The Effect of Dose Rate on Composite Durability When Exposed to a Simulated Long-Term Lunar Radiation Environment

Kristina Rojdev\textsuperscript{a,b}, Mary Jane O’Rourke\textsuperscript{a}, Charles Hill\textsuperscript{b}, Steven Nutt\textsuperscript{b}, William Atwell\textsuperscript{c}

\textsuperscript{a}NASA-Johnson Space Center
\textsuperscript{b}University of Southern California
\textsuperscript{c}The Boeing Company

Human exploration of space beyond low Earth orbit (LEO) requires a safe living and working environment for crew. Composite materials are one type of material being investigated by NASA as a multi-functional structural approach to habitats for long-term use in space or on planetary surfaces with limited magnetic fields and atmosphere. These materials provide high strength with the potential for decreased weight and increased radiation protection of crew and electronics when compared with conventional aluminum structures. However, these materials have not been evaluated in a harsh radiation environment, as would be experienced outside of LEO or on a planetary surface. Thus, NASA has been investigating the durability of select composite materials in a long-term radiation environment.

Previously, NASA exposed composite samples to a simulated, accelerated 30-year radiation treatment and tensile stresses similar to those of a habitat pressure vessel. The results showed evidence of potential surface oxidation and enhanced cross-linking of the matrix\textsuperscript{1}. As a follow-on study, we performed the same accelerated exposure alongside an exposure with a decreased dose-rate. The slower dose-rate is comparable to a realistic scenario, although still accelerated. Strain measurements were collected during exposure and showed that with a fast-dose rate, the strain decreased with time, but with a slow-dose rate, the strain increased with time\textsuperscript{2}. After the radiation exposures, samples were characterized via tensile tests, flexure tests, Fourier Transform Infrared Spectroscopy (FTIR), and Differential Scanning Calorimetry (DSC). The results of these tests will be discussed.


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NASA-JSC/USC: Kristina Rojdev
NASA-JSC: Mary Jane O’Rourke
NASA-JSC: Charles Hill
NASA-JSC: John Alred
The Boeing Company: William Atwell
University of Southern California: Steve Nutt
Outline

• Introduction
• Exposures – test setup and in-situ results
• Sample characterization and analysis
• Ongoing Work
• Questions
INTRODUCTION
Motivation/Purpose

• Long-term surface habitation requires large structures that must withstand the environment for the duration of the missions

• Fiber reinforced composites have gained interest
  – Potential weight savings
  – Potential enhanced radiation protection for the crew and electronics
  – Potential for infusing cutting edge research
Problem/Objectives

• Problem: composite materials have not been characterized for the space radiation environment, which is known to cause damage to polymeric materials

• Objective: assess composite durability in a simulated long-term lunar radiation environment
Assumptions

- The habitat is unshielded from radiation on the exterior
  - There is some multi-layer insulation and micrometeorite/surface ejecta shielding, but no galactic cosmic ray shielding (i.e. covering the habitat under regolith)
- The habitat will remain on the surface and be in service for 30 years
- The habitat is pressurized with air at an elevated oxygen concentration
- The habitat is exposed to one large solar particle event during each solar cycle and constant galactic cosmic ray exposure
Previous Work (2009)

• Investigated two materials
  – Carbon fiber + epoxy composite (CF)
  – Boron/Carbon fiber + epoxy composite (BF-CF)
• Exposure groups: control, tension only, radiation only, radiation + tension
• Conclusions: material properties changing, but inconsistent results
  – Need to validate repeatability of data
  – Increase data set for statistical significance
• Work presented at NSMMS 2010:
  
  Presented at National Space and Missile Materials Symposium, Scottsdale, AZ. June 28-July 1, 2010
Current work (2010)

- Repeated parts of 2009 study
  - Repeatability
  - Increase statistical significance
- Added exposure group to study the **dose-rate effects** on the material
  - Fast dose-rate (0.1478 krad/s) vs. Slow dose-rate (0.0139 krad/s)
- Added in a study to look at how proton-radiation, stress, and dose-rate affect the materials’ performance during hypervelocity impacts
Test setup and In-situ results

