NOTICE

The invention disclosed in this document resulted from research in aeronautical and space activities performed under programs of the National Aeronautics and Space Administration. The invention is owned by NASA and is, therefore, available for licensing in accordance with the NASA Patent Licensing Regulation (14 Code of Federal Regulations 1245.2).

To encourage commercial utilization of NASA-owned inventions, it is NASA policy to grant licenses to commercial concerns. Although NASA encourages nonexclusive licensing to promote competition and achieve the widest possible utilization, NASA will consider the granting of a limited exclusive license, pursuant to the NASA Patent Licensing Regulations, when such a license will provide the necessary incentive to the licensee to achieve early practical application of the invention.

Address inquiries and all applications for license for this invention to NASA Patent Counsel, Langley Research Center, Mail Code 279, Langley Station, Hampton, VA 23665. Approved NASA forms for application for nonexclusive or exclusive license are available from the above address.
This invention relates generally to pultrusion die assemblies, and more particularly, to a pultrusion die assembly which incorporates a plurality of functions in order to produce a continuous, thin composite fiber-reinforced thermoplastic material.

As illustrated in the drawings, a pultrusion die assembly 12 includes a profile die portion 14 and a crosshead die portion 16. The profile die portion includes an impregnation cavity 22 which begins at the confluence of upper and lower resin channels 32 and 34 and a central passageway 30 associated with a collimator 36. Through-holes 90, 92, 94, and 96 are provided downstream of the impregnation cavity 22 so as to cool impregnated fiber tows as they leave the outlet 28 of the profile die portion 14. Various heating elements are provided in a collimator 36 which is located in the crosshead die portion 16 of the pultrusion die assembly 12. The heating elements maintain a temperature of the fiber tows at the temperature of the injected molten thermoplastic material.

The invention is useful for making high performance thermoplastic composite materials in sheets which can be coiled on a spool and stored for further processing.

Inventor: Maywood L. Wilson
Home Address: 403 Calthrop Neck Road
Tabb, Virginia 23602
SS
Employer: NASA-Langley Research Center

Inventor: Gary S. Johnson
Home Address: 361 Kinsmen Way
Hampton, Virginia 23666
SS
Employer: NASA-Langley Research Center

Inventor: Mark W. Frye
Home Address: 1500 Hampton Highway
Hampton, Virginia 23666
SS
Employer: NASA-Langley Research Center
Inventor: Clarence E. Stanfield
Home Address: 1E Southampton Avenue
           Hampton, Virginia  23669
Employer: NASA-Langley Research Center
Initial Evaluator: James W. McNamara, Jr.
Serial No.: 239,260
Filed: September 1, 1988
PULTRUSION DIE ASSEMBLY AND METHOD

Origin of the Invention

The invention described herein was made by employees of the United States Government and may be manufactured and used by or for the Government for governmental purposes without the payment of any royalties thereon or therefor.

Background of the Invention

Field of the Invention

The present invention relates generally to pultrusion die assemblies, and more specifically, to pultrusion die assemblies capable of fabricating continuous high performance thermoplastic composite materials in thin, single ply, uni-axis sheets which can be rolled on a spool and stored for further laminating and molding procedures.

Description of the Related Art

Continuous graphite reinforced high performance thermoplastic composite materials have many uses. However, these materials are not widely available. Phillips Chemical Company offers a polyphenylene sulfide (PTS) product in a graphite reinforced prepreg. Imperial Chemical Industries offers a similar product using polyetheretherketone (PEEK), and others are
conducted research and development for other thermoplastic materials, including polyamide-imide (PAI) and polyetherimide (PEI).

Currently available extrusion methods and apparatus are not well suited for manufacturing continuous graphite reinforced thermoplastic composite materials.

U.S. Patent No. 4,541,884 to Coaswell et al. discloses a series of process steps that are used to produce thermoplastic impregnated carbon fiber tapes by a pultrusion process. The steps includes spreading the fibers, heating the fibers, and impregnating the fibers in molten thermoplastic. However, no die assembly or extrusion means is disclosed.

U.S. Patent No. 4,486,373 to Kurauchi et al. discloses a method for producing reinforced thermoplastic resin compositions utilizing an extrusion molding machine. The molding machine spreads the fibers prior to molding and uses water to cool the thermoplastic materials.

U.S. Patent No. 4,439,387 to Hawley discloses a die for making compound composite structures including thermoplastic resin with fibers embedded therein. The patent discloses a die having side entry ports for introducing fiber strands.

