Quarterly Progress Report No. 4

Development of Nondestructive Tests for
the Evaluation of Bonded Materials

by

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Submitted by

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Avco Systems Division
Lowell, Massachusetts 01851
I. INTRODUCTION

This report covers the work performed and the results obtained under the subject contract during the quarter 1969 October 20 through 1970 January 19. Data obtained in the surface condition study are presented. Electric field reflectometry, at 1 KHz and 9.8 GHz, and gas-phase ultrasonic transmission methods are detailed as possible methods for nondestructively evaluating substrate surfaces.

II. PROGRESS ACCOMPLISHED

A. Surface Condition Study

All experimental laboratory preparations and evaluations have been completed up to the point of data correlation and analysis. The information has been grouped for each specimen type: butt tensile, core shear, and lap shear. One representative surface, code 3A, for each of 13 different surface conditions within each group, was thoroughly characterized both in the as prepared condition and immediately following sulfuric acid sodium dichromate etch just prior to adhesive bonding. All substrates were measured for bond-line thicknesses. Nondestructive test and destructive test data for the bonded specimens are summarized.

1. Surface Finishes

A total of 390 substrate surfaces were prepared as indicated in Table I. This was sufficient to provide five specimens at two substrates per specimen for each surface condition and type of specimen. Of these, three specimens per surface condition were bonded and tested.

Photomicrographic, surface roughness, and contact angle data are presented on the following pages in grouped fashion for the most beneficial comparisons.

The photomicrographs were taken with a Burke and James "Rembrandt" model camera using an Agfa solinar lens (focal length 5 cm., \( f = 3.5 \)). The film used was Polaroid type 52, 4" X 5".

A Taylor-Hobson Tallysurf Recording Profilometer, Model 3, provided the Center-line-average (CLA) and strip chart profile data. Interpretation of the strip charts is aided by Table II.

Contact angle measurements were performed on the Langmuir-style device pictured in Figure 1. The surface to be measured was brought into the plane of the table. A few small drops of distilled water were placed on the surface to form one highly-domed drop. While sighting along the juncture of the droplet and the surface via a surface mirror attached to the protractor, the protractor was adjusted until the axis of the arm was aligned exactly with the three-phase point. This operation was found to be most important to accuracy and reproducibility. The observed light-extinction angle was read from the protractor.
TABLE I

SURFACE ROUGHNESS PREPARATION

<table>
<thead>
<tr>
<th>NOMINAL ROUGHNESS</th>
<th>BUTT TENSILE</th>
<th>CORE SHEAR</th>
<th>LAP SHEAR</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 RMS</td>
<td>Paper Lapped</td>
<td>Paper Lapped</td>
<td>Paper Lapped</td>
</tr>
<tr>
<td>5 RMS</td>
<td>Paper Lapped</td>
<td>Paper Lapped</td>
<td>Paper Lapped</td>
</tr>
<tr>
<td>7 RMS</td>
<td>Paper Lapped</td>
<td>Paper Lapped</td>
<td>Paper Lapped</td>
</tr>
<tr>
<td>10 RMS</td>
<td>Paper Lapped</td>
<td>Paper Lapped</td>
<td>Paper Lapped</td>
</tr>
<tr>
<td>20 RMS</td>
<td>Turned</td>
<td>Paper Sanded</td>
<td>Paper Sanded</td>
</tr>
<tr>
<td>40 RMS</td>
<td>Paper Sanded</td>
<td>Paper Sanded</td>
<td>Turned</td>
</tr>
<tr>
<td>80 RMS</td>
<td>Turned</td>
<td>Paper Sanded</td>
<td>Turned</td>
</tr>
<tr>
<td>110 RMS</td>
<td>Turned</td>
<td>Turned</td>
<td>Turned</td>
</tr>
<tr>
<td>150 RMS</td>
<td>Turned</td>
<td>Turned</td>
<td>Turned</td>
</tr>
<tr>
<td>Fine Grit Low Pressure</td>
<td>140 Mesh Silica 20 psi</td>
<td>140 Mesh Silica 20 psi</td>
<td>140 Mesh Silica 20 psi</td>
</tr>
<tr>
<td>Fine Grit High Pressure</td>
<td>140 Mesh Silica 80 psi</td>
<td>140 Mesh Silica 80 psi</td>
<td>140 Mesh Silica 80 psi</td>
</tr>
<tr>
<td>Coarse Grit Low Pressure</td>
<td>80-100 Mesh Al₂O₃ 20 psi</td>
<td>80-100 Mesh Al₂O₃ 20 psi</td>
<td>80-100 Mesh Al₂O₃ 20 psi</td>
</tr>
<tr>
<td>Coarse Grit High Pressure</td>
<td>80-100 Mesh Al₂O₃ 80 psi</td>
<td>80-100 Mesh Al₂O₃ 80 psi</td>
<td>80-100 Mesh Al₂O₃ 80 psi</td>
</tr>
<tr>
<td>Position</td>
<td>Magnification</td>
<td>Full Scale Chart 2 ins. or 5 cm.</td>
<td>C.I.A. Index</td>
</tr>
<tr>
<td>----------</td>
<td>---------------</td>
<td>---------------------------------</td>
<td>--------------</td>
</tr>
<tr>
<td></td>
<td>Micro-inches</td>
<td>Microns</td>
<td>Microns</td>
</tr>
<tr>
<td>1</td>
<td>1,000</td>
<td>1,000</td>
<td>1,000</td>
</tr>
<tr>
<td>2</td>
<td>2,000</td>
<td>2,000</td>
<td>2,000</td>
</tr>
<tr>
<td>3</td>
<td>5,000</td>
<td>5,000</td>
<td>5,000</td>
</tr>
<tr>
<td>4</td>
<td>10,000</td>
<td>10,000</td>
<td>10,000</td>
</tr>
<tr>
<td>5</td>
<td>20,000</td>
<td>20,000</td>
<td>20,000</td>
</tr>
<tr>
<td>6</td>
<td>50,000</td>
<td>50,000</td>
<td>50,000</td>
</tr>
</tbody>
</table>

