the molten metal, the oxygen and nitrogen of the air contained within the container and within the pores of the porous material will react with the magnesium to form small quantities of solid MgO and Mg_3N_2. This reaction creates a low pressure or a substantial vacuum within the container and within the porous particles whereby they become filled with molten magnesium. As the perforated end of the container is again rotated toward an upright position, the pressure returns to normal and the molten magnesium which has not infiltrated the pores of the porous material passes back through the perforations in the container cover and returns to the molten metal bath. The forces of surface tension retain the molten metal within the pores until the metal is cooled and solidified. In the same manner, molten aluminum will react with the air in the container and in the pores of the porous solid to create a vacuum and thereby produce impregnation.

The time required for metal to be drawn by the self-generated vacuum into the pores of the porous material will, of course, depend on the reactivity of the gas and the metal, upon the size of the pores, the temperature, and the like. However, impregnation is usually accomplished in from about 5 to about 15 minutes. Once impregnation is complete, the perforated end of the container is removed from the molten metal, excess molten metal is drained from around the impregnated solids, and impregnated solids are removed therefrom either before or after they are cooled to solidify the molten metal. The composite articles thus produced may be used as castings having particular characteristics or, in the case of those infiltrated with magnesium, may be used as treating agents to nodularize iron or desulfurize steel.

For impregnation with most metals, it is advantageous to heat the container and the porous material to about the temperature of the molten metal prior to contact with the molten metal bath. This prevents the possibility of premature freezing of the metal and usually improves the rate and degree of impregnation.

Most metals, and particularly magnesium, are subject to oxidation in the molten state when in contact with air. It is a usual practice, therefore, to cover the exposed surface of such molten metals with a protective layer or flux. For magnesium, typical fluxes consist of salt mixtures containing NaCl, KCl, $MgCl_2$ and minor amounts of alkali or alkaline earth fluorides. When impregnating the metals into a porous solid, however, it is desirable to have as little contmaination of the metal as possible. The process of this invention permits the use of such a flux and, at the same time, keeps contamination to a very low level. As the container is rotated into the bath, the edge of the container sweeps away a substantial portion of the flux and allows the perforations in the cover to contact relatively pure molten metal below the flux layer. However, direct immersion of the perforated end of the container into the metal after the parting the flux is also suitable and provides less contamination than presently available methods.

The following examples are provided to more fully illustrate the invention but are not to be construed as limiting to the scope thereof.

EXAMPLE 1

A two inch diameter container was provided made of black iron, having a welded bottom, an open top and an internal volume of 6.5 cubic inches. Into the container was placed 60 grams of coke having a particle size of about ¾-inch. A perforate cover having multiple openings of about ½-inch in size was then fastened to the container. Both the container and the coke were heated to about 1300° F. The container was positioned over a molten magnesium bath having a temperature of about 1300° F. and covered by a thin layer of standard flux (Dow 234). As positioned in the upright position, the bottom of the container did not touch the surface of the magnesium bath. As the container was rotated, the upper edge of the cover skimmed away a major portion of the flux in the area of the contact and the perforated end of the container was immersed well below the surface of the molten magnesium. The container was maintained in this position for a period of 5 minutes during which time the air in the container and in the pores of the coke reacted with the magnesium, creating a substantial vacuum whereupon the molten magnesium filled the container and the pores of the coke. At the end of this period, the container was rotated to the original upright position, the cover was removed, excess molten metal was drained from around the coke particles, and the impregnated coke removed from the container. The container was then filled with fresh coke and the process was repeated. Four batches of magnesium-impregnated coke were prepared in this manner. Examination of representaive pieces in cross-section revealed that the pores were substantially completely filled with magnesium. Analysis showed the product batches to contain 41, 44, 45 and 41 weight percent of magnesium, respectively. No detrimental contamination of the particles by the flux was found.

In a like manner, a two inch diameter container is filled with coke particles having a diameter of about ¾-inch and a cover containing ½-inch perforations is attached thereto. After attaching the cover, the container is inverted and directly immersed in a bath of molten magnesium. After draining of the excess magnesium from the container, the coke particles were found to be impregnated with magnesium.

In a manner similar to the above, particles of sponge iron and porous particles produced by compacting small irregular pieces of iron scrap or turnings were impregnated with magnesium and aluminum.

Various modifications can be made in the present invention without departing from the spirit or scope thereof for it is understood that I limit myself only as defined in the appended claims.

I claim:

1. A process for impregnating porous solids with metals which comprises
   (a) providing a container open to the atmosphere only at one end,
   (b) providing a molten bath of a metal,
   (c) introducing a particulate porous solid into said container and covering said open end with a cover containing perforations smaller than the particle size of said porous solid, said container cavity and the pores of the porous solid containing an atmosphere which is at least partially reactive with said molten metal,
   (d) immersing the perforated end of the container below the surface of the molten metal,
   (e) maintaining the perforated end of the container submerged in the molten metal for a predetermined period of time sufficient that the reactive atmosphere in the vessel and in the pores of the porous solid reacts with a portion of the molten metal thereby forming a reduced pressure and drawing the molten metal into the container and into the pores of the porous solid,
   (f) removing the container from the molten metal, and
   (g) removing the metal impregnated solids from the container.

2. The process as defined in claim 1 including the additional step of rotating the covered container to place the perforated cover below the surface of the molten metal.

3. The process as defined in claim 1 including the step wherein the container and the porous solid are heated to a temperature about that of the molten metal prior to contacting the perforated end of said container with said molten metal.

4. The process defined in claim **1** wherein the porous solid is coke.

5. The process as defined in claim **1** wherein the porous solid is sponge iron.

6. The process as defined in claim **1** wherein the porous solid is compressed into a compact of irregular iron particles.

7. The process as defined in claim **1** wherein the molten metal is magnesium.

8. The process as defined in claim **1** wherein the porous solid is coke and the molten metal is magnesium.

9. The process as defined in claim **1** wherein the molten metal is aluminum and the atmosphere in the container and in the pores is air.

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