EXPOSURES
Radiation Exposures

- Indiana University Cyclotron Facility
  - Total dose: 500 krads (200 MeV protons)
  - Fast dose rate: 147.8 rad/s
  - Slow dose rate: 13.9 rad/s

<table>
<thead>
<tr>
<th>Exposure #</th>
<th>Dose Rate</th>
<th># of Samples</th>
<th>Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>Exposure 1</td>
<td>Slow</td>
<td>10</td>
<td>BF-CF</td>
</tr>
<tr>
<td>Exposure 2</td>
<td>Slow</td>
<td>10</td>
<td>CF</td>
</tr>
<tr>
<td>Exposure 3</td>
<td>Fast</td>
<td>2</td>
<td>CF</td>
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<td>Exposure 4</td>
<td>Fast</td>
<td>2</td>
<td>CF</td>
</tr>
<tr>
<td>Exposure 5</td>
<td>Slow</td>
<td>8</td>
<td>4 – BF-CF, 4 – CF</td>
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<tr>
<td>Exposure 6</td>
<td>Fast</td>
<td>8</td>
<td>4 – BF-CF, 4 – CF</td>
</tr>
</tbody>
</table>
Example Radiation Run

5 materials with exposure “radiation”

5 test stands with material exposure “radiation + tension”
Radiation Test Set up

Strain gauge in center of sample – gather strain during the radiation exposure (also included thermocouple for sample temperature, not shown)

Sample in Test Stand

Beam Exit

Radiation Beam
In-Situ Strain Measurements

Details presented at Ionizing Radiation and Polymers (IRaP) 2011, College Park, MD.

Strain vs. Time (Fast Dose Rate)

Strain vs. Time (Slow Dose Rate)
SAMPLE CHARACTERIZATION AND ANALYSIS
Characterization Completed

- Manufacturing
  - C-scan
- Chemistry
  - Fourier Transform Infrared Spectroscopy (FTIR): bulk chemical composition
- Mechanical Properties
  - Tension: tensile stress, strength, strain, ultimate strain, chord modulus, poisson’s ration, stress vs. strain
  - Flexure: Flexural stress, strength, offset yield strength, chord modulus, strain, tangent modulus of elasticity, secant modulus, stress vs. strain
- Thermal Properties
  - Differential Scanning Calorimetry (DSC): heat capacity as a function of temperature, and changes in glass transition temperature
- Surface Properties and Edges
  - Scanning Electron Microscopy (SEM): look at surface for visual changes
  - Post-Fracture Analysis: Scanning Electron Microscopy (SEM): look at fracture edge after tension/flexure tests
Scanning Electron Microscopy (SEM) of Surfaces (Example: BF-CF Sample #5)

No visible surface morphology changes due to radiation

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Madison, WI
June 27-30
Fourier Transform Infrared Spectroscopy (FTIR) Procedure

- Before radiation exposure, each sample was characterized by FTIR in 9 locations.
- After radiation exposure, each sample was again characterized by FTIR in the same 9 locations.
- Analysis focused on center region.
- Post-radiation absorbance values were subtracted from pre-radiation absorbance values.
FTIR Analysis (example)

- OH peak: Wavenumber 3373
- C-H stretch: Wavenumber 2921
- Aromatic peak: Wavenumber 1229
Both dose rates saw an increase in the peaks of interest. However, the fast dose rate had a greater increase in the peaks.
Fast vs. Slow

Fast dose rate saw a decrease in the peaks of interest, whereas the slow dose rate saw a general increase in the peaks of interest.
Differential Scanning Calorimetry (DSC): BF-CF sample

Similar peaks seen in CF samples, but not consistently in slow dose rate samples.

