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G. B. TAYLOR

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METHOD OF CASTING SYNTHETIC ORGANIC COMPOUNDS

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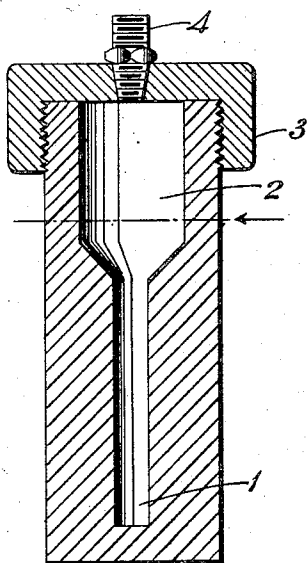


Fig. 1

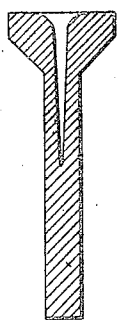


Fig. 2

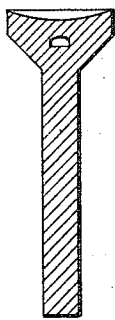


Fig. 3



Fig. 4



Fig. 5

Guy B. Taylor

INVENTOR

BY

J. M. Castle
ATTORNEY

UNITED STATES PATENT OFFICE

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METHOD OF CASTING SYNTHETIC
ORGANIC COMPOUNDS

Guy B. Taylor, Wilmington, Del., assignor to E. I.
du Pont de Nemours & Company, Wilmington,
Del., a corporation of Delaware

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6 Claims. (Cl. 18—58)

This invention relates to a method of casting synthetic organic compounds and, more particularly, to a method of casting molten synthetic linear superpolymers.

The term "superpolymer" is used herein as in Carothers U. S. Patent 2,071,250 to refer to synthetic polymers capable of being formed into useful fibers. As disclosed in the aforementioned patent, such polymers include polyesters, polyamides, polyethers, and polyanhydrides. These polymers are microcrystalline in character and can be converted into fibers showing by characteristic X-ray patterns orientation along the fiber axis.

The term "superpolyamides" is used herein to refer to polyamides capable of being formed into useful fibers. Such polyamides and their preparation are disclosed in Carothers U. S. Patents 2,071,253 and 2,130,948, and are designated generically in the art as nylon. In these polyamides the amide groups form an integral part of the main chain of atoms in the polymer.

Generally, these superpolymers are not plastics but, rather, have definite melting points near their decomposition temperatures, and are subject to oxidation when exposed to air at temperatures approaching their decomposition temperatures; further, there is in many cases a large contraction in volume of material as the molten superpolymers solidify. It will be obvious that these superpolymers cannot be cast by the usual methods heretofore employed in the plastics art. Moreover, the molding-powder technique of the plastics art is not practical with these superpolymers not only for the reasons above but also because they are not easily reduced to pulverulent form.

In order to cast these superpolymers acceptably they must be cast at or above their melting point. An additional requirement in the case of many superpolymers is the rigid exclusion of air to prevent oxidation. Many of these superpolymers, particularly the superpolyamides, present a further problem in that they tend to decompose slightly at temperatures high enough to melt them and, as a result, bubbles may be formed thereby preventing a sound casting. Not all of the superpolyamides are subject to this bubble formation when in molten condition but in many instances the tendency to bubble formation is sufficiently great to present a serious problem.

An object of the present invention is to provide a satisfactory method of casting these superpolymers. A further object is to provide a

method wherein superpolyamides subject to bubble formation in the molten state may be satisfactorily cast. A still further object is to provide a method of casting flawless rods of superpolymers for use in melt spinning according to the process disclosed in U. S. Patent No. 2,253,089, in the name of Roland R. Nydegger, entitled "Spinning apparatus and method," assigned to the assignee of the present application. A further object of the invention is to provide a method of casting superpolymers in turnery shapes adapted for machining, and the like. Other objects will be apparent from the description of the invention given hereinafter.

