Development of Ionic Liquid Based Epoxies for Carbon Fiber Composite Cryogenic Tanks

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Introduction

Ionic Liquids:

Basically "salts" that are liquid at some subjective low temperature, say < 100°C; Largely composed of ions.

Their discovery dates back to the 1800's.

Considerable advances made since then, applications/uses include:

- Battery Electrolytes
- Cellulose Processing
- Metallic Ore Refining
- Biofuel Processing
- Rocket Fuels
- Epoxies
- Many Others

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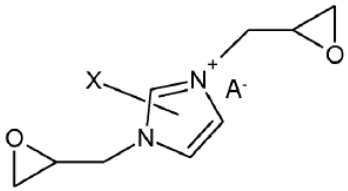
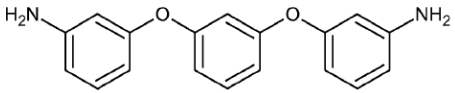
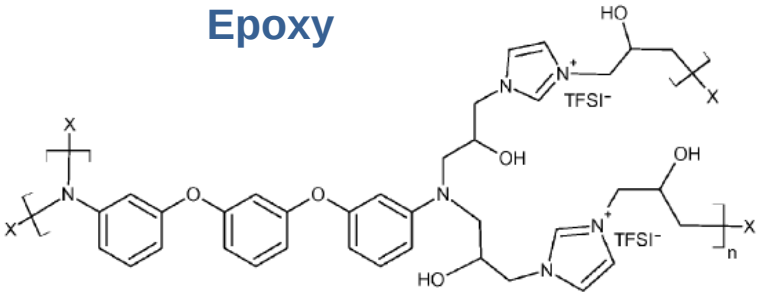
Epoxy Monomer + **Curing Agent** = **Epoxy**



1,3-bisglycidylimidazolian



Bis-Aminophenoxybenzene (APB)



ILEP-2

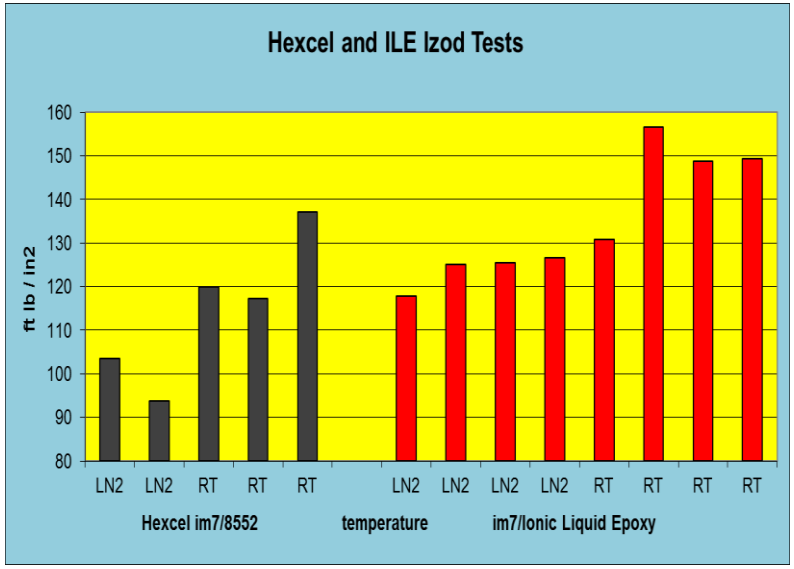
Ionic Liquids as a Base for Epoxy Resins

Number of advantages for NASA Applications:

- Extremely Low Vapor Pressures
- Comparatively Better Low Temperature Strength
- Low Flammability
- Low Hydrogen Permeability
- Hydrophobic
- Low Coefficient of Thermal Expansion
- Comparatively "Greener" Manufacturing Process

