Jan. 7, 1964

GRAIN REFINEMENT OF BERYLLIUM WITH TUNGSTEN
CARBIDE AND TITANIUM DIBORIDE

959

Filed Dec. 15, 1959

2 Sheets-Sheet 1

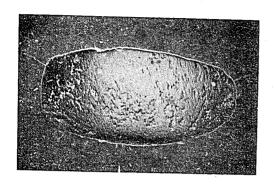
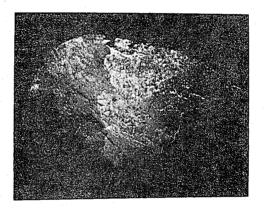
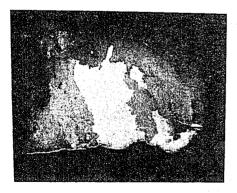


Fig-1

Fig-2



F1=3



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2 Sheets-Sheet 2

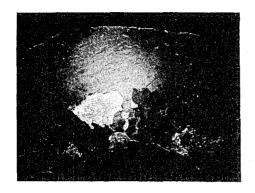
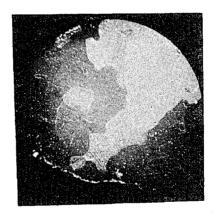


Fig-4





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## **United States Patent Office**

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3,117,001
GRAIN REFINEMENT OF BERYLLIUM WITH TUNGSTEN CARBIDE AND TITANIUM DIBORIDE Frank A. Crossley, Chicago, Ill., assignor to IIT Research Institute, a corporation of Hilnois Filed Dec. 15, 1959, Ser. No. 859,617 3 Claims. (Cl. 75—150)

The present invention relates to the grain refinement of as-cast beryllium and more particularly relates to an inoculation process whereby beryllium of fine grain structure is readily and conveniently produced. Even more specifically, my invention relates to the inoculation of molten beryllium with tungsten carbide or titanium diboride, or mixtures thereof to result in a fine grain structure in the as-cast metal

It is known by those skilled in the art to which the present invention most closely pertains that as-cast beryllium is characterized by a coarse grain structure, and that because of this, a melt thereof so prepared is characterized by poor physical and metallurgical properties. In fact, for example, the brittleness of the metal may be substantially directly correlated with the relative coarseness of the grains comprising the as-cast ingot. Not only does the brittleness result, but furthermore, there also are present serious strength defects. It is to the reduction of beryllium grain size and the refinement of the grains thereof, along with the physical and metallurgical improvements of the as-cast material that my invention is primarily directed, and as is set out in greater detail below, by practicing the invention as herein taught and claimed, marked improvements in beryllium structure and properties are readily achieved. Such improvements and the utility of my invention are illustrated in the photographs accompanying the present specification.

I have discovered that as-cast beryllium which is characterized by exceptionally fine grain structure and improved physical properties resulting therefrom may be produced by the inoculation of beryllium prior to the casting thereof with small amounts of tungsten carbide or titanium diboride or mixtures thereof. Such inoculating agents act as seeds for crystal growth phenomena as the molten beryllium cools from a liquid to a solid. Such seeds are selected as to be capable of being wetted by 45 the molten beryllium. The inoculants as hereinafter set out are added to the molten beryllium prior to the solidi-

fication thereof in the refined grain structure.

Accordingly, a primary object of my invention is to provide a process for the grain refinement of as-cast beryl- 50 lium.

A more specific object of my invention is to improve the grain structure of as-cast beryllium by inoculating molten beryllium metal with small amounts of tungsten carbide, titanium diboride, or mixtures thereof.

A further object of my invention is to provide a novel composition of matter, namely fine grain beryllium metal inoculated with small amounts of material selected from the group consisting of tungsten carbide, titanium di-

boride and mixtures thereof.

Other objects, features and advantages of my invention will become apparent to those skilled in this particular art from the following detailed disclosure thereof and the attached photographs which illustrate the beneficial results of my invention and wherein:

FIGURE 1 is a photograph of an unalloyed beryllium button showing a single beryllium grain in cross section. Such photograph was taken with polarized light after the button was etched in 10% hydrofluoric acid solution in 95% ethanol. The magnification is  $5.5\times$ ;

FIGURE 2 is a photograph of an unalloyed beryllium fragment illustrating the typical coarse grains found

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therein. The photograph was taken by polarized light, but in this particular instance the button was unetched. Magnification is  $5.5 \times$ ;

FIGURE 3 is a photograph of a beryllium button inoculated at the bottom surface thereof with titanium

diboride. Polarized light, unetched, 5.5×;

FIGURE 4 is a photograph of a beryllium button, inoculated at the bottom surface thereof with tungsten carbide. Polarized light, unetched,  $5.5 \times$ ;

FIGURE 5 is a photograph of the same material as set out in FIGURE 4 except that the magnification is 25 x.

Referring first to FIGURES 1 and 2, it is seen that the as-cast beryllium made in accordance with the teachings of the prior art has a coarse grain structure and is known to skilled metallurgists, such coarseness of grains is indicative of poor physical and metallurgical properties of the melt

In contradistinction to the coarseness of the grain structure of the unalloyed as-cast beryllium, referring next to FIGURES 3, 4, and 5 which disclose the inoculated beryllium, it is seen that there results a considerable improvement and refinement of the beryllium grains at the inoculated surface.

The inoculants may be added to the beryllium in the molten state in amounts from 0.1% to 3% by weight to provide the grain refinement improvements hereinabove indicated. I have most successfully employed such inoculants in particulate form of about —300 mesh. Ultrafine powders should not be employed since the materials should not completely dissolve in the molten beryllium if they are to act as seeds thereof.

The seed powders may be added to the molten beryllium in a number of ways. For example, in making the melts demonstrated in FIGURES 3-5, commercial grade beryllium pieces were first consolidated into a button by resistance melting. The button was then positioned atop the inoculant powder and the composite remelted again by resistance melting. This gave the results as illustrated.

Another means of inoculating comprises pouring the molten beryllium into a mold which is lined with inoculant powder. Other equivalent means are deemed within the scope of my invention.

It will be understood that modifications and variations may be effected without departing from the spirit or scope of the novel concepts of my invention.

I claim as my invention:

- 1. A composition consisting essentially of from 0.1% to 3% by weight of a material selected from the group consisting of tungsten carbide, titanium diboride and mixtures thereof, balance beryllium.
- 2. A composition consisting essentially of from 0.1% to 3% by weight of tungsten carbide, balance beryllium.
- 3. A composition consisting essentially of from 0.1% to 3% by weight of titanium diboride, balance beryllium.

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