1 Micro-inch = 0.001 in. 1 Micron = 0.001 mm. = 40 micro-inches
FIGURE 1. CONTACT ANGLE MEASURING DEVICE DEVELOPED AT AVCO FOR SURFACE CHARACTERIZATION SPECIMENS (AFTER LANCHEUR)
For highly polished, pure-element surfaces, a single contact angle is reported in the literature. In less-perfect cases advancing and receding contact angles are reported. Investigation of the hydrodynamic and gravitational forces involved in the cited measurements, as they relate to the distinctly rough surfaces being studied under this contract, revealed that many important forces act to modify the basic surface-free-energy attractions. Our own early observations revealed other parameters which affect the light extinction point determination, such as droplet shape and "lay" of the surface texture.

A satisfactory technique for performing the contact angle measurements, in consideration of both the theoretical and practical aspects, was developed. By adding water to an existing droplet in small increments the droplet was brought to its maximum contact angle, beyond which, the droplet would "jump" outward to a lower contact angle. Conversely, by removing small increments of water from an existing droplet, a minimum contact angle could be obtained, just prior to the droplet retracting in a "jump" motion. In all cases the test surfaces were horizontal. The results were found to be reproducible and properly related to surface energetics. The data is reported in a group of three values, θ maximum, θ minimum, and the angular difference between them, Δθ.
Photo Mag. = 4.4X

BLA = 1.2
RMS = 1.3

θ MAX = 71.5
θ MIN = 21.5
Δθ = 50.0

1 BT-3A (M) Before Etch

Photo Mag. = 4.5X

BLA = 10.0
RMS = 11.1

θ MAX = 45.5
θ MIN = 9.0
Δθ = 36.5

1 BT-3A (E) After Etch
Photo Mag. = 4.4X

5BT-3A (M) Before Etch

CLA = 6.0
RMS = 6.6

θ MAX = 79.5
θ MIN = 25.0
Δθ = 54.5

Photo Mag. = 4.5X

5BT-3A (E) After Etch

CLA = 14.0
RMS = 15.5

θ MAX = 35.5
θ MIN = 9.5
Δθ = 26.0
Photo Mag. = 4.4X

7 BT-3A (M) Before Etch

CLA = 9.2
RMS = 10.2

θ MAX = 82.5
θ MIN = 12.5
Δθ = 70.0

Photo Mag. = 4.5X

7 BT-3A (E) After Etch

CLA = 15.0
RMS = 16.7

θ MAX = 52.5
θ MIN = 15.5
Δθ = 37.0
Photo Mag. = 4.4X

10 BT-3A (M) Before Etch

CLA = 44.4
RMS = 16.0

θ MAX = 83.5
θ MIN = 9.0
Δθ = 74.5

Photo Mag. = 4.5X

10 BT-3A (E) After Etch

CLA = 24.0
RMS = 26.6

θ MAX = 54.0
θ MIN = 8.5
Δθ = 45.5
**40 BT-3A (M) Before Etch**

- Photo Mag. = 4.4X
- CLA = 37.5
- RMS = 41.6
- $\theta$ MAX = 63.5
- $\theta$ MIN = 4.0
- $\Delta \theta$ = 59.5

**40 BT-3A (E) After Etch**

- Photo Mag. = 4.5X
- CLA = 35.0
- RMS = 38.8
- $\theta$ MAX = 19.0
- $\theta$ MIN = 5.0
- $\Delta \theta$ = 14.0
150 BT-3A (M) Before Etch

Photo Mag. = 4.4X

CLF = 84
RMS = 93.2

\[ \theta_{\text{MAX}} = 83.5 \]
\[ \theta_{\text{MIN}} = 27.0 \]
\[ \Delta \theta = 56.5 \]

150 BT-3A (E) After Etch

Photo Mag. = 4.5X

CLF = 77.0
RMS = 85.5

\[ \theta_{\text{MAX}} = 46.5 \]
\[ \theta_{\text{MIN}} = 12.0 \]
\[ \Delta \theta = 34.5 \]
FGLP-BT-3A (M) Before Etc