Also Adheres well to Aluminum

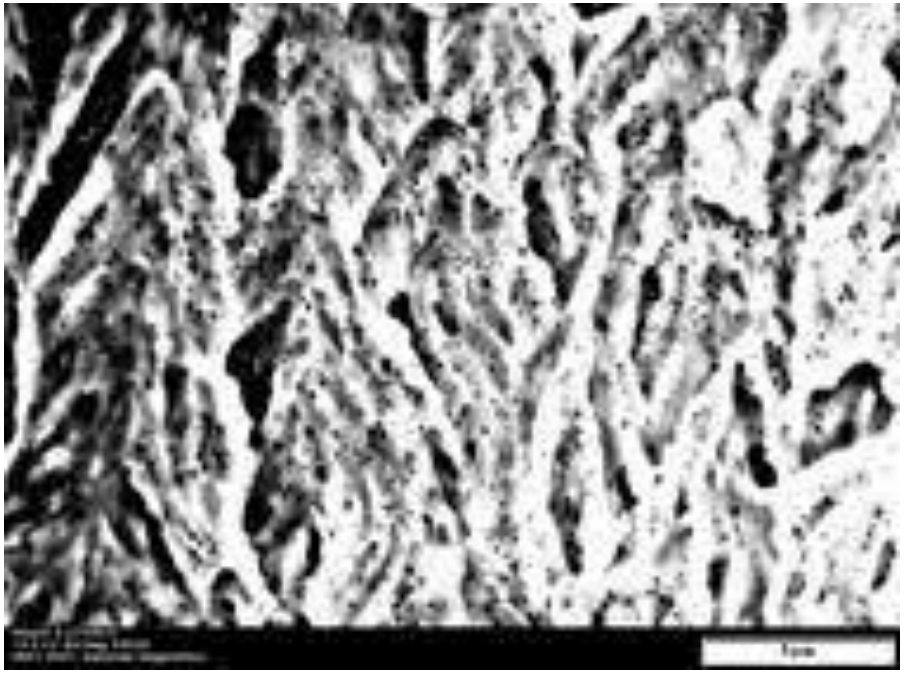
Mechanical Testing: Initial Results



EPOXY	TEST VALUE	TEMP
Button adhesion		
ILEP-2	8400psi	RT
ILEP-2	9100psi	LN2
Lap shear		
ILEP-2	1833psi	RT
Epon 828	1350psi	RT
Tensile		
ILEP-2	8675psi	RT
Epon 828	6004psi	RT
Epotek 301-2	3751psi	RT
Hysol 9361	1153psi	RT
Tra-Con F 113	2539psi	RT
Epotek 301-2	6783psi	100 K
Hysol 9361	4225psi	100 K
Tra-Con F 113	7092psi	100 K

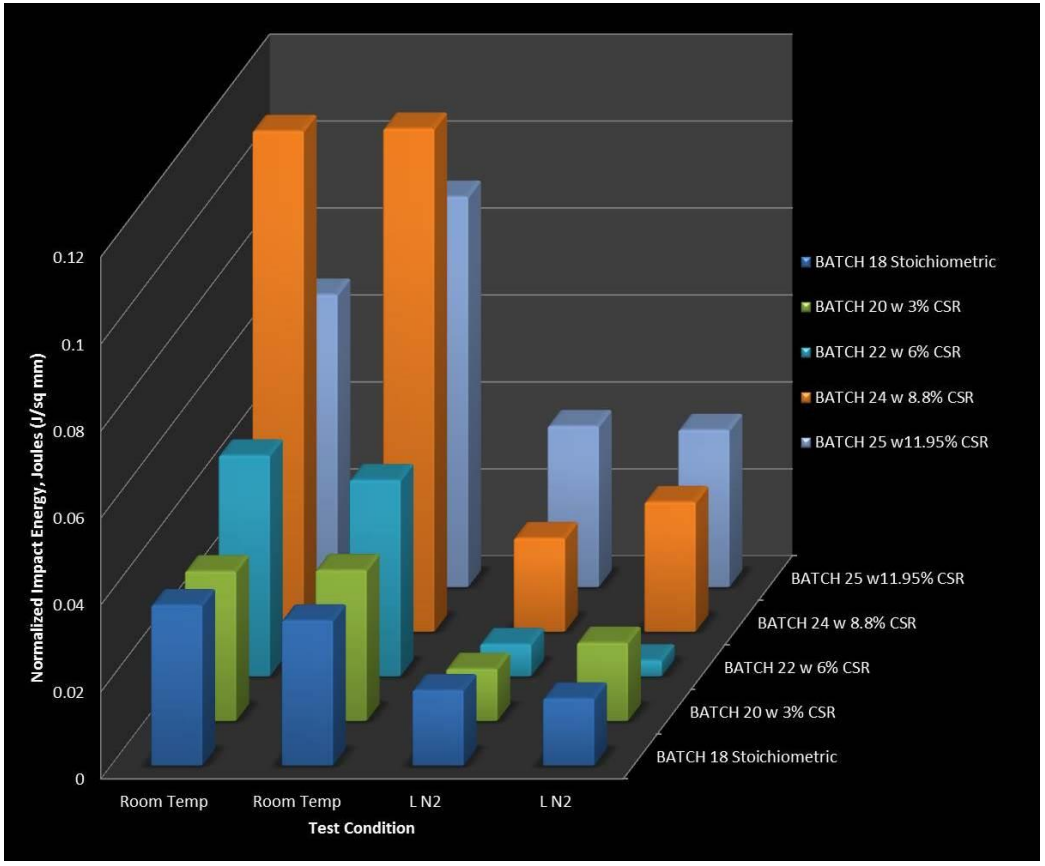
Property Improvement: Addition of Core Shell Rubber (CSR) Particles

