**Report Documentation Page**

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Standard Form 298 (Rev. 8-98)  
Prescribed by ANSI Std Z39-18
Overview

- Background
- Materials and Processes
- Tests and Test Methods
- Lap Shear and Peel Data
- Wedge Test Data
- Conclusions
- Future Work
Background

- Large Screening Effort to Evaluate Low-VOC Bond Primers
- Primary Focus is to Eliminate Chromated Bond Primers
- Working with Industry-Government Non-Cr Adhesive Bond Primer Team
  - Effort is part of the larger picture but also has USAF focus
- Desire Performance Equivalent to that Currently Obtained with Chromated Primers
  - Cytec’s BR 127 and BR 6747-1, primarily
- Bond Strength and Moisture Durability are Both Important
- Looking for Primer that Works Well with Multiple Adhesives on Adherends Prepared Using Grit-Blast/Sol-Gel (GBSG)
  - Currently, GBSG is used exclusively with Cytec’s BR 6747-1 primer
  - Cytec's FM 73 adhesive is not recommended for use with GBSG
  - 3M’s AF 163-2 adhesive tends to fail at its interface with BR 6747-1
Materials and Processes

• **Five Waterborne Adhesive Bond Primers**
  – Cytec’s BR 6747-1 (zero-VOC, chromated)
  – Cytec’s BR 6747-1NC (zero-VOC, nonchromated - no inhibitor)
  – Cytec’s BR 6700-1 (low-VOC, nonchromated corrosion inhibitor)
  – 3M’s EW 5000 (low-VOC, chromated)
  – 3M’s EW 5000 AS (low-VOC, nonchromated corrosion inhibitor)

• **Spray Applied Using Conventional Air Gun**
  – Dry film thicknesses: 0.0025 mm - 0.0063 mm (0.0001 - 0.00025 in);
    measured using Fischer Isoscope MP30E-S with ETA3.3H probe
    (directly on PAA adherends and on travelers for GBSG adherends)

• **Cured in an Air-circulating Oven at 121°C (250°F) for 60 Mins after Air Dry at Ambient Conditions for 30 Mins**
• **Seven Adhesives Evaluated with Each Primer**
  - Henkel’s Hysol EA 9696 and Hysol EA 9628
  - Cytec’s FM 73M, FM 209M, and FM 300-2M
  - 3M’s AF 163-2M and AF 500M

• **Mat Carrier**

• **290g/m² (0.06 psf) Areal Weight**

• **Cured per Manufacturers’ Recommendations Under Positive Pressure**
  - 121°C (250°F) for 60-90 Minutes
• 2024-T3 (bare) Aluminum Adherends

• Two Surface Preparations
  – Phosphoric Acid Anodize (PAA) per ASTM D 3933
    • First choice for aluminum prebond surface preparation
    • Used in USAF for rework and preparation of repair doublers
  – Grit-Blast/Sol-Gel (GBSG)
    • $\text{Al}_2\text{O}_3$ grit-blast followed by application of AC Tech AC-130 (Boegel-EPII) sol-gel solution; no rinse and ambient dried
    • Used in USAF for preparation of on-aircraft adherends
Tests and Test Methods

• **Tensile Lap Shear (ASTM D 1002)**
  - Ambient temperature at 22°C (72°F)
  - Hot/Wet at 82°C (180°F) after conditioning for 90 days at 60°C (140°F) and 95-100% relative humidity (RH)

• **Metal-to-Metal Climbing Drum Peel (ASTM D 1781)**
  - Ambient temperature at 22°C (72°F)

• **Wedge Test (ASTM D 3762)**
  - Conditioned at 60°C (140°F) and 95-100% RH for 28 days
  - Conditioned at 49°C (120°F) and 95-100% RH for 28 days*

• **Five Specimens per Test for Each Adhesive/Primer/Surface Preparation Combination**

* These data are not reported in the paper and will not be presented; will be in a comprehensive report
The Wedge Test

- Used Extensively by AFRL for Screening to Assess Bonded Joint Moisture Durability

