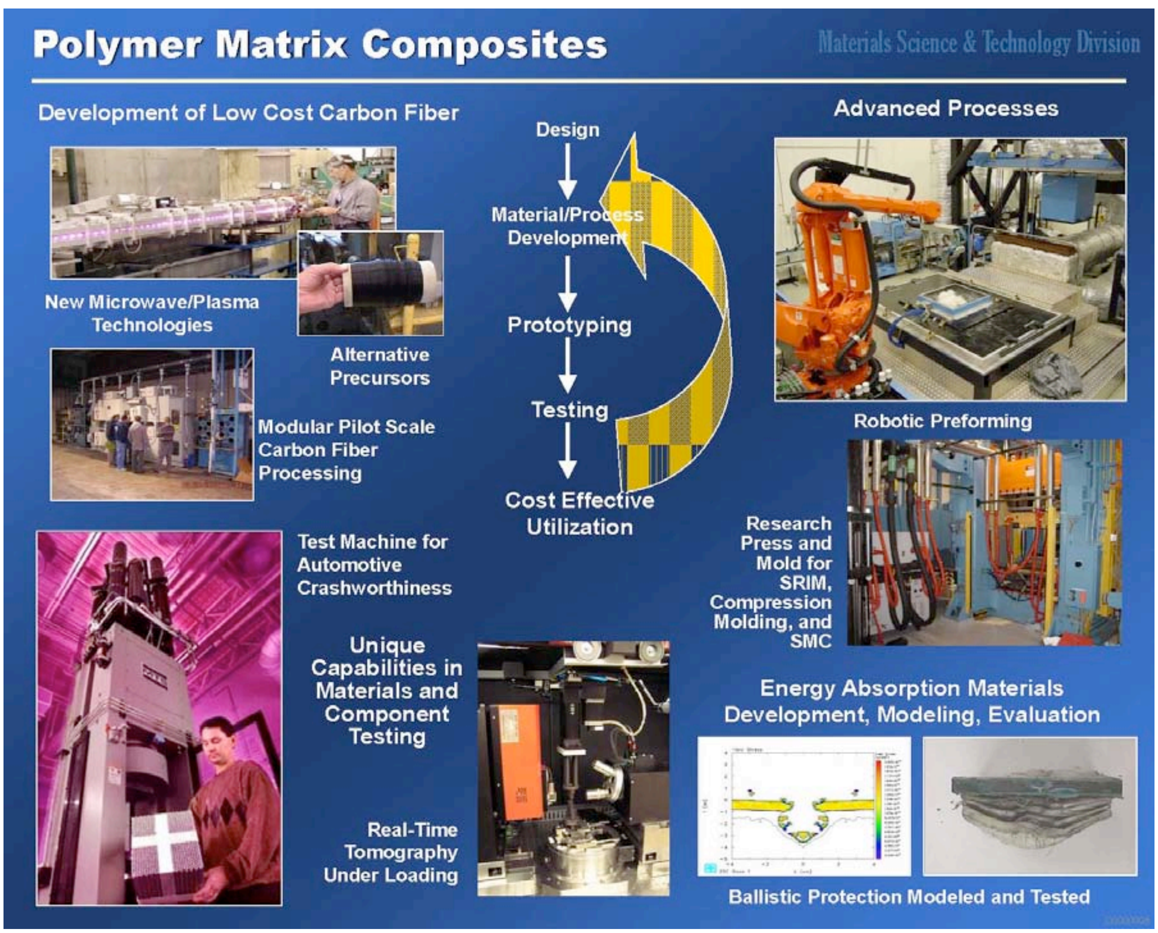
ORNL MS&T Division's Polymer Matrix Composites Group Capabilities and Expertise

Mission Overview: The Oak Ridge National Laboratory's Polymer Matrix Composites (PMC) Group provides comprehensive capabilities for the design, analysis, development, evaluation, and demonstration of affordable advanced composite materials and structures. The PMC Group functions as a key resource for research, development, prototyping, and industrial deployment of advanced composite materials applications and manufacturing technology. Working with industry, government, and academia, the PMC Group serves as a focal point for the development and demonstration of solutions to critical issues surrounding the manufacture of cost-effective composite materials, structures and assemblies.