Nano-scale Core-Shell-Rubber (CSR) particles designed to toughen polymers have been successfully and uniformly incorporated into the epoxy matrix.



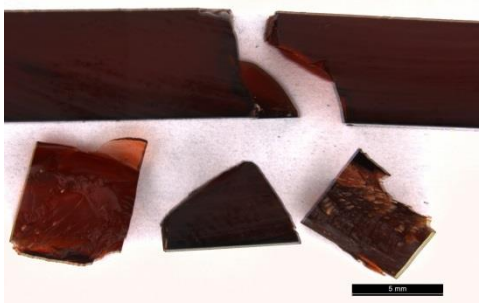
SEM micrograph of an epoxy impact test showing uniform dispersion of CSR particles over a torturous fracture path.

CSR: Impact test results

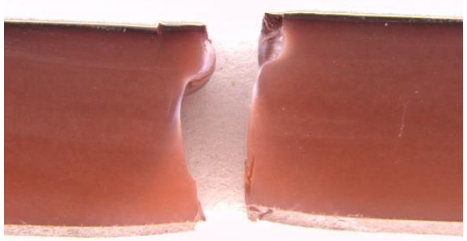


Plot of impact test results with increasing percentages of CSR for room and liquid nitrogen temperatures.

Batch 18
0% CSR RT



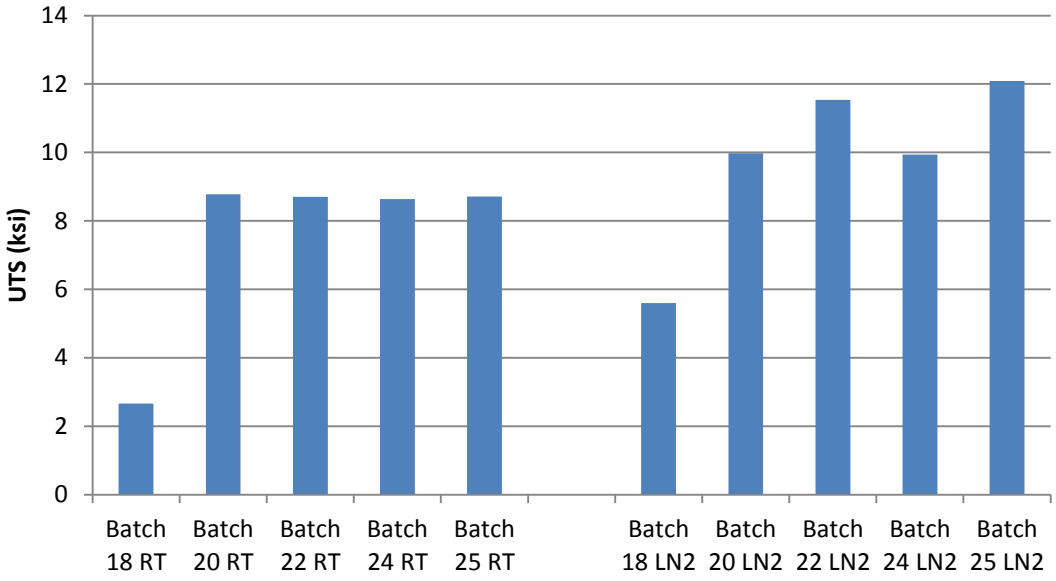
Batch 18
0% CSR LN2



Batch 22
8.8% CSR RT

Batch 22
8.8% CSR LN2

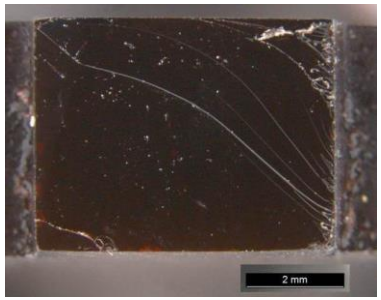
CSR: Tensile Test Results



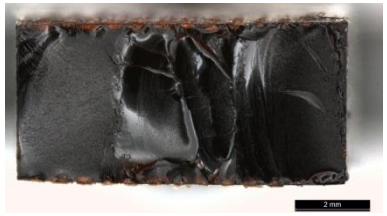
Plot of tensile test results with increasing percentages of CSR for room and liquid nitrogen temperatures.

Batch 22 8.8% CSR LN2

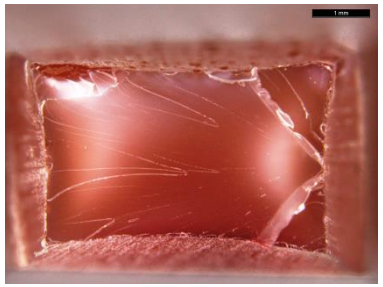
Note: Above LN2 Tensile Data is Misleading!



