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PRODUCTION OF ARTIFICIAL MATERIALS

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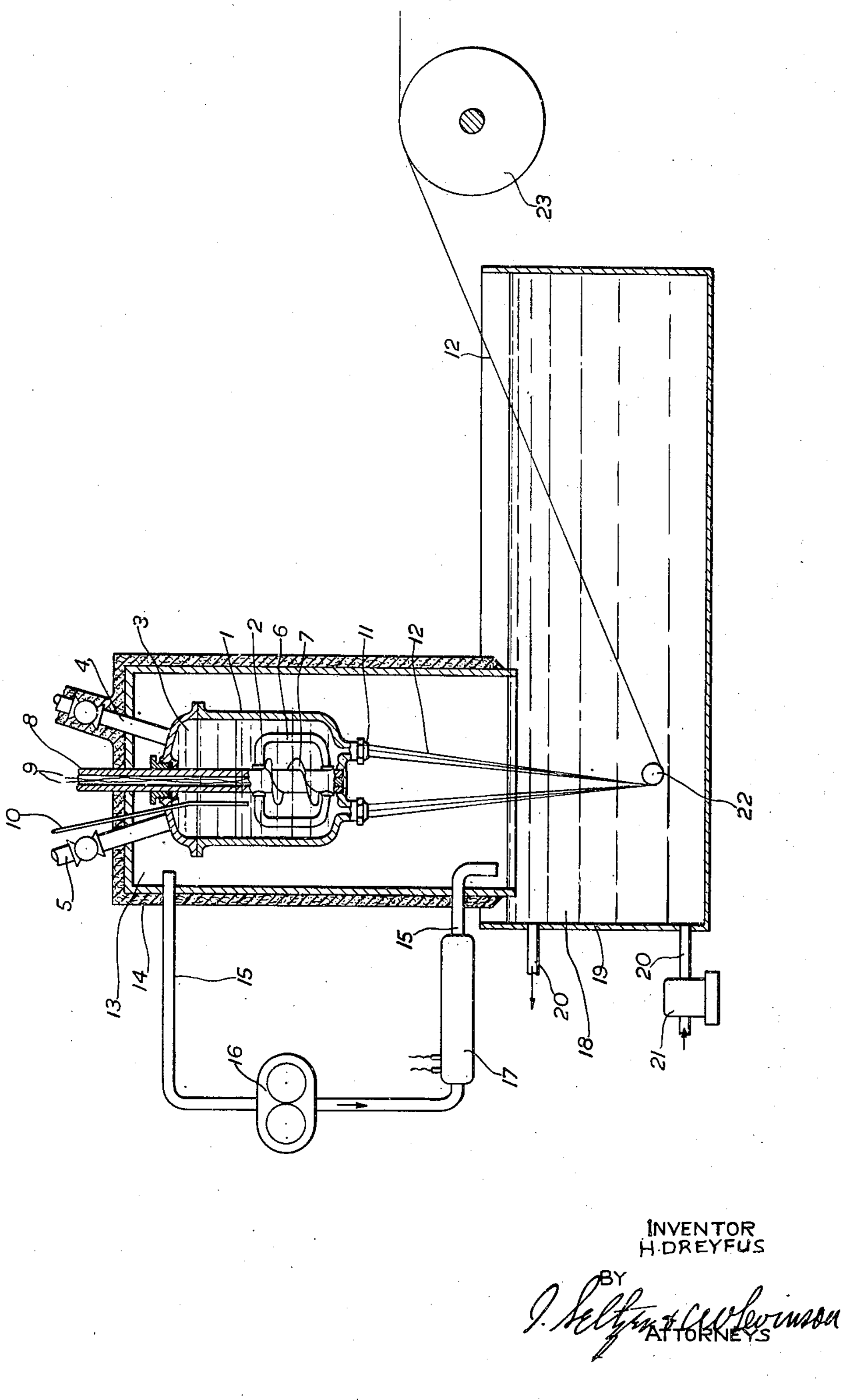

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PRODUCTION OF ARTIFICIAL MATERIALS

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This invention relates to the production of artificial materials and more particularly to the production of shaped materials having a basis of highly polymeric substances.

