Effect of Surface Contamination on Composite Bond Integrity and Durability

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Motivation and Key Issues

- Past research has focused on determining/understanding acceptable performance criteria using the initial bond strength of composite bonded systems.
- There is significant interest in assessing the durability of composite bonded joints and the how durability is effected by contamination.

Objective

- Develop a process to evaluate the durability of adhesively bonded composite joints
- Investigate undesirable bonding conditions by characterizing the initial performance at various contamination levels
- Characterize the durability performance of the system using the same contamination levels
- Support CMH-17 with the inclusion of content for bonded systems
Durability Assessment Procedure

- Pristine Specimen
- Contaminated Specimen

  Surface Characterization

  - Initial Strength Characterization DCB
  - Mechanical Fatigue DCB
  - Environmental Exposure DCB
  - Mechanical Fatigue & Environmental Exposure DCB

  Assessment of Contamination Effects on Long Term Durability
Bonding System Materials

- Material type and curing procedure for specimens: unidirectional carbon-epoxy system, film adhesive, secondary curing bonding and contaminants.

- Materials utilized:
  - Toray P 2362W-19U-304 T800 Unidirectional Prepreg System (350F cure)
  - 3M AF 555 Structural adhesive film (7.5x2 mills, 350F cure)
  - Precision Fabric polyester peel ply 60001
  - Freekote 700-NC from Henkel Corporation

- Specimen Conditioning:
  - Environmental Chamber: 50° C, 95% RH, for 8 weeks and 1.5 years
  - Fatigue Loading: 3 point bending arrangement, 1 inch double amplitude, 2.6 million cycles
Fatigue Fixture and Contamination Procedure

- Stamp Procedure
- Fixture Loaded in Environmental chamber
Double Cantilever Beam (DCB) tests are conducted to determine the adhesive critical energy release rate ($G_{IC}$).

Reveals data for the energy release rate, crack propagation mechanism and provide the dominant mode of failure.

End Notch Flexure (ENF) tests are conducted *in-situ* to determine the initiation and propagation of damage.

Reveals mechanisms of damage propagation via crack growth progression and crack opening profiles.

Configuration: Loading rate - 5.0 mm/min in the direction perpendicular to the specimen from one of the edges.
Quantification of Modes of Failure

Image J software was utilized to quantify failure modes

- Baseline (no contamination)
- A1 contaminated (low pressure)
- A1 contaminated (high pressure)
- A3 contaminated

Modes of failure:
- Cohesive
- Adhesive/Interlaminar failure
Recent Results

- Mode of failure analysis and how that correlates with bond quality
- Assessment of damage initiation and propagation using *in situ* microscopy
- Analytical modeling of a contaminated bondline using Linear Elastic Fracture Mechanics (LEFM).
Bond Quality Assessment

Dual Cantilever Beam (DCB) Specimen

$G_{IC}$ (kJ/m$^2$) vs. Cohesive Failure Ratio %

- NC Baseline
- A1 Stamp Low Pressure
- A1 Stamp High Pressure
- A3 Stamp
Bond Quality Assessment

Dual Cantilever Beam (DCB) Specimen

Varying Stamp Size
Similar Cohesive Area
Similar Bond Quality

A1L-06
$G_{IC} = 0.78 \text{ kJ/m}^2$
COH % - 68.38

A3-05
$G_{IC} = 0.78 \text{ kJ/m}^2$
COH % - 71.07

A3-05
$G_{IC} = 0.78 \text{ kJ/m}^2$
COH % - 71.07

A3-07
$G_{IC} = 0.33 \text{ kJ/m}^2$
COH % - 39.30

Similar Stamp Size
Varying Cohesive Area
Significant Change in Bond Quality
Environmental Conditioning

Graph showing the relationship between $G_{1c}$ (kJ/m$^2$) and Cohesive Area Ratio %.
Fatigue in Ambient Air

Cohesive Area Ratio % vs. $G_{1C}$ (kJ/m²)

- $G_{1C}$ values range from 0.20 to 1.00 kJ/m².
- Cohesive Area Ratio % ranges from 0 to 100%.
- The graph shows a trend where $G_{1C}$ increases with increasing Cohesive Area Ratio %.

