PROCESS FOR THE IMPREGNATION OF FIBER STRANDS AND TAPES

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Filed: Jan. 17, 1972

Appl. No.: 218,115

Foreign Application Priority Data
Jan. 15, 1971 Germany........................................... 2101756

U.S. Cl. .................. 427/172; 118/6; 118/228; 427/175; 427/339; 428/367; 428/902

Int. Cl.2.......................... B05D 1/28; B05D 1/40

Field of Search .............. 117/7, 68, DIG. 11; 118/6; 118/33, 227, 228; 242/43

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ABSTRACT
A process for impregnating fiber strands, especially carbon fiber strands, and similar reinforcing fibrous materials having substantial length which comprises winding a carbon fiber strand off of a take-off means at a low take-off tension, impregnating the strand with the resin, and thereafter winding up the resin impregnated strand under a high winding tension.

13 Claims, 2 Drawing Figures
PROCESS FOR THE IMPREGNATION OF FIBER STRANDS AND TAPES

This invention relates to a process for impregnating lengths of reinforcing fibers such as carbon, boron and glass fibers and especially carbon fibers with a synthetic resin and an apparatus for carrying out this process.

It is known and considered state of the art to embed fibrous reinforcing materials of various substances in synthetic resin or metal matrix material in order to reinforce matrix materials. The effects obtained in this type of reinforcement are dependent, to a great extent, on the properties and characteristics of the reinforcing material.

Fibrous materials having a high tensile strength and a comparatively high modulus of elasticity (tensile strength $S_u$ up to 460 kpsi/m$^2$, modulus of elasticity $E = 7,000$ to $10,000$ kpsi/m$^2$ where kpsi represents kiloponds) made of various glasses have been utilized for many years for plastic reinforcements in the form of mats, fabrics, rovings, etc.

A special method for the production of glass-fiber reinforced composite materials is the filament winding process. As is known, rotational bodies can be manufactured in accordance with this procedure; the reinforcing materials used in this procedure are individual strands or rovings as well as roving ribbons of greater length. As the matrix materials, unsaturated polyester resins (UP) and epoxy resins (EP), are employed in many cases.

For several years now the procedure has been adopted to manufacture and to produce reinforcing fibers for composite materials suitable for the increased demands of industry which exhibit higher strength characteristics in comparison to glass fibers. Those fibrous materials particularly worth mentioning are boron filaments and carbon fibers. Although suitable for impregnation of other reinforcing fibers, the present invention is particularly concerned with a special processing method for carbon fibers and the following description is directed primarily to this fiber.

Since the successful production of carbon fibers having sufficient uniformity in great lengths (e.g., 1,000 m.) has been accomplished, this reinforcing fiber has become of interest in those fields of application wherein such great lengths are absolutely necessary, such as, for example, in the winding of containers, etc. However, as compared to the conventional glass fiber, carbon fibers exhibit considerable disadvantages during processing. Thus, carbon fiber is, for example, much more sensitive to breaking than the glass fiber. This is due to the fact that the modulus of elasticity of the carbon fiber is about 3 to 5 times as high. The number of broken and/or damaged fibers in a strand of, for instance, 10,000 monofilaments is substantially larger than in a comparable glass fiber strand. This tendency must be considered to a great extent when processing carbon fiber strands according to the filament winding method, particularly if a wound body is to be produced wherein a high percentage of the strength values inherent in carbon fibers is to be reached.

Customarily, the carbon fibers to be employed are wound onto spools or take-off means in cross laps, i.e., at an angle of about 45° to 70° with respect to the longitudinal axis of the spool, in lengths starting with about 500-600 m. If the strand of carbon fibers is now taken off from the spool with a take-off force of about 500 ponds (the unit pond being a standard measure of force, otherwise known as a gram-force) and thereabove, it is unavoidable that the carbon fiber strand, wound in cross laps, can be considerably damaged during the take-off procedure.

It is an object of this invention to provide a process for the impregnation of a carbon fiber strand which is characterized by a high uniformity of the resin distribution in the fiber strand and which insures extensive reduction in the proportion of damaged monofilaments in the strand.