- Considered by AFRL to be a Semiquantitative Test
  - Initial crack lengths provide important information (w.r.t. test severity)
  - Crack extensions can be useful, but are not the bottom line
  - Most consideration is given to failure modes after conditioning
  - Desire failure modes are 95% or greater cohesive within the adhesive

- Caveats
  - Useful for comparing surface preparations (including primers)
  - Many factors affect the test; must only vary the factor being evaluated
  - Several adhesive characteristics affect the test: modulus, bondline thickness, carrier, degree of cure, hot/wet properties
  - Test cannot be quantitatively correlated with in-service performance
EA 9696 Adhesive
22°C (72°F) Lap Shear

DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.
EA 9696 Adhesive
82°C (180°F) Wet Lap Shear

Lap Shear Strength (MPa) vs. Lap Shear Strength (psi)

- BR 6747-1 (84% cohesive)
- BR 6747-1 NC (46% cohesive)
- BR 6700-1 (66% cohesive)
- EW 5000 (48% cohesive)
- EW 5000 AS (34% cohesive)

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JMJ; AFRL/RX SAC
EA 9696 Adhesive
22°C (72°F) Climbing Drum Peel

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JMJ; AFRL/RXSAC 11
EA 9628 Adhesive

22°C (72°F) Lap Shear

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EA 9628 Adhesive
82°C (180°F) Wet Lap Shear

![Graph showing Lap Shear Strength for different compositions and types of adhesives]
EA 9628 Adhesive
22°C (72°F) Climbing Drum Peel

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JMJ; AFRL/RXSAC 14
FM 73M Adhesive
22°C (72°F) Lap Shear

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FM 73M Adhesive
82°C (180°F) Wet Lap Shear

DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.
FM 73M Adhesive
22°C (72°F) Climbing Drum Peel

FM 73M Tech Data Sheet

CDP Torque (N*m/m)

CDP Torque (in*lbs/in)

BR 6747-1
BR 6747-1NC
BR 6700-1
EW 5000
EW 5000 AS

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FM 209M Adhesive
22°C (72°F) Lap Shear

DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.
**FM 209M Adhesive**

82°C (180°F) Wet Lap Shear

<table>
<thead>
<tr>
<th>Material</th>
<th>PAA Lap Shear Strength (MPa)</th>
<th>GBSG Lap Shear Strength (psi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BR 6747-1</td>
<td>88% cohesive</td>
<td>3,000</td>
</tr>
<tr>
<td>BR 6747-1NC</td>
<td>93% cohesive</td>
<td>3,500</td>
</tr>
<tr>
<td>BR 6700-1</td>
<td>85% cohesive</td>
<td>4,000</td>
</tr>
<tr>
<td>EW 5000</td>
<td>68% cohesive</td>
<td>4,500</td>
</tr>
<tr>
<td>EW 5000 AS</td>
<td>76% cohesive</td>
<td>5,000</td>
</tr>
</tbody>
</table>

*DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.*
FM 209M Adhesive
22°C (72°F) Climbing Drum Peel

DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.
FM 300-2M Adhesive
22°C (72°F) Lap Shear

DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.
FM 300-2M Adhesive
82°C (180°F) Wet Lap Shear

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DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.
FM 300-2M Adhesive
22°C (72°F) Climbing Drum Peel

DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.
AF 163-2M Adhesive
22°C (72°F) Lap Shear

DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.
AF 163-2M Adhesive
82°C (180°F) Wet Lap Shear

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AF 163-2M Adhesive
22°C (72°F) Climbing Drum Peel

AF 163-2M Tech Data Sheet

CDP Torque (N*m/m)

<table>
<thead>
<tr>
<th>Material</th>
<th>PAA</th>
<th>GBSG</th>
</tr>
</thead>
<tbody>
<tr>
<td>BR 6747-1</td>
<td>350</td>
<td>350</td>
</tr>
<tr>
<td>BR 6747-1NC</td>
<td>350</td>
<td>350</td>
</tr>
<tr>
<td>BR 6700-1</td>
<td>350</td>
<td>350</td>
</tr>
<tr>
<td>EW 5000</td>
<td>350</td>
<td>350</td>
</tr>
<tr>
<td>EW 5000 AS</td>
<td>350</td>
<td>350</td>
</tr>
</tbody>
</table>

DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.
AF 500M Adhesive
22°C (72°F) Lap Shear

AF 500M Tech Data Sheet

Lap Shear Strength (MPa)

<table>
<thead>
<tr>
<th>Material</th>
<th>Lap Shear Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>BR6747-1</td>
<td>35</td>
</tr>
<tr>
<td>BR6747-1NC</td>
<td>37</td>
</tr>
<tr>
<td>BR6700-1</td>
<td>40</td>
</tr>
<tr>
<td>EW5000</td>
<td>45</td>
</tr>
<tr>
<td>EW5000AS</td>
<td>50</td>
</tr>
</tbody>
</table>

Lap Shear Strength (psi)

PAA
GBSG

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AF 500M Adhesive
82°C (180°F) Wet Lap Shear

![Graph showing lap shear strength comparison for different materials.]

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AF 500M Adhesive
22°C (72°F) Climbing Drum Peel

AF 500M Tech Data Sheet

CDP Torque (N*m/m) vs. CDP Torque (in*lbs/in)

- BR 6747-1
- BR 6747-1 NC
- BR 6700-1
- EW 5000
- EW 5000 AS

PAA vs. GBSG
EA 9696 Adhesive with PAA
60°C (140°F) Wedge Test

DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.
EA 9696 Adhesive with GBSG
60°C (140°F) Wedge Test

DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.
EA 9628 Adhesive with PAA
60°C (140°F) Wedge Test

![Graph showing crack length over conditioning time for different adhesives.]

- BR 6747-1
- BR 6747-1NC
- BR 6700-1
- EW 5000
- EW 5000 AS

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EA 9628 Adhesive with GBSG
60°C (140°F) Wedge Test

DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.
FM 73M Adhesive with GBSG

60°C (140°F) Wedge Test

<table>
<thead>
<tr>
<th></th>
<th>BR 6747-1</th>
<th>BR 6747-1NC</th>
<th>BR 6700-1 (4% coh)</th>
<th>EW 5000 (84% coh)</th>
<th>EW 5000 AS (64% coh)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.50</td>
<td>1.45</td>
<td>1.35</td>
<td>1.35</td>
<td>1.35</td>
</tr>
<tr>
<td>7</td>
<td>1.65</td>
<td>1.55</td>
<td>1.40</td>
<td>1.40</td>
<td>1.40</td>
</tr>
<tr>
<td>14</td>
<td>1.75</td>
<td>1.65</td>
<td>1.55</td>
<td>1.50</td>
<td>1.50</td>
</tr>
<tr>
<td>21</td>
<td>1.80</td>
<td>1.70</td>
<td>1.60</td>
<td>1.55</td>
<td>1.55</td>
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<tr>
<td>28</td>
<td>1.85</td>
<td>1.75</td>
<td>1.65</td>
<td>1.60</td>
<td>1.60</td>
</tr>
</tbody>
</table>

Conditioning Time (Days)

DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.
FM 209M Adhesive with GBSG
60°C (140°F) Wedge Test

Graph showing crack length (cm) and conditioning time (days) for different materials:
- BR 6747-1 (14% coh)
- BR 6747-1NC (50% coh)
- BR 6700-1 (0% coh)
- EW 5000 (0% coh)
- EW 5000 AS (0% coh)
FM 300-2M Adhesive with PAA
60°C (140°F) Wedge Test

<table>
<thead>
<tr>
<th></th>
<th>BR 6747-1</th>
<th>BR 6747-1NC</th>
<th>BR 6700-1</th>
<th>EW 5000</th>
<th>EW 5000 AS</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 Days</td>
<td>4.60</td>
<td>4.55</td>
<td>4.50</td>
<td>4.45</td>
<td>4.35</td>
</tr>
<tr>
<td>7 Days</td>
<td>4.60</td>
<td>4.55</td>
<td>4.50</td>
<td>4.45</td>
<td>4.35</td>
</tr>
<tr>
<td>14 Days</td>
<td>4.60</td>
<td>4.55</td>
<td>4.50</td>
<td>4.45</td>
<td>4.35</td>
</tr>
<tr>
<td>21 Days</td>
<td>4.60</td>
<td>4.55</td>
<td>4.50</td>
<td>4.45</td>
<td>4.35</td>
</tr>
<tr>
<td>28 Days</td>
<td>4.60</td>
<td>4.55</td>
<td>4.50</td>
<td>4.45</td>
<td>4.35</td>
</tr>
</tbody>
</table>