Photo Mag. = 4.4X

- CLA = 22
- RMS = 24.4

\[ \theta_{\text{MAX}} \] = 56.0°
\[ \theta_{\text{MIN}} \] = 20.0°
\[ \Delta \theta \] = 36.0°

FGLP-BT-3A (E) After Etch

Photo Mag. = 4.5X

- CLA = 25.0
- RMS = 27.7

\[ \theta_{\text{MAX}} \] = 44.5°
\[ \theta_{\text{MIN}} \] = 9.5°
\[ \Delta \theta \] = 35.0°
Photo Mag. = 4.4X

FGHP-BT-3A (M) Before Etc

CLA = 29
RMS = 32.2

θ MAX 56.0
θ MIN 20.0
Δθ 36.0

Photo Mag. = 4.5X

FGHP-BT-3A (E) After Etc

CLA = 50.0
RMS = 55.5

θ MAX 59.0
θ MIN (<< 31.0)
Δθ (>> 28)
<table>
<thead>
<tr>
<th>Photo Mag. = 4.4x</th>
<th>CGHP-BT-3A (M) Before Etch</th>
</tr>
</thead>
<tbody>
<tr>
<td>CLA = 55.0</td>
<td>θ MAX = 78.0</td>
</tr>
<tr>
<td>RMS = 61.0</td>
<td>θ MIN = 13.0</td>
</tr>
<tr>
<td></td>
<td>Δθ = 65.0</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Photo Mag. = 4.5x</th>
<th>CGHP-BT-3A (E) After Etch</th>
</tr>
</thead>
<tbody>
<tr>
<td>CLA = 70.0</td>
<td>θ MAX = 40.5</td>
</tr>
<tr>
<td>RMS = 77.7</td>
<td>θ MIN = 9.0</td>
</tr>
<tr>
<td></td>
<td>Δθ = 31.5</td>
</tr>
</tbody>
</table>
Photo Mag. = 4.4X

1 C5-3A (M) Before Etch

\[
\begin{align*}
\text{CLA} & = 2.2 \\
\text{RMS} & = 2.4
\end{align*}
\]

\[
\begin{align*}
\theta \text{ MAX} & = 88.0 \\
\theta \text{ MIN} & = 44.5 \\
\Delta \theta & = 43.5
\end{align*}
\]

Photo Mag. = 4.7X

1 C5-3A (E) After Etch

\[
\begin{align*}
\text{CLA} & = 2.5 \\
\text{RMS} & = 2.8
\end{align*}
\]

\[
\begin{align*}
\theta \text{ MAX} & = 24.5 \\
\theta \text{ MIN} & = 4.5 \\
\Delta \theta & = 20.0
\end{align*}
\]
Photo mag. = 4.4X

5 CS-3A (M) Before Etch

CLA = 4.1
RMS = 4.5

θ MAX = 74.0
θ MIN = 15.0
Δθ = 59.0

Photo Mag. = 4.5X

5 CS-3A (E) After Etch

CLA = 6.5
RMS = 7.2

θ MAX = 15.0
θ MIN = 4.5
Δθ = 10.5
Photo Mag. = 3.7X

CLA = 23.0
RMS = 25.5

20 CS 3A (M) Before Etch

θ MAX = 73.5
θ MIN = 14.0
Δ θ = 59.5

Photo Mag. = 4.5X

CLA = 17.0
RMS = 18.9

20 CS-3A (E) After Etch

θ MAX = 28.5
θ MIN = 6.0
Δ θ = 22.5
<table>
<thead>
<tr>
<th>CLA</th>
<th>RMS</th>
<th>θ MAX</th>
<th>θ MIN</th>
<th>Δθ</th>
</tr>
</thead>
<tbody>
<tr>
<td>27.3</td>
<td>30.3</td>
<td>76.0</td>
<td>12.5</td>
<td>63.5</td>
</tr>
</tbody>
</table>

**Photo Mag. = 3.7X**

40 CS-3A (M) Before Etch

---

<table>
<thead>
<tr>
<th>CLA</th>
<th>RMS</th>
<th>θ MAX</th>
<th>θ MIN</th>
<th>Δθ</th>
</tr>
</thead>
<tbody>
<tr>
<td>26.0</td>
<td>28.8</td>
<td>25.0</td>
<td>(6.5)</td>
<td>(18.5)</td>
</tr>
</tbody>
</table>

**Photo Mag. = 4.5X**

40 CS-3A (E) After Etch

---

Scale Mag. = 3

Scale Mag. = 2
Photo Mag. = 3.7X

80 CS-3A (M) Before Etch

CLA = 68.0
RMS = 75.5

θ MAX = 77.5
θ MIN = 11.5
Δθ = 66.0

Photo Mag. = 4.5X

80 CS-3A (E) After Etch

CLA = 70.0
RMS = 77.7

θ MAX = 31.0
θ MIN = (5.0)
Δθ = (26.0)
Photo Mag. = 3.7X

<table>
<thead>
<tr>
<th>CLA</th>
<th>RMS</th>
</tr>
</thead>
<tbody>
<tr>
<td>60.0</td>
<td>66.6</td>
</tr>
</tbody>
</table>

