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[54] **PROCESS FOR CONTROLLING ORIFICE SIZE WHEN EXTRUDING MOLTEN MATERIALS**

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[52] **U.S. Cl.** **164/66,** 164/82

[51] **Int. Cl.** **B22d 11/12,** B22d 23/00

[58] **Field of Search** ....164/66, 67, 82, 259; 264/176 F

[56] **References Cited**

UNITED STATES PATENTS

2,907,082	10/1959	Pond	164/66
2,976,590	3/1961	Pond	164/82
3,216,076	9/1965	Alber et al.	164/82 X

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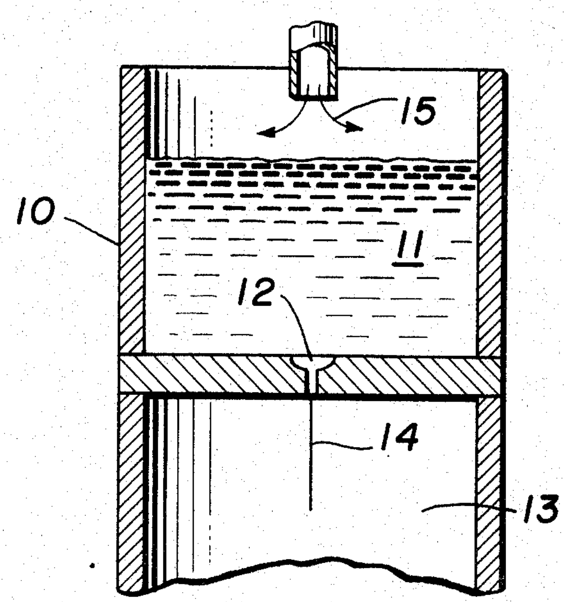
[57] **ABSTRACT**

The size of the orifice in a process for extruding low viscosity melts is controlled by maintaining at a predetermined level the partial pressure of a gas which enters into chemical reaction with the molten material contained within the crucible assembly, the materials comprising the crucible assembly, and the impurities contained in the melt.

4 Claims, 4 Drawing Figures



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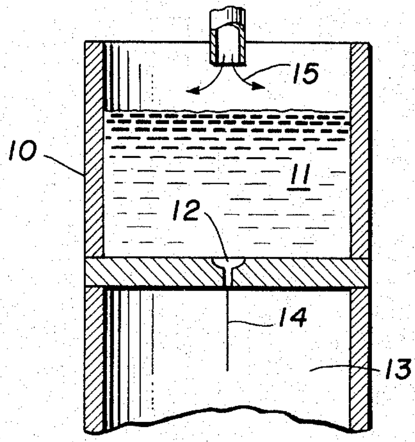

FIG. 1.

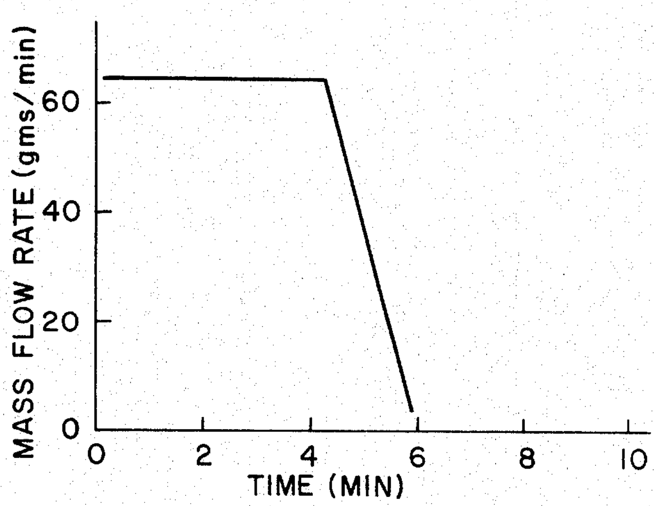

FIG. 2.

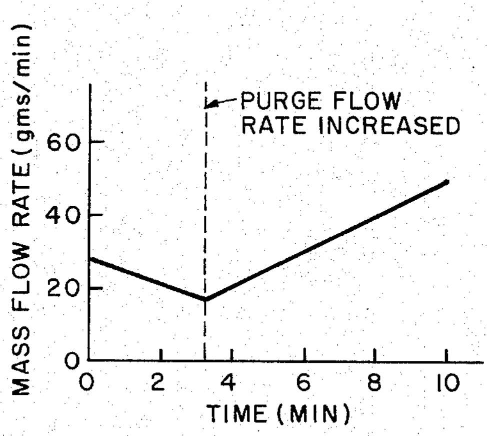

FIG. 3.