Note: The graph is a scatter plot with error bars indicating variability.
Combined Fatigue & Env. Exposure

$G_{IC}$ (kJ/m$^2$) vs. Cohesive Failure Ratio %

- $G_{IC} = 0.20$
- $G_{IC} = 0.40$
- $G_{IC} = 0.60$
- $G_{IC} = 0.80$
- $G_{IC} = 1.00$
- $G_{IC} = 1.03$
- $G_{IC} = 0.72$
In-situ Micro-scale Evaluation
End Notch Fracture (ENF)

Description
In situ load frame for simultaneous loading and imaging of samples within the FIB chamber.

Capabilities
High resolution strain measurement
Programmable loading programs
Very low strain rate are achievable

Testing modes
Tension
Compression
Fatigue
3 point bending
4 point bending
Fracture
Compact tension

Specifications

<table>
<thead>
<tr>
<th></th>
<th>Load Capacity</th>
<th>Max. Strain Travel</th>
<th>Load Cell Accuracy</th>
<th>Linear Scale Accuracy</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>4500N</td>
<td>30 mm</td>
<td>0.2%</td>
<td>20 nm resolution</td>
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</tbody>
</table>

Grips
Extensometer
Actuator
Cross head
Load cell
In-situ Micro-scale Evaluation
End Notch Fracture (ENF)
In-situ Micro-scale Evaluation
End Notch Fracture (ENF)

Prior to Loading
At Peak Load (1000N)

Damage confined to localized crack tip region
In-situ Micro-scale Evaluation
End Notch Fracture (ENF)
In-situ Micro-scale Evaluation
End Notch Fracture (ENF)

Contaminated bond line to create undesirable bonding conditions

Composite Lay-up

Adhesive Layer

Composite Lay-up

30 kV  x33  500 μm

32 50 SEM_SEI
Verification and Validation

Non-Contaminated

Contaminated
Linear Elastic Fracture Mechanics to Model Effects of Contamination

Penny Shaped Crack embedded in a solid

Solid subjected to remotely applied stress

2a is the diameter of the penny shaped crack

Stress Intensity Factor at the crack plane, $K_C = \sigma_y \sqrt{\pi a}$

$\sigma_y$ - Remotely applied stress
Developmental Framework

Penny shaped cracks = Contaminated sites.

Modifications to the theory:

a) RVE Unit Cell considerations
b) Crack size as varied in a RVE Unit Cell
Approach

RVE Unit Cell

cohesive adhesive

A1L-06
$G_{1C} = 0.78 \text{ kJ/m}^2$
COH % - 68.38

A3-05
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Stress Intensity factor $K_c$ for RVE Unit Cell

Relationship between Stress Intensity Factor, $K_C$ and Fracture Toughness, $G_C$

\[ K_C = \sqrt{\frac{E}{G_C}} \]

\[ G_C = \frac{K_C^2}{E} \]

\[ G_C = \frac{(A_{\text{cohesive}} \cdot \sigma_s \sqrt{\pi a})^2}{E} \]

Typical Structural Adhesives possess
  - Young’s modulus between 3 GPa
  - Tensile Strength between 4-6 Mpa
  - Varying Crack Flaw Size

The relationship between critical energy release rate and cohesive area
$y = 0.0076x + 0.1609$

$y = 0.011x - 0.1124$

Experimental vs Predicted

$G_{IC}$ (kJ/m$^2$)

Cohesive Failure Ratio %

Tensile Strength – 4.6 MPa & Young Modulus, $E=3$ GPa
• Durability assessment was conducted by conditioning of specimens using a 3-point bending fixture for mechanical fatiguing in air and in environmental chamber.

• Adhesion/Cohesion failure mode patterns were observed with the Freekote contamination.

• $G_{IC}$ properties correlate well with cohesive area ratio

• Line Profile analysis and area analysis of the failure surface are used to quantify the areas of contamination.

• Micro-scale fracture testing revealed location of initial damage and damage propagation in contaminated specimen.

• LEFM was used to model the behavior of contaminated regions
Future Work:

• In situ analysis of fatigued and environmentally exposed samples to examine fracture properties and damage initiation.

• Investigate additional contamination procedures to change surface chemistry and determine fracture properties of additional cases.

• Change contaminate application locations and dimensionality to investigate additional morphologies.

Benefit to Aviation:

• Better understanding of durability assessment for adhesively bonded composite joints.

• Assisting in the development of bonding quality assurance procedures.
Composite Bond Integrity/Long-Term Durability of Composite Bonds

Questions?