

[54] **ELEVATION OF MELT IN THE MELT EXTRACTION PRODUCTION OF METAL FILAMENTS**

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[51] Int. Cl. **B22d 11/06**

[58] Field of Search..... **164/87, 276, 283 M; 264/8, 264/165**

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[57]

ABSTRACT

A method and apparatus for the production of metal filaments which are extracted using melt extraction techniques and quenched on a quench wheel, in which a discrete amount of the melt is elevated to contact the quench wheel thereby substantially increasing the quench rate.

12 Claims, 10 Drawing Figures

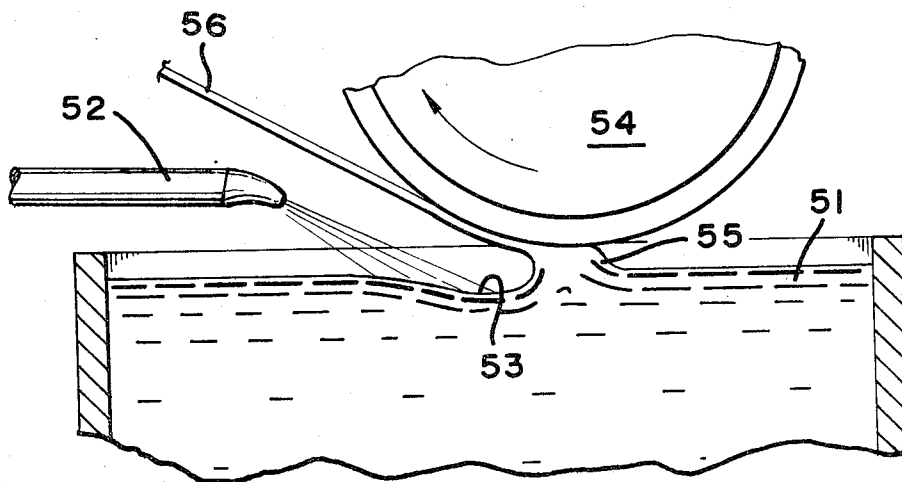


FIG. 1

PRIOR ART

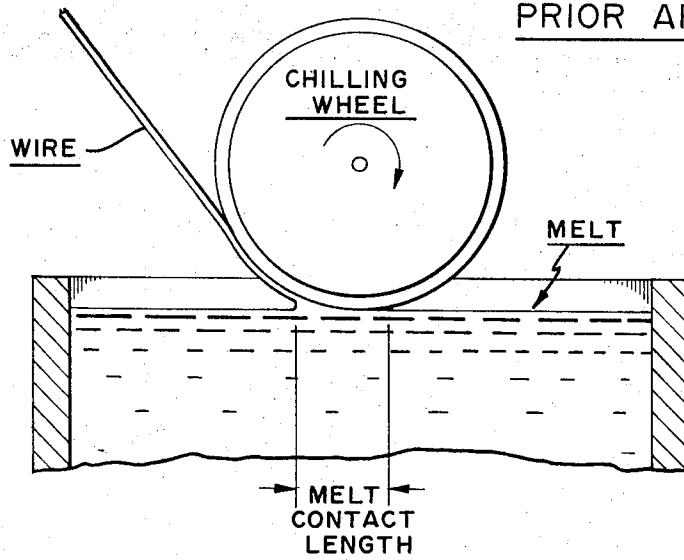


FIG. 2

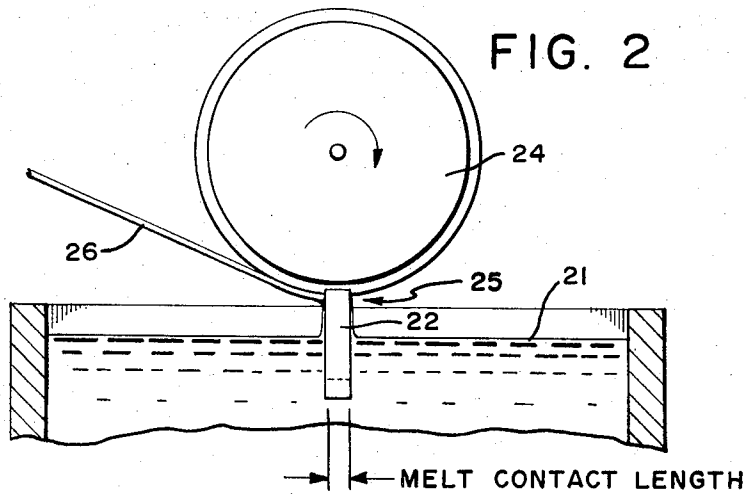


FIG. 2a

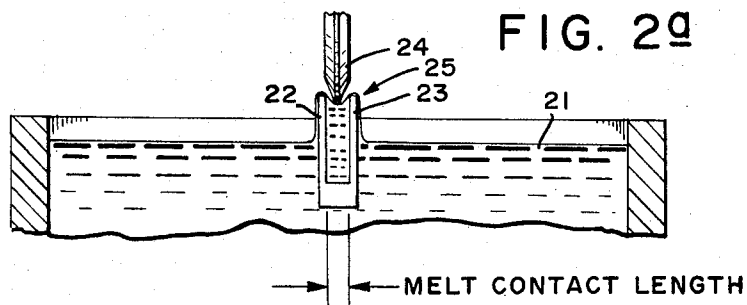


FIG. 3

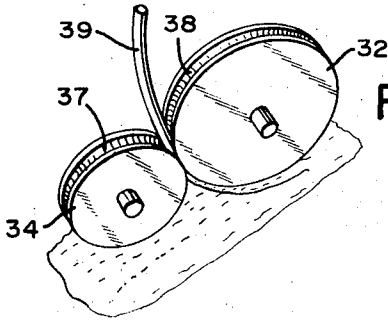
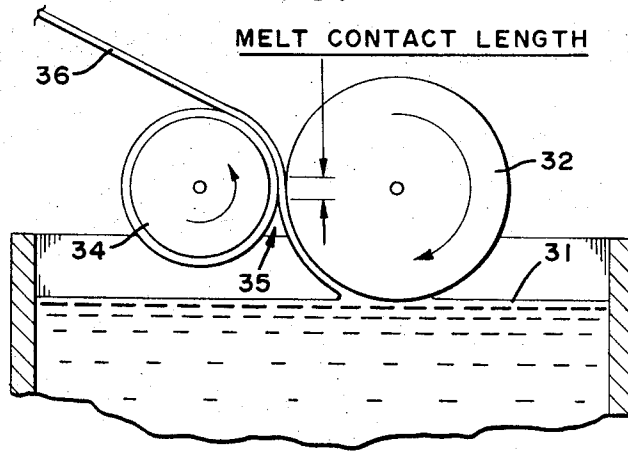