The above objects are accomplished according to the present invention by cooling, in the presence of an inert atmosphere, a molten superpolymer disposed in a mold having a mold cavity and a superposed reservoir cavity directly connecting with the mold cavity, the reservoir cavity being greater in horizontal cross sectional area than the upper portion of the mold cavity, under such conditions that a core of molten superpolymer connecting with the mold cavity is maintained in the reservoir cavity until the superpolymer in the mold cavity is solidified, the amount of superpolymer disposed in the mold being at least sufficient to fill the mold cavity and a part of the reservoir cavity after it has been allowed to solidify.

More particularly, the present invention contemplates casting superpolymers which are subject to bubble-formation when molten, by increasing the pressure upon the molten material in the mold until all bubbles formed are suppressed, that is, collapsed or dissolved in the melt, and thereafter cooling the molten superpolymer as above.

The invention generally is carried out by introducing superpolymer, usually in the form of flakes or lumps, into the mold which is provided with means for introducing an inert gas, heating the mold until the superpolymer melts, and, then, subjecting the molten superpolymer to pressure to suppress any bubbles formed in it and cooling the mold in the manner described.

Alternatively, the monomeric ingredients may be introduced into the mold, the polymerization reaction carried out in such a way to end with the superpolymer in molten condition, and then the subsequent procedure carried out in the same manner as if superpolymer in the solid state had been introduced into the mold and melted.

Referring to the drawing accompanying the present application:

Fig. 1 is a vertical section of a mold suitable for casting rods according to the present invention; and

Figs. 2-5, inclusive, are vertical sections of castings made in the mold illustrated in Fig. 1, under varying conditions.

Referring to Fig. 1, the mold there shown comprises a mold cavity 1 adapted for casting rods and a reservoir cavity 2 superposed on the mold cavity 1 and communicating directly therewith. It will be noted that the horizontal cross sectional area of the reservoir cavity 2 exceeds that of the mold cavity 1. In this instance, the horizontal cross sectional area of the mold cavity 1 is substantially identical throughout its height but, in the event that it varied, it would be sufficient for the cross sectional area of the reservoir cavity simply to exceed that of the upper portion of the mold cavity.

The upper portion of the mold is externally threaded and the cap 3 is adapted to be screwed thereon. This cap 3 is provided with the pipe connection 4 which may be connected with a vacuum line or with any source of gaseous pressure whereby air in the mold can be displaced by an inert gas and/or the mold contents placed under pressure.

The method of cooling the molten superpolymer is an important feature of the present invention and, in order to obtain a sound casting, must be carried out in such a manner that a core of molten superpolymer connecting with the mold cavity is maintained in the reservoir cavity until the superpolymer in the mold cavity is solidified. To accomplish this, it is obvious that care must be taken in the manner in which the mold is cooled, the manner in which the mold has been heated influencing, to some extent, the manner in which the mold may be cooled to obtain the desired result.

If the mold is filled with superpolymer which on being melted and resolidified fills the mold cavity and the reservoir cavity to the level indicated by the arrow in Fig. 1, the method of heating the mold to melt the superpolymer will greatly affect the results when the cooling is carried out in certain ways. For example, the molten superpolymer will be heated to the same temperature throughout whether the mold is heated by (1) immersion in a liquid bath to a level just above that of the molten superpolymer, or (2) immersing the whole mold including the cover in a bath. However, in case (1) on cooling the mold in air, a pipe appears in the casting as shown in Fig. 2, thus giving an unsatisfactory casting whereas, in case (2) on spontaneous cooling in the air, no pipe appears in the casting proper but frequently a void will occur in the cone of superpolymer which solidified in the reservoir cavity, as shown in Fig. 3. As the object is to secure only the rod as a sound casting, the result in this instance is satisfactory.