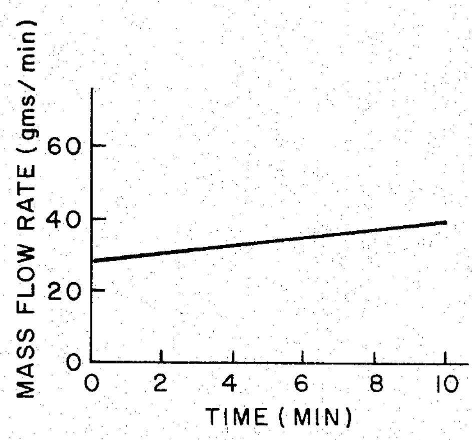

FIG. 4.

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PROCESS FOR CONTROLLING ORIFICE SIZE WHEN EXTRUDING MOLTEN MATERIALS

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to a method of forming fibers directly from melts of low viscosity, and, more particularly, to a method of controlling orifice size when extruding molten materials of low viscosity through the orifice.

2. Discussion of the Prior Art

The recent introduction of new techniques of forming fibers from melts of extremely low viscosity, i.e., less than about 10 poises, has proven to be highly satisfactory and economically competitive to older processes such as, for example, wire drawing. One of the new techniques is described and claimed in detail in U.S. Pat. No. 3,216,076 issued on Nov. 9, 1965, to Alber et al. Described therein is a method by which metals may be extruded as a free filamentary molten stream into a gaseous atmosphere and solidified into a fiber. To prevent surface tension from breaking the molten stream up into discrete spherical bodies, the gaseous atmosphere reacts with the stream to form an oxide film about the stream. The oxide film has sufficient strength to prevent surface tension-induced disruptions in the stream continuity prior to solidification of the stream into a fiber. This technique is called "stabilization"d.

The Alber patent further states that certain metal oxide films are soluble in the molten metal stream and therefore are not suitable to prevent or retard stream disruptions due to surface tensions. The patentees teach the addition to the melt of a small quantity of a second metal having an oxide not soluble in the stream. Thus, for example, iron (or steel) has oxides which are soluble in the molten metal, but through the addition of a small quantity of aluminum, an insoluble aluminum oxide film may be formed about the stream to preclude continuity disruptions.

In the referenced application, it is taught that molten streams may also be stabilized by forming films other than oxides, such as, for example, nitride or carbon films. The method claimed therein has the additional advantage of not requiring the presence of a second metal in those instances wherein the predominant metal has a soluble oxide. The application further demonstrates that stabilization techniques are not limited to metals but may be used in forming fibers from other low viscosity materials such as metalloids, metal oxides, and salts.

THE PROBLEM

It has been observed that when certain molten materials, particularly the metals, are extruded through a small opening such as an orifice, precipitates often form in or near the orifice. This problem becomes highly significant when forming fibers in accordance with the stabilization techniques described above. The precipitate products in or near the orifice may cause undesirable lateral deviations of the molten stream after it has been extruded through the orifice and generally culminates in complete orifice plugging.

Chemical reactions occurring during the extrusion process are largely responsible for the partial or complete plugging of the orifice with the precipitate

products. For example, in the extrusion of steel, the reaction is generally believed to be a refractory metal-oxygen-refractory metal oxide reaction. Because it is necessary to use crucible assemblies comprised of materials which have both a high melting point and good thermal shock properties, certain refractory metal oxides such as alumina (Al_2O_3) and beryllia (BeO) are often employed. Aluminum itself is often used in small quantities as a second metal to stabilize a molten stream of steel. Reactions occur between these materials, the oxygen, and the carbon in the molten steel. A gas such as carbon monoxide is released and begins to accumulate above the melt. Eventually, oxides such as chrysoberyl and alumina precipitate in or near the orifice causing the plugging problem. Chrysoberyl is beryllium aluminate ($BeO . Al_2O_3$) and generally is found in a mixed system where both alumina and beryllia parts contact the melt.

It is therefore desirable to prevent or control changes in the orifice size due to the chemical reactions occurring between the melt, the impurities in the melt and the materials comprising the crucible assemblies.

