

## Effect of Surface Contamination on Composite Bond Integrity and Durability

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## **Composite Bond Integrity/Long-Term Durability of Composite Bonds**

- 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**





## **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





## Assessment of Bond Quality

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



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

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.





## 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



```
Adhesive/
Interlaminar
   failure
```



Interlaminar failure







- 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**





# **Bond Quality Assessment**

### **Dual Cantilever Beam (DCB) Specimen**





A1L-06 G<sub>1C</sub> - 0.78 kJ/m<sup>2</sup> COH % - 68.38

A3-05 G<sub>1C</sub> - 0.78 kJ/m<sup>2</sup> COH % - 71.07

Varying Stamp Size Similar Cohesive Area

**Similar Bond Quality** 



A3-05 G<sub>1C</sub> - 0.78 kJ/m<sup>2</sup> COH % - 71.07

A3-07 G<sub>1C</sub> - 0.33 kJ/m<sup>2</sup> COH % - 39.30

Similar Stamp Size

Varying Cohesive Area

**Significant Change in Bond Quality** 



## **Environmental Conditioning**





## Fatigue in Ambient Air





# **Combined Fatigue & Env. Exposure**





#### 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



| Load Capacity      | 4500N | Max. Strain Travel    | 30 mm            |  |
|--------------------|-------|-----------------------|------------------|--|
| Load Cell Accuracy | 0.2%  | Linear Scale Accuracy | 20 nm resolution |  |

Specifications









15kV



## Prior to Loading

At Peak Load (1000N)







| Contaminated bond line to create   | undesirable bonding conditions | Unidirectional Composite |
|------------------------------------|--------------------------------|--------------------------|
| Composite Lay-up                   | Contaminated bond region       |                          |
| Adhesive Layer<br>Composite Lay-up |                                | 50/W x13 1mm 36 50 SEM.  |
|                                    |                                |                          |
| 30kV x33 500µm                     | 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





#### Stress Intensity factor $K_c$ for RVE Unit Cell

Relationship between Stress Intensity Factor, K<sub>C</sub> and Fracture Toughness, G<sub>C</sub>





## **Experimental vs Predicted**



Tensile Strength – 4.6 MPa & Young Modulus, E= 3 GPa



## Conclusions/Summary

- 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<sub>IC</sub> 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



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## **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?**