110 CS-3A (X) Before Etch

θ MAX = 73.0
θ MIN = 21.0
Δ θ = 52.0

Photo Mag. = 4.5X

<table>
<thead>
<tr>
<th>CLA</th>
<th>RMS</th>
</tr>
</thead>
<tbody>
<tr>
<td>70.0</td>
<td>77.7</td>
</tr>
</tbody>
</table>

110 CS-3A (E) After Etch

θ MAX = 32.0
θ MIN = (2.8)
Δ θ = (29.0)
**150 GS-3A (M) Before Etch**

- **CLA**: 95.0
- **RMS**: 105.4
- **θ MAX**: 72.5
- **θ MIN**: 15.0
- **Δθ**: 57.5

**150 GS-3A (E) After Etch**

- **CLA**: 70.0
- **RMS**: 77.7
- **θ MAX**: 37.5
- **θ MIN**: 7.5
- **Δθ**: 30.0
Photo Mag. = 3.7X

FGLP-CS-3A (M) Before Etch

CLA = 22.0
RMS = 24.4

θ MAX = 52.0
θ MIN = 11.0
Δθ = 41.0

Photo Mag. = 4.5X

FGLP-CS-3A (E) After Etch

CLA = 41.0
RMS = 45.5

θ MAX = 8.0
θ MIN = 2.0
Δθ = 6.0
Photo Mag. = 37.5X

FGHP-CS-3A (M) Before Etch

CLA = 45.0
RMS = 50.0

θ MAX = 47.5
θ MIN = 14.5
Δθ = 33.0

Photo Mag. = 4.5X

FGHP-CS-3A (E) After Etch

CLA = 48.0
RMS = 53.3

θ MAX = 0.5
θ MIN < 0.5
Δθ = 0.5
Photo Mag. = 3.7X

- CLA = 25.0
- RMS = 27.7

CGLP-CS-3A (M) Before Etch

- $\theta_{\text{MAX}}$ = 61.0
- $\theta_{\text{MIN}}$ = 27.0
- $\Delta \theta$ = 44.0

Photo Mag. = 4.5X

- CLA = 35.0
- RMS = 38.8

CGLP-CS-3A (E) After Etch

- $\theta_{\text{MAX}}$ = 7.0
- $\theta_{\text{MIN}}$ = 2.0
- $\Delta \theta$ = 5.0
Photo Mag. = 2.7X

5 LS-3A (M) Before Etch

CLAX = 4.5
RMS = 5.0

θ MAX = 87.0
θ MIN = 36.0
Δθ = 51.0

Photo Mag. = 2.7X

5 LS-3A (E) After Etch

CLAX = 7.0
RMS = 7.8

θ MAX = 74.0
θ MIN = 19.0
Δθ = 55.0

Scale Mag. = 3
Photo Mag. = 2.9X

7 LS-3A (A) Before Etch

CLA = 7.2
RMS = 8.0

θ MAX = 70.0
θ MIN = 15.5
Δθ = 54.5

Photo Mag. = 2.7X

7 LS-3A (B) After Etch

CLA = 9.0 - 9.5
RMS = 10.0 - 10.6

θ MAX = 64.0
θ MIN = (7.0)
Δθ = (57.0)
Photo Mag. = 2.7X

10 LS-3A (M) Before Etch

<table>
<thead>
<tr>
<th>CLA</th>
<th>RMS</th>
<th>θ MAX</th>
<th>θ MIN</th>
<th>Δθ</th>
</tr>
</thead>
<tbody>
<tr>
<td>8.5</td>
<td>9.4</td>
<td>62.0</td>
<td>19.0</td>
<td>43.0</td>
</tr>
</tbody>
</table>

Photo Mag. = 2.7X

10 LS-3A (E) After Etch

<table>
<thead>
<tr>
<th>CLA</th>
<th>RMS</th>
<th>θ MAX</th>
<th>θ MIN</th>
<th>Δθ</th>
</tr>
</thead>
<tbody>
<tr>
<td>10.8 - 11.0</td>
<td>12.0 - 12.2</td>
<td>79.0</td>
<td>10.5</td>
<td>69.5</td>
</tr>
</tbody>
</table>
Photo Mag. = 2.7X

20 LS-3A (X) Before Etch

CLA = 17.0
RMS = 18.9

θ MAX = 69.0
θ MIN = 19.5
Δθ = 49.5

Photo Mag. = 2.7X

20 LS-3A (E) After Etch

CLA = 32.0
RMS = 35.5

θ MAX = 62.0
θ MIN = 16.0
Δθ = 46.0
### 40 LS-3A (M) Before Etch

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>CLA</td>
<td>21.0</td>
</tr>
<tr>
<td>RMS</td>
<td>23.3</td>
</tr>
</tbody>
</table>

\[ \theta_{\text{MAX}} = 75.0 \]
\[ \theta_{\text{MIN}} = 14.0 \]
\[ \Delta \theta = 61.0 \]