Crack Length (cm) vs. Conditioning Time (Days)

DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.
FM 300-2M Adhesive with GBSG
60°C (140°F) Wedge Test

DoD Distribution Statement A: Approved for Public Release; Distribution is Unlimited.
AF 163-2M Adhesive with PAA
60°C (140°F) Wedge Test

* Visually appeared to fail at the primer-adhesive interface, but a thin layer of AF 163-2M remains on the primer.
AF 163-2M Adhesive with GBSG
60°C (140°F) Wedge Test

* Visually appeared to fail at the primer-adhesive interface, but a thin layer of AF 163-2M remains on the primer
AF 500M Adhesive with PAA
60°C (140°F) Wedge Test

![Graph showing crack length over conditioning time](image)

- **BR 6747-1**
- **BR 6747-1NC**
- **BR 6700-1**
- **EW 5000**
- **EW 5000 AS**

**Conditioning Time (Days)**

**Crack Length (cm)**

**Crack Length (in)**
AF 500M Adhesive with GBSG
60°C (140°F) Wedge Test

Conditioning Time (Days)
Crack Length (cm)
Crack Length (in)

- BR 6747-1 (94% coh)
- BR 6747-1NC
- BR 6700-1 (14% coh)
- EW 5000 (42% coh)
- EW 5000 AS (32% coh)

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Typical Failure Modes Observed in 60°C (140°F) Wedge Tests

- Typical Primer-Aluminum Interfacial Failure with FM 209M Adhesive
- Typical AF 163-2M “100 Percent Cohesive Failure” Near Primer-Adhesive Interface
- Typical Good “100 Percent” Cohesive Failure
Summary of Failure Modes with GBSG in 60°C (140°F) Wedge Tests

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Conclusions

- Large Amount of Data Generated to Guide Future Efforts
- No Clear Path Forward for Non-Cr Primer Qualification
  - Based on initial screening with only 5 specimens per test
- PAA Test Results were Overall Superior to GBSG
  - PAA is the premier aluminum surface preparation
  - PAA can be used with more combinations of surface preps & primers
- Hot/Wet Lap Shear Results when Using GBSG Exhibit Poor Failure Modes
  - Routinely seen even with good wedge tests data for same combination
  - A bit concerning
- Wedge Test Results Must be Used with Caution
  - Screening w/ controlled variables; no quantitative correlation to service
Conclusions (cont.)

- BR 6747-1NC (No Corrosion Inhibitor) Yielded Amazingly Good Results
- BR 6700-1 Wedge Test Results with GBSG were Surprisingly Poor and Not Consistent with Previous AFRL (Good) Data
- FM 209M Wedge Test Data were Poor when with GBSG Prep
- FM 73M Generated Surprisingly Good Wedge Test Results with GBSG/BR 6747-1; Contradicts Previous USAF Data
- FM 300-2M Adhesive Generated Larger Initial Cracks in the Wedge Test and Tended to Produce Less Crack Growth
  - Wedge test interrogates interface less stringently using FM 300-2M
- AF 163-2M Adhesive Exhibits Failure Modes At or Near the Primer-Adhesive Interface with BR 6747-1 & Others
Future Efforts

• USAF Will Work with Industry-Government Non-Cr Adhesive Bond Primer Team to Further Evaluate Some of the Products Screened, as well as Promising New Candidates

• Another Industry-Government Team was Awarded a SERDP Project Aimed at Understanding the Requirements for Bond Primers and their Corrosion Inhibitors
  – Investigate failed bonds and relationship to primer
  – Evaluate various types/levels of corrosion inhibitor in bond primers
  – Develop a risk assessment tool for bonded joints
  – Investigate alternative accelerated aging protocols

• Much Work is Required to Implement any New Primer
  – Laboratory testing beyond screening
  – Generation of data based on field-level processing