FIG. 3A

FIG. 4

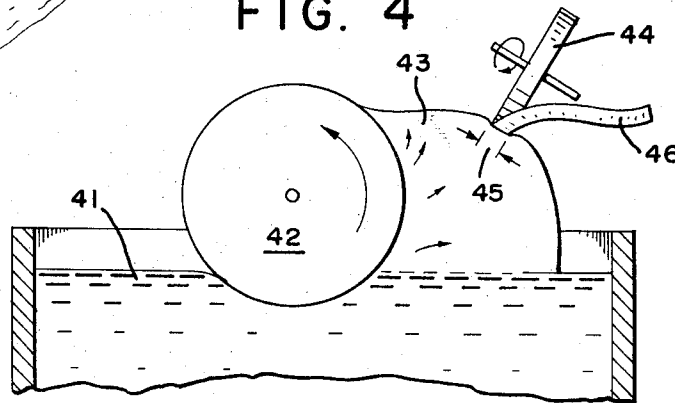


FIG. 5

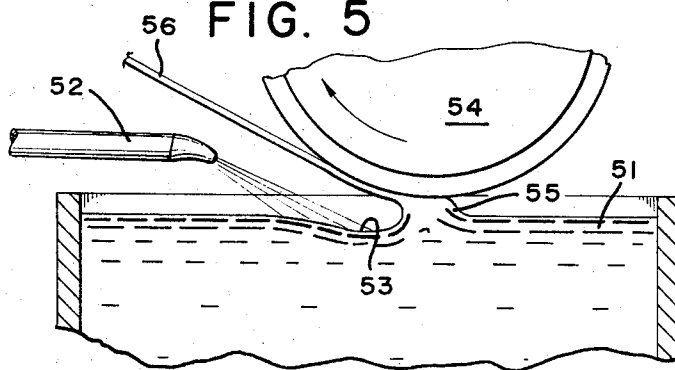


FIG. 6

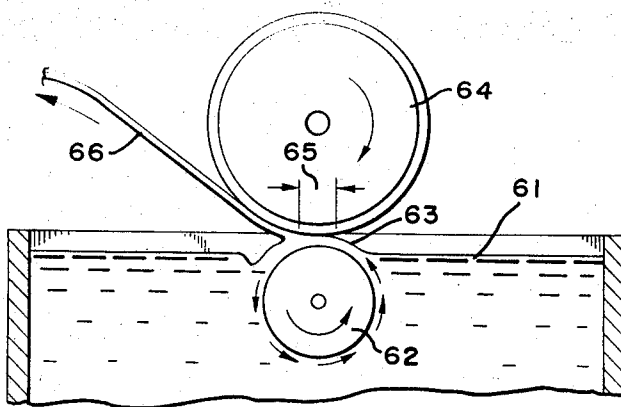


FIG. 7

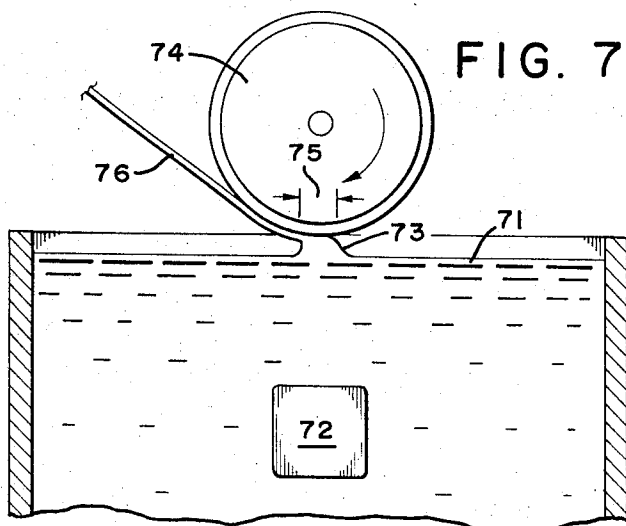
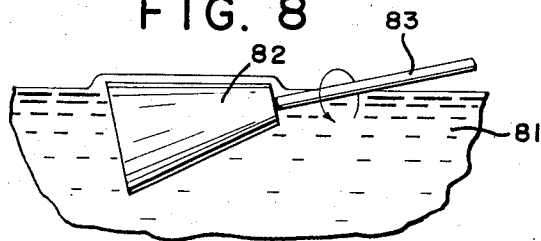


FIG. 8



ELEVATION OF MELT IN THE MELT EXTRACTION PRODUCTION OF METAL FILAMENTS

BACKGROUND OF THE INVENTION

Metal filaments may be produced by extracting from a molten metal bath and quenching on a chill or quench wheel. This invention is directed to an apparatus and method for improving the quench rate of the extracted molten metal by elevating the melt so that only a discrete amount of molten metal comes into contact with the quench wheel at any one time.

For purposes of the invention, the term "filament" is meant to include any slender metallic body whose transverse dimensions are much less than its length. These filaments may be ribbon, sheet or wire or may have an irregular cross-section.

