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[54] ORIFICE PLATE FOR SPINNING FINE DIAMETER WIRE

3 Claims, 1 Drawing Fig.

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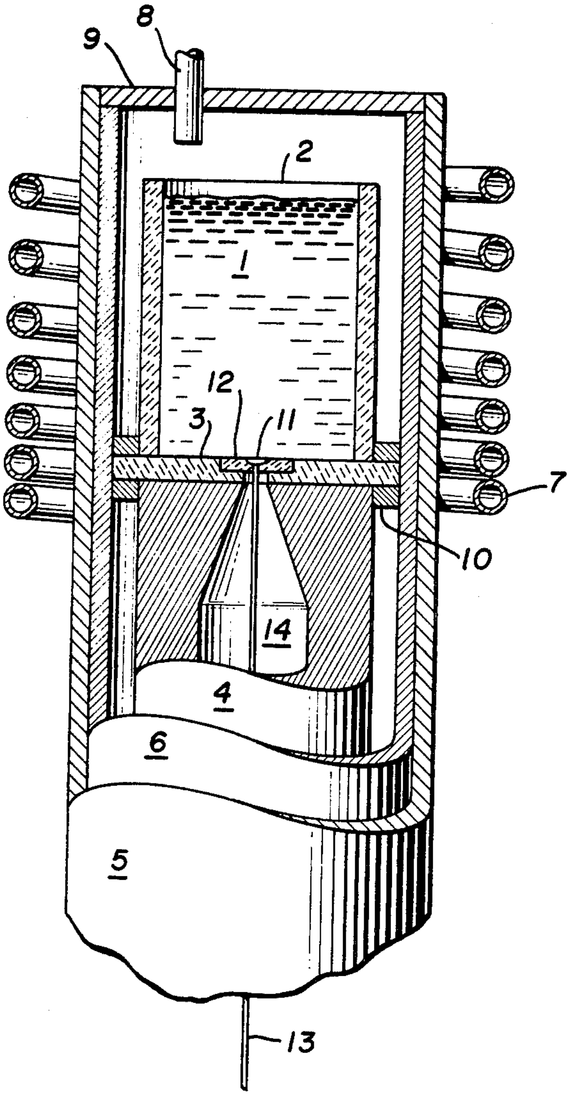
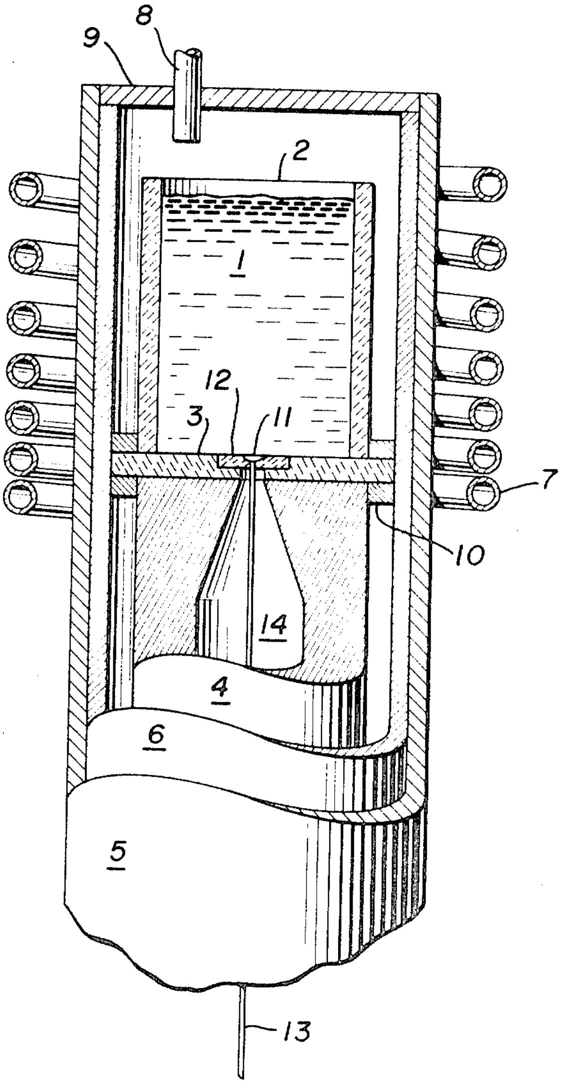
ABSTRACT: High density, high purity polycrystalline and single crystal beryllium oxide orifice plates have been successfully employed in wire manufacturing processes which comprise spinning molten metals through fine diameter orifices as molten metallic jets.