A number of proposals have been put forward for converting superpolymers, particularly superpolyamides produced, for example, from aminocarboxylic acids or from diamines and dicarboxylic acids, into filaments and other shaped articles. Among these is a proposal to extrude molten superpolymers into a cool atmosphere whereby the extruded materials are set. I have now found that shaped articles, e. g. filaments, films and the like of high tenacity, having a basis of a superpolymer may with advantage be produced by shaping a molten superpolymer and passing the shaped material into an inert medium, e. g. an inert atmosphere or an inert liquid non-solvent, which is maintained at a temperature which is substantially the same as, or only slightly lower than, that of the molten material.

Articles may be produced according to this process which possess a particularly satisfactory regularity of dimensions and which display a surprising constancy of physical characteristics, e. g. tenacity and extensibility, over their length.

In general the same types of agents may be employed either as liquid or as vaporous media into which the molten materials are passed, due regard being paid to the physical characteristics and stability of the agents. Among such are, for example, hydrocarbons, e. g. the paraffins and arylated paraffins, acenaphthene, anthrocene and its alkyl and aryl derivatives, benzene, diphenyl, toluene, the benzyl toluenes, including dibenzyl, naphthalene and its alkyl and aryl derivatives, including the dinaphthyls, phenanthrene, and compounds containing partially or fully hydrogenated aromatic nuclei, e. g. tetralin; ethers, e. g. isoamyl ether, the dimethyl ether of triethylene glycol, anisol, ethyl benzyl ether, the dinaphthyl ethers, the alkyl naphthyl ethers, and diphenyl ether; and halogen-substitution derivatives of hydrocarbons and of ethers. Apart from such organic compounds, the medium may comprise an atmosphere of heated gas such as, for example, nitrogen, hydrogen or carbon dioxide, the last-mentioned, in particular, being thoroughly free from traces of moisture.

The present invention is particularly applicable to the production of filamentary materials. For the production of such materials the fused superpolymer may be extruded through orifices into an inert liquid, vaporous or gaseous medium, which, as indicated above, is maintained in the neighbourhood of the orifices at a temperature which is substantially the same as, or only slightly lower than, that of the molten superpolymer. Preferably this hot medium has a temperature at least as high as the fusion point of the superpolymer, so that the extruded material remains in a fluid condition for a short time after leaving the orifices. The time during which this fluid condition is maintained need be only a small fraction of a second, e. g. 0.05–0.1 or 0.25 sec., after which time the materials may be passed into an inert medium maintained at a lower temperature adapted to set the materials. This transfer to a cooler medium may be gradual, a substantially regular temperature drop being maintained over the length of travel of the filaments, or it may result in a sudden temperature drop, e. g. a drop of 80–100 or 200° C. The latter alternative generally gives rise to products which possess a higher tenacity and may therefore be preferable. If desired, cooling may take place in a series of stages or in part gradually and in part by one or more sudden drops in temperature. The cooler medium may consist of the liquid of which the vapour constitutes the hotter medium. On the other hand, it is not necessary for the composition of the cooler medium to be uniform with that of the hot medium. For example, the hot medium may comprise a gas or vapour and the cooler medium may comprise water or some other liquid with which advantageously the gas or vapour is substantially immiscible. Again, the hot medium may comprise a liquid and the cooler medium some other immiscible liquid.

When it is desired to subject the extruded materials to a sudden temperature drop during their passage from the orifices, a vessel may be provided to contain both the hot and the cooler media and fitted with a plate of non-absorbent heat insulating material, e. g. glass, mica or glazed porcelain, bored with orifices to allow the passage of the filaments, the plate being mounted transversely to the direction of travel of the filaments to separate the two media. Each of the media may be circulated and their temperatures readjusted by suitable means outside the vessel.

After passing through a suitable length of the cooler medium so as to complete the setting of the materials, the latter may be continuously collected on rotating drums or the like, preferably at a rate in excess of that at which they emerge from the orifices, so that draw-down is applied while they are still in a plastic or fluid condition.

Extrusion may advantageously be effected under pressure of an inert liquid or vapour, as described in U. S. application S. No. 333,952 filed May 8, 1940, in which a suitable apparatus for fusion and extrusion of a thermoplastic material is described. Such an apparatus may, with advantage, be immersed in the hot medium, so as to ensure that the temperature of the fused superpolymer and of the medium into which it is extruded are substantially identical. When employing this method it is found possible to employ orifices mounted on tubular elements projecting outwardly from the reservoir containing fused superpolymer, without the necessity of providing electrical winding or other heating means on such elements. It will be evident that the invention is not limited to this particular apparatus or to the method particularly described in U. S. application S. No. 333,952, but that many other methods of extrusion may be employed.