### 40 LS-3A (E) After Etch

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>CLA</td>
<td>22.0</td>
</tr>
<tr>
<td>RMS</td>
<td>24.4</td>
</tr>
</tbody>
</table>

\[ \theta_{\text{MAX}} = 23.5 \]
\[ \theta_{\text{MIN}} = 7.5 \text{ (irregular droplet)} \]
\[ \Delta \theta = 16.0 \]
110 LS-3A (M) Before Etch

Photo Mag. = 2.9X

<table>
<thead>
<tr>
<th>CLA</th>
<th>RMS</th>
<th>θ MAX</th>
<th>θ MIN</th>
<th>Δθ</th>
</tr>
</thead>
<tbody>
<tr>
<td>47.0</td>
<td>52.2</td>
<td>66.5</td>
<td>26.5</td>
<td>40.0</td>
</tr>
</tbody>
</table>

110 LS-3A (E) After Etch

Photo Mag. = 2.8X

<table>
<thead>
<tr>
<th>CLA</th>
<th>RMS</th>
<th>θ MAX</th>
<th>θ MIN</th>
<th>Δθ</th>
</tr>
</thead>
<tbody>
<tr>
<td>58.0</td>
<td>64.4</td>
<td>17.0</td>
<td>2.5 (Irregular droplet)</td>
<td>14.5</td>
</tr>
</tbody>
</table>
150 LS-3A (X) Before Etch

Photo Mag. = 2.9X

<table>
<thead>
<tr>
<th>CLA</th>
<th>72.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>RMS</td>
<td>80.0</td>
</tr>
</tbody>
</table>

θ MAX 79.0
θ MIN 22.0
Δθ 57.0

150 LS-3A (E) After Etch

Photo Mag. = 2.8X

<table>
<thead>
<tr>
<th>CLA</th>
<th>72.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>RMS</td>
<td>80.0</td>
</tr>
</tbody>
</table>

θ MAX 86.0
θ MIN 19.0
Δθ 67.0
Photo Mag. = 2.7X

FGLP-LS-3A (N) Before Etch

CLA = 20.0
RMS = 22.2

θ MAX = 58.5
θ MIN = 10.5
Δθ = 48.0

Photo Mag. = 2.8X

FGLP-LS-3A (E) After Etch

CLA = 20.0
RMS = 22.2

θ MAX = 74.0
θ MIN = 18.5
Δθ = 55.5
Photo Mag. = 2.7X

FGHP-LS-3A (B) Before Etch

CLA = 32.5
RMS = 35.6

θ MAX = 39.0
θ MIN = 14.0
Δθ = 25.0

Photo Mag. = 2.8X

FGHP-LS-3A (E) After Etch

CLA = 45.0
RMS = 50.0

θ MAX = 63.5
θ MIN = 25.0
Δθ = 38.5
<table>
<thead>
<tr>
<th>Photo Mag. = 2.7X</th>
<th>CGLP-LS-3A (N) Before Etch</th>
</tr>
</thead>
<tbody>
<tr>
<td>CLA = 34.0</td>
<td>θ MAX = 81.5</td>
</tr>
<tr>
<td>RMS = 38.8</td>
<td>θ MIN = 18.0</td>
</tr>
<tr>
<td></td>
<td>Δθ = 63.5</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Photo Mag. = 2.8X</th>
<th>CGLP-LS-3A (E) After Etch</th>
</tr>
</thead>
<tbody>
<tr>
<td>CLA = 36.0</td>
<td>θ MAX = 13.0</td>
</tr>
<tr>
<td>RMS = 39.9</td>
<td>θ MIN = 3.0</td>
</tr>
<tr>
<td></td>
<td>Δθ = 10.0</td>
</tr>
</tbody>
</table>

Scale Mag. = 2
Photo Mag. = 2.7X

CGHP-LS-3A (X) Before Etch

CLA = 55.0
RMS = 61.0

θ MAX = 83.5
θ MIN = 25.0
Δθ = 58.5

Photo Mag. = 2.8X

CGHP-LS-3A (E) After Etch

CLA = 60.0
RMS = 68.6

θ MAX = 88.0°
θ MIN = 31.0°
Δθ = 57.0°
2. Preparation for Bonding

The schedule for bond preparation, etched surface evaluation, and adhesive bonding was carefully arranged to benefit both data-taking and bonding. Three hours after etching had been completed, the curing agent was added to the adhesive resin mixture, with the bonds being completed within 30 minutes from the start of that mixing. Twelve specimens were prepared each day for two days, with fifteen specimens being prepared the third day, for a given type of specimen. Three such series covered the butt tensile, core shear, and lap shear specimen types.

The substrate cleaning and etching was conducted according to Avco RAD Specification G100004-4, "Immersion Cleaning and Etching of Aluminum and Aluminum Alloy, Process for" except that manual solvent wiping with methyl-ethyl ketone (MEK) replaced the recommended vapor degreasing. Distilled water was used for the final rinse.