Batch 18
0% CSR RT



Batch 18
0% CSR LN2

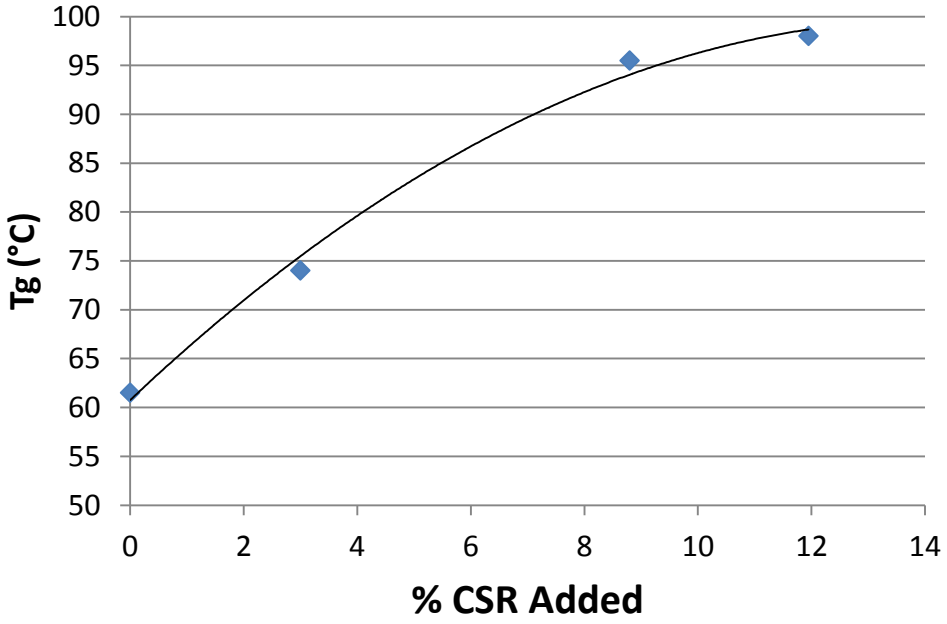


Batch 22
8.8% CSR RT

Batch 22
8.8% CSR LN2

Other Property Improvements due to Addition of CSR Particles

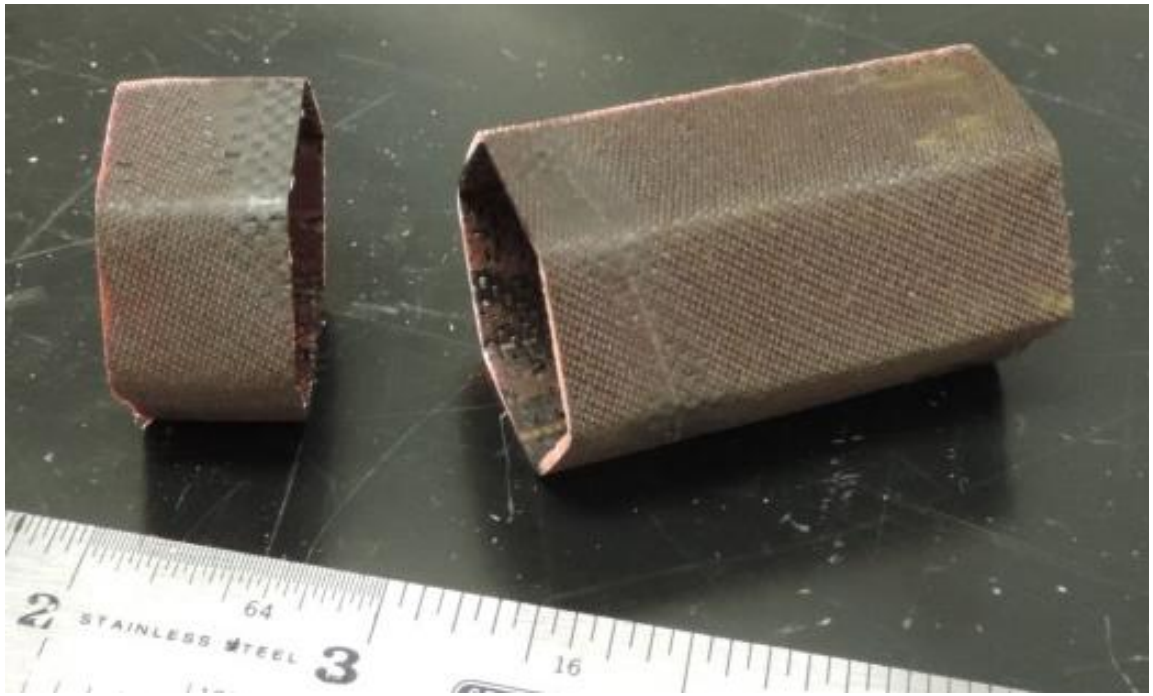
1) Measured improvement in the glass transition temperature



2) Measurement of the IL epoxy Coefficient of Thermal Expansion from cryogenic to room temperature was very favorable at 36 ± 2 ppm (36 ± 2 x 10^{-6}) which places amongst the lowest of the common polymers.

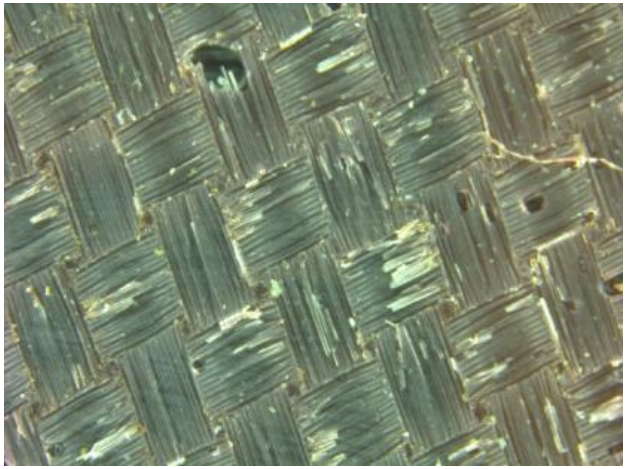
Composite Article Fabrications using CSR IL Epoxy

