Effects of Core-Shell Rubber(CSR) nanoparticles on the Cryogenic Fracture Toughness of CSR modified epoxies

J. Wang, D. Magee, J.A.Schneider
Department of Mechanical Engineering
Mississippi State University

ABSTRACT

This study investigated the effects of core-shell rubber (CSR) nanoparticles on the mechanical properties and fracture toughness of an epoxy resin at ambient and liquid nitrogen (LN_2) temperatures. Varying amounts of Kane Ace® MX130 and Kane Ace® MX960 toughening agent were added to a commercially available EPON 862/Epikure W epoxy resin. Elastic modulus was calculated using quasi-static tensile data. Fracture toughness was evaluated by the resulting breaking energy measured in Charpy impact tests conducted on an instrumented drop tower. The size and distribution of the CSR nanoparticles were characterized using Transmission Electron Microscopy (TEM) and Small Angle X-ray Scattering (SAXS). Scanning Electron Microscopy (SEM) was used to study the fracture surface morphology. The addition of the CSR nanoparticles increased the breaking energy with negligible change in elastic modulus and ultimate tensile stress (UTS). At ambient temperature the breaking energy increased with increasing additions of the CSR nanoparticles up to 13.8wt%, while at LN_2 temperatures, it reached a plateau at much lower CSR concentration.

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Jun Wang, D. Magee, Judy Schneider

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Outline

1. Background
2. Experimental Procedures
3. Results
4. Summary

Polymeric composites are candidates for storage of high pressure, cryogenic fuels

- LH_2 Storage (20K)
- LOX Storage (90K)
- Liquefied Natural Gas (113K)
- LN_2 (77K)

Objective

Investigate low cost testing methods to evaluate and identify promising material properties of interest in the fabrication of cryogenic composite overwrapped pressure vessel (COPV)

Screening methods for selection of suitable materials include:

- **Quasi-static mechanical properties**
- **Dynamic impact test**

Improving fracture toughness of polymeric resins at cryogenic temperatures

Fracture toughness of polymeric resins has been improved at ambient temperatures by the addition of nanoparticles such as:

- exfoliated clays
- carbon nanotubes
- core-shell rubber nanoparticles

This study investigates the effectiveness of CSR particles on the fracture toughness of an epoxy based resin at cryogenic temperatures.

EPON 862/ Epikure W epoxy resin was cast with varying amounts of Kane Ace® MX130 toughening agent

Specimens:

1. *0 wt. % - Neat Resin*
2. *1 to 13.8 wt.% addition of*

- CSR rubber tougheners

Specimens cast for:

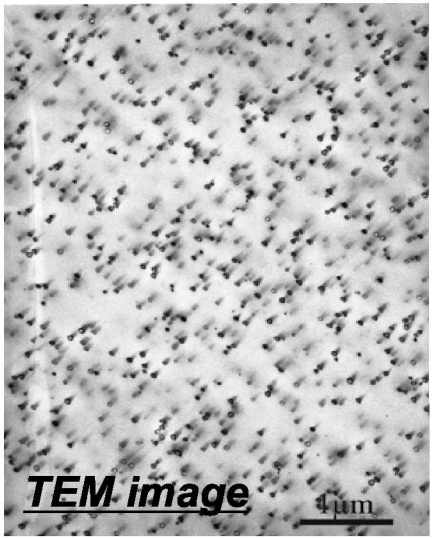
Uniaxial tensile tests (3 mm thick)

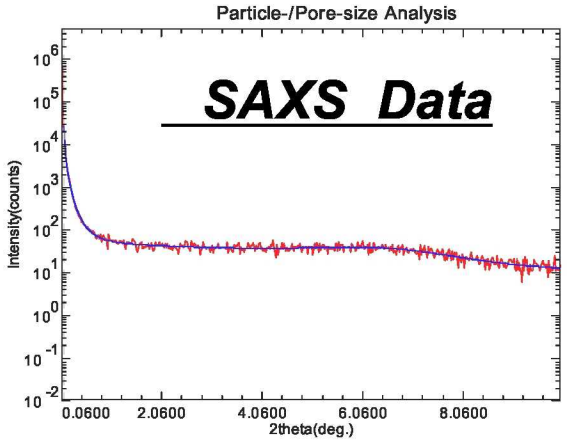
Charpy impact tests (10 mm thick)