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ORIFICE PLATE FOR SPINNING FINE DIAMETER WIRE

This invention relates to improvements in the spinning of molten metals and alloys thereof.

More particularly, this invention relates to improvements in spinning processes and assemblies employed for melt spinning of metals and alloys thereof in the manufacture of fine diameter wire.

Processes for the formation of fine diameter wire by spinning molten metals through an orifice as a free molten filamentary stream have been disclosed in U.S. Pat. No. 3,216,076. According to these processes a molten filamentary metallic jet is spun into a film-forming atmosphere whereupon a thin stabilizing film is rapidly formed on the surface of the molten filamentary stream. The film negates the effects of surface tension and prevents breakup of the molten stream for a sufficient period of time to allow the molten stream to freeze or solidify in filamentary form. The apparatus employed to facilitate spinning of molten filamentary jets in forming fine diameter wire generally comprises a crucible having an orifice in its base, either as a part of the crucible base, or preferably, as an orifice insert. Typically, the crucible is provided with means for melting the metallic charge and maintaining a desired degree of superheat for the molten metal charge contained in the crucible and, additionally, means for applying a positive pressure to the head of the molten charge to force the molten metal through the orifice at desirable jet velocities.

FIG. 1 is a cross-sectional view of a representative unit useful for spinning fine diameter wire where a molten metal is contained in crucible 2 having base plate 3, the crucible and base plate being supported on pedestal 4 and enclosed within an insulating cylinder 5 and a susceptor 6 employed in conjunction with induction heating coils 7. The unit is pressurized within by a pressure source 8 through top 9. Sealing rings 10 serve to maintain the pressure within the enclosure and prohibit leakage past the base plate. The molten metal 1 is forced through orifice 11 in orifice plate 12 by the force of pressure supplied by pressure source 8 at sufficient velocities to provide a filamentary shaped molten jet 13. The nascent jet passes through a film-forming atmosphere contained within the cavity 14 generally provided by the pedestal 4.

Although materials science has developed at a rapid pace in recent years there nevertheless appears to be limited data reported on the strength, solubilities and chemical reactivities of materials at temperatures above 1000° C. Thus, in the manufacture of copper and steel wire, for example, directly from their respective melts considerable time and effort have been spent defining the nature and performance of materials used to handle molten metals at high temperature as well as the effect of molten metals on such materials.

Perhaps the most critical element of the spinning assemblies used to make wire according to the process outlined above is the member defining the orifice through which molten metal is spun under pressure. While the glass fiber art has developed its "bushings" and the synthetic fiber art its "spinnerets," little useful information has been reported in the literature relating to orifice defining members employed for spinning molten metals. Because of the nature of the charge and conditions employed in the manufacture of fine diameter wire (below about 35 mils) by melt spinning processes process, development has been seriously handicapped by repeated failure of the materials used to define the spinning orifice. Materials such as high density alumina, sapphire, zirconia (calcium oxide stabilized), $Al_2O_3 \cdot Cr_2O_3$ ceramic and others frequently employed to contain molten metals, such as steel, in metallurgical processes fail to perform satisfactorily as orifice defining members through which molten metals can be spun as fine diameter filamentary jets. The cause of such failures can be variously attributed to insufficient strength, low thermal fracture resistance and poor chemical stability under the conditions of temperature and pressure required to spin molten metals from orifices having diameters below about 35 mils. Such failures have been noted to be particularly aggravated at temperatures above about 1000° C. and with decreases in orifice diameter, particularly where the orifice diameter is below about 15 mils.