This invention is characterized in that a take-off means, e.g., a spool, reel or the like, is positioned in a pivotal manner, namely at an angle corresponding to the winding angle of the strand of carbon fibers. Furthermore, since the take-off means is braked during the unwinding procedure, (so that a controlled small take-off force is built up, which ranges advantageously between about 50 and about 250 ponds), this invention ensures that the carbon fiber strand is gently taken off from the take-off means without exerting friction on the laps disposed therebelow and thus without damaging the fiber. This angular adjustment or orientation is maintained until the strand has reached one end of the take-off means. Once this is the case, the take-off means is swiveled by photoelectric control means to such an extent that the strand, which now runs back on the take-off means in the direction of its longitudinal axis, is taken off from the winding-up coil in an extensively frictionless manner. The take-off means is once again pivoted once the strand has reached the other end of the take-off means.

The dry strand of carbon fibers now travels over rollers to the first impregnating device or means. This means includes a thermostatically controlled container having a capacity of about 300 to about 1,000 cc which contains the impregnating resin which has been brought to the most favorable impregnating temperature. At right angles to its longitudinal axis the container or bath contains a metallic roll immersed to about 2 to about 5 centimeters into the resin bath. The carbon fiber strand travels, controlled by guide rollers from the take-off device, with a certain contact pressure over the impregnating roll, which, in turn, is set into rotation thereby. Due to this rotation, the impregnating roll conveys impregnating resin from the bath on its cylindrical outer surface.

In this manner, a resin film is produced through which the strand of carbon fibers travels. The thickness of the resin film is regulated by means of stripping plate or doctor blade means, e.g., by the provision of a replaceable stripping plate means between the resin level in the bath and the carbon fiber strand traveling over the impregnating roller.

The thickness of the resin film to be selected is predominantly dependent on the viscosity of the impregnating resin at the predetermined temperature.

By the contact pressure exerted by the strand of carbon fiber on the impregnating roll, a certain fanning out of the strand is attained, ensuring an improved penetration of the resin into the strand.

It has been found that a single impregnating step is insufficient, due to the poor affinity of the surface of the carbon fibers to epoxy resins or the like resins advantageously employed as the impregnating resin. Therefore, the strand of carbon fibers is guided over a second impregnating device or means, constructed sim-
ilar to the impregnating device described previously. The strand is advantageously conducted or transported in such a manner that the surface of the strand which is on top on the first impregnating roll contacts the second impregnating roll directly when traveling over this roll.

The thus-impregnated strand is now guided over several guide or deflecting rollers (advantageously at least three) to the threading eye of the winding machine. These guide rollers can be braked so that an adjustable maximum take-off force or filament tension can be built up in the impregnated carbon fiber strand.

It is possible, for example, to accomplish a build-up of the winding force or filament tension of at least 5 kpsi for a strand of 10,000 monofilaments, by the combination of the aforesaid described reel-off device, impregnation means, and braked guide rollers, with the strand of carbon fibers being handled with maximum care. This high winding force or filament tension is absolutely necessary for obtaining a uniformly constructed wound body wherein the distribution of fibers within the resin is as homogeneous as possible, i.e., wherein it is ensured that the filaments are impregnated with the resin without any air bubbles and in an extensively homogeneous manner, and conversely, the resin surrounds the strand in the same homogeneous manner, so that optimum property characteristics can be attained.

This invention is particularly directed to the combination of pivotable delivery spool or take-off means provided with a braking motor with two impregnating means and the braked guide rollers for providing a maximally attainable take-off force or filament tension. Thus it has been found that the combination of the above-described devices leads to optimum results and ensures the highest possible degree of utilization of the filament characteristics in the wound laminate.

By the combination of take-off means and double-sided impregnation with a controllable resin film on the impregnating rollers, an impregnation of the carbon fiber strand is obtained which exhibits the following advantageous characteristics:

- a high uniformity of the resin/fiber ratio;
- a product practically free of air bubbles;
- a high uniformity of the resin distribution in the strand;
- a substantial reduction in the proportion of damaged monofilaments in the strand.

Strands, ribbons, tapes, laminates or the like fibrillary products of carbon fibers impregnated in this manner are particularly suitable for the production of wound articles in accordance with the known filament winding method, or for the manufacture of pre-impregnated carbon fiber strands and/or carbon fiber threads.

One embodiment of the apparatus of this invention for the impregnation of carbon fiber strands or threads is schematically illustrated in the drawings, wherein:

FIG. 1 shows an elevational view of an impregnating apparatus according to this invention; and

FIG. 2 shows a swiveling device for the delivery spool.