SUMMARY OF THE PRESENT INVENTION

Briefly, the present invention involves preventing undesirable changes in the size of an orifice or alternately controlling the size of the orifice while extruding a molten material contained in the crucible assembly through the orifice. In one aspect of the present invention, the partial pressure of a gas species which enters into the chemical reaction with the molten material contained within the crucible assembly, the materials comprising the crucible assembly, and the impurities contained in the melt is maintained at a predetermined level sufficient to "thermodynamically equilibrate" the chemical reaction. For example, in the extrusion of molten steel contained in an alumina crucible, with carbon monoxide being released above the melt, a flow of inert gas is used to remove the carbon monoxide as it is formed thereby thermodynamically equilibrating the chemical reaction.

Still another aspect of the present invention is the alteration of the size of the orifice during the extrusion process. Thus, by increasing or decreasing the partial pressure of the gaseous species to a selected predetermined level, orifice size may be increased or decreased as desired.

In describing the present invention, it is convenient to couch the description in terms of extruding molten steel. It will be apparent after a reading of the subject matter contained herein that the invention is adapted to the extrusion of other metals and other low viscosity materials in which a gaseous species enters into the reaction. Thus, the features believed to be characteristic of the present invention are set forth in the appended claims. The invention with further objects and advantages thereof may be best understood by reference to the following description taken in connection with the accompanying drawings in which:

FIG. 1 is a vertical cross-section of a typical spinning apparatus;

FIG. 2 is a graph depicting mass flow rate as a function of extrusion time without an inert gas purge;

FIG. 3 is a graph depicting the mass flow rate as the purge flow rate is varied;

FIG. 4 is a graph of the mass flow rate as a function of time for a constant inert gas purge.

DESCRIPTION

In the extrusion of molten steel, the process may be conducted either as "batch" or "continuous" extrusion. Batch extrusion is primarily the melting and extrusion of a single charge of steel as opposed to the continuous addition of charges in continuous extrusion. In batch extrusion, there is a general increase in the oxygen level as the process continues. There is also an increase in the amount of carbon monoxide in contact with and above the melt. It is believed that the following reactions take place in the melt:

$$MeO_{(s)} \rightleftharpoons Me + O \qquad (1)$$

$$O + C \rightleftharpoons CO_{(g)} \qquad (2)$$

where Me represents a metal such as aluminum, for example, and the underlined components represent components that are in solution. Combining Reactions (1) and (2),

$$MeO_{(s)} + C \rightleftharpoons Me + C + O \rightleftharpoons Me + CO_{(g)} \qquad (3)$$

where $MeO_{(s)}$ is the refractory oxide which under thermodynamically non-equilibrated conditions causing reaction (3) to proceed to the left may continually precipitate out of solution.

Because the orifice is open to a stabilizing chamber, thus usually cooler than the surrounding melt, precipitation when it occurs generally is in the vicinity of the orifice. At best, the presence of undesired precipitate particles in the orifice may affect the stream flow patterns which in turn affects stabilization, ultimately culminating in an inferior fiber. Generally, precipitate particles collect in the orifice until the orifice is filled or plugged, preventing stream flow.

We have found, however, that the orifice size may be kept essentially constant by thermodynamically equilibrating reaction (3). In other words, precipitation may be significantly reduced or eliminated. In general, the carbon and refractory metal concentrations in a single charge of molten steel are essentially constant. The melt temperature and carbon monoxide pressure level, however, are independent but interrelated variables. By controlling these two variables, reaction (3) may be kept thermodynamically equilibrated.

In accordance with one feature of the present invention, the gas species, for example, carbon monoxide, was removed by a flow of an inert gas, argon, for example, as it formed. By varying the rate of the carbon monoxide removal, the orifice size was altered as desired. That is, reaction (3) proceeds to the left when the carbon monoxide level is greater than the value at which reaction (3) is equilibrated. Under these conditions, the metal oxide precipitates in or near the orifice. On the other hand, we have also found that by increasing the inert gas purge, i.e., reducing the partial pressure, the metal oxide tends to go into solution causing the orifice to increase in size due to erosion.

FIG. 1 illustrates schematically the above operation. Crucible assembly **10** contains a melt **11** which is extruded through orifice **12** into a chamber **13** containing a coolant gaseous atmosphere reactive to form a stabilizing film about stream **14**. In this operation, an inert gas **15** is caused to flow across the surface of melt **11** so as to sweep away the gas formed due to the melt-crucible assembly reaction such as reaction (3). The rate of flow of the gas is controlled as desired to either maintain or alter orifice size. Other operations may use bubbling techniques to control orifice size. The term "-purging" is therefore employed generically to define selective controlling of the gas species formed by the reaction to provide the proper partial pressure of the gas species. Thus, the term purging is unrestricted by the manner in which the gas species is removed.