3. Adhesive Bonding

The adhesive formula for each of nine batches was:

<table>
<thead>
<tr>
<th>Component</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Epoxy resin</td>
<td>50.00 grams</td>
</tr>
<tr>
<td>Viscosity adjuster</td>
<td>1.25 grams</td>
</tr>
<tr>
<td>Epon 828</td>
<td></td>
</tr>
<tr>
<td>Cab-o-sil</td>
<td></td>
</tr>
<tr>
<td>Total prepared resin</td>
<td>51.25 grams</td>
</tr>
<tr>
<td>Hardener</td>
<td>5.00 grams</td>
</tr>
<tr>
<td>DETA</td>
<td></td>
</tr>
<tr>
<td>Total mixture</td>
<td>56.25 grams</td>
</tr>
</tbody>
</table>

The epoxy resin and fumed silica were mixed together in two master batches, each consisting of 750.00 grams of resin and 18.75 grams of Cab-o-sil. Prior to mixing, the Cab-o-sil was dried for four hours at 230°F. After mixing the batches were placed under full vacuum for at least 10 minutes at room temperature to remove air and volatiles. The master batches were stored at room temperature. A day’s batch of prepared resin and hardener was mixed by hand for two minutes and then spread thin to maximize evolution of reaction exotherm heat. This procedure minimized mixture aging over the 30-minute application period. Curing in all cases was conducted at room temperature (72° ±4) for a minimum of 24 hours in order to avoid thermal expansion variability.

The cylindrical substrates were aligned and cured in Vee-blocks using substrate length measurements to set the 0.006 inch bond-line thickness. The lap-shear specimens, however, required a special set-up procedure. Because differing surface finishes were machined, lapped, fly-cut on the specimen surfaces, more or less material was removed from some specimens, yielding specimens which varied in the thickness by 3-4 mils from the original blanking stock.
All specimens were dry fitted, shimming individual specimens with plastic (mylar) film such that a 1 mil feeler gauge blade could not be inserted between the mating halves at the point to be bonded.

Previous experiments with the bonding system (adhesive) used in this program permitted bonds of approximately 6 mil thickness to be obtained on lap shear specimens when a "dead weight" of 100 grams per sq. in. was applied to the battered halves. The weight-adhesive viscosity combination allowed the substrates to seek that equilibrium which, in previous experiments, yielded an approximately 6 mil thick bond line. Individual lap shear substrates were pre fitted in a bonding fixture specifically designed to maintain the alignment and overlap required. Bonding fixtures were made to bond six individual lap shear specimens simultaneously. One substrate of each of six specimens was placed onto the fixture and spring loaded. The second or mating substrate was placed on the fixture and a straight edge was placed across the 0.50 in. overlap area of all six specimens. Mylar and/or polyethylene film shim stock was added as necessary under the lower individual substrates so that less than one mil clearance was noted between the suspended straight edge and any singular substrate (in the overlap area). It should be noted that the fixtures were initially designed for mating 0.064 in. nominal thickness substrates yielding an approximate 5 mil parallel bond line. Following surface preparation the adhesive was prepared and spread into a thin film. Each substrate was wet with adhesive, covering the sample width and approximately 0.625 in. of the bonding edge. Samples were permitted to set at room temperature about 20 minutes to permit application of adhesive to all samples, allow excess air to escape and ensure a more uniform consistency (adhesive). Specimens were then assembled and a 100 gram per square inch (overlap area) load was applied by means of a pre weighed bar. Room temperature cure of 24 hours was effected before specimens were removed from the fixtures.

4. Nondestructive Test Evaluation

a. Radiography

A few butt tensile specimens were radiographed at an oblique angle. A sensitivity of 2-1T with an aluminum penetrometer (0.015" thk, 0.008" dia. hole) was obtained. A polyethylene film step wedge (0.005" and 0.010" thick) was constructed and placed across the bond area. The polyethylene film could not be adequately discriminated in the radiographs. Similar lack of ability to radiographically observe bubbles in the bond line was experienced with the lap shear and core shear specimens.

Radiographic conditions:

Using a Philips-Norelco 50-150 KV X-ray tube with a 2.5 mm focal spot operated at 100 KV, 10 Ma for 1 minute and a focus to film distance of 48 inches, representative radiographs were obtained on Eastman Kodak type "M" film. Films were hand processed (developed) for 8 minutes at 68°F. The tube was positioned at an angle of 30° off the perpendicular such that the area of interest was projected onto the film for the butt tensile specimens.
b. Ultrasonic Pulse-echo

The centering holes and lead-pin holes in the butt tensile specimens made it impossible to obtain unambiguous bond/unbond indications.

The lap shear specimens were all inspected for bond/unbond, and no unbonds were detected. Reference bond/unbond specimens were prepared from two pairs of substrates using coupling agent to represent the bonded case.