In case (2) above, if cooling is hastened by lowering the mold into water to a level about half the height of the mold cavity 1, a sharper temperature gradient is established so that the top is forced to solidify last and the casting is sound throughout, see Fig. 4; this result may also be obtained in case (1) if the cooling by means of water is carefully done to avoid premature solidification of the center of the mass in the reservoir cavity.

It will be understood that, in the above illustrations, the reason for the difference in results between case (1) and (2) is largely attributable

to the fact that the mold, particularly that portion of it forming the walls of the reservoir cavity, absorbs more heat in case (2) and, hence, tends to keep the superpolymer in the reservoir cavity molten for a period sufficient to permit solidification of the superpolymer in the mold cavity.

The reason that the reservoir cavity 2 is an essential feature of a mold suitable for carrying out the present invention is illustrated in Fig. 5 which shows a section of a rod cast in a straight tube mold not having a superposed reservoir cavity. In this instance the solidification of the superpolymer adjacent the walls in the upper portion of the mold leaves no molten material to fill the pipe formed due to contraction of the material as it solidifies and this is true no matter what method of cooling is used. By having the reservoir cavity considerably greater in horizontal cross sectional area than the mold cavity and, also, of appreciable height, a molten core of superpolymer in the reservoir cavity is quite readily maintained and permits considerable variation of the conditions under which the mold may be cooled to obtain a sound casting. On the other hand, if cooling of the mold is carried out with great care to keep a substantial temperature differentiation between the material in the mold cavity and in the reservoir cavity, then it is not necessary that the horizontal cross sectional area of the reservoir cavity be substantially greater than that of the upper portion of the mold cavity, in fact, they could be substantially the same.

Obviously, the cover of the mold, when heated, assists in maintaining the molten core of superpolymer in the reservoir cavity by restricting loss of heat through radiation from the upper surface of the superpolymer in the reservoir cavity.

The following examples are given to illustrate specific embodiments of the present invention:

Example I.—A mold adapted for casting rods and having the relative dimensions as the mold shown in Fig. 1 was used. The diameter of the reservoir cavity was 3" and the diameter of the mold cavity was 1½". This mold was charged with flake polymeric hexamethylene adipamide in an amount sufficient to give a melt level at the point indicated by the arrow in Fig. 1. The air in the mold was displaced by nitrogen, the mold sealed by closing a valve in the gas line connecting with the mold, and the mold was then immersed entirely in a fused salt bath maintained at 280° C. until all of the polymer was melted.

The melt within the mold at this stage contains numerous small bubbles due to slight decomposition of the polymer. To suppress these bubbles the nitrogen pressure within the mold was raised to 300 pounds per square inch. The mold was then removed from the bath and allowed to cool in the air, the pressure being relieved after the polymer solidified. When the mold has cooled to room temperature, it is opened and the meniscus contact of the polymer with the walls of the mold is cut with a knife. The molded piece then drops freely out upon turning the mold upside down. A sound casting was thus obtained.

Example II.—The same mold as used in Example I was charged with 230 grams of hexamethylene diammonium adipate and 100 grams of water and set in an electric furnace with heating elements extending only as far as the

cap for the mold. Heating was started and shortly after steam appeared, showing displacement of the air, a valve cutting off the pipe connection to the atmosphere was closed. Thereafter, heating was continued until 250 pounds per square inch gage pressure was reached. Steam was bled out at this pressure until the mold contents reached a temperature of 270° C., then the pressure was slowly released at constant temperature down to atmospheric pressure. The temperature was raised to 280° C. and held for one hour, an auxiliary heater being applied to heat the cap cover to the same temperature during this period. Nitrogen pressure of 200 pounds per square inch was applied and the mold then lifted from the furnace and allowed to cool in air. A casting was obtained such as illustrated in Fig. 3, that is, there was a void in the cone of material which solidified in the reservoir cavity but this in no way affected the soundness of the rod casting.