Such filaments are presently formed by melt extraction techniques rather than by the previous casting or extrusion methods. Melt extraction is a process wherein a cold wheel rotates at high velocity in "kissing" contact with a liquid metal surface. The molten metal wetting or contacting the wheel is carried up out of the molten bath, solidifies, shrinks away from the wheel and is flung off by centrifugal action. This technique is to be distinguished from the essentially casting technique described in U.S. Pat. Nos. 1,025,848 and 2,074,812 in which a cold wheel is substantially immersed in the liquid metal and in which the rotational velocity of the wheel is appreciably lower than in the melt extraction system.

In this melt extraction system, contact between the quench wheel and the large surface area of the high temperature molten metal reservoir occurs over an appreciable distance as shown in FIG. 1. This extended contact causes some turbulence, sizeable exposure to melt and therefore considerable heat exchange between the wheel and the melt without adequate temperature reduction. The amount of heat absorbed from the melt by the wheel is well in excess of the necessary amount which must be absorbed from the discrete portion of the melt which is to be quenched to form the filament. This excess heat absorbed by the wheel reduces the quenching capability of the wheel thereby decreasing the effective rate of chill by the wheel in the area of molten contact. The efficiency of this thermal transfer can be expressed as

$$\text{Efficiency} = \frac{\text{heat removed by wheel from melt extract product}}{\text{heat removed by wheel from melt}}$$

Due to the low efficiency of the system and the excess thermal exchange, the metal often is not quenched at the desired rate or to the desired temperature before centrifugal and gravitational forces cause it to depart from the wheel. This incomplete quenching leads to products possessing some undesired properties including relatively large grain size in polycrystalline metals and some crystalline structure in otherwise amorphous metals. Thus, in the case of polycrystalline metals, rapid and thorough solidification is advantageous since it produces filaments of finer grain size with better attendant physical properties and also avoids the problems, such as embrittlement, associated with oxidation. Moreover, in order to achieve completely amorphous or glassy metals or ceramics, it is essential that the molten stream be quenched below the characteristic glass

transition temperature at a sufficiently high quench rate to avoid nucleation and growth of crystalline material in the amorphous structure.

It would be desirable to try to limit the thermal exchange between the extracted material and the chill wheel to only that amount required for adequately quenching the discrete portion of the material to be formed into a filament and thereby achieve a maximum quenching capability in the quench wheel. In order to do this, it is necessary to regulate to a minimum the amount of heat picked up from the melt by that quantity of molten metal being extracted for quenching; to regulate to a minimum the heat picked up by the quench wheel from the body of the melt; and to regulate the residence time of the melt on the wheel to that time required for adequate quenching of the extracted material. In an ideal situation, the thermal exchange would be confined to the heat exchanged between the quench wheel and the exact quantity of melt being quenched and there would be no heat exchange with the remaining body of the melt.

SUMMARY OF THE INVENTION

It is obvious that there is a need for a method to reduce the maximum thermal exchange in the melt extraction system.

It is therefore an object of this invention to provide a method of this kind for enhancing the thermal exchange.

It is a further object to provide a method which will increase the quench rate of the system.

Additionally, it is an object of the invention to provide a method which will substantially increase the quench rate so as to provide that the amorphous metal product be quenched below its characteristic glass transition temperature before departure from the wheel.

It is also an object of the invention to minimize the turbulence present during the melt extraction and subsequent freezing of the metal, thereby minimizing transfer of additional heat from the unfrozen melt to the wheel.

It is another object of the invention to provide an improved apparatus for the melt extraction and chill wheel quenching of metal filaments.

These and other objects will become apparent from the following description and examples.

These objects are accomplished in a melt extraction system wherein the quench wheel is positioned contiguous to the molten bath, by elevating a portion of the molten metal to make contact with the wheel in a more restricted zone, the melt contact length being correspondingly reduced, from that shown in FIG. 1 to that of FIG. 2, so only a discrete amount of molten metal comes into contact with the wheel at any one time, thus the capability of the wheel to quench, as well as the effective quench rate of the filament on the wheel is thereby substantially increased.

In accordance with the present invention, the melt may be elevated in a variety of ways. Included among these are elevation by using a hot wheel which draws the melt into contact with the chill wheel at a position remote from the molten bath or by use of capillary action, submerged wheels, gas jets, gas bubbled through the melt, or pumps. Any of these devices for elevating a discrete portion of the melt can be readily adapted for use in conventional melt extraction apparatus to pro-

duce an improved apparatus for melt spinning of metal filaments.