As already indicated the requirements for materials used to define fine diameter orifices in melt spinning processes are critical to the successful operation of prolonged spinning. Initially, the material must be resistant to thermal fracture generally caused by elevating the temperature and by temperature gradients across the face of the disc during the spinning operation. Insofar as it is desirable to employ orifices having aspect ratios of between about 1 and 20 the orifice defining plate or disc from which fine diameter wire is spun is usually quite thin in the vicinity of the orifice and yet its strength at the spinning temperature, of steel for example, must be of such magnitude that it can withstand the high pressure necessary to force the molten metal through the small orifice at reasonably high velocities. Cracked orifice defining discs caused by thermal fracture or by mechanical failure under pressure result in abortive operations in wire manufacturing process. Chemical reactivity or solubility with the molten metallic charge can result in erosion of the orifice or deposition of occlusions in the fine diameter orifice, neither of which can be tolerated in an industrial spinning process. Moreover, chemical reactivity of the ceramic material with graphite assembly parts presents serious problems. For example, both alumina and zirconia readily react with graphite at elevated temperatures.

Thus, a suitable orifice defining member of a spinning assembly must adhere to a combination of physical and chemical requirements.

While the property requirements of orifice plate are severe in high temperature spinning, the crucible assembly as a whole should adhere to the chemical reactivity and solubility requirements.

It has now been discovered that polycrystalline beryllium oxide having a density of greater than 95 percent of the theoretical density and purity of at least 99.5 percent and single crystal beryllium oxide serve well as orifice defining materials in the spinning of molten metals without significant problems of thermal or mechanical failure, chemical reactivity, or orifice erosion or occlusion due to the solubility characteristics of beryllia.

Plates or discs of single crystal or polycrystalline beryllia can be readily machined to suitable size, shapes by means known to those skilled in the art. Fine diameter orifices are generally provided by machining a countersink in the feed face of the orifice plate and thereafter drilling and polishing an orifice of desired diameter concentric with the countersink. Additionally, orifice plates composed of polycrystalline beryllia can be conveniently prepared by a bisque process wherein the plate is shaped and the orifice drilled before the final firing. The greenware plate is thereafter fired to appropriate densities, after which the orifice is polished to reduce minor crystal imperfection.

As above indicated the orifice defining member may additionally serve as the base plate of the crucible assembly. However, it has been found desirable to employ orifice plate inserts in the base plate of the crucible as indicated in FIG. 1. The insert type or orifice plate is conveniently circular and may, if desired, be secured in the base plate using a clamp or holddown ring. Multiple orifice insert discs may, of course, be employed in the base plate of the crucible. The orifice should have an aspect ratio of between 1 and 20, preferably less than 10, exclusive of the countersink and, although slightly tapered orifices have been used, straight bore orifices are preferred because of greater ease in fabrication.

Where spinning assemblies incorporating the beryllia orifice plates of this invention are used it has been found convenient to use beryllia crucible base plate members insofar as the chemical reactivity and solubility characteristics are suitable for high temperature spinning. Additionally, it has been discovered that beryllia crucible assembly members are inert to graphite parts of the spinning units.

The following examples illustrate the utilization of low viscosity melt spinning orifices constructed of beryllium oxide (BeO) and zirconium oxide (ZrO_2) for spinning molten metals.

EXAMPLE I

An apparatus, similar in construction to that depicted in FIG. 1, was employed to produce metal filaments from a composition comprising 99 weight percent type 304 stainless steel and 1 weight percent type 1345 aluminum.

The metal charge was placed in the crucible which had been prefitted with a beryllium oxide (BeO) orifice insert 12 having a tapered orifice construction wherein the taper, adjacent the melt, had an included angle of 12°. The orifice capillary diameter was 6 mils and an aspect ratio (length/diameter) of approximately 2.5.

After placing the metal charge in the crucible it was elevated in temperature to 1670° C. under the influence of a vacuum. Subsequent to melting an inert gas (argon) under 20 p.s.i.g. pressure was applied to the melt whereby streaming of the melt through the orifice was effected. Upon the initiation of streaming the melt temperature was reduced to 1620° C. (to reduce the amount of superheat).

