# Interfacial Properties of Electron Beam Cured Composites

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# Project Overview

## Objectives:

- **Understand and resolve material property deficiencies, specifically -**
  - **interlaminar shear strength**
  - **toughness**
- **Understand processing effects; define a desired process envelope; develop process control protocols and proven processing procedures**

  *In carbon fiber reinforced, electron beam cured, cationic epoxy matrix composites*

- **Budget: $3.3M ($1.1M government, $2.2M industrial)**
- **Schedule: 3 years commencing April 1999**

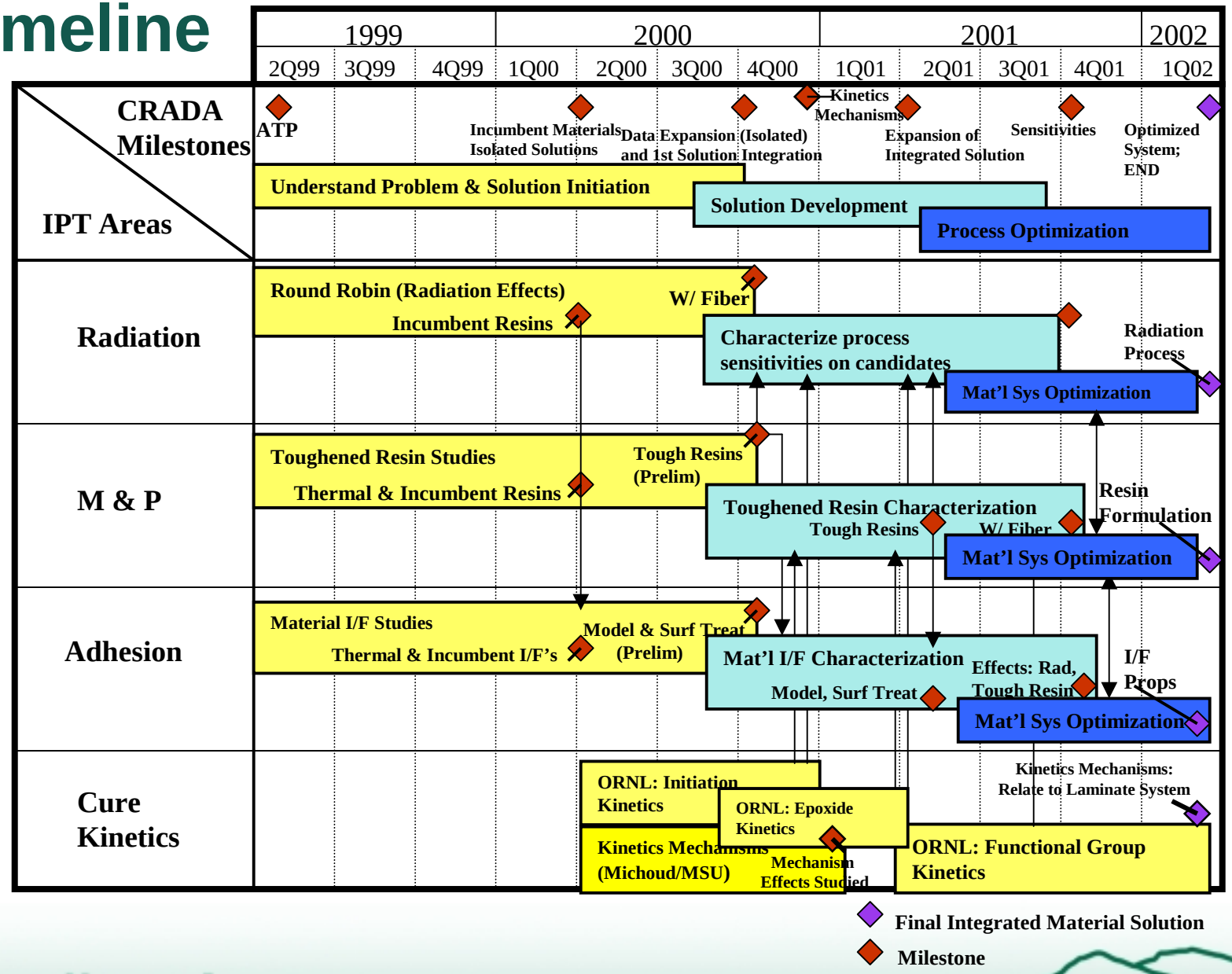
# Project Team

- Government Sponsors
  - US Dept. of Energy, Office of Science, Laboratory Technology Research Program
  - Air Force Research Laboratory
  - Army Research Laboratory
  - NASA Langley
- Research Institutes
  - Oak Ridge National Laboratory
  - Michigan State University
  - Texas A&M University
- End Users
  - Boeing (Phantom Works)
  - Lockheed Martin
    - Aero - Fort Worth (lead)
    - Michoud Space Systems
- Fiber Manufacturers
  - BP Amoco
  - Hexcel
- Irradiators
  - Acsion Industries
  - E-BEAM Services
  - STERIS
- Resin Suppliers
  - Applied Poleramic
  - UCB Chemicals
- Specialty Materials, Prepregs
  - Adherent Technologies
  - YLA