Composites Competencies

Much of the ORNL composites activities are built around world-class capabilities in the following composites disciplines:

- **Design and Analysis -** Benchmarked design and analysis tools combined with material property databases and empirical data from manufacturing processes are used to produce composite part designs with predictable quality and performance.
- **Materials Characterization** – Utilization of equipment and facilities for establishing both constituent material properties and as-manufactured laminate design parameters. Material property data for fiber, resin, and lamina properties are then correlated with manufacturing process effects to yield predictable behavior and design allowables.
- **Materials Development** – Program and technological leadership in development of low cost carbon fibers, alternative precursor materials, formulation of radiation-processable materials, tailored sizings for carbon fibers, materials for energy absorption, etc.
- **Processing Technology** - Facilities and process equipment combine the flexibility to support development of new manufacturing concepts with the capacity to produce full-scale demonstration components. Advanced processing techniques utilizing electron-beam and microwave energy, plasmas, robotic-preforming, high performance filament winding, etc.
- **Testing and Evaluation -** Fabrication and testing of state-of-the-art composite materials for the characterization of material systems from the receipt of starting materials through finished products..

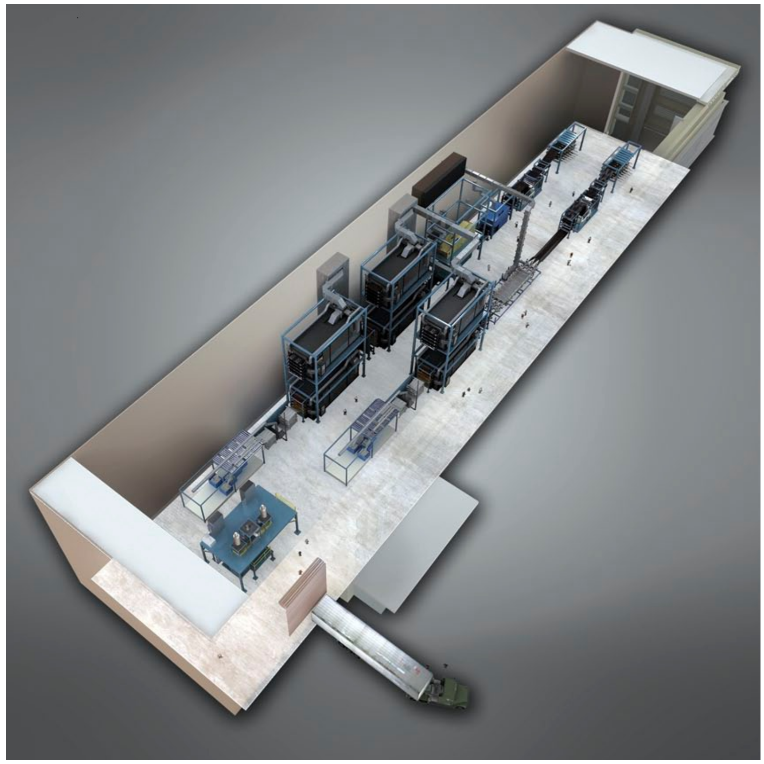
Relevant Technologies and Programs

Carbon Fiber Development

ORNL is presently leading a major DOE initiative to develop several technologies key to producing low cost carbon fiber. An effort is underway to develop precursors from renewable resources and other alternative source materials while other projects utilize microwave energy in conjunction with plasmas in conversion of precursors to carbon fiber. The objective is to produce carbon fiber on a production scale using these technologies. If successful, carbon fiber cost may be significantly reduced and use in automotive and other industrial and commercial applications may be increased many fold.

This effort is part of a larger Department of Energy (DOE) program to promote the use of lighter weight materials in automotive structures - the Lightweight Materials Program which is encompassed in the Freedom Car initiative. The availability of low cost carbon fiber is a necessary condition for the widespread use of lightweight composite materials in automotive structures.

ORNL was recently awarded $34.7M in ARRA funding from DOE to design, construct, and operate a highly flexible, highly instrumented Low-Cost Carbon Fiber (LCCF) Technology Demonstration Facility for demonstrating and evaluating new low-cost manufacturing processes and technologies at pilot scale. This Facility will increase the nation's investment in energy technology and move the United States toward a cleaner and more sustainable energy future. The Facility will promote successful LCCF commercialization, which has the potential to save energy and advance the development of renewable energy. This facility will strengthen the capabilities of ORNL and develop the necessary infrastructure to enable key scientific and engineering breakthroughs in LCCF, an area of strategic interest to the U.S. economy. Centerpiece of this initiative is acquisition of a custom-designed thermal (conventional) carbon fiber conversion line and a melt-spinning precursor fiber production line to be housed in a dedicated demonstration Facility. In addition, the Facility will have the capabilities to:



- Demonstrate the scalability of the science and technology for lowering the cost of carbon fiber by at least 50%
- Produce and make available up to approximately 25 tons/year of conventionally converted fibers (made from low-cost precursors) to potential end users and upper-tier suppliers in multiple industries
- Accommodate the future addition of an advanced technology conversion line
- Produce precursor fibers made from a variety of precursor materials, e.g., lignin and polyolefin, on a melt-spinning line in sufficient quantity to feed the conversion lines
- Enable industrial and university collaborations to effectively leverage expertise of Laboratory personnel in developing low-cost manufacturing techniques for carbon fiber components
- Educate and train a highly skilled future workforce for LCCF implementation

In related work, ORNL is also working to develop lower cost carbon fiber alternatives to standard modulus, but relatively high strength products utilized in construction of high pressure tankage and an advanced fiber with properties (especially tensile strength) exceeding state-of-the-art high performance carbon fiber.

Composites Design And Analysis

Computing platforms and analysis codes for micromechanics, laminate theory, and ply-by-ply stress analysis are available in the design center. Also available are custom design methodologies for correlation of fiber, resin, and lamina properties with processing effects and failure models to assess predictable behavior and design allowances. ORNL-developed design codes based on closed-form analytic solutions are available for analyzing axisymmetric composite structures for both axisymmetric and nonaxisymmetric loads. Special codes are also available for stress analysis of laminated plates and buckling analysis of laminated shells. The design tools based on closed-form solutions are complimented by special purpose and commercial, general purpose finite element codes. An integrated experimental and analytical approach is often used for designing lightweight composite structures.

Structural Molding

ORNL maintains the capability to mold complex three-dimensional composite hardware by an assortment of manufacturing methods, which include vacuum/pressure-assisted resin transfer molding (RTM). Experience includes collaboration with an industrial partner to mold low-cost fiberglass replicas of Apache helicopter transmission components for a training simulator. These components provide substantial cost savings over the actual helicopter components while still being functionally adequate for the training simulator application.

Material And Structural Testing and Test Development

ORNL has extensive capabilities for characterizing metals, ceramics, polymers and their composites from coupon level to structural components. Typical mechanical testing equipment is available for static, dynamic, creep, fatigue, and impact testing at room, elevated, and low temperatures. Additional capabilities are utilized for testing the composite constituents (i.e., fiber, matrix) independently. Unique test capabilities include a strand farm for stress life characterization of fibers; split-disk stress life machines; flywheel spin test facility; photomechanics laboratory for high sensitivity, full-field deformation measurements; ORNL also develops custom test fixtures and facilities for unique materials of test situations.

Electron Beam Curing of Composites

ORNL has been a national leader in Electron Beam Curing of Composites, using high energy electrons and/or X-rays to cure polymers at controlled rates. Curing is volumetric versus diffusion controlled and the degree of cure more related to absorbed radiation than temperature environment at cure. In addition to very fast curing the process allows long pot life and selectable cure temperature lending itself to unique parts/processes, inexpensive tooling, and capability for curing thick sections via X-ray. ORNL won an R&D 100 award for advancements in this technology.

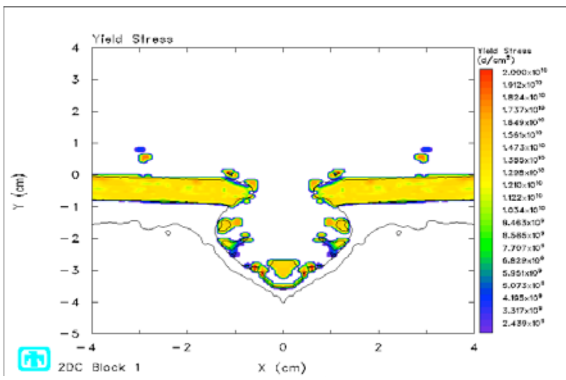
	Traditional Thermal Curing	E-Beam Curing
Resin System	Resin and Hardner	Radiation-Sensitive Resin No Hardener
Cure Temperature	High	User Selectable
Cure Speed	Slow	Fast

Reversible Microwave Bonding

ORNL has developed reversible microwave bonding techniques for a number of applications. The objective is to bond composite structures using microwave energy to process the adhesive, then to use microwave energy to soften the adhesive while maintaining the integrity of the composite materials. This will allow for construction of fastenerless composite material structures that can be disassembled and reassembled for inspection or repair.