Equipment used:

Branson Sonoray, Model 51C

AI 5.0 MHz/.312 dia. type SFZ transducer

| Delay 5 | Damping 9 |
| Range 6 | C Gain A |
| Reject 1 o'clock | Extended Range 3 o'clock |

The core shear specimens were all inspected for bond/unbond, and no unbonds were detected. Reference bond/unbond specimens were prepared from two pairs of substrates using coupling agent for the bonded case.

Equipment used:

Branson Sonoray, Model 51C

AI 5.0 MHz/.312 dia. SFZ transducer

| Delay 5 | Damping 10 |
| Range 6 | C Gain A |
| Reject 1 o'clock | Extended Range 3 o'clock |

Measured bond-line thicknesses are presented in Table III for comparison.

5. Destructive Test Evaluation

Results of destructive tests are presented in Table IV. The specimens were all pulled at room temperature, 75°F ± 2°F and 50% RH, on the Instron universal testing machine. Crosshead speed was adjusted to provide an applied stress rate of 600 to 700 psi per minute.

6. Correlation and Analysis

No correlation and analysis studies have been conducted to date. The entire content of data and experience with these specimens will be sifted via correlations to extract the meaningful relationships.
| Surface Finish Nominal | BUTT TENSILE | | | | CORE SHEAR | | | | LAP SHEAR | | |
|------------------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
|                        | 1A | 2A | 3A | 1A | 2A | 3A | 1A | 2A | 3A | 1A | 2A | 3A |
| 1 RMS                  | 5.5 | 3.8 | 4.9 | 5.7 | 4.8 | 4.2 | 2.1 | 3.9 | 2.7 |       |       |   |
| 5 RMS                  | 4.4 | 4.2 | 4.4 | 5.5 | 6.3 | 4.6 | 2.6 | 1.0 | 3.2 |       |       |   |
| 7 RMS                  | 10.0 | 4.7 | 6.7 | 5.2 | 4.9 | 3.9 | 5.0 | 2.0 | 1.3+  |       |       |   |
| 10 RMS                 | 7.1 | 6.6 | 4.2 | 5.1 | 3.6 | 5.0 | 1.2 | 2.3 | 3.5 |       |       |   |
| 2 RMS                  | 7.6 | 6.5 | 5.5 | 6.3 | 9.7 | 7.4 | 3.9 | 3.1 | 1.9 |       |       |   |
| 40 RMS                 | 6.1 | 6.5 | 5.5 | 8.8 | 8.2 | 7.9 | 4.5 | 4.6 | 3.6 |       |       |   |
| 80 RMS                 | 4.7 | 6.4 | 6.7 | 7.4 | 5.5 | 5.2 | 7.2 | 6.1 | 4.4 |       |       |   |
| 110 RMS                | 8.3 | 7.5 | 8.1 | 5.6 | 5.2 | 8.2 | 3.6 | 5.9 | 6.4 |       |       |   |
| 150 RMS                | 7.7 | 4.9 | 6.0 | 6.0 | 7.5 | 6.4 | 5.0 | 2.7 | 2.8 |       |       |   |
| FGPL                   | 6.1 | 5.6 | 4.5 | 8.1 | 6.6 | 5.8 | 3.7 | 4.4 | 3.9 |       |       |   |
| FGHP                   | 6.6 | 5.8 | 5.5 | 5.5 | 4.6 | 4.9 | 3.7 | 3.0 | 4.8 |       |       |   |
| CGLP                   | 7.3 | 6.0 | 4.6 | 6.0 | 7.3 | 5.7 | 2.2 | 1.3 | 2.7 |       |       |   |
| CGHP                   | 4.4 | 2.7 | 5.3 | 3.9 | 5.4 | 4.0 | 3.0 | 5.2 | 5.3 |       |       |   |
### TABLE IV

ADHESIVE BOND ULTIMATE STRENGTH

<table>
<thead>
<tr>
<th>Surface Finish</th>
<th>BUTT TENSILE</th>
<th>CORE SHEAR</th>
<th>LAP SHEAR</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1A</td>
<td>2A</td>
<td>3A</td>
</tr>
<tr>
<td>1 RMS</td>
<td>3900</td>
<td>3750</td>
<td>3200</td>
</tr>
<tr>
<td>5 RMS</td>
<td>5770</td>
<td>4780</td>
<td>5540</td>
</tr>
<tr>
<td>7 RMS</td>
<td>3600</td>
<td>3920</td>
<td>5190</td>
</tr>
<tr>
<td>10 RMS</td>
<td>4100</td>
<td>5040</td>
<td>2660</td>
</tr>
<tr>
<td>20 RMS</td>
<td>5580</td>
<td>6930</td>
<td>4750</td>
</tr>
<tr>
<td>40 RMS</td>
<td>4070</td>
<td>3920</td>
<td>4240</td>
</tr>
<tr>
<td>80 RMS</td>
<td>5300</td>
<td>4210</td>
<td>5100</td>
</tr>
<tr>
<td>110 RMS</td>
<td>4450</td>
<td>4080</td>
<td>3630</td>
</tr>
<tr>
<td>150 RMS</td>
<td>5750</td>
<td>5510</td>
<td>5320</td>
</tr>
<tr>
<td>FGLP</td>
<td>7590</td>
<td>5210</td>
<td>7300</td>
</tr>
<tr>
<td>FGHP</td>
<td>6930</td>
<td>7160</td>
<td>5560</td>
</tr>
<tr>
<td>CGLP</td>
<td>5110</td>
<td>6720</td>
<td>4710</td>
</tr>
<tr>
<td>CGHP</td>
<td>6110</td>
<td>5760</td>
<td>6770</td>
</tr>
</tbody>
</table>
B. Exo-Electron Emission