Example III.—The procedure of Example II was followed except that the cooling was done by immersing the lower half of the mold in water. In this case the casting was sound throughout and as indicated in Fig. 4.

The above examples illustrate the invention as applied to superpolymers that are not plastics, have a fairly sharp melting point, a large contraction on solidification, and exhibit no tendency for the casting to stick to the mold. Other specific superpolymers of this type include hexamethylene adipamide-terephthalamide interpolymer and hexamethylene-beta-methyl adipamide polymer.

There are some superpolymers which do not so clearly exhibit the properties mentioned above and, as a consequence, tend to stick in the mold. The present invention is equally applicable to such superpolymers except that a glass or thin metal liner is inserted in the mold and broken or machined off the finished casting. The following example illustrates this:

Example IV.—An interpolymer was prepared from an equi-molar mixture of the salts hexamethylene diammonium adipate and decamethylene diammonium sebacate by the procedure of Example II in a mold provided with an aluminum liner. A flawless casting was obtained and the thin metal liner removed therefrom by machining in a lathe.

Amide-ester interpolymers frequently require a liner in the mold to prevent sticking; hexamethylene diammonium isophthalate is another material that tends to stick in the mold and generally necessitates the use of a liner.

The above examples are merely illustrative both as to the specific superpolymers disclosed and the specific method employed. The invention is applicable to superpolymers broadly and is especially advantageous in the casting of superpolymers that have relatively definite melting points not far removed from their decomposition temperatures, that have a tendency toward bubble-formation when molten, and have an unusually large shrinkage factor in solidifying. The superpolyamides in general are frequently desired in the form of castings and the present invention is particularly useful for the production of castings of this class of superpolymers.

The herein disclosed procedure is not restricted to castings of cylinders. Any shape may be molded providing the mold is constructed so that there is a reservoir adapted to feed down molten

superpolymer to avoid the formation of a pipe. Where a shape having other than straight sides is to be cast, it is necessary, of course, to strip the mold off the casting.

The present invention is useful in the preparation of cast articles from superpolymers, especially where turnery shapes are desired and flawless rods for use in the procedure disclosed in the aforementioned Nydegger Patent No. 2,253,089.

An advantage of the present invention is that it provides a practical method of producing castings under gas pressure from a melt that requires handling in a narrow temperature range set by the melting point and decomposition temperature. A further advantage of the invention is that it permits the production of flawless castings adapted for turnery purposes and other uses where flaws cannot be tolerated. A still further advantage of the present invention is that it does not involve complicated or costly apparatus nor exceedingly fine adjustment of conditions such as would be beyond the scope of an ordinary laborer.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that the invention is not limited to the specific embodiments thereof except as defined in the appended claims.

I claim:

1. A process of casting voidless shaped articles of synthetic superpolymers which comprises disposing in a mold in the presence of an inert gas an amount of molten superpolymer sufficient when subsequently solidified to fill a cavity of the desired shape at the bottom of said mold and to at least partially fill a directly connecting superposed reservoir cavity having a relatively larger horizontal cross-sectional area than the upper portion of said shaped cavity, and in the presence of said inert gas cooling the molten superpolymer in the cavity of the desired shape more rapidly than the superpolymer in the reservoir cavity by subjecting the lower portion of said mold to a cooling medium whereby a core of molten superpolymer connecting with the cavity of desired shape is maintained in the reservoir cavity until the superpolymer of the desired shape is solidified.

2. A process of casting voidless shaped articles of synthetic superpolyamides which comprises disposing in a mold in the presence of an inert gas an amount of molten superpolyamide sufficient when subsequently solidified to fill a cavity of the desired shape at the bottom of said mold and to at least partially fill a directly connecting superposed reservoir cavity having a relatively larger horizontal cross-sectional area than the upper portion of said shaped cavity, increasing the pressure of inert gas in the mold until bubbles formed by decomposition of the molten superpolyamide are suppressed, in the presence of said inert gas under pressure cooling the molten superpolyamide in the cavity of the desired shape more rapidly than the superpolyamide in the reservoir cavity by subjecting the lower portion of said mold to a cooling medium whereby a core of molten superpolyamide connecting with the cavity of desired shape is maintained in the reservoir cavity until the superpolyamide of the desired shape is solidified, then releasing the pressure of inert gas in said mold.