When the melt is elevated, the distance between the molten bath and the quench wheel is increased, and the amount of melt coming into contact with the wheel at any one moment is decreased. These factors of distance and reduced contact combine to minimize the thermal exchange, thus permitting the quench wheel to effectively act on a small quantity of melt.

In the case of polycrystalline metal products, when the quench rate is increased in this manner, cooling can be controlled to assure sufficient quench temperature to achieve a product possessing the desired properties. In particular, when polycrystalline metal filaments are formed using the method of this invention, the resulting filaments are of very fine grain size in the range of about 0.01 micron to 1 micron. Such fine grain size gives a material possessing superior physical properties. By way of example, the ductility of the narrow filament is improved since one grain does not bridge as much of the filament diameter as in larger grained filaments. Thus the movement along the slipplanes of the grain does not cause total yield to deformation or breakage through a single grain. Most importantly, when the procedure of the present invention is followed, amorphous metal filaments can be readily produced since the cooling of the amorphous metal at the required quench rate is assured.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 represents the effective melt contact length of the chill wheel extraction technique as it is currently practiced.

FIG. 2 illustrates the use of capillary action to raise the melt. This figure also illustrates the decreased melt contact length achieved using the novel method of the present invention.

FIG. 2a is a side view of the system of FIG. 2.

FIG. 3 represents an embodiment of the invention employing a hot wheel to elevate the melt.

FIG. 3a illustrates a method of contouring the wheels to produce a shaped metal filament.

FIG. 4 illustrates an alternate positioning of the chill wheel, particularly useful in the production of metallic ribbons or sheets.

FIG. 5 is a diagram of the action of gas jets to raise the molten metal.

FIG. 6 illustrates a rotating wheel submerged within the melt.

FIG. 7 represents a pump situated within the melt.

FIG. 8 is a representation of a drum which is rotated by an inclined drive shaft.

PREFERRED EMBODIMENT

The following embodiments are suggested means for elevating the melt and the invention in its broadest scope is not intended to be limited to these particular methods.

The melt may be elevated by capillary action. One of the methods for producing this action is shown in FIGS. 2 and 2a. In this embodiment, the melt 21 is elevated between two solid members 22 and 23 which are wetted by the melt to form an elevated concave meniscus; the quench wheel 24, positioned contiguous to the bath, then runs between the two solid members thereby contacting only that discrete portion 25 of the melt

which is raised and quenching this portion to form a filament 26.

In addition to the advantages inherent in this invention, the use of capillary action is advantageous in that it maintains a fairly constant height of liquid to be contacted by the wheel, thus stabilizing the filament size against effects caused by changes in the melt level such as those due to turbulence or to fluctuations in melt volume. The use of capillary action also permits the molten material to be raised from the melt thereby reducing the complications of thermal geometry introduced by the wheel. For example, when high melting ceramic liquids, such as yttrium-alumina-garnet are spun, the high temperatures required for melting suggests a lid on the crucible to conserve against radiation heat losses. A wire or ribbon arrangement, which serves to produce a capillary column, thereby raising the melt to the wheel, also can be used to generate heat by electrical resistance in order to keep the melt in a molten state.

Another way to raise the molten metal from the melt before contact with the quench wheel is shown in FIG. 3. Here, a hot wheel 32 is employed to extract and raise the molten metal from the melt 31, thereby retaining a layer of molten metal at its periphery. This molten layer is then conveyed to a quenching wheel 34 where a discrete portion 35 is rapidly quenched and then spun off as a solid wire or ribbon 36. The hot wheel of this embodiment is preferably made of a material which is wetted by the melt. Otherwise, some means may be needed to retain the molten metal at the surface of the hot wheel; such methods would include a split through the wheel conveying a vacuum to the zone of contact with the metal or else shaping of the hot wheel to permit it to carry molten metal as by capillarity.