Composites for Energy Absorption

ORNL has developed and demonstrated a variety of materials and materials systems for energy absorption applications including ballistic armor, blast protection for aircraft, and mitigation of sympathetic detonation. For this work, ORNL develops and employs start of the art composites materials and processing and structural integration techniques. Computer models of high-rate load events are utilized to guide initial approach and system optimization.

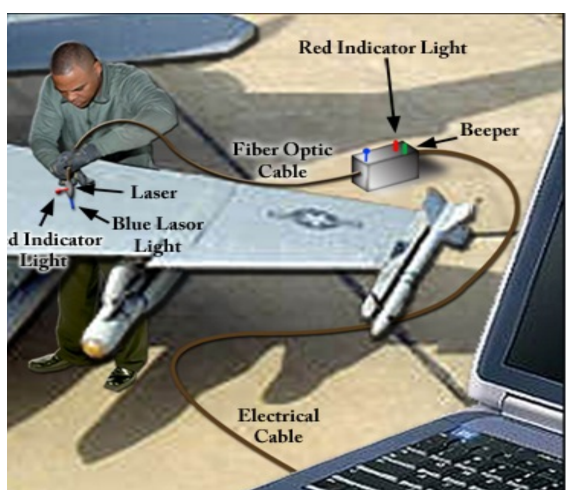


Deepwater Structures

ORNL designed, developed, and built a positive buoyancy graphite-epoxy pressure hull for the U.S. Navy Advanced Unmanned Search System. The composite pressure hull, which has a thickness of 2-1/2 inches and has titanium end caps, is qualified for repeated dives to 20,000 feet.

Demonstrated Detection/Quantification of Composite Heat Damage

ORNL has developed and demonstrated a reliable, nondestructive method for heat damage detection. It consists of portable LIF (laser induced fluorescence) instrument (9” x 12” x 8”) and laptop computer. It is lightweight (< 15 lbs.), uses Class 3A laser (same Class as laser pointer) and provides real-time heat damage detection response (10-50 measurements/second). The device can evaluate large, small, and limited access areas and can be operated by a single technician. The



current design is for depot level use, but a robust, field deployable unit is possible. This technology won a 2008 R&D 100 Award.

Advanced Preforming Development

ORNL is working collaboratively with the Automotive Composites Consortium and other industrial and academic partners to further develop, demonstrate, and implement advanced fiber preforming processes such as the Programmable Powdered Preforming Process (P4) initially introduced by Owens Corning.

ORNL's Advanced Preforming Machine

Adhesive Bonding For Mass Production Applications

ORNL, in collaboration with the Automotive Composites Consortium (ACC), is developing adhesive bonding and hybrid joining technologies for current and future automotive materials. Capabilities are multidisciplinary and include researchers who are chemists, polymer scientists, and mechanical engineers. Current research activities include test method development, bulk materials characterization, modeling, process control and non-destructive evaluation, and advanced processing. Successes include:

- Fracture mechanics based test methods have been developed for similar and dissimilar materials. Test procedures been transferred for use by the automotive industry and their suppliers.
- Three rapid-cure technologies have been demonstrated which reduce adhesive curing times by 60 to 90% (2 patents granted).
- Adhesive additives have been developed to enhance cure rates with alternative processing techniques.

Materials Characterization

The following physical resources are available in ORNL's Materials Science and Technology Division and are frequently employed in characterization of composite materials. Numerous additional, state-of-the-art scientific instruments are available elsewhere throughout the laboratory. Much of the listed equipment and data acquisition software has been custom designed and built for transportation composite materials research.