Glass Transition Temperature ($T_g$)

Unknown Exothermic Peaks

Control
Fast
Slow
Trends show that radiation decreases the Tg and that the fast dose rate sees the largest decrease in Tg.
Tensile Test

- 1 coupon was cut per sample
- Coupons were cut perpendicular to 0° plys
  - to highlight any matrix sensitivities in tensile properties
- Each tensile coupon included
  - tabs to protect the material during test
  - single strain gauge in the center to collect tensile data
## Average Tensile Modulus

### BF-CF Tensile Modulus

<table>
<thead>
<tr>
<th></th>
<th>Avg. Modulus (Gpa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>no rad</td>
<td>64</td>
</tr>
<tr>
<td>fast</td>
<td>66</td>
</tr>
<tr>
<td>slow</td>
<td>70</td>
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</tbody>
</table>

### CF Tensile Modulus

<table>
<thead>
<tr>
<th></th>
<th>Average Modulus (Gpa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>no rad</td>
<td>58</td>
</tr>
<tr>
<td>fast</td>
<td>62</td>
</tr>
<tr>
<td>slow</td>
<td>65</td>
</tr>
</tbody>
</table>
Flexure Test

- 1 coupon was cut per sample
- Coupons were cut perpendicular to 0° plys
  - to highlight any matrix sensitivities in the properties
Average Flexural Strength

**BF-CF Flexural Strength**

- Average Flexural Strength (Mpa)
  - no rad
  - fast
  - slow

**CF Flexural Strength**

- Average Flexural Strength (Mpa)
Average BF-CF Flexural Modulus

BF-CF Flexural Modulus

- Avg. Flexural Modulus (Gpa)

CF Flexural Modulus

- Average Flexural Modulus (Gpa)
## Summary of Results

<table>
<thead>
<tr>
<th></th>
<th>in-situ strain</th>
<th>SEM of surface</th>
<th>FTIR</th>
<th>DSC Tg</th>
<th>Tensile Modulus</th>
<th>Flexure strength</th>
<th>Flexure modulus</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>BF-CF</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>fast</td>
<td>decreasing strain with time</td>
<td>no change</td>
<td>increase in species</td>
<td>decreased Tg, smaller than slow</td>
<td>no change</td>
<td>smallest strength</td>
<td>decreased from control</td>
</tr>
<tr>
<td>slow</td>
<td>increasing strain with time</td>
<td>no change</td>
<td>increase in species</td>
<td>decreased Tg</td>
<td>no change</td>
<td>decreased from control</td>
<td>decreased from control, slightly smaller than fast</td>
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<tr>
<td><strong>CF</strong></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>fast</td>
<td>decreasing strain with time</td>
<td>no change</td>
<td>decrease in species</td>
<td>decreased Tg, smaller than slow</td>
<td>no change</td>
<td>smallest strength</td>
<td>decreased from control and smaller than slow</td>
</tr>
<tr>
<td>slow</td>
<td>increasing strain with time</td>
<td>no change</td>
<td>increase in species</td>
<td>decreased Tg</td>
<td>no change</td>
<td>decreased from control</td>
<td>decreased from control</td>
</tr>
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</table>
## Potential Mechanisms

<table>
<thead>
<tr>
<th></th>
<th>in-situ strain</th>
<th>SEM of surface</th>
<th>FTIR</th>
<th>DSC Tg</th>
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<td><strong>BF-CF</strong></td>
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<td>Crosslinking</td>
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<td>Scission</td>
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<td>slow</td>
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<td>-</td>
<td>?</td>
<td>Scission</td>
<td></td>
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<tr>
<td><strong>CF</strong></td>
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<tr>
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<td>Scission</td>
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<td>?</td>
<td>Scission</td>
<td></td>
<td>Scission</td>
<td>Scission</td>
</tr>
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Future Work

- Analyze the fracture mechanisms in the tensile coupons using SEM
- Repeat radiation exposures
  - Gather more data on in-situ strain
    - Investigating potential sources of error
- Repeat tensile tests
  - Changing method of coupon manufacture to attempt to reduce the error bars
- Collect tensile data at +3 months, +6 months, and +9 months after radiation
Acknowledgements

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• Avionics Division at NASA-JSC
• Indiana University Cyclotron Facility
• The Boeing Company
• University of Southern California
Questions

