In-Situ SEM Investigation of Microstructural Damage Evolution and Strain Relaxation in a Melt-Infiltrated SiC/SiC Composite

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Motivation – Experimental Data for Modeling

• Robust CMC life prediction capabilities require experimental data for inputs and validation
• Several groups working on environmental degradation models that incorporate matrix cracking and interface debonding
  – few studies report measured crack opening displacements
• NASA GRC characterizing CMCs to support environmental modeling
  – Sylramic fiber reinforced, slurry cast MI SiC/SiC
  – CODs predicted to be very small – too small for traditional DIC
  – Apply SEM-DIC using small tensile loading stage in SEM

Image Courtesy of NASA GRC
• Load in 5 ksi increments to 30 ksi using small tensile stage

• Measure COD using SEM-DIC and manual methods

Approach

**Young’s Modulus**

\( \sim 238.4 \text{ GPa (34.6 Msi)} \)

**Ultimate Tensile Stress:**

\( \sim 459.7 \text{ MPa (66.7 ksi)} \)

**Estimated Proportional Limit:**

\( \sim 119 \text{ MPa (17.3 ksi)} \)
Digital Image Correlation

Non-contact “optical” method
White light, SEM, AFM

Requires surface to have a random tracking pattern
Isotropic, high-contrast, random

Surface pattern analyzed in small subsets
Grayscale intensity within subsets is tracked as sample is deformed.


In-SEM Miniature Tension/Compression Stage

SEM-DIC applied to CMCs

Area analyzed was a 200 µm x 500 µm rectangle located ≈ 1.4 mm left and 0.7 mm below centroid of gage section
Initial Fields of View (FOVs)

FOVs selected to sample microstructure and catch a matrix crack
• Sample loaded in tension at ~5 ksi stress increments

• Loading paused at each stress increment to capture SEM images

• Images captured after load relaxed

• Matrix cracks formed between 20 and 25 ksi of initial load cycle, but outside of imaging area.

• Sample unloaded/reloaded to capture matrix crack openings displacements in new AOI
Damage Evolution Before Matrix Cracking

- Strain localization seen in all FOVs
- Strain localization observed ~ 10 ksi
Strain Relaxation Adjacent to Matrix Crack

Prior to first matrix cracking

After first matrix cracking

Outside original area of interest during first loading cycle
Cracks observed across the cross-section
Crack 1

- All high mag FOVs are 10 µm
- High mag FOVs shown at ~30ksi
Crack 2

- All high mag FOVs are 10 µm
- High mag FOVs shown at ~30ksi
Crack 3

• All high mag FOVs are 10 µm
• High mag FOVs shown at ~30ksi
Matrix Crack Opening Exhibits Variability
Interface Opening Exhibits Variability

- Expect opening max along direction of stress max
- Stress component along opening direction $= \sigma \cos(\theta)$
Cracking Along Interfaces

- Some openings follow max global stress, some do not
- Cannot see the entire opening in the FOV
Multiple Fibers Along Crack 1

- Again some openings follow max global stress, some do not
- Local stress state is unknown
**Future Work**

**In-situ Testing and Analysis**

- Couple macroscale DIC with SEM-DIC to examine the multiscale nature of damage evolution and the influence of microstructure on crack growth
  - Couple high speed imaging with macroscale DIC to examine and quantify the distances over which matrix cracks influence neighboring cracks
- SEM-DIC at ultrafine length scales (FOVs < 5 µm) to probe mechanical response in matrix constituents – available constituent properties are mostly approximations
- Examine environmental effects on subcritical crack growth
  - Investigate the effects of fatigue, humidity, combustion gases on crack growth in both coatings and matrix
  - SEM/ESEM (microscale) or an environmental chamber (macroscale)

**Modeling**

- Statistical modeling of the influence/impact of microstructural features on damage evolution (for data collected in all of the above studies)
  - Quantify and correlate measurements of microstructural features with damage observations
  - Use results to develop models describing the influence of microstructure on damage evolution.
Summary and Conclusions

• A slurry cast MI SiC/SiC sample was loaded to a global stress of 30 ksi in a small tensile stage within an SEM

• SEM-DIC and traditional analysis was used to quantify damage

• Damage at fiber/matrix interfaces at global stresses as low as 5 ksi

• After initial matrix cracking, strain relaxation was observed adjacent to matrix cracks

• Crack opening displacements varied from 0.2 to 1.5 µm at a global stress of 30 ksi

• Interface openings exhibited angular variability where maximum opening was not always along the global loading axis - opening may follow a local maximum