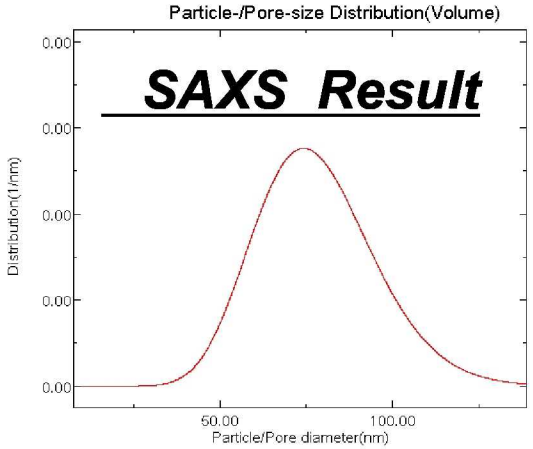
Microstructure Characterization

- **Transmission Electron Microscopy (TEM):**
  - Representative bulk samples
- **Small Angle X-ray Scattering (SAXS):**
  - Larger sample size to investigate homogeneity
- **Scanning Electron Microscopy (SEM):**
  - Fracture surface of Charpy impact test specimens

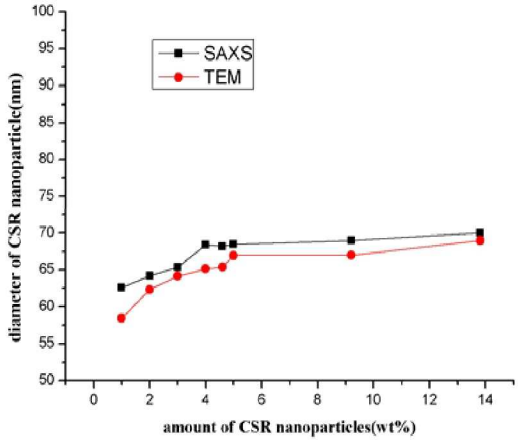
SAXS analysis confirmed CSR nanoparticles size and distribution