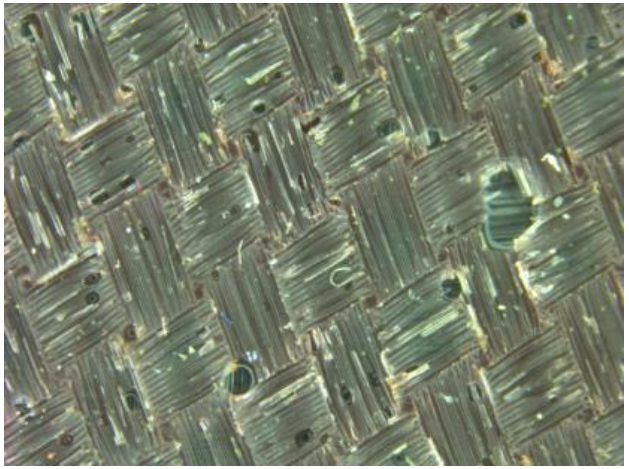
Some carbon-fiber test articles utilizing CSR containing IL epoxy have been successfully made.



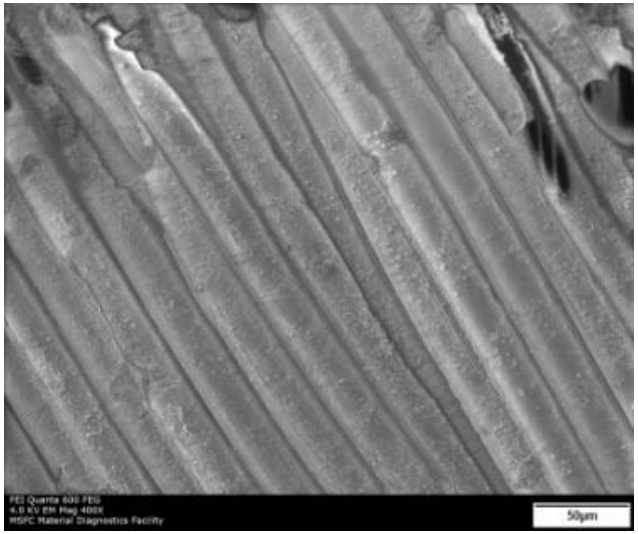
Fabricated 5-layer carbon cloth hexagonal shaped rollup made with IL epoxy containing 8.8% CSR

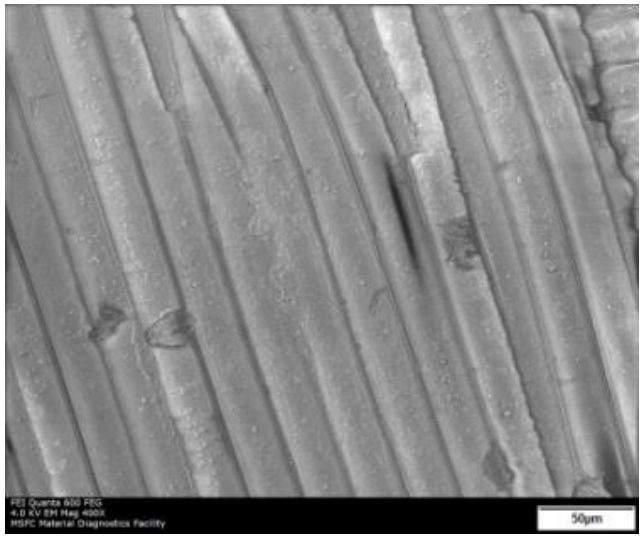
Cycling ILE - Carbon Fiber in LN2 shows no evidence of cracking/delamination





Left: Macrograph of initial surface. Right: Surface after 10 cycles between room and LN2 temperatures.





