INTEGRATED FACILITY FOR CARBON FIBRES AND PREPREGS

A pilot plant for the development of carbon fibres and prepregs has been set up at National Aerospace Laboratories, Bangalore. The various facilities set up under this programme, and their nominal design capacities, are:

- Synthesis of polyacrylonitrile (PAN) copolymer (60 TPA)
- Continuous spinning of special acrylic fibres [SAF] (40 TPA)
- Conversion of SAF to carbon fibres [CF] (20 TPA)
- Preparation of 600 mm wide unidirectional prepregs (30 TPA).

The pilot plant is equipped with centralized utilities like the demineralised and soft water plant, steam boiler, water and brine chiller, nitrogen plant, etc. The pilot plant is equipped with fire safety systems, effluent treatment plant, solid incinerator, process exhaust systems etc. as per standard industrial norms.

The various processes like polymerisation, spinning, heat treatment etc. at this facility are controlled using a distributed control system (DCS). An ABB Freelance 2000 distributed control system with 840 I/O counts and operating on Digivis software has been installed for the purpose.

The pilot plant for the polymerization, spinning and heat treatment were designed based on information from literature and the group's expertise in the field of polymer science, fibre science and chemical engineering. The development of the processes for intermediate and final products, namely polyacrylonitrile copolymers, special acrylic fibres and the carbon fibres has been carried out directly on the above-mentioned scales without going through laboratory or bench scale experiments.

The facility has a well-equipped quality control and testing laboratory.

TECHNOLOGIES DEVELOPED

- Synthesis of polyacrylonitrile [PAN] copolymer
- Wet spinning of special acrylic fibres [SAF]
- Heat treatment of acrylic fibres to carbon fibres
- Resin formulation and prepreg preparation.

Synthesis of polyacrylonitrile copolymer

The PAN copolymer is synthesized by various processes. Standard industrial processes are aqueous slurry polymerization or solution polymerization. The aqueous redox polymerisation process is being used at NAL. The semi batch reactions are very flexible and offer good control of the reaction rate. The polymerization reactor has been designed for either batch or semi batch process. The semi batch process is being used for the synthesis of PAN copolymers. The kinetic equations for continuous polymerisation reactions have been modified to arrive at the process parameters like monomer concentrations, dozing rates, initiator and activator concentrations etc. for semi batch operation to prepare polymers of required molecular weight and molecular weight distribution.

The polymerisation process is carried out through the distributed control system where the flow rates and temperatures are closely controlled during the reaction, monomer stripping, and drying. Polymers with acrylonitrile content between 95 to 98-wt % have been prepared.

Over 100 polymerization reactions have been conducted and ~10 tonnes of the polymer has been prepared so far. The process conditions have been standardized to produce polymers with the desired composition, molecular weight, intrinsic viscosity and molecular weight distribution. These polymers have been spun into special acrylic fibres.

Wet spinning of special acrylic fibres [SAF]

The acrylic fibres are prepared from PAN polymer by the wet spinning process from polymer solutions. Dimethylacetamide is used as the solvent for spinning the acrylic fibres.

The fibre spinning unit consists of the dope preparation unit, extrusion unit, coagulation bath, washing and stretch baths, spin finish unit, driers, steam stretch unit and the winders. Auxiliary circulation systems are used to maintain the concentrations and the temperatures of the baths.

Acrylic fibres have been spun from the polymers prepared at IFCAP. 750-1600 litres of spin dope having solid content 16-22% have been prepared and spun into fibres. Fibre tows with 3000, 6000 and 12000 filaments have been prepared.

About 30 spinning operations, each of 100-150 hours duration, have been conducted. The effect of various process conditions, like dope temperature, dope concentration, coagulation conditions, stretches and stretching conditions, drying and collapsing conditions have been optimized. All the process conditions like stretch ratios and bath temperatures are controlled through the distributed control system.

Fibres with deniers 0.8-1.5 per filament [equivalent to 10-13.5 μM diameters] have been prepared. The tows have a tenacity of 4-4.5 gpd and the single filament strengths of these tows range from 6-8 gpd. Uninterrupted spinning for more than 100 hours has been carried out. The SAF prepared is being used to prepare carbon fibres.

Heat treatment of acrylic fibres to carbon fibres

The carbon fibres are prepared by the heat treatment of

acrylic fibres. The acrylic fibres are initially made infusible in the stabilization oven in an oxidizing atmosphere in the temperature range 200^{0}C-280^{0}C and then subjected to higher temperatures in the precarbonization and carbonization furnaces under nitrogen atmosphere to obtain carbon fibres.

The carbon fibre line consists of a 96 position let off creel for SAF fibres spools, an oxidative stabilization oven [multizone oven], a six zone low temperature [LT] precarbonization furnace with a maximum temperature up to 900^{0}C, a two zone high temperature [HT] carbonization furnace with a maximum temperature up to 1500^{0}C, a furnace with a maximum temperature up to 2600^{0}C for producing high modulus fibres, surface treatment bath, vertical dryer, an epoxy sizing unit, a second vertical dryer to cure the sizing agent and a 96 position winding unit. The gaseous products released during the different stages of pyrolysis are treated using a catalytic cracker located atop the stabilization oven, a fuel fired incinerator attached to the LT furnace and a lute pot along with the HT furnace. The gases produced during the pyrolysis are exhausted using a process exhaust duct. A HVAC system supplies clean filtered air into the heat treatment bay. The line has been designed to process 96 SAF spools simultaneously at speeds up to 2.0 meters / minute.

The carbon fibres are collected on the 96 position-winding unit. The winders are designed for handling the low elongation to failure of the carbon fibres. The winding speed should not deviate much from the feeding speed of the fibres to the winders.

About 30 continuous heat treatment operations have been conducted. The carbon fibres with tensile strength 2.8 to 3.8 GPa and tensile modulus up to 240-340 GPa have been prepared.

Resin formulation and prepreg preparation

A prepreg is a precursor to composites in which the collimated fibres are preimpregnated with the precise amount of formulated resin system. The resin, the curing agent and additives are formulated to get the required tack, toughness, processing and performance characteristics. Continuous fibre prepregs can be prepared by two impregnation methods. The first, by dissolving the resin in a suitable low boiling solvent and the other by a hot-melt, solventless process. In the hot melt process the additional process of solvent removal is eliminated but the resins used must have adequate chemical stability during formulation and impregnation and low viscosity at the impregnation temperature. The hot melt process is being used at NAL for making prepregs.

The hot melt process for making unidirectional carbon fibre prepregs consists of four steps. These are the production of a uniform resin film, impregnation of the collimated fibres, reduction of the prepreg temperature after impregnation, and take up. In the preparation of the thin resin film, the formulated resin is coated on a silicon coated release paper to desired thickness. This film is transferred onto the carbon fibres by sandwiching the fibres between two layers of film in the impregnating zone at a suitable temperature and pressure. After the film transfer, the prepreg is quenched and wound on rollers, packed, sealed and stored under refrigeration.

Standard prepregs of 300mm width using 175^{0}C curing epoxy resin systems with at least 60 days out of refrigeration life at 22^{0}C have been prepared. These prepregs have been characterized exhaustively for qualification and type approval.The characterizations include physicochemical analysis for prepregs and mechanical properties of the cured laminates.

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Project sponsored by DRDO and ADA

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