# Integrated Project Teams (IPT's)

| *IPT* | *Leader* |
| --- | --- |
| • Adhesion | M. Wilenski (Boeing) |
| • Materials & Processing | C. Janke (ORNL) |
| • Irradiation | V. Lopata (Acsion) |
| • Kinetics | J. Choi (Lockheed Martin) |
| • Leadership | P. Kirn (Lockheed Martin)<br>C. Eberle (ORNL) |

# Timeline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CRADA Milestones
IPT Areas
ATP
Incumbent Materials Isolated Solutions
Data Expansion and 1st Solution
(Isolated) Integration
Kinetics Mechanisms
Expansion of Integrated Solution
Sensitivities
Optimized System; END
Understand Problem & Solution Initiation
Solution Development
Process Optimization
Radiation
Round Robin (Radiation Effects)
Incumbent Resins
W/ Fiber
Characterize process sensitivities on candidates
Radiation Process
Mat'l Sys Optimization
M & P
Toughened Resin Studies
Thermal & Incumbent Resins
Tough Resins (Prelim)
Toughened Resin Characterization
Tough Resins
W/ Fiber
Resin Formulation
Mat'l Sys Optimization
Adhesion
Material I/F Studies
Thermal & Incumbent I/F's
Model & Surf Treat (Prelim)
Mat'l I/F Characterization
Model, Surf Treat
Effects: Rad, Tough Resin
I/F Props
Mat'l Sys Optimization
Cure Kinetics
ORNL: Initiation Kinetics
ORNL: Epoxide Kinetics
Kinetics Mechanisms (Michoud/MSU)
Mechanism Effects Studied
ORNL: Functional Group Kinetics
Kinetics Mechanisms: Relate to Laminate System
Final Integrated Material Solution
Milestone

# Exit Criteria (Performance)

| Level | Performance Criteria | Exit Supporting Data Type | Target Value |
|---|---|---|---|
| 1 | Thermal Performance | Wet Tg by DMA<br>(DMA from –67F to 400F+) | 290F, min; 375F target<br>No change in E' from –67 to RT |
| 1 | Resin Toughness | K1c via Single Edge Notch Bend @ RT<br>(ASTM D5045-93 SENB) | .82 ksi*in^.5 min<br>1.6 ksi*in^.5 target<br>3.2 ksi*in^.5 (8551-7) |
| 1 | Resin Tensile Behavior | Neat resin tensile test @ RT | 12 ksi min, 16 ksi target |
| 1 | Resin Weight | Resin Density | <= 1.4 g/cc |
| 1 | Quality laminates: Fiber Volume, Void Content, Resin Content | RC/FV/VV by Acid Digestion<br>SEM of laminate cross-section | VV <= .5%, FV = 60+/-3% |
| 1 | Quality laminates: Resin is sticking to the fiber | SEM of a mechanical coupon after failure | Compare to SEM of thermals |
| 1 | Good Resin props and good fiber resin interaction | 90 Flex OR Transverse Tension | 9 ksi target for transverse tension |
| 1 | Good Interlaminar Shear | 0 Short Beam Shear (SBS) Test @ RT | 15 ksi min, 18 ksi target |
| 1 | Good Interlaminar Shear and compressive resin props | 0 Flex (look for fiber tension failure before compressive resin failure) | TBD |
| 2 | Performance after exposed to jet fuel | 0 SBS after jet fuel soak | 95 % retention, min |
| 2 | Performance after exposed to solvents during assembly | 0 SBS after IPA soak<br>0 SBS after Acetone soak | 95 % retention, min |
| 2 | Performance after exposed to hydraulic fluid | 0 SBS after hydraulic fluid soak | 95 % retention, min |
| 2 | Tension Performance<br>(typically fiber dominated) | 0 Tension @ RTA, HW<br>(Strength/Modulus ksi/msi) | IM7: 375/23(RTA), 340/21 (HW)<br>AS4: 315/20.5(RTA), 280/20 (HW) |
| 2 | Compression Performance<br>(resin and some fiber dominated) | 0 Compression @ RTA, HW<br>(Strength/Modulus ksi/msi) | IM7: 255/22(RTA), 185/18 (HW)<br>AS4: 230/21(RTA), 175/18 (HW) |
| 2 | In-plane shear Performance<br>(resin-fiber inter action) | IPS (+/- 45) Tension @ RTA, HW<br>(Strength/Modulus ksi/msi) | IM7: 16/.8 (RTA), 13/.5 (HW)<br>AS4: 13/.8 (RTA), 11/.5 (HW) |
| 2 | Robustness with holes (required for typical assemblies) | (OHC) Open Hole Compression (QI lay-up) @ RTA,HW<br>Compression (QI lay-up) @ RTA | 70% retention |