Subsequent investigations by Dr. George Martin have shown the need for incident electromagnetic energy to stimulate exo-electron emission during fatigue cycling. (Reference 1). In these experiments his staff used the Channeltron electron multiplier by itself, without the cylindrical electrostatic mirror. Results with this technique were satisfactory for his purposes.

These successes, coupled with the deduced advantages this approach holds for substrate surface characterization, serve to encourage further development in the face of restrictions imposed by high vacuum.

The equipment necessary for creating the desired high vacuums, while allowing sufficient working room to perform point-to-point and scan measurements, must be designed and costed. Remaining effort on this approach within the current funding period, will be to analyze such designs and costs and the cost of the electron-detection system.

C. NDT Surface Characterization

1. Defining a Surface

Having observed that the various literature definitions for a "surface" are either too idealized, observationally vague, or mathematically restricted, we have sought to define a surface which has the capability to perform the material-energy interactions already cataloged in the sciences of physics and chemistry. This view prepares the way for selection of energy forms useful to the nondestructive characterization of surfaces. Our definition is presented in Figure 2. It is compatible with current thinking in quantum mechanics, particularly with reference to chemical bonding (Reference 2).

A surface is defined here as, "A region between two different phases which exists in an excited, high-energy state relative to the energy states normally associated with each of the bulk phases, where dimensional aspects are quasi-stable but energy aspects are highly mobile."

With this definition we have expanded and clarified, to some degree, the definition of "surface" as stated in Quarterly Report No. 3. Most important in this new definition is that no attempt has been made, as yet, to identify the frequencies and locations (i.e. atomic orbitals, molecular orbitals) of specific energy quantities, such as those which control the wetting phenomenon. Although pinpointing such energy states is beyond present quantum mechanical computational capabilities, awareness of the existence of such states is extremely valuable to this program.

2. Gas-phase Ultrasonic Transmission

Following the concept of deBroglies' "pilot waves" associated with total-internal light reflection, the analogous "pilot waves" in ultrasonic acoustics serve as the basis for the "gas-phase ultrasonic transmission method." It is known in acoustics that a solid-gas interface (surface) presents a huge acoustic impedance (\(Z\)) mismatch, resulting in total internal reflection of the acoustic wave (or pulse) in the solid. This serves as the basis for pulso-echo measurements in nondestructive testing. The "total" reflection is for all practical purposes total energy return, yet a small but significant amount of the acoustic energy is actually transmitted through the surface into the
FIGURE 2. CONCEPT OF "SURFACE" IN TERMS OF QUANTUM MECHANICAL ENERGY STATES
gas phase as pilot waves or wave fronts. That amount, and its frequency/phase/amplitude character, must depend upon the energy state (excitement) of the surface. Thus, by measuring the gas-phase transmitted acoustic energy, information concerning the state of the surface will be obtained. Means to experimentally accomplish such measurements are currently being developed.

3. Electric Field Reflectometry

Just as we characterize surface-reflected light in terms of its intensity, color (frequency), polarization plane angle, etc. to describe the character of a surface (i.e. mirror, paint-job, velvet, frost), the reflections of electromagnetic energy at other frequencies will carry information related to surface energetics. By using single-frequency (coherent) energy forms proper characterizations of incident and reflected energy may be readily accomplished. Selection of the frequency provides the means for isolating individual surface material-energy interaction relationships specifically related to adhesive bonding.

By way of a starting point with existing equipment, electric field reflectometric measurements are being conducted at 9.8 GHz (microwaves) and 1 KHz (low frequency capacitance) Figures 3 and 4. The wavelengths associated with these frequencies are orders of magnitude greater than light wavelengths, such that a rough surface does not create the "optical illusion" diffraction effects usually perceived by the casual observer in looking at such surfaces. The specific means of separating first order interactions from the second and third-order interactions of interest are now being investigated.

III. CONCLUSIONS AND RECOMMENDATIONS

None

IV. FUTURE WORK PLANNED

The following work is planned for the quarter 1970 January 20 through 1970 April 19:

1. Complete correlation and analysis of data from surface character study.

2. Explore feasibility of gas-phase ultrasonic transmission method through initial experiments.

3. Develop means for performing electric field reflectometry at 9.8 GHz and 1 KHz.

John Burbick
Principal Investigator

E. A. Proudfoot
Program Manager
FIGURE 3. LOW-FREQUENCY CAPACITANCE REFLECTOMETRY, 1 kHz
REFERENCES