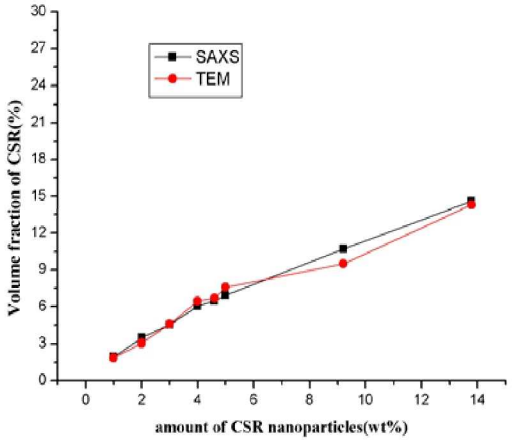




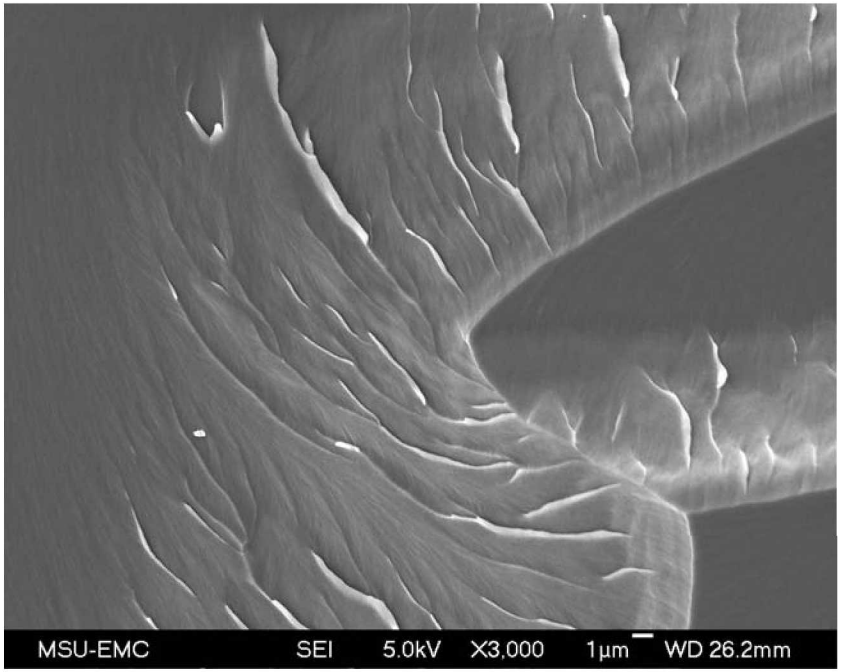


EPON 862/W MX130 with 9.2 vol.% CSR additives

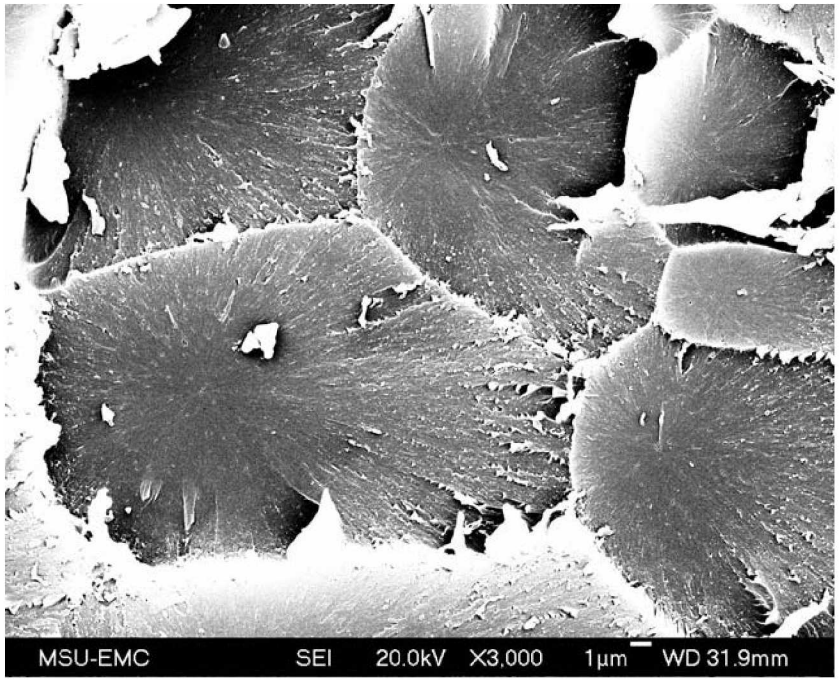




Fracture mechanisms were studied by SEM images



neat resin
featureless wave-like fracture steps