3. A process of casting voidless shaped articles of synthetic superpolymers which comprises dis-

posing in a mold an amount of solid superpolymer sufficient when subsequently melted and resolidified to fill a cavity of the desired shape at the bottom of said mold and to at least partially fill a directly connecting superposed reservoir cavity having a relatively larger horizontal cross-sectional area than the upper portion of said shaped cavity, displacing the air in said mold by an inert gas, then heating substantially the entire mold until all of said superpolymer is melted, and in the presence of said inert gas cooling the molten superpolymer in the cavity of the desired shape more rapidly than the superpolymer in the reservoir cavity by subjecting said mold to a cooling medium whereby a core of molten superpolymer connecting with the cavity of desired shape is maintained in the reservoir cavity until the superpolymer of the desired shape is solidified.

4. A process of casting voidless shaped articles of synthetic superpolymers which comprises disposing in a mold an amount of solid superpolymer sufficient when subsequently melted and resolidified to fill a cavity of the desired shape at the bottom of said mold and to at least partially fill a directly connecting superposed reservoir cavity having a relatively larger horizontal cross-sectional area than the upper portion of said shaped cavity, displacing the air in said mold by an inert gas, then heating said mold until all of said superpolymer is melted, and in the presence of said inert gas cooling the molten superpolymer in the cavity of the desired shape more rapidly than the superpolymer in the reservoir cavity by subjecting the lower portion of said mold to a cooling medium whereby a core of molten superpolymer connecting with the cavity of desired shape is maintained in the reservoir cavity until the superpolymer of the desired shape is solidified.

5. A process of casting voidless shaped articles of synthetic superpolyamides which comprises disposing in a mold an amount of solid superpolyamide sufficient when subsequently melted and resolidified to fill a cavity of the desired shape at the bottom of said mold and to at least par-

5 tially fill a directly connecting superposed reservoir cavity having a relatively larger horizontal cross-sectional area than the upper portion of said shaped cavity, displacing the air in said mold by an inert gas, then heating substantially the entire mold until all of said superpolyamide is melted, increasing the pressure of inert gas in the mold until bubbles formed by decomposition of the molten superpolyamide are suppressed, in the presence of said inert gas under pressure cooling the molten superpolyamide in the cavity of the desired shape more rapidly than the superpolyamide in the reservoir cavity by subjecting said mold to a cooling medium whereby a core of molten superpolyamide connecting with the cavity of desired shape is maintained in the reservoir cavity until the superpolyamide of the desired shape is solidified, then releasing the pressure of inert gas in said mold.

20 6. A process of casting voidless shaped articles of synthetic superpolyamides which comprises disposing in a mold an amount of solid superpolyamide sufficient when subsequently melted and resolidified to fill a cavity of the desired shape at the bottom of said mold and to at least partially fill a directly connecting superposed reservoir cavity having a relatively larger horizontal cross-sectional area than the upper portion of said shaped cavity, displacing the air in said mold by an inert gas, then heating said mold until all of said superpolyamide is melted, increasing the pressure of inert gas in the mold until bubbles formed by decomposition of the molten superpolyamide are suppressed, in the presence of said inert gas under pressure cooling the molten superpolyamide in the cavity of the desired shape more rapidly than the superpolyamide in the reservoir cavity by subjecting the lower portion of said mold to a cooling medium whereby a core of molten superpolyamide connecting with the cavity of desired shape is maintained in the reservoir cavity until the superpolyamide of the desired shape is solidified, then releasing the pressure of inert gas in said mold.

GUY B. TAYLOR.