Proceedings of the Second Annual Symposium for Nondestructive Evaluation of Bond Strength

Compiled by
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Proceedings of a symposium sponsored by the National Aeronautics and Space Administration, Washington, D.C., and held at Langley Research Center, Hampton, Virginia, November 6, 1998

National Aeronautics and Space Administration
Langley Research Center
Hampton, Virginia 23681-2199

May 1999
Fifteen nondestructive evaluation (NDE) experts met for the Second Annual Review of NASA's NDE of Bond Strength Program at LaRC, NDE Sciences Branch on November 6, 1998. The goal of this research is to nondestructively determine quantitative strength levels in structural bonds. The Symposium was held to review both "in house" NDE research and work performed by sponsored university grantees. The grants reviewed were: "Investigation of Adhesive Bond Cure Conditions using Nonlinear Ultrasonic Methods", The Johns Hopkins University (Dr. Robert Green and Mr. Tobias Berndt); "An Assessment of Adhesive Bond Deterioration by Detection of Nonlinear Effects", Northwestern University (Dr. Jan D. Achenbach and Mr. Zhengeng Tang); "Characterization of Adhesive Bonds Using Nonlinear Ultrasound", The Georgia Institute of Technology (Dr. Jianmin Qu and Mr. Larry Jacobs). Invited presentations were given by Drs. Stan Rokhlin and Lazslo Adler of the Ohio State University & Adler Consultants, and Dr. Donald C. Price of the Computational Industrial Research Organization (CSIRO, Sydney, Australia). Several technologies and approaches were presented including "Microwaves for Bondline NDE" by Dr. Mark Roberts, ARMY-VTC, "Surface Contamination Monitoring using Optical Simulated Electron Emission (OSEE)" by Mr. Daniel Percy, NASA, and "Computational Chemistry of Bondlines" by Dr. Donald Phillips, NASA. Nonlinear ultrasonics is currently the leading technology for nondestructively determining bond strength. The Symposium proceedings are published in this NASA Conference Publication.
Bond Strength Program

Dr. Mark J. Roberts

Friday, 6 November 1998
NDE BRANCH WELCOME
8:30-8:45 Dr. Edward Generazio (Head)

NASA BOND STRENGTH
8:45-9:00 Overview and Status of Program: Dr. Mark Roberts
9:00-9:10 Microwaves for Bondline NDE : Dr. Mark Roberts
9:10-9:30 Computational Chemistry of Bonds: Dr. Donald Phillips
9:30-9:50 Surface Contamination Monitoring using OSEE: Mr. Dan Perey

GRANTEE PRESENTATIONS
9:50-10:15 The Johns Hopkins University: Dr. Bob Green & Tobias Berndt
   Investigation of Adhesive Bond Cure Conditions using Nonlinear Ultrasonic Methods
10:15-10:30 BREAK
10:30-11:15 Georgia Technology Institute: Dr. Jianmin Qu
   Characterization of Adhesive Bonds Using Nonlinear Ultrasound
11:15-11:45 Northwestern University: Dr. Jan Achenbach - An Assessment of Adhesive Bond Deterioration by Detection of Nonlinear Effects
11:45-1:15 LUNCH
EXTERNAL PROGRAMS
1:15-1:45 CSIRO Sydney, Australia: Dr. Donald Price - Progress Towards a Theoretical Model of Nonlinear Guided Wave Propagation in Bonded Joints

1:45-2:45 The Ohio State University & Adler Consultants: Dr. Stan Rokhlin & Dr. Laszlo Adler - Characterization of Adhesive Bond Integrity Using Angle Beam Spectroscopy

2:45-3:00 COFFEE BREAK

3:00-4:00 GENERAL DISCUSSION

4:00 ADJOURN
# Sign-in Sheet

<table>
<thead>
<tr>
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<th>Phone</th>
<th>Org</th>
<th>Email</th>
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</thead>
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</table>
NEDB SUPPORT OF PROGRAMS

- OFFICE OF SAFETY AND MISSION ASSURANCE (AGENCY LEAD)
  - SOLID ROCKET COMPOSITE NOZZLE INTEGRITY
  - SSME COMBUSTOR LINER INTEGRITY
  - MARS MICROPROBE MICROELECTRONICS PACKAGING INTEGRITY
  - SPACE STATION WINDOW INTEGRITY

- COMPOSITE WING TECHNOLOGY
  - NDE MEASUREMENTS ON WING COMPOSITE COMPONENTS
  - NDE ASSESSMENT OF BONDED STRUCTURES
  - MANUFACTURING PROCESS CONTROL

- HIGH SPEED RESEARCH
  - NDE FOR DURABILITY
  - IN-SITU MEASUREMENTS (DENSITY, STIFFNESS, AND FLAW LOCATION)

- AIRFRAME STRUCTURAL INTEGRITY/AIRWORTHINESS ASSURANCE
  - NDE FOR DISBOND, CORROSION, AND CRACKS IN AIRFRAME STRUCTURE
  - CRACKS IN THICK METALLIC COMPONENTS, QUANTITATIVE RESIDUAL STRESS, FATIGUE SENSING