There are additional advantages inherent in the particular embodiment of the invention. If the hot wheel surface is of low thermal conductivity, low heat transfer between the metal to be frozen and the hot wheel will occur and there will be even more rapid quenching when the metal reaches the quench wheel. Moreover, as shown in FIG. 3a, it is possible to achieve shaping of both sides of the wire product by contouring both the hot wheel 38 and the quench wheel 39 faces in a complementary manner so that the entire metal filament can be "molded" into the desired shape. In particular, this contouring could be adapted to produce round wire products. In addition, it is possible to "meter" the proper quantity of molten metal which is to be quenched by adjustment of parameters at the hot wheel, i.e. surface contour, wheel temperature, wheel velocity and wheel diameter. Proper metering is useful in controlling uniformity and shape of the chilled product. Optionally, the hot wheel 38 may be heated using a torch, induction coil, or the like to facilitate the wetting of the wheel during the initial stages of the operation. Once filament formation has begun, the heat from the molten reservoir will be sufficient to insure adequate wetting and no additional heat is needed.

FIG. 4 shows another embodiment of the hot wheel elevating technique, this embodiment being particularly adaptable to the production of metallic sheets though it is obvious that all the techniques herein described can be used to produce any filamentary form. In this situation, a discrete sheet or fountain of metal 43 is elevated by means of one or more rotating hot wheels 42 which are slightly more immersed in the melt

than the wheel of FIG. 3. As the molten metal 43 fans out to the side of the wheel, a second wheel 44, positioned approximately perpendicular to the sheet and serving as the quench wheel, then contacts the elevated metal along a very narrow length 45 thus producing the desired product 46.

Other methods of elevating the melt in a discrete region include bubbling gas up through the melt or directing at least one gas jet 52, as shown in FIG. 5, against the melt surface causing a depression 53 as well as a rise 55 in the surface sufficient to force the melt into contact with the quench wheel 54 to produce filament 56.

FIG. 6 illustrates another alternative. Here, a hot wheel or drum 62 is submerged at such a position in the melt 61 that when it rotates, it drags a layer 63 of melt over the upper surface and above the level of the molten reservoir. The quench wheel 64 then contacts the melt in a narrow contact length 65 to produce the desired product 66.

In FIG. 7 an insulated pump 72 located within the melt 71 is used to elevate a surge or discrete amount 73 of molten metal into contact with the quench wheel 74 to form the filament 76. A large variety of pumps may be employed, in particular those of the induction or electromagnetic thrust variety.

FIG. 8 illustrates a manner in which the rotating submerged wheel 62 of FIG. 6 or the submerged pump 72 of FIG. 7 can be driven from above the melt without gears, belts or other linkages but rather by using an inclined drive shaft 83. In the embodiment illustrated, a drum 82 is shown which has a conical shape so that the uppermost surface is horizontal. However, it is apparent that a similar mechanism could be employed to drive a mechanical pump.

The following are illustrative examples and the invention in its broadest scope is not intended to be limited thereto. Parts are in atomic percent unless otherwise specified.

EXAMPLE 1

Using the apparatus of FIG. 4, a Type 304 stainless steel wheel, 5.08cm in diameter by 1.7cm thick was mounted on a 0.96cm horizontal stainless steel shaft, and, while rotating at 40 to 50 revolutions per minute, was lowered approximately 0.32cm into a bath of molten solder alloy, consisting of 65 weight percent lead, 30 weight percent tin and 5 weight percent antimony, at $320^{\circ}\text{F.} \pm 20^{\circ}\text{F.}$, the bath being overlaid with a fluxing skin of boric anhydride powder. Initially, some of the bath was chilled by the wheel, and a frozen layer of solder became attached to the wheel. With continued rotation and absorption of heat from the bath the temperature of the wheel increased until the frozen layer has melted back into the bath.

The wheel was then lowered to an immersion depth of about 1.7cm and the rotation speed of the wheel was increased until molten solder was carried up from the melt and flung outward in a thin sheet from the mid line of the periphery of the wheel, on the upward-travelling side of the wheel. The sheet of molten metal thus generated was 0.17 to 0.254cm thick, and was elevated 2.54 to 4.2cm above the melt surface.