By way of example an arrangement of apparatus suitable for carrying out the invention and employing the devices described in U. S. application S. No. 333,952 will now be described in greater detail with reference to the accompanying drawing, wherein the figure shows a form of apparatus that may be used.

In the drawing, 1 is a pressure vessel containing in its lower part a body 2 of fused polyamide to be extruded and in its upper part a body of liquid 3 by means of which the polyamide 2 is being extruded by displacement. The displacing liquid 3 enters the vessel 1 by the pipe 4, being raised to the same temperature as the polyamide 2 before being introduced. The pipe 5 serves for the escape of the liquid 3 when it is desired to replenish the vessel 1 with polyamide material while introducing further polyamide through the pipe 4.

As described in U. S. application S. No. 333,952, the molten polyamide is circulated and stirred in the vessel 1 by means of stirrer arms 6 and a screw 7 mounted on a rotating shaft 8, the stirrer arms 6 and screw 7 and shaft 8 being electrically heated when required through leads 9. A thermostatic device 10 is provided, having its head within the vessel 1, and adapted to maintain the temperature of the fused polyamide at the desired value.

The displacing liquid 3 entering through the pipe 4 forces the fused polyamide through nozzles 11 in the form of filaments 12. The entire pressure vessel 1 is enclosed within a chamber 13 provided with suitable lagging 14 and filled with a heated gaseous atmosphere such as nitrogen at substantially the same temperature as the vessel 1 and its contents. Since the vessel 1 is thus immersed in an atmosphere of the required temperature, it is not necessary, as in U. S. application S. No. 333,952, to lag the vessel 1 or to provide the vessel or the spinning jets 11 with electrical heating means. The nitrogen or other gaseous medium in the chamber 13 is circulated by means of pipes 15 and a blower 16, and its temperature is regulated as it enters the chamber 13 by means of a heater 17 to bring it to the required temperature.

The filaments 12, after passing through the heated atmosphere within the chamber 13 pass below the level of a liquid cooling medium 18 such as water, which is circulated in a bath 19 by means of pipes 20 and a pump 21 and maintained at a temperature substantially lower than that of the atmosphere within the chamber 13 for the purpose of cooling and setting the filaments 12. The filaments 12 pass round a guide 22 below the surface of the liquid 18 in the vessel 19 and then pass obliquely upwards out of the vessel 19 to a roller 23.

The filaments pass completely round the roller 23 before leaving it and the roller 23 is rotated at a suitable speed so as to stretch the filaments 12. Since the filaments 12 are set soon after they enter the liquid 18 the stretching action runs back to that part of the filaments 12 where they are in a more fluid condition, i. e. within the chamber 13. The filaments 12 may either be collected on the roller 23 itself, or, as shown in the drawing, may proceed from the roller 23 to any other suitable collecting device.

The shaped materials may be subjected to a stretching operation after they have been set, whether or not they have been stretched during their travel from the shaping devices. Before such a stretching operation the materials may be treated with a softening agent, e. g. a solution of a solvent or plasticiser or a solvent vapour and/or they may be softened by heat. Advantageously the materials are stretched in the presence of a hydroxylic non-solvent, e. g. water. Thus, they may, with advantage, be stretched while immersed in hot water or while in a steam chamber. In this way the tenacity of the products may be increased.

Alternatively, or in addition, the materials may be subjected to the action of a shrinking agent, e. g. a solution of a solvent for the superpolymer, in the substantial absence of tension so as further to modify their properties. Such a treatment is described more fully in U. S. application S. No. 318,710 filed February 13, 1940.

While the compositions which are shaped according to the invention may consist wholly of one or more superpolymers, it is generally preferable to employ compositions which also contain a softening agent for the superpolymers. For example, particularly in the case of superpolyamides, a proportion of phenols, especially high-boiling phenols, e. g. diphenylol propane, or of amides, e.g. acetamide, formamide, benzamide or sulphonamide, may be incorporated in the compositions. Other agents such as, for example, quinoline and acetophenone are also useful for a similar purpose. Such softening agents may be employed in proportions of 5 or 10 up to 35 or 40% or even more, based on the weight of the superpolymer. The compositions may also contain other agents in order to modify the properties of or to give various effects in the products. They may contain, for example, pigments, dyes and/or fire retardants.

Filamentary products produced according to the invention may be associated together, e. g. by twisting, to form yarns, and they may then be employed in the manufacture of woven, knitted or other fabrics. Filaments of relatively high denier may be employed directly as artificial horsehair or bristles. Filamentary products may also be coalesced under the action of solvents or plasticisers to form unitary products suitable for use as bristles or the like.

