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METHOD OF PRODUCING GERMANIUM PELLETS

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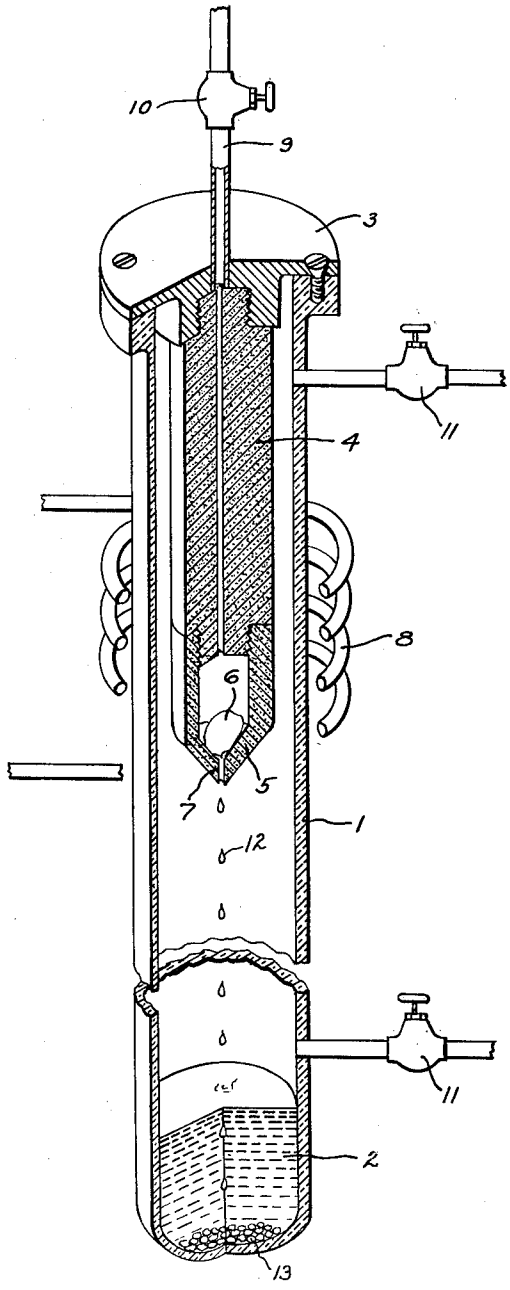

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METHOD OF PRODUCING GERMANIUM PELLETS

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2 Claims. (Cl. 18—47.3)

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My invention relates to methods for producing metallic pellets and more particularly to "shot-tower" methods for producing germanium pellets suitable for use in asymmetrically conductive devices, such as described and claimed in application Serial No. 134,826 of Harper Q. North, filed concurrently with the present application and assigned to the same assignee.

The above-mentioned North application discloses that tiny pellets of properly prepared germanium are suitable for use in asymmetrically conductive devices. Accordingly, a principal object of my invention is to provide an improved shot-tower method for quickly and prolifically producing such small germanium pellets of substantially uniform predetermined size and composition with minimum handling or care.

Another more specific object of my invention is to provide an improved shot-tower method for converting an ingot of highly purified germanium into tiny pellets with little danger of chemical contamination from undesirable impurities.

For performing my invention I provide a shot-tower apparatus which enables a continuous gas pressure to be supplied across an ingot of germanium while it is being melted within a crucible. This gas pressure ejects the germanium in the form of droplets through a small hole in the crucible just as soon as the germanium is melted with the result that the molten germanium is exposed to the contaminating influence of the crucible for only an extremely short period of time. In addition, I have found that by properly regulating the gas pressure with reference to the size of the crucible hole, the approximate size and rate of production of the pellets may be controlled to a considerable extent.

The novel features which I believe to be characteristic of my invention are set forth with particularity in the appended claims. My invention itself, however, together with further objects and advantages thereof can best be understood by reference to the following description taken in connection with the accompanying drawing in which the sole figure is a sectional view of a shot-tower apparatus for performing my invention and illustrating a germanium ingot in a partially melted state.

Referring to the drawing, I have shown, apparatus for performing my invention comprising a closed container 1 preferably in the form of a cylindrical tube of transparent material, such as quartz or glass, at the bottom of which a liquid bath 2 is located. Suspended within the upper end of the container 1 from a lid or cover 3 of

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the container 1 is a cylindrical hollow rod 4, preferably of graphite, which constitutes a gas conduit. Detachably secured to the bottom of rod 4 is a crucible 5 in which an ingot of germanium 6 may be placed. The crucible 5 is composed of a material, such as graphite which does not readily combine with molten germanium and has a small hole or aperture 7 at the bottom thereof. The sides of the crucible 5 leading to hole 7 are preferably tapered, as illustrated, and the size of the hole 7, as will be more fully explained hereinafter, determines to a great extent the size of the pellets that will be produced. Crucible holes in the neighborhood of .020 inch in diameter have been found to give excellent results although considerable variation of the diameter may be tolerated.