U.S. Patent No. 2,682,292 to Nagin discloses the use of various shaped dies to produce a variety of molded products. The molded products are made of thermoplastic materials reinforced with fiber. The thermoplastic material is pressurized and delivered to a mold cavity.
Summary of the Invention

An object of the present invention is to provide a pultrusion die assembly which is capable of being attached to a commercially manufactured extrusion machine and used to fabricate continuous graphite reinforced high performance thermoplastic composite materials.

Another object of the invention is to provide a pultrusion die assembly capable of producing thin, one ply, uni-axis sheets which can be wound on a spool and stored for further laminating and molding procedures.

Another object of the invention is to provide a pultrusion die assembly which can perform a variety of functions while multiple tows of high modulus graphite are pulled through its chambers which are simultaneously injected with a thermoplastic material.

Yet another object of the present invention is to provide a pultrusion die assembly capable of performing fiber spreading, collimation, heating, impregnation, consolidation, compaction, resin metering, and curing.

In a preferred embodiment, a pultrusion die assembly for attachment to an extrusion machine includes a die body having a profile die portion with an inlet end and an outlet end and a crosshead die portion having an inlet end and an outlet end, collimator means for spreading and collimating fiber tows entering the inlet end of the crosshead die portion of the die body, heater means for heating the crosshead die portion and the profile die portion of the die body and thus the fiber tows collimated by the collimator means, an impregnation cavity within the
profile die portion of the die body, in fluid communication with the crosshead die portion of the die body, for impregnating the heated and collimated fiber tows with molten resin injectable into the crosshead die portion of the die body thereby forming a composite, and cooling means near the outlet end of the profile die portion for cooling the composite to a temperature below a transition temperature of the resin, thereby forming a thin sheet which exits the outlet end of the profile die portion of the die body.

The collimator means cooperates with the crosshead die portion of the die body to form a central passageway which passes fiber tows to the impregnation cavity. Also, converging resin channels are formed above and below the central passageway for fiber tows thereby forming a confluence of the resin channels and the central passageway at a rearward end of the impregnation cavity. The impregnation cavity is tapered in steps to have a reduced height which becomes smaller towards the outlet end of the profile die portion of the die body.

In another embodiment of the invention, a pultrusion die process includes the steps of feeding fiber tows into an impregnation cavity of a die assembly, injecting a molten thermoplastic material into the die assembly, heating the fiber tows while feeding to a temperature of the molten thermoplastic material, impregnating the fiber tows with the molten thermoplastic material in the impregnation cavity of the die assembly, and cooling the impregnated fiber tows after leaving the impregnation cavity to a temperature below a glass transition temperature of the
thermoplastic material, thereby forming a sheet of fiber reinforced thermoplastic material.

These objects, together with other objects and advantages which will be subsequently apparent reside in the details of construction and operation of the pultrusion die assembly as more fully hereinafter described and claimed, reference being had to the accompanying drawings forming a part hereof, wherein like reference numerals refer to like parts throughout.

Brief Description of the Drawings

FIG. 1 is a top view, partially in section, of one half of the pultrusion die assembly according to the present invention;

FIG. 2 is a cross-sectional view of a pultrusion die assembly showing both halves thereof;

FIG. 3 is a rear view of the pultrusion die assembly of FIG. 2;

FIG. 4 is a front view of the pultrusion die assembly of FIG. 2;

FIG. 5 is a partial, detailed cross-sectional view showing the impregnation cavity of the pultrusion die assembly of FIG. 2.

FIG. 6 is a top view of the collimator which forms a part of the pultrusion die assembly of FIG. 2;

FIG. 7 is a side view of the collimator of FIG. 6;

FIG. 8 is a front view of the collimator of FIG. 6;

FIG. 9 is a top plan view of the pultrusion die assembly according to the present invention; and
FIG. 10 is a side view, partly in section, of the pultrusion die assembly of FIG. 9.

**Detailed Description of the Preferred Embodiments**

Referring to FIGS. 1 and 2, a pultrusion die assembly is referred to generally by the numeral 12 and includes a profile die portion 14 and a crosshead die portion 16. The profile die portion 14 is made of two profile die halves 18 and 20. FIG. 1 is a top view of the profile die half 20 which is disassembled from the half 18 illustrated in FIG. 2.

The profile die portion 14 includes an impregnation cavity 22 which is formed between die halves 18 and 20 and tapered in steps so that the height of the cavity decreases in the direction of the outlet end 24 of the profile die portion 14.