A carbon fiber strand 1 of a greater length, wound crosswise in several layers on a delivery spool or take-off means 2 disposed in a spool swiveling device (as shown in FIG. 2), is guided over a roller system consisting of guide rollers, resin impregnating rolls, and braking rollers and is wound up under tension on a winding core or roll. The traveling direction of the fiber strand, as well as the direction of rotation of the rolls, is indicated with arrows.

The roller system is mounted on a vertically disposed mounting plate 3, the axes of all rollers being in parallel to one another, and forming a right angle with respect to the plate. The strand of carbon fibers is conducted from the delivery spool or reel 2 over a first guide roller 4 arranged directly in front of the first resin impregnating roll 5. Thereafter, the strand is fed, by a second guide roller 6 disposed at a somewhat lower level, to a second resin impregnating roll 7. Both resin impregnating rolls 5 and 7 are immersed in resin baths 8a, respectively. These resin baths can be heated by thermostatically heated heating baths 9a, respectively. The layer thickness of the resin entrained from the resin bath by the rolls during rotation thereof is adjustable with the aid of the stripper plate or doctor blades 10 and 10a disposed directly in front of the rolls; the gap between the impregnating rolls and the stripper plates represents a measure for the layer thickness and saturation of the carbon fiber strand. The guide roller 6 ensures that the carbon fiber strand, initially impregnated on one side by the roll 5, is also impregnated on the other side during contact with the second impregnating roll 7. By the presence of the carbon fiber strand on the impregnating rolls, the rotation of the latter is effected. After being impregnated on the second resin impregnating roll 7, the strand passes over a series-connected guiding system consisting of a number of braking rollers 11 (in this case five rollers are used) through which the strand is conducted in an approximately sinusoidal path. The braking force of the braking rollers can be adjusted, so that the carbon fiber strand can be wound up at the windup core 12 with a counter-tension of about 3 kpsi.

One embodiment of the spool swiveling device is described with reference to FIG. 2. On a cross strut 13a of the frame 13, the cross-wound delivery spool 2 is mounted in the plane of the delivery of the fiber strand on the plate 15 to be pivotable about the pivot axis 14. The delivery spool 2 is coupled, via a belt drive, with a variable-speed motor 16, likewise mounted on the plate 15. The torque of the motor is adjustable. The plate 15 is connected with an eccentric disk 18 via a lever 17 and the disk is set into rotation by a drive motor 19. During operation, the eccentric disk is rotated by an angle of 180° whenever a pivoting of the delivery spool is necessary after winding off one layer of the cross-wound carbon filament strand 1, namely by the angle formed in each case by the fiber strand with the axis of the delivery bobbin. The pivoting of the delivery spool 2 is controlled photoelectrically by means of reversing switches 20, 20a provided with a light contact.

The photoelectric control shown uses two light emitters installed on either side of frame (13) in FIG. 2. Each emitter has its own receiver mounted on a place opposed to it (not shown on the drawing). The emitter produces a light ray which is continuously received by its own receiver. Now if any object as for instance a strand enters the space between emitter and receiver, the latter no longer receives the light ray and interrupts an electrical circuit, thus giving a signal to activate the switch 20 or 20a to switch the driving motor of the spool swiveling device from "forward" to "reverse." It will also be understood that the braking rollers 11 each employ conventional braking devices. For example, one filament carrying roller may be mounted on
one side and a braking wheel of similar diameter on the other side of the frame of the impregnating device, both being on a common shaft, which in turn is carried by bearing means in the wall. Braking is effected by means of braking shoes which are pressed against the braking wheel by an adjustable spring or a pneumatically or hydraulically operated piston. It is also possible to provide an automatic control means for better controlling the braking torque in relationship to the winding tension.

It will be appreciated the process and apparatus of this invention may also be used to impregnate reinforcing fibrous materials with various resinous materials including unsaturated polyester resins, epoxy-novolaks, epoxy resins hardened at elevated temperatures and epoxy resins hardened at ambient temperatures. Thus it will be understood that control of the temperature of the impregnated resin in the baths is dependent on the choice of resin. For example it has been found that the temperature should be about 50°C for an epoxy-novolak resin.

The process of this invention will be further understood by reference to the following example:

**EXAMPLE**

By using an apparatus as illustrated in the drawings a carbon fiber strand made up of 10,000 monofilaments and having a denier of 6,000 was drawn off a take-off spool at a speed of about 5 meters per minute at a winding-force of 100 grams. The strand was passed through a first impregnating bath of epoxy resin at a temperature of 50°C, to impregnate that carbon fiber strand from one side. The strand was then passed over a second impregnating roller and thereby fully impregnated from the other side. The strand was then passed over five braking rollers operated at speeds of in the region of 20 rpm and then wound onto a winding core under a tension of 5 kiloponds. The resulting product after solidification of the resin was found to be a very good and worthwhile carbon article.