862/W-1 vol.%
jagged and scale-liked edges

SEM images of Fracture surfaces of Cryogenic Charpy Impact test specimens

Mechanical properties evaluated using quasi-static, uniaxial tension testing

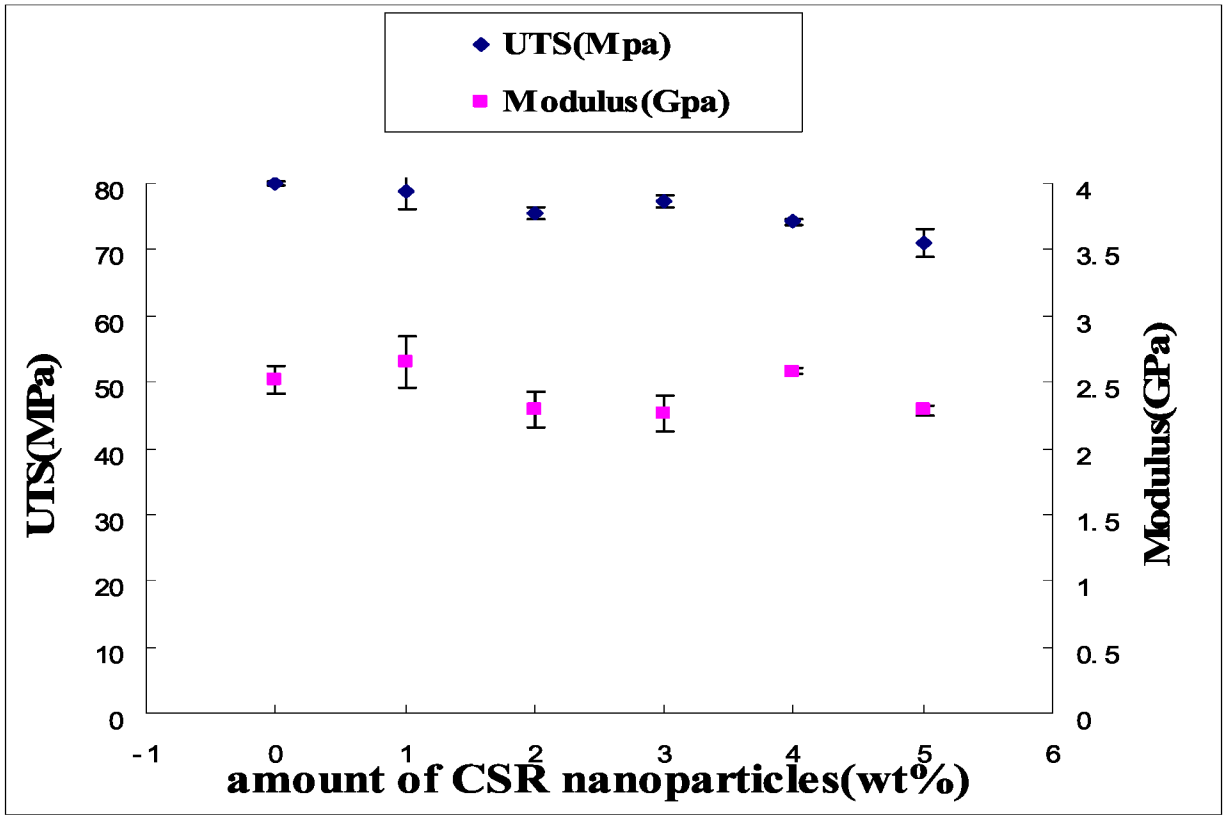
MTS extensometer attached to specimen mounted in grips.