Kristina Rojdev
Kristina.rojdev-1@nasa.gov
BACKGROUND
Background – Radiation Environment

• Primary Radiation Exposure
  – Galactic Cosmic Rays (GCR)
    • Consists of stripped nuclei (hydrogen to uranium)
  – Solar Particle Events (SPE)
    • Consists of mainly high energy protons
• Concerned with high energy particle radiation
Discussion of the Sun

- 11 year cycle
  - Caused by the changing magnetic field of the sun
  - Solar maximum
    - Sunspots
    - Coronal mass ejections
    - Flare phenomenon
  - Solar minimum
    - Minimal activity
- Solar wind always present

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Solar Particle Events

- Coronal Mass Ejections (CME)
- Fast moving, very high energy particles
  - Bow shock at the front accelerates the particles
  - Mainly protons

![Proton Fluences Graph](image-url)
Band Fit for Large SPE

Largest fluence below 100 MeV
Solar Particle Events and Dose

- Absorbed dose (D): change in mean energy imparted to matter over a discrete mass (dm)
- Mean energy (ε): the change in the number of particles emitted, transferred, or received multiplied by the energy of the particles plus the change in rest energy

\[ D = \frac{d\varepsilon}{dm} = \frac{d(R_{in} - R_{out} + \sum Q)}{dm} \]

\[ R = NE \]
Galactic Cosmic Rays

Climax Neutron Monitor Data

Year


Monthly Count/100

3000 3200 3400 3600 3800 4000 4200 4400

Monthly Mean Sunspot Count

0 100 200 300

red – solar cycle
Blue - GCR

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Focus of study is on proton radiation
Doses Material will See Due to this Radiation Exposure


Mission Lifetime (years)

- 10 year
- 20 year
- 30 year

Visible damage begins to occur in some plastic materials

Dose (cGy)

- 1.E+00
- 1.E+01
- 1.E+02
- 1.E+03
- 1.E+04
- 1.E+05
- 1.E+06
- 1.E+07
- 1.E+08
- 1.E+09
- 1.E+10

- 10 year
- 20 year
- 30 year

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Doses Material will See Due to this Radiation Exposure

Conventional composites failure

Radiation Effects on Polymeric Materials

- Previous radiation research on polymers is mainly electron, neutron, or gamma radiation
- Previous research on materials different from today
- Effects discussed in literature
  - Cross-linking – bonds that link one polymer chain to another through chemical reaction
    - Pro: increases stiffness of material, potentially making it stronger
    - Con: if the stiffness is increased too much, the material becomes brittle and easily fractured
  - Chain scission – a chemical reaction that breaks the bonds of the backbone polymer chain
    - Con: weakens the polymer strength
Stresses on a Pressure Vessel

Longitudinal Stress

\[
\sigma = \frac{p_i \left( \frac{\pi R^2}{2 \pi R t} \right)}{2t} = \frac{p_i R}{2t}
\]

\( P_0 = 0 \)

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Stresses on a Pressure Vessel – Hoop Stress

\[ \sigma_h = \frac{p_i (2R \Delta x)}{2t \Delta x} = \frac{p_i R}{t} \]
Stress on a Pressure Vessel

- Due to the internal pressure of the pressure shell (~ 8psi) and the potential thickness of the material, there will be two tensile stresses imparted on the material:
  - Hoop stress (2x longitudinal stress)
  - Longitudinal stress

- Based on the minimum gauge necessary for the habitat, these stresses are the following:
  - Sandwich structure
    - Hoop stress: 5.43 MPa
    - Longitudinal stress: 2.71 MPa
  - Skin-stiffened structure
    - Hoop stress: 40.72 MPa
    - Longitudinal stress: 20.36 MPa
Material Design/Manufacture

- Material #1 (boron/carbon + epoxy)
- Material #2 (carbon + epoxy)

- 6 plies - quasi-isotropic, balanced, and symmetric layup
- \([+60^\circ,-60^\circ,0^\circ,0^\circ,-60^\circ,+60^\circ]\)

- Material #1 (boron/carbon) – press cure
- Material #2 (carbon) – autoclave cure