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
Approach

- Load in 5 ksi increments to 30 ksi using small tensile stage
- Measure COD using SEM-DIC and manual methods
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.

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

20 ksi

25 ksi

matrix crack

Longitudinal Strain, $\varepsilon_{xx}$ (µε)
Cracks observed across the cross-section

Crack 1

Crack 2

Crack 3

500 μm
Crack 1

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

Location 3

Location 2

Location 1

• 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
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.