For the production of fibres according to the invention, filamentary products produced as described above may be cut after winding or while travelling, and such fibres are advantageously produced from filamentary materials which have been crimped, e. g. by twisting as yarn, setting the twist by steaming or similar treatment, and untwisting. Alternatively, or in addition, the cut fibres may be crinkled by treating them while in a loose condition with an agent having a plasticising, solvent or swelling action on the materials, preferably diluted with a relatively inert liquid, the concentration of the agent and the temperature of treatment depending upon the degree of crinkle desired. The fibres so produced may be spun into yarn.

Films, foils and the like, produced according to the invention, may be employed for any of the purposes to which thermoplastic films, foils and the like have been previously applied. For example they may be employed for wrapping purposes, as bases for photographic materials, as glass substitutes and for the manufacture of splinterless glass.

Having described my invention what I desire to secure by Letters Patent is:

1. Process for the production of shaped articles, which comprises shaping a molten superpolymer, passing the shaped material into an inert medium which is maintained at a temperature which is substantially the same as that of the molten material and setting the molten material.

2. Process for the production of shaped articles, which comprises extruding a molten superpolymer, passing the extruded material into an inert medium which is maintained at a temperature which is substantially the same as that of the molten material and setting the molten material.

3. Process for the production of shaped articles, which comprises extruding a molten superpolymer and passing the extruded material into an inert medium which is maintained at a temperature which is substantially the same as that of the molten material, drawing down the extruded material during its passage through the said medium and setting the molten material.

4. Process for the production of shaped articles, which comprises extruding a molten superpolymer and passing the extruded material into an inert medium which is maintained at a temperature which is substantially the same as that of the molten material, and then passing the extruded material into an inert medium maintained at a lower temperature adapted to set the material by cooling.

5. Process for the production of shaped articles, which comprises extruding a molten superpolymer and passing the extruded material into an inert medium which is maintained at a temperature which is substantially the same as that of the molten material, and then passing the extruded material into an inert medium maintained at a lower temperature adapted to set the material by cooling, and drawing down the extruded material in the course of its passage through the said media.

6. Process for the production of shaped articles, which comprises extruding a molten superpolymer into an inert gas maintained at a temperature which is substantially the same as that of the molten material, and then passing the extruded material into an inert liquid maintained at a lower temperature adapted to set the material by cooling.

7. Process for the production of shaped articles, which comprises extruding a molten superpolymer into an inert gas maintained at a temperature which is substantially the same as that of the molten material, and then passing the extruded material into an inert liquid maintained at a lower temperature adapted to set the material by cooling, and drawing down the extruded material during its passage through the said gas.

8. Process for the production of shaped articles, which comprises extruding a molten superpolymer into an inert vapour maintained at a temperature which is substantially the same as that of the molten material, and then passing the extruded material into the liquid which corresponds to said vapour maintained at a lower temperature adapted to set the material by cooling.

9. Process for the production of shaped articles, which comprises extruding a molten superpolymer into an inert vapour maintained at a temperature which is substantially the same as that of the molten material, and then passing the extruded material into the liquid which corresponds to said vapour maintained at a lower temperature adapted to set the material by cooling, and drawing down the extruded material during its passage through the said vapour.

10. Process for the production of shaped articles, which comprises extruding a molten superpolymer into an inert gas maintained at a temperature which is substantially the same as that of the molten material, and then passing the extruded material into an inert liquid, with which the said gas is substantially immiscible, maintained at a lower temperature adapted to set the material by cooling.

11. Process for the production of shaped articles, which comprises extruding a molten superpolymer into an inert gas maintained at a temperature which is substantially the same as that of the molten material, and then passing the extruded material into an inert liquid, with which the said gas is substantially immiscible, maintained at a lower temperature adapted to set the material by cooling, and drawing down the extruded material during its passage through the said gas.

12. Process for the production of shaped articles, which comprises extruding a molten superpolymer into an inert substantially water-immiscible gas maintained at a temperature which is substantially the same as that of the molten material, and then passing the extruded material into water maintained at a lower temperature adapted to set the material by cooling.

13. Process for the production of shaped articles, which comprises extruding a molten superpolymer into an inert substantially water-immiscible gas maintained at a temperature which is substantially the same as that of the molten material, and then passing the extruded material into water maintained at a lower temperature adapted to set the material by cooling, and drawing down the extruded material during its passage through the said gas.

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