# Exit Criteria (Processing)

| Level | Processing Criteria | Exit Supporting Data Type | Target Value |
|---|---|---|---|
| 1 | Debulking Temp and sensitivity (if any) to elevated temp prior to cure | Viscosity vs. Temp<br>Viscosity at debulk temp vs. time | <=160F debulk |
| 1 | Resistant to Hazardous Exotherming | DSC of uncured (end-users need to know what NOT to do with uncured materials)<br>Temp profile as a function of cure parameters | No hazardous exotherming |
| 1 | No hazardous by-products – do not exceed normal shop protection req'ts (latex gloves, little or minimal use of respirator) | TBD | TBD |
| 1 | Processing: resistance to creating in-situ porosity | Volatile content of resin | <= 1% |
| 1 | Processing: Out time – exposing pre-preg to normal light and humidity (65%RH, 75F) | SBS as a function of pre-preg moisture exposure<br>SBS as a function of light exposure | 95% retent<br>16 hours |
| 1 | Processing: Cure profile; Process Window | Properties (DMA, SBS) as a function of cure profiles | |
| 1 | Availability | Supplier chain list | Na |
| 2 | Shelf Life | TBD: Accelerated Age Testing (SBS)<br>Note: Save material at end of CRADA for real time age evaluation (SBS) | 3 years @ RT |
| 2 | Quality: Good Wet-out | Discussed in April '00 M&P telecon | Qualitative |
| 2 | Quality: Good Tack | TBD (vertical stick type test) | Qualitative |
| 2 | Quality: Good Drape | TBD | Qualitative |
| 2 | Capability to B-stage | DMA as a function of delivered dose<br>Visual appearance as a function of dose | B-stageable is desirable |

# Adhesion Status

- **Fiber-matrix interface chemistry has been well characterized**
- **Fiber-matrix adhesion characterization nearing completion**
- **Fiber-matrix adhesion has been shown to significantly contribute to low interlaminar shear strength**
- **Several methods for re-engineering the fiber-matrix interface/interphase are currently under investigation**
- **Progress toward improving fiber-matrix adhesion is encouraging, with some techniques increasing interfacial shear strength by 25% - 50%**

# Materials & Processing Status

- Toughening results are quite encouraging; toughness appears to be comparable to that of SOA thermally cured, toughened  resins
- Data validation is underway
- Further toughening will be attempted
- Evolution of laminate fabrication techniques continues
- Resin tensile strength demands further attention

# Irradiation Status

- Irradiators support the other IPT's
- Irradiation parametric effects is a topic of intense study and interest in all IPT's
- Preliminary "end user" predictive code, beta version, completed and tested against various accelerators
- Irradiation "round robin" executed on neat resin specimens; data is still being evaluated
- More focused experiments are planned

# Kinetics Status

- Kinetics studies commenced recently
  - Product identification and reaction kinetics
    - Effects of various parameters on reaction products
  - Reactive intermediates
    - Identify reactive intermediates, map reaction pathways and rates
  - Epoxide consumption
    - Monitor change in concentration of reactive sites and correlate with various parameters
- Initiation and consumption studies this CY
- Studies are being coordinated with kinetics work performed outside the CRADA
- Long term goals (beyond the CRADA) are to inform the creation of robust analytical curing and processing models

# Summary

- Fiber-matrix adhesion has been shown to significantly contribute to low interlaminar shear strength
- Interfacial shear strength has been increased by 25% - 50%
- Recent data indicates greatly increased toughness; validation is underway
- Reaction mechanics and kinetics have emerged as important parameters that will be studied intensively over the next year
- CRADA is ahead of milestone schedule; transitioning from characterization to solution development phase
- Current progress provides reason for optimism that project goals will be achieved