Mechanical Test Systems

- 1 – Controlled strain rate, servohydraulic axial test system

- constant velocity via iterative learning mode
- compression or tension
- 110 kip static load capacity
- 60 kip capacity @ 200 inches/sec
- Unloaded @ 300 inches/sec
- 100 kip side load capacity
- Specimens up to ~30" long x ~18" dia
- 10" maximum actuator stroke
- Modular, high speed data acquisition
  - 4 synchronous analog inputs @ 12 bit resolution and 10 MHz sampling rate
  - 16 multiplexed analog inputs @ 16 bit resolution and 333 kHz sampling rate
- High speed video
  - < 10 μs shutter speed
  - 1,000 – 10,000 frames per second

- 1 – High velocity impact test facility
- 1 – Low velocity impact test facility
- 1 – 50/10 kip servohydraulic axial test system
- 2 – 25 kip hydraulic testing machine: tension, compression, fatigue, -300° to +250°F
- 3 – 10 kip hydraulic testing machine: tension, compression, fatigue
- 3 – 50 kip/25 kip-in axial torsional test systems
- 2 – 110 kip axial torsional machines
- 3 – 5 kip servohydraulic test systems
- 1 – 100 kip servohydraulic test system, axial-compression specific, thermal shock capable
- 1 – high resolution crosshead tensile testing machine w/ 300 gram load cell
- large diameter pipe test facility
- 5 –- tension creep test stands
  - 4 – 0° to 600°F, 1200 lbs
  - 1 – 0° to 400°F, 600 lbs
- 180 – strand testing fixtures
- 10 – split D ring stress life stands, maximum temperature 350°F, vacuum, lever loaded to 20,000 lbs
- 1 – Internal/external hydraulic pressure system, 8,000 psi static, 7,000 psi fatigue

Testing Capabilities

- Controlled strain rate tensile, compression – strength, stiffness, yield stress
- Controlled crush characterization of energy management components at intermediate and high rates
- Static tensile, compression, shear – strength, stiffness, yield stress
- Combined axial/torsional loading

- Fatigue – machines capable of 0-50 Hz depending on specimen displacement with in-house developed data systems and control. Allows automation of test procedure, data acquisition and analysis.
- Automated creep and creep-rupture
- Fracture – Mode I, II and mixed-mode fracture toughness tests employing DCB, ENF, compact tension, and center notch tension specimens. Fractomat automates crack growth and specimen compliance measurements. Hi-Res video for in-situ high magnification crack monitoring.
- Impact and strength after impact
- Biaxial
- Stress-life of strands and split-D rings
- Internal and external pressurization
- High/Low Temperature – RF inductance heaters, 5 @ 50 kW
  - LN2 to 800°F oven
  - portable LN2 chambers
  - quartz furnaces, to 2800°F
- Environmental Testing
  - various liquid submerged systems
  - 3 humidity chambers: 0 to 100%, 32° - 300°F, 17" x 23" x 18"
  - dry chambers to 5%
  - hygrothermal environments
- Dynamic Mechanical Analysis
- Dynamic Scanning Calorimetry
- Electrical resistivity of fibers
- Ultrapycnometer density measurement
- Dynamic Contact Angle measurement
- Optical microscope, w/image processing capability, 50X – 750X magnification
- Viscometers
- Non-Destructive Evaluation (numerous proven and developmental techniques)
- Structural Health Monitoring
  - Fiber optics
  - Modal analysis
  - Heat damage detection
  - Thermography

Photomechanics Laboratory

- Two portable compact moiré interferometers (full-field deformation measurement w/ spatial resolution ~1μm, displacement resolution ~400nm)
- Holography, photoelasticity, speckle
- Assorted optical tables, optics, lasers and associated hardware

Data Acquisition/Control

- Custom data acquisition/control systems employing
  - National Instruments PCI cards
    - 20 – 100 kHz, 16-bit A/D and 12- or 16-bit D/A conversion
    - Data acquisition and control software, programmed in LabView, customized to experiment needs
- Standard software packages written and currently used in house include:
  - Stress/strain/strength/stiffness data acquisition and analysis
  - Load-displacement data acquisition
  - Fatigue with compliance vs. cycles (axial/torsional) data acquisition
  - Creep/recovery data acquisition
  - MTS Microprofiler control
  - MTS 407 control
  - General programs to monitor transducers including temperature, strain, load, displacement, etc.