It will be appreciated that the process of this invention is especially suitable for impregnation of carbon fiber strands. However, as noted other reinforcing materials may also be processed. Thus it is advantageous that the take-off tensions may vary from 0.01 through 5 kp and the winding tensions may vary between 0.1 and 20 kp, the tension being controlled in each case to depend on the fiber employed and the product desired.

While the novel embodiments of the invention have been described, it will be understood that various omissions, modifications and changes in these embodiments may be made by one skilled in the art without departing from the spirit and scope of the invention.

What is claimed is:

1. A process for the impregnation of carbon strands having substantial length, which process comprises winding carbon fiber strands off of a pivotal take-off means at a low take-off tension by controlling the take-off force to minimize damage to the carbon fiber strand, impregnating all sides of the strand with a resin, passing the resin impregnated strand through a guiding means including a plurality of rollers to achieve a predetermined adjustable maximum tension on the impregnated strand, and thereafter winding up the resin impregnated strand under said adjustable maximum tension onto a rotatable support means to provide a high winding tension, so that the carbon fiber strand is constructed uniformly and homogeneously impregnated with minimal damage of the carbon fiber, wherein the strand is cross-wound onto said take-off means and the take-off means is pivoted so that its longitudinal axis is at an angle approximately equal to the wind-up angle of the cross-wound strand on said take-off means whereby damage to the reinforcing fibers of said strand is avoided.

2. The process of claim 1, wherein the high winding tension is on the order of about 5 kp.

3. The process of claim 1, wherein opposite sides of a carbon fiber strand are impregnated with said resin by successively contacting one side of the strand with a first resin impregnating means and then contacting the other side of the strand with a second resin impregnating means.

4. The process of claim 1, wherein the strand is impregnated with said resin at an adjustable layer thickness by successive impregnating rolls.

5. The process of claim 1, wherein the carbon fiber strand is at least 1,000 m.

6. The process of claim 1, wherein control of the take-off force is by braking said take-off means.

7. The process of claim 1, further comprising the step of adjusting the thickness of the resin impregnating the carbon fiber strand.

8. The process of claim 3, wherein the carbon fiber strand contacts said first and second resin impregnating means by means of guide rollers.

9. The process of claim 1, wherein said predetermined maximum tension on the impregnated strand is achieved by building-up the tension on said impregnated strand.

10. The process of claim 9, wherein said tension on said impregnated strand is built-up by braking respective ones of said plurality of rollers.

11. The process of claim 1, wherein low take-off tension has a minimum value such that friction is prevented from being exerted between respective laps of said carbon fiber strand disposed on said pivotable take-off means.

12. A process for the impregnation of carbon strands having substantial length, which process comprises winding carbon fiber strands off of a pivotal take-off means at a low take-off tension by controlling the take-off force to minimize damage to the carbon fiber strand, impregnating all sides of the strand with a resin, passing the resin impregnated strand through a guiding means including a plurality of rollers to achieve a predetermined adjustable maximum tension on the impregnated strand, and thereafter winding up the resin impregnated strand under said adjustable maximum tension onto a rotatable support means to provide a high winding tension, so that the carbon fiber strand is constructed uniformly and homogeneously impregnated with minimal damage of the carbon fiber, wherein the low take-off tension in said carbon strand is controlled to be in the range of about 50 to about 250 ponds.

13. A process for the impregnation of carbon strands having substantial length, which process comprises winding carbon fiber strands off of a pivotal take-off means at a low take-off tension in the range of
about 50 to about 250 ponds by controlling the take-off force to minimize damage to the carbon fiber strand, wherein the strand is cross-wound onto said take-off means and the take-off means is pivoted so that its longitudinal axis is at an angle approximately equal to the wind-up angle of the cross-wound strand on said take-off means whereby damage to the reinforcing fibers of said strand is avoided, impregnating all sides of the strand with a resin, passing the resin impregnated strand through a guiding means including a plurality of rollers to achieve a predetermined adjustable maximum tension on the impregnated strand, and thereafter winding up the resin impregnated strand under said adjustable maximum tension onto a rotatable support means to provide a high winding tension, so that the carbon fiber strand is constructed uniformly and homogeneously impregnated with minimal damage of the carbon fiber.