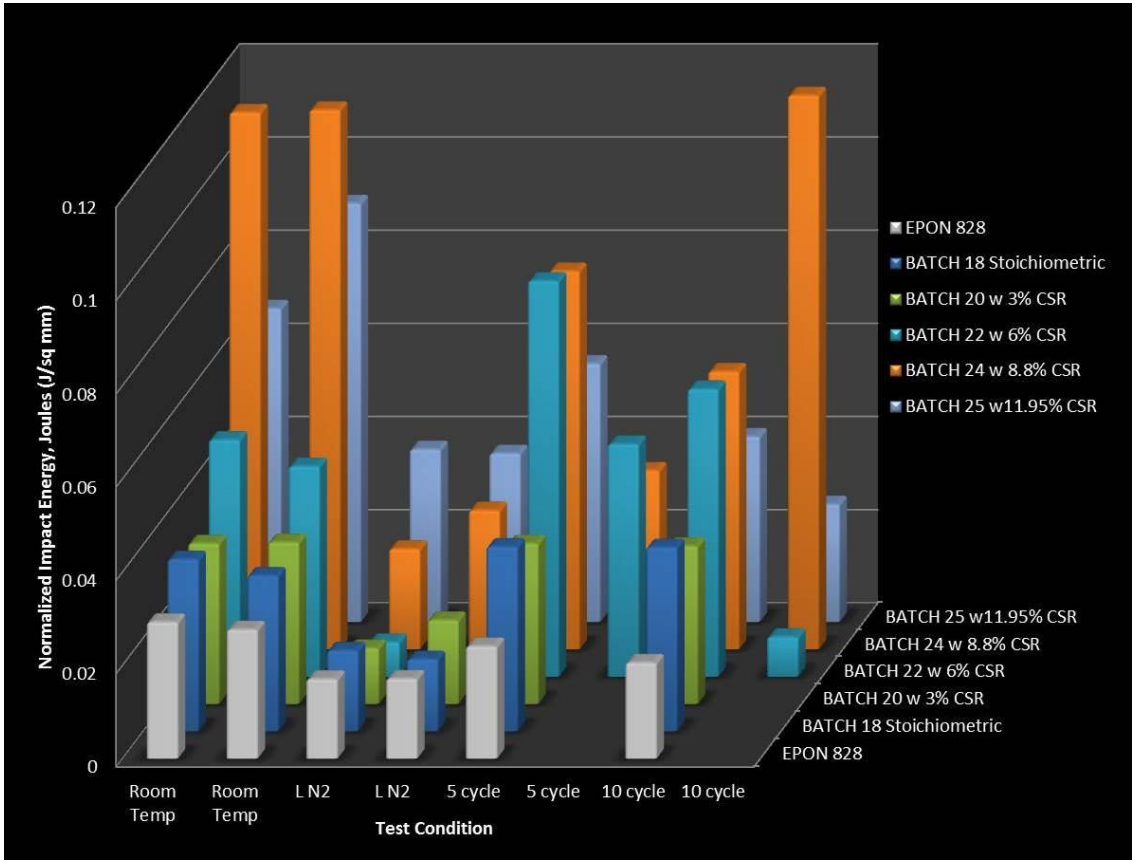
Interim Summary

1) Properties of ILE Resins Strongly Support NASA Applications

2) The Addition of CSR Particles Significantly Improves Properties

3) Viable Carbon Fiber Composites made with ILE Resins

Commercial Comparison



Plot of impact test results comparing ILE's to Epon 828 resin – Huntsman T-403 curing agent epoxy resin

Future Work

- Continue to Improve IL Chemistry
- Continue Testing
  - ◊ Composite Overwrap Pressure Vessel (COPV) Burst Test
  - ◊ Evaluate IL Performance in LOX and LH2 Environments

Epon 828 Resin with Huntsman T-403 Curing Agent

IL Resin (no CSR) with APB Curing Agent

Samtech SK1229 aluminum liner COPV burst test bottles with identical Hexcel IM-10 Carbon Fiber wraps

Acknowledgements

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