Materials Processing

The following processing equipment is employed in carbon fiber development and composites projects at ORNL:

Precursor Evaluation Line

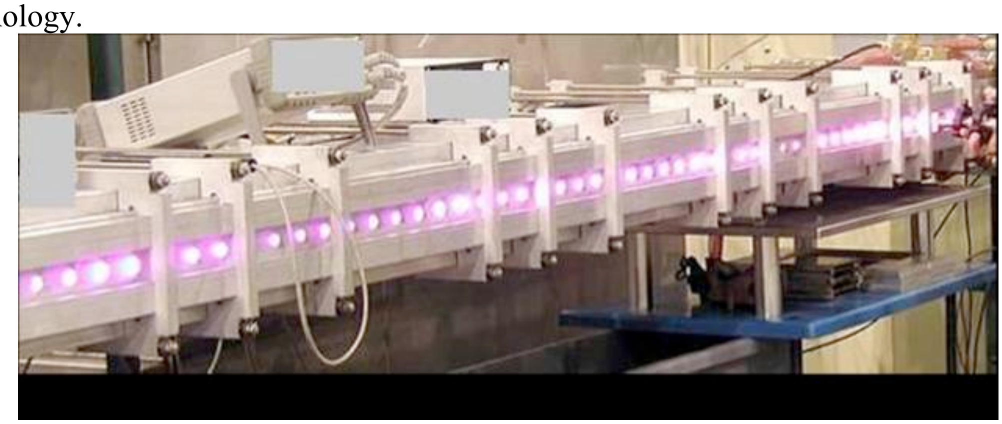
This system is designed for development of conventional processing recipes (oxidative stabilization, carbonization, and graphitization) with limited quantities of precursor from single filaments up to thousands of filaments. Residence time, temperature, atmospheric composition, and tension are independently controlled in each furnace can process and precise tension control allows tensioned processing of ~20-filament tows.

Carbon Fiber Pilot Line

This system is 1:20 scale of a commercial grade production line and has capacity for 8 tows. Upgrades are underway for automated operation and production of high strength carbon fiber. This system is nominally capable of producing a ton per year of carbon fiber and is a unique capability among FFRDC's and universities.

Carbon Fiber Demonstration Line
Recently funded by the Department of Energy, implementation of this facility will enable demonstration of the scalability of the science and technology for lowering the cost of carbon fiber by at least 50%. The facility can produce and make available up to approximately 25 tons/year of conventionally converted fibers (made from low-cost precursors) to potential end users and upper-tier suppliers in multiple industries. It also can accommodate the future addition of an advanced technology conversion line and produce precursor fibers made from a variety of precursor materials, e.g., lignin and polyolefin, on a melt-spinning line in sufficient quantity to feed the conversion lines. This capability will enable industrial and university collaborations to effectively leverage expertise of Laboratory personnel in developing low-cost manufacturing techniques for carbon fiber components and educate and train a highly skilled future workforce for LCCF implementation.

Microwave-Assisted-Plasma (MAP) Developmental System
System is used to carbonize carbon fiber at high rates utilizing traditional (vacuum) plasmas generated and controlled via microwave energy. In its current configuration, we have demonstrated single large tow line speed of ~ 5 m/min and 3-tow processing at > 1 m/min. System is currently being scaled to larger bandwidths and higher line speeds. This advanced process has significantly reduced fiber residence time versus conventional technology.



Advanced Oxidative Stabilization Developmental System
System is utilized to oxidize and stabilize carbon fiber, primarily via atmospheric pressure plasma. This advanced process has also significantly reduced fiber residence time versus conventional technology.

Surface Treatment and Sizing Modules
In a recently funding project, ORNL is developing equipment and technological means to tailor surface treatments and sizings of carbon fiber to specific resin systems of interest for automotive applications. Surface treatment enhances fiber-matrix bonding and reduces quantity of fiber needed in the structure. Preliminary ORNL plasma surface treatment results are far superior to conventional treatment results in terms of targeted surface chemistry. Capabilities being developed in this activity will be useful to other resin systems and for other applications.

Filament Winding

ORNL has world-class filament winding capability, typically producing high fiber fractions with low void content resulting in exceptional performance. Our primary developmental unit is computer-controlled, 4-axis, and can currently produce parts up to 30inches in diameter and 12 ft long.

Preforming

Built by Aplicator, this machine has capability to process a wide variety of materials from thermoplastics to thermosets, carbon, glass, and/or hybrid reinforcement, and various chopping configurations including continuously variable programmed cut fiber lengths over a broad range. Powdered binder can also be sprayed in at various rates. With a developmental chopper, disposition rates approaching 20 kg/min have been achieved.

Press/Molding Capability

A Williams White 750-ton net ton press with intelligent leveling is used for molding operations. A highly instrumented 24in X 24in plaque mold is utilized for demonstration and property determination. This system can be utilized for compression molding of SMCs and thermoplastics in addition to injection/compression of SRIM.