The molten metal streamed into the cavity 14 which was occupied by an atmosphere of carbon monoxide (CO) whereby the molten stream was stabilized against breakup until solidification thereof took place. Very long filaments were produced having nodes spaced approximately at 8 to 10 inch intervals.

Subsequent to the completion of the spinning run the apparatus was cooled down and the beryllium oxide orifice insert was removed from the crucible whereupon it was microscopically examined for structural defects such as surface flaws and/or cracks and for orifice erosion. The examination disclosed no imperfections nor any measurable degree of orifice erosion.

EXAMPLE II

The orifice insert of example I was reinstalled in the apparatus thereof and an identical metal composition was melted and extrudes at a melt temperature of 1690° C. into an atmosphere of carbon monoxide. The melt extrusion pressure was increased to 30 p.s.i.g. and that in combination with the increase in the melt temperature provided extremely long filaments having nodes spaced at approximately 15 to 20 inch intervals.

After the spinning run was completed the beryllium oxide insert was again microscopically examined as in example I with no detectable structural failure nor measurable degree of orifice erosion.

The results obtained in example I and II were unexpected because thermodynamic data indicated that beryllia would dissolve in molten steel to such an extent that it would not be satisfactory orifice material.

EXAMPLE III

The spinning run of example I was repeated with the exception that the orifice insert 12 was constructed of zirconium oxide (ZrO_2) and included a tapered orifice inlet having a 27° included angle. The orifice capillary diameter was 7 mils and the orifice aspect ratio was 1.48.

Melt streaming was initiated through the orifice but ceased after 1 minute duration.

Subsequent microscopic examination of the orifice disclosed that plugging had occurred due to the formation of an oxide deposit therein.

EXAMPLE IV

The apparatus of Example I with the exception that a one-piece beryllium oxide crucible was employed for the production of copper filaments.

The crucible was provided with an orifice having a diameter of 4 mils and a capillary length of 6 mils which was fabricated in the base of the crucible.

The crucible along with a charge of electrolytic grade copper was placed in the melt spinning heat whereupon it was melted and extruded at a temperature of 1200° C. by means of an inert gas pressure of 40 p.s.i.g. into a stabilizing atmosphere of propane.

Subsequent to the complete melt extrusion of the copper charge the spinning apparatus was cooled down and disassembled whereupon the crucible and orifice were examined under a microscope. The examination revealed that no apparent erosion of the orifice had occurred.

EXAMPLE V

A single crystal ZrO_2 orifice insert was fabricated having a 4 mil diameter orifice with an aspect ratio of 5. The orifice insert was placed in the melt spinning apparatus and the assembly was elevated in temperature was maintained for 5 hours and after cooling down the equipment the orifice insert was removed therefrom and microscopically examined.

The examination revealed that the orifice was completely plugged with a clear crystalline material. The bottom of the insert in contact with the graphite had undergone severe reaction resulting in loss of gas pressure seals.

A similar study using $A1_2O_3$ single crystal orifice insert substantially the same conditions as above resulted in reaction with graphite to thereby destroy the gas seals and severely damaged the orifice exit.

I claim:

1. In an apparatus for spinning molten metals as filamentary shaped jets comprising a crucible assembly for containing a molten metallic charge, said assembly having an orifice defining member, means for forcing molten metal contained in said assembly through an orifice in said orifice defining member and heating means for maintaining said charge in a molten condition, the improvement wherein said orifice defining member is composed of single crystal beryllium oxide or polycrystalline beryllium oxide having a purity of at least 99.5 percent and a density which is greater than 95 percent of the theoretical density for beryllium oxide.

2. An orifice plate having a countersink on one face thereof and an orifice concentric with said countersink extending through said disc normal to the transverse plane thereof, said disc being composed of single crystal beryllium oxide or polycrystalline beryllium oxide being at least 99.5 percent pure and having a density of at least 95 percent of the theoretical density of beryllium oxide, said orifice being less than 35 mils. in diameter and having an aspect ratio of less than 20

3. The orifice defining member of claim 2 wherein the orifice has a diameter of less than 15 mils and an aspect ratio of less than 10.