The profiled die portion 14 is provided with a plurality of bores 70 which receive threaded fasteners in order to assemble the two halves 18 and 20. Smaller bores 72 and 74 (not threaded) are provided for dowel pins (not shown) which help align the two halves for assembly. In addition, threaded bores 76, 78, 80 and 82 are provided for receiving screw jacks 84 which are used to push the two die halves 18 and 20 apart in the event of sticking due to the thermoplastic resin setting in the die. The screw jacks 84 are counter-bored so that ends of the screw jacks 84 which are normally disposed in one of the two halves 18, push against the opposite half 20 when screwed downwardly.

FIG. 4 illustrates the bolting pattern for bolting the two halves 18 and 20 of the profile die portion 14
to an opposite end of the crosshead die block 42. It should be noted that the two halves 18 and 20 are provided with flanges 86 and 88 similar to the flanges 62 and 64 of the halves 38 and 40 of the collimator 36.

As shown in FIG. 10, transducer-type pressure probes 152 and 154 are received in bores adjacent the largest segment of the impregnation cavity 22. The pressure probes 152 and 154 feed data to a controller (not shown). The controller guarantees the proper ratio of fiber to resin by controlling the rate of pull of the fiber tows and/or other process parameters in order to achieve a desired ratio.

Referring to FIG. 5, each segment 22a, 22b, 22c, 22d and 22e of the impregnation cavity 22 becomes progressively smaller until the smallest segment 22e merges into a thin passageway 26 which leads from the impregnation cavity 22 to an outlet 28 of the profile die portion 14. The largest segment 22a of the impregnation cavity 22 is at the confluence of a central passageway 30 and upper and lower resin channels 32 and 34 associated with the crosshead die portion 16 (FIG. 2).

The crosshead die portion 16 includes a collimator 36 which, like the profile die portion 14 is formed in two axially split halves 38 and 40. The collimator 36 is received in a crosshead die block 42 which is provided with a port 44 which connects an extrusion machine (not shown) to the pultrusion die assembly 12. Molten thermoplastic resin is injected through the port 44 and is guided by a radial channel 46 to the upper and lower resin channels 32 and 34 of the collimator 36. Details of the collimator 36 are illustrated in
FIGS. 6-8. The two halves 38 and 40 are assembled by threaded fasteners received in bores 48, 50, 52, 54, 56, and 58. Dowel pins 60 are used to align the two halves 38 and 40 prior to bolting the two halves together. Each collimator half 38 and 40 includes a flange portion 62 and 64, respectively, which define therebetween an inlet 66 for receiving the fiber tows. The flanges 62 and 64 are bolted to an end of the crosshead die block 42.

The outlet 68 (FIG. 8) of the central passageway 30 is disposed at a point of confluence between the central passageway 30 and the upper and lower resin channels 32 and 34. This facilitates impregnation of the fiber and consolidation and compaction of the impregnated fiber. FIG. 3 illustrates the bolting pattern for bolting the flanges 62 and 64 of the collimator halves 38 and 40 to the crosshead die block 42.

The inlet 66 is approximately three inches wide in one embodiment of the invention which is particularly suited for making a composite tape. The tape is preferably 40% resin and 60% fiber.

To begin the manufacturing process, the die assembly may be disassembled to feed the fiber tows into the central passageway 30 of the collimator 36 and to the thin passageway 26 of the profile die portion 14. In an alternative method, the pultrusion die is assembled and the fiber tows are then fed into the inlet 66 by placing the ends of the fiber tows between two pieces of thin tape having adhesive surfaces adhering to each other and to the ends of the fiber tows. Then, the tape is fed into the inlet 66 and
pushed through the pultrusion die assembly 12 until the tape extends through the outlet 28. The tape is then pulled through the die so that the fiber tows can be placed in an appropriate pulling apparatus (not shown). The tape, and the taped ends of the fiber tows, are discarded and the die is ready for injection of resin. The fiber tows are each yarns of about 1200 filaments. In the embodiment where the finished product or tape is about three inches wide, about twelve of these yarns are fed through the die.

Referring now to FIGS. 9 and 10, the pultrusion die assembly 12 is shown in views which illustrate heater ports and thermocouple mountings, as well as coolant ports. Near the outlet end 24 of the profile die portion 14 a series of four through-holes 90, 92, 94, and 96 are provided in two pairs above and below the thin passageway 26. These through-holes 90, 92, 94 and 96 may be tapped in order to attach cooling water supply lines so as to cool the lower portion of the profile die portion which corresponds to an area downstream of impregnation cavity 22. Thus, after the fiber tows have been impregnated in the impregnation cavity 22, the coolant passing through the through-holes 90, 92, 94 and 96 cools the composite by heat transfer below the glass transition temperature of the thermoplastic constituent of the composite prior to exiting the die through outlet 28.