EXAMPLE 1

A steel charge having about 1 percent aluminum was melted in a beryllia crucible assembly and held at a temperature of about 1,650° C. The melt was then extruded through a 195 micron orifice at about 20 PSI into an oxide forming atmosphere such as, for example, air, carbon dioxide, or carbon monoxide. As Table 1 below illustrates the flow rate initially was about 63.4 gms/minute.

TABLE 1

Elapsed Time (min.)	Flow Rate (gms/min.)
0	63.4
2.0	63.3
3.0	64.9
4.0	61.9
5.0	34.9
5.5	8.6
6.0	Flow stopped

The flow rate significantly decreased during the fourth minute and ceased at six minutes as demonstrated by the graph of FIG. 2. An examination of the precipitate which plugged the orifice determined that it was primarily composed of chrysoberyl.

EXAMPLE 2

A steel charge having about 1 percent aluminum was melted in a beryllia crucible assembly and held at a temperature of about 1,605° C. The melt was then extruded through a 160 micron orifice at an initial flow rate of about 23.0 gms/minute into an oxide forming atmosphere. Argon was used to purge the carbon monoxide which formed above the melt. Table 2 depicts the various elapsed time intervals, rate of argon purge, and mass flow rates of the molten steel. The graph of FIG. 3 illustrates the mass flow rate for the various elapsed time intervals.

TABLE 2

Elapsed Time (min.)	Argon Purge (cc/min.)	Flow Rate (gms/min.)
2.0	800	22.4
2.5	800	20.4
4.0^- (slightly less)	>>800	18.4
4.0^+ (slightly greater)	>>800	18.4
6.0	>>800	22.4
7.0	> >800	25.6
8.0	>>800	27.2
9.0	>>800	29.2
10.0	>>800	32.8
11.0	>>800	34.0

Example 2 demonstrates that the flow rate of the molten material may be controlled by the rate of purge. As seen in FIG. 3, the flow decreased steadily for an argon purge of approximately 800 cc/min. This indicates that precipitation was occurring in the orifice. When the argon purge was increased to a value much greater then 800 cc/min., erosion of the orifice began to take place.

EXAMPLE 3

The charge of steel was melted in a beryllia crucible assembly and held at a temperature of about 1,550° C. The orifice diameter was approximately 155 microns. The initial mass flow rate was about 28.4 gms/minute. An argon purge of about 800 cc/min. was used to carry away the carbon monoxide gas as it was formed. As indicated by Table 3 and the graph of FIG. 4, the mass flow rate slowly increased during the extrusion run, but the charge was completely extruded through the orifice in contrast to the run of Example 1.

TABLE 3

Elapsed Time (min.)	Flow Rate (gms/min.)
1.75	28.8
2.50	31.2
3.25	30.8
4.00	30.8
4.75	32.4
5.50	32.0
6.25	33.6
7.00	33.2
7.75	33.6
8.50	34.8
9.25	34.0
10.0	34.0
10.75	34.0
11.50	33.6
12.25	33.6
13.00	33.6
13.75	34.4
14.50	34.4
15.25	35.2
19.75	36.8
21.50	streamed out

From a reading of the description contained herein, it is clear that the present invention may be practiced with various molten materials being extruded through an orifice. Depending upon the result desired, the concentration of the gaseous species above the melt is maintained at a predetermined level resulting in close control of the orifice size. It will also be apparent that there are various ways in which the concentration of the gaseous species may be changed. It is to be understood that changes may be made in the particular features of the present invention which are within the full intended scope of the invention as defined in the following claims.

What we claim is:

1. In a process for extruding molten steel contained in a refractory metal oxide crucible assembly through an orifice wherein carbon monoxide enters into a chemical reaction occurring between the molten steel, the crucible assembly and impurities contained in the molten steel, the improvement which comprises maintaining the partial pressure of the carbon monoxide at a level which essentially thermodynamically equilibrates the said reaction.

2. The improvement of claim 1 wherein carbon monoxide is purged with an inert gas to maintain equilibrium.

3. The improvement of claim 1 wherein the refractory metal oxide is beryllia.

4. The improvement of claim 1 wherein the refractory metal oxide is alumina.

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