Interior of cryostat.

Cryostat mounted on Instron load frame.

- Instron Model 5869 EM
- 50 kN load cell
- MTS Model 634 extensometer
- Constant velocity 0.13 cm/min
- ASTM Standard D638

Negligible Elastic Modulus and UTS of small additions of CSR at ambient temperature

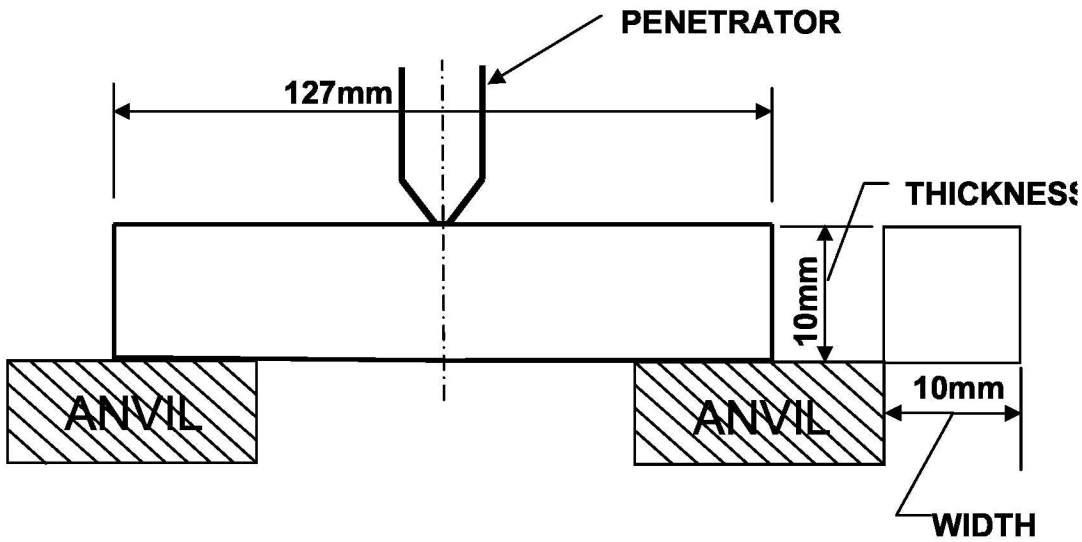


Future test will include the cryogenic temperatures

Charpy impact test to evaluate fracture toughness

INSTRON Dynatup 9250HV
Instrumented Drop Tower

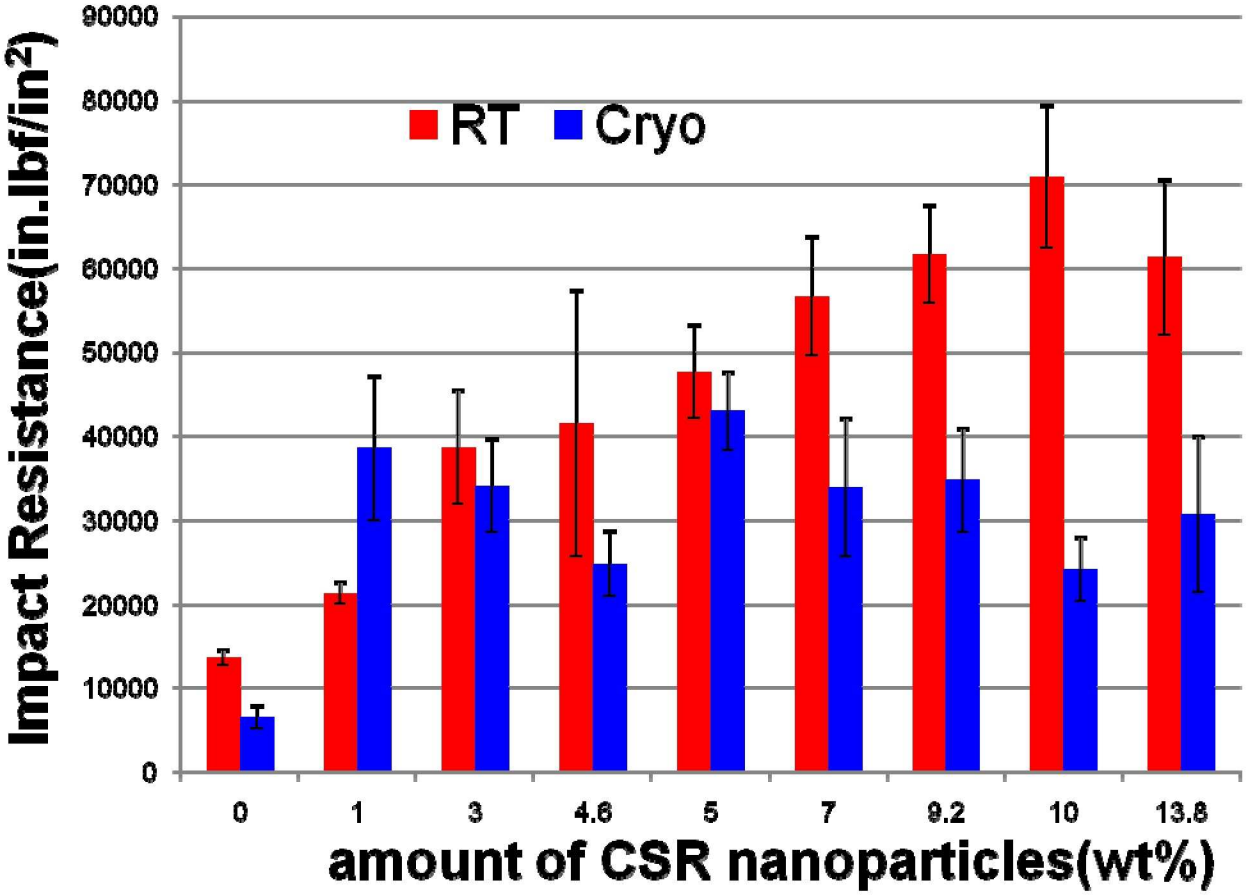
Un-Notched Specimen



ASTM Standard D6110

- **Specimen Temperature: 77-100K**
- **Impact speed: 216 m/min**
- **Energy: 58 Nm**

Charpy impact test results for MX130



EPON 862/Epikure W

Summary

- ***Using Kane Ace® MX130, a 1 wt.% addition of CSR nanoparticles resulted in the maximum increase in the impact resistance measured during an instrumented Charpy impact test at cryogenic temperature.***
- ***TEM and SAXS characterization of these specimens verified the particle size distribution and volume fraction of the CSR in the epoxy matrix.***
- ***SEM images showed differences in the fracture surface morphology which corresponded with the increased impact resistance associated with the additions of CSR nanoparticles.***
- ***Future work included Kane Ace® MX960 will be tested for the comparing.***
  - ***Kane Ace® MX960 may have better performs at cryogenic temperature.***

Acknowledgements

- Mr. Doug Sober of Kaneka Texas for providing the evaluation materials.

- Funding provided by the MSU Hearin (JW) and Mississippi Space Grant Consortium (DG).

- X-ray diffractometer obtained with NSF-MRI Grant # DMR-0619773 (2006) and FE-SEM was purchased under NSF-IMR Grant #DMR.0216703 and 02070615.