- X-33/34 REUSABLE LAUNCH VEHICLE
  - STRUCTURAL HEALTH MONITORING & SHUTTLE UPGRADES

- BASE: QUANTITATIVE NDE OF BOND STRENGTH, MORPHING

Structures & Materials Research Group
NASA Langley Research Center
Program Issues

- Results of Successful Program

- Could eliminate or reduce rivet / fastening technology
- Production of higher strength bonds
- Higher confidence in bonded structures
- Measurement technique to quantitatively measure strength and assign quality
- Reduce operational downtime of aircraft significantly
- Prototype instrument developed would have potential in any industry requiring "on the spot" bond analysis
Program Issues

- Customer Needs & Requirements
  - Aircraft operational safety
  - General structural integrity (Composite Structures)
  - Cost reduction using bonded structures
- Nondestructive Evaluation Methods
  - Ultrasonics
  - Microwaves
  - OSEE (Q & A)
- Mechanical Testing Methods / Destructive
  - Fatigue test
  - Load tests
Goals & Approach

- **MAIN GOALS**
  - Develop and optimize NDE method(s) for measuring bond strength & bond quality levels
  - Develop instrumentation for prototype system capable of measuring bond strength & quality nondestructively

- **APPROACH**
  - Industry / NASA / University collaboration into third year of work
  - Selected NDE technologies best suited for the bond strength problem solution: Ultrasonics (Overall), Microwaves (Moisture)
  - Perform strength measurements using selected NDE methods
  - Begin system prototype design of bond strength instrument
Degradation, Damage and Failure

**Degradation**
- Adhesive: crosslink density, moisture absorption, plasticization
- Interphase: corrosion, oxidation
- Adherend: deformation

**Damage Accumulation**
- creep and fatigue
- microvoids
- microcracks

**Failure**
- fracture
- crazing
- debonding
- delamination
- fatigue and fracture

**Graph**
- Remaining Life vs. Time
- Phases: I, II, III
BONDING KEY ISSUES

- Adhesive bond strength & bond quality effected by
  - Joint type & geometry
  - adherends & adhesives used
  - quality control in bond creation process
  - primer type
  - surface roughness of adherends
  - wettability of adherends

- Modes of failure: Cohesive / Adhesive / Mixed Mode

- Cohesive Properties (Bulk)
  - adhesive chemistry & mechanical properties
  - bondline thickness
  - cure state

- Adhesive Properties (Surface)
  - Interfacial Boundary Properties / mechanical interlocking
  - Existence of weak boundary layers
  - Contamination / Environmental Effects
  - Moisture / water in the bondline
Collaboration / Resources

• NASA / VTC Army
  • 2 NASA CSs & 1 Army - Funding $600 thru FY01

• University Grantees
  • Dr. Jianmin Qu - Georgia Institute of Technology
  • Dr. Robert Green - The Johns Hopkins University
  • Dr. J.D. Achenbach - Northwestern University

• Private Industry
  • Dr. Wayne Woodmansee - Boeing Airplane Group
    Seattle, WA
  • Dr. Donald Price - Computational Sciences Industrial
    Research Organization (CSIRO)
    Division of Applied Physics
    Sydney, Australia

• ARL Rodman
  • Dr. Steven McKnight - Adhesive Bonding Microfactory
    (Polymers Research Branch)
Collaborative Work with NASA

- University grantees unanimously conclude that nonlinear ultrasonics best method for bond strength analysis: Johns Hopkins, Northwestern, Georgia Tech
  - ALL Grantees are in their 3rd year of research to conclude 6/30/99

- Industrial partners Boeing & CSIRO Sydney continue work on "weak" bond conditions using peel ply insertion into bonds
  - Report submitted: Static Stress Effects on Guided Waves in Adhesive Bonds (Dr. Don Price of CSIRO)

- Army Research Lab: Rodman Facility Aberdeen (Polymers Research Branch), Adhesive Bonding Microfactory
  Possible Collaboration Opportunity Exists
  Visit to ARL occurred in October '98
In House Research

- Computational Chemistry Approaches to Bondline Analysis (Dr. Donald Phillips)

- Laser ultrasonic methods to analyze adhesively bonded specimens proposed (Madaras / Roberts)

- Microwaves for Moisture Detection in Nonmetallic Bonds (Roberts)

- Optically Stimulated Electron Emission (OSEE) (Perey)
Accomplishments FY98

- Nonlinear ultrasonic methods was decided as the best way to potentially attain the nondestructive measurement of both bond quality and strength in adhesive bonds.

- Nonlinear ultrasonics proved capable of clearer detection of disbond areas in aluminum lap joint.

- Nonlinear stress / strain relationship determined to occur before an adhesive bond breaks.

- High amplitude ultrasound generates increased harmonics as bonds age