In order to heat the germanium without the contaminating contact of an adjacent heat element, induction heating means are preferably provided external to the crucible 5. In the instant preferred apparatus for performing my invention an induction heating coil 8 is arranged to surround the container in planar alignment with the crucible and functions in a well-known manner to melt the germanium ingot within the crucible by the application of a high frequency magnetic field therethrough.

Means are also provided whereby the entire system may be flushed with a chemically inactive gas, and the molten germanium within the crucible may be subjected to a controlled gas pressure to force the germanium out of the crucible hole 7 in the form of droplets comprising a stream or spray. A chemically inactive gas which may, for example, be dry nitrogen or helium which does not readily combine with molten germanium is fed into the container 1 through a gas inlet 9 containing a pressure controlling valve 10 and extending through the lid 3 to communicate with the gas conduit graphite rod 4. The crucible 5 is preferably secured to the gas conduit 4 such as by threading, as illustrated, so that the conduit completely covers the mouth of the crucible leaving the crucible hole 7 as the only outlet into container. Gas outlet valves 11 are preferably provided in the top and bottom of the container 1 in order to allow the entire container 1 to be flushed out by the pressure of the entering gas. It will be readily appreciated that the gas entering through inlet 9 will pass through the gas conduit 4 and through the crucible hole 7 into the container 1 in order to enable this flushing action.

In the operation of my invention, the prepared

ingot 6 of highly purified germanium is placed within the crucible 5 and the entire container flushed with a chemically inactive gas as described above. A high frequency electrical current of sufficient magnitude to melt the germanium within the crucible 5 is passed through the coil 8, and the gas pressure is regulated by adjustment of the inlet and outlet valves to a proper value as more fully explained below. As the germanium melts it runs down the sides of the crucible 5 and is forced out the hole 7 in the form of droplets 12 by the pressure of the applied gas. The droplets 12 are forced out of the crucible hole 7 in the form of a stream or spray and fall through the previously established chemically inactive atmosphere within the container 1 into the liquid bath 2 below. If the bath 2 is one foot or more below the crucible 5, the droplets 12 cool sufficiently as they fall so that they are not normally contaminated by the liquid bath 2 although distilled water is preferably employed as the bath in order to insure against such contamination. The droplets 12 are quickly quenched by the bath 2 and solidify into tiny pellets 13 of germanium, as indicated at the bottom of the container 1.

It is evident that because of the small size of crucible hole 7, the force of gravity alone is insufficient to overcome the surface tension of the molten germanium and to pull the germanium through the hole. For each size hole there is a certain threshold pressure which barely overcomes the surface tension in order to force the germanium through the hole. This threshold gas pressure ranges from a pressure of approximately 9 centimeters of mercury for a hole of .015 inch to a pressure of approximately 5 centimeters of mercury for a hole of .040 inch. I have found, however, that upon the application of a gas pressure immediately above this threshold pressure, the germanium droplet which is forced through the hole 7 tends to hang momentarily at the orifice and to grow slightly larger before it drops off, with the result that pellets are produced of variable diameter considerably larger than the diameter of the hole. As the pressure is further increased the droplets are blown through the hole more rapidly and the diameter of the resultant pellet becomes smaller and more uniform as it approaches the diameter of the hole. Once the size of the droplet becomes comparable with the size of the hole, any further increase of gas pressure merely increases the rate of production. It will, of course, be appreciated that the rate of production of these pellets is further limited by the speed of melting of the germanium ingot. With a strong heating

current causing rapid melting of the ingot and a high gas pressure, over 20,000 pellets of relatively uncontaminated germanium have been produced by this method within a few minutes after the germanium ingot begins to melt.

It is to be understood that while I have described a particular embodiment of my invention, many modifications can be made, and I, therefore, intend by the appended claims to cover all such modifications as fall within the true spirit and scope of my invention.

What I claim as new and desire to secure by Letters Patent of the United States, is:

1. The method of producing germanium pellets, which method comprises placing a germanium ingot within a crucible having a small hole at the bottom, passing under predetermined pressure a gas chemically inactive to germanium around the ingot and through the crucible hole, and then heating the germanium ingot above its melting point while it is subjected to the influence of the encompassing gas to form molten droplets of germanium blown out of the hole by the force of said gas while the germanium is melting.

2. The method of producing germanium pellets suitable for use in asymmetrically conductive devices, which method comprises placing an ingot of germanium within a crucible having a small hole at the bottom, forcing under predetermined pressure a flow of gas chemically inactive to germanium around the germanium and through the crucible hole, and then passing through the ingot a high frequency magnetic field of sufficient intensity to melt the ingot while the ingot is subjected to the encompassing gas flow, said gas flow forcing germanium through the hole while it is being melted to form germanium droplets having little chemical contamination from the crucible.

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