Heater through-holes or ports 98, 100, 102, 104, 106, and 108 are provided along the two halves 18 and 20 of the profile die portion 14 in order to help control the temperature of the composite. Since the holes extend through the profile die portion 14, a
heating (or cooling) fluid may be circulated through the holes to achieve a predetermined temperature for a particular region of the die, or heating elements may be inserted into the holes. The crosshead die portion 16 is also provided with through-holes 110, 112, and 114 which allow the passage of a heating medium or fluid or insertion of heating elements, so as to raise the temperature of the graphite fiber tows to the temperature of the molten thermoplastic. Thus, the fiber tows are heated as the tows travel from the crosshead die portion inlet 66 to the thermoplastic impregnation cavity 22. Additional through-holes 116, 118, 120 and 122 are provided in the crosshead die block 42 for controlling heat transfer.

FIGS. 10 and 11 illustrate placement of heater ports 124, 126, 128, 130, 132, 134, 136, 138 and 140 which extend axially into the collimator 36 and the crosshead die block 42. The heater ports are capable of receiving either heating elements or a heater fluid. Thermocouples are disposed in bores 142 and 144 which extend into the collimator 36. The thermocouples provide the means for controlling the heating of the crosshead die portion 16.

The various heating elements can be controlled by a central controller to provide precise temperatures at different stages of the pultrusion die assembly. Basically, temperature is controlled such that the collimator 36 and thus the fiber tows passing therethrough, are heated to a temperature of the molten thermoplastic material which flows through the inlet port 44, through the radial channel 46, through the upper and lower resin channels 32 and 34 of the
collimator and into the impregnation cavity 22. Additional thermocouples are provided in the profile die portion 14 at bores 146, 148 and 150 in order to control the heating capabilities of the die. These thermocouples may be selectively energized in order to achieve a desired temperature. A thermocouple in bore 150 would be used to monitor the temperature at the exit end of the profile die portion 14. However, the pultrusion die assembly 12 may be adapted for use with a thermosetting resin, in which through-holes and thermocouples in the profile die portion 14 would be used to control the heat to a selective thermosetting temperature. Heater ports or thermocouples associated with the crosshead die block 42 and/or the collimator 36 would be converted for cooling purposes in order to prevent premature setting of the thermosetting resin.

The various heater ports and through-holes may likewise be adapted to receive heater cartridges instead of a heating medium or fluid. Again, the exact type of heating elements used depends on the particular type of hot-melt thermosetting resin to be used. In a preferred embodiment, the pultrusion die assembly 12 is capable of producing a hot-melt thermoplastic impregnated graphite prepreg tape to be used subsequently for laminating and molding. Other resin systems such as hot-melt epoxies could be pultruded using the same die assembly and methods. Fiber reinforcement other than graphite could also be used, i.e., fiberglass or aramid fiber. The extruder (not shown) could be used to deliver hot-melt epoxy into the impregnation cavity under pressure.
While the pultrusion die assembly described herein produces a thin flat tape having a width of about three inches and a thickness of about 0.011 inches, other shapes could be accommodated by altering the shape of the passageway 26.

Solvent resin systems consisting of thermoplastic or thermosets could also be accommodated by the present invention. Application of solvent resin systems would require attaching an air driven resin pump to the die instead of an extrusion machine for delivering resin to the impregnation chamber 22.

The many features and advantages of the present invention are apparent from the detailed specification, and thus, it is intended by the appended claims to cover all such features and advantages of the pultrusion die assembly which fall within the true spirit and scope of the invention. Further, since numerous modifications and changes will readily occur to those skilled in the art based upon the disclosure herein, it is not desired to limit the invention to the exact construction and operation illustrated and described. Accordingly, all suitable modifications and equivalents may be resorted to falling within the scope and the spirit of the invention.

What Is Claimed Is:
PULTRUSION DIE ASSEMBLY AND METHOD

Abstract of the Disclosure

A pultrusion die assembly includes a die body having a profile die portion and a crosshead die portion. A collimator spreads and collimates fiber 5 tows entering the crosshead die portion of the die body and heating elements are provided for heating the crosshead die portion and the profile die portion of the die body and thus the fiber tows which are collimated by the collimator. An impregnation cavity within the profile die portion of the die body is in fluid communication with the crosshead die portion of the die body and impregnates the heated and collimated fiber tows with molten thermoplastic resin injectable into the crosshead die portion of the die body thereby forming a composite. A portion of the die body downstream of the impregnation cavity is cooled so that the composite is cooled below a transition temperature of the resin after leaving the impregnation cavity. Thus, a thin sheet is formed which exits an outlet end of the profile die portion of the die body.