The elevation of the melt was thus maintained while a second wheel was introduced to the system. This second wheel was of copper, 1.7cm thick by 10.16cm in diameter, rotating on its circular axis at the end of a

shaft on a flexible driving cable. The copper wheel, which was initially at room temperature, was rotated at about 1,500 revolutions per minute and, by holding its shaft manually, its peripheral face was brought into contact with the sheet of molten metal near to a point of maximum elevation of the metal. The copper wheel, when in this position, has its axis in a perpendicular direction to the direction of the axis of the stainless steel wheel which was partially immersed in and elevated the molten solder alloy. The axis of the copper wheel also was approximately in the plane of the sheet of molten elevated alloy. The plane of rotation of the copper wheel was approximately perpendicular to the average path of travel of molten metal in the vicinity in which the copper wheel contacted the molten alloy. The copper wheel intersected the outer edge of the molten sheet by approximately 0.32cm. As the path of the molten metal in the molten sheet was intersected, that portion of the molten metal whose path was intersected by the body of the copper wheel became frozen at the copper wheel's peripheral surface and was carried perpendicularly from its prior free-falling path, temporarily attached to the copper wheel. This frozen material, in the form of a solid metal ribbon, then departed from the copper wheel after travelling 1.7 to 7.6cm from the place of contact between the molten sheet and the copper wheel. Continuous metal ribbons of approximately ten to twenty foot lengths were thus generated, which were 0.025 to 0.046cm thick; they were shown, using x-ray diffraction techniques to possess very fine grain size.

EXAMPLE 2

Using a tungsten capillary and an apparatus similar to that of FIG. 2, copper was charged and melted to $1,180^{\circ}\text{C.}$ in an atmosphere containing 5 percent hydrogen and 95 percent argon. The molten metal wetted the capillary and formed a concave meniscus within the capillary. A rotating copper wheel was allowed to contact and quench the elevated portion to form a fine grained polycrystalline copper filament.

EXAMPLE 3

Another alloy, formulated to be amorphous on quenching, and comprising 48% Ni, 30% Fe, 14% P, 6% B and 2% Al, was melted at $1,020^{\circ}\text{C.}$ in a melt extraction system similar to that depicted in FIG. 6. Gaseous jets of argon were impinged upon the area of contact between the wheel and the molten reservoir at a flow rate of $3 \text{ m}^3/\text{min.}$ through a .63cm orifice, causing only a discrete amount of melt to contact the rotating copper quench wheel. Upon analysis, the resulting filament was found to be completely amorphous.

EXAMPLE 4

A gray cast iron alloy containing about 3.4% C, 2.2% Si, 10.6% Mn, 0.2% P and 0.1% S was melted at $1,200^{\circ}\text{C.}$ in an apparatus similar to that depicted in FIGS. 6 and 8. A conical shaped alumina drum was submerged within the molten reservoir, rotated using an inclined drive shaft, thereby producing an elevated dragged metal film which contacted a rotating copper quench wheel which was driven in a direction opposite to that of the submerged drum. The resulting filament was analyzed and found to be of very fine grain size.

EXAMPLE 5

An alloy formulated to be amorphous on quenching and containing 76% Fe, 15% P, 5% C, 3% Al and 1% Si was charged in an insulated reservoir and melted in vacuum at 1,100°C. A ceramic tube was placed in the melt through which 15 gm/cm² of gaseous argon was bubbled, thereby raising a discrete portion of the molten metal into contact with the quenching wheel. Discrete segments of amorphous metal were thus produced.

EXAMPLE 6

In a device similar to that of FIG. 7, an alloy formulated to be amorphous and containing 47% Ni, 30% Fe, 14% P, 6% B, 1% Si and 2% Al was melted at 1,000°C. An insulated induction coil submerged in the melt and pumping with a force of 5gm/cm² elevated a portion of the molten metal a height of about 0.60cm to contact the quench wheel. X-ray diffraction analysis showed the resulting filament to be totally amorphous.

EXAMPLE 7

A solder alloy was charged and heated as in Example 1. A heated contoured steel wheel, similar to that shown in FIGS. 3 and 3a was rotated so as to extract a small stream of the molten alloy from the reservoir. The molten alloy remained in the groove on the surface of the hot wheel and was raised so as to contact a correspondingly contoured copper quench wheel. The metal filament ejected from the quench wheel was circular in shape with a diameter of 0.03cm and x-ray diffraction analysis indicated fine grain size.

We claim:

1. In a melt extraction method for the production of metal filaments from a molten reservoir wherein a quenching wheel is positioned contiguous to the melt and extracts a filament directly from the melt reservoir, the improvement comprising elevating a discrete amount of the melt directly from the melt reservoir to contact the quenching wheel by means of an auxiliary rotating hot wheel which is partially immersed within the melt thereby utilizing substantially the entire quenching capacity of said quenching wheel to quench said discrete amount of the melt.

2. The method of claim 1 wherein the rotating hot wheel and the quench wheel are complementarily contoured so as to produce a correspondingly shaped metal filament.

3. In an apparatus for the production of metal filaments from a molten source using a quenching wheel as a quenching element, the improvement which comprises employing a rotating hot wheel which is partially immersed within the melt to elevate a discrete amount of the molten melt into contact with the quench wheel.

4. The apparatus of claim 3 wherein the hot wheel and quench wheel are complementarily contoured.

5. In a melt extraction method for the production of metal filaments from a molten reservoir wherein a quenching wheel is positioned contiguous to the melt and extracts a filament directly from the melt reservoir,

the improvement comprising elevating a discrete amount of the melt directly from the melt reservoir to contact the quenching wheel by bubbling gas through the melt contiguous to said quenching wheel, thereby utilizing substantially the entire quenching capacity of said quenching wheel to quench said discrete amount of the melt.

6. In a melt extraction method for the production of metal filaments from a molten reservoir wherein a quenching wheel is positioned contiguous to the melt and extracts a filament directly from the melt reservoir, the improvement comprising elevating a discrete amount of the melt directly from the melt reservoir to contact the quenching wheel by contacting the melt surface with a gas jet, thereby utilizing substantially the entire quenching capacity of said quenching wheel to quench said discrete amount of the melt.

7. In a melt extraction method for the production of metal filaments from a molten reservoir wherein a quenching wheel is positioned contiguous to the melt and extracts a filament directly from the melt reservoir, the improvement comprising elevating a discrete amount of the melt directly from the melt reservoir to contact the quenching wheel by a pump located within the body of the melt, thereby utilizing substantially the entire quenching capacity of said quenching wheel to quench said discrete amount of the melt.

8. In a melt extraction method for the production of metal filaments from a molten reservoir wherein a quenching wheel is positioned contiguous to the melt and extracts a filament directly from the melt reservoir, the improvement comprising elevating a discrete amount of the melt directly from the melt reservoir to contact the quenching wheel by a rotating hot wheel which is submerged within the melt, thereby utilizing substantially the entire quenching capacity of said quenching wheel to quench said discrete amount of the melt.

9. In an apparatus for the production of metal filaments from a molten source using a quenching wheel as a quenching element, the improvement which comprises employing a gas bubbling means to elevate a discrete amount of the molten melt into contact with the quench wheel.

10. In an apparatus for the production of metal filaments from a molten source using a quenching wheel as a quenching element, the improvement which comprises employing a gas jet to elevate a discrete amount of the molten melt into contact with the quench wheel.

11. In an apparatus for the production of metal filaments from a molten source using a quenching wheel as a quenching element, the improvement which comprises employing a pump to elevate a discrete amount of the molten melt into contact with the quench wheel.

12. In an apparatus for the production of metal filaments from a molten source using a quenching wheel as a quenching element, the improvement which comprises employing a rotating hot wheel which is submerged within the melt to elevate a discrete amount of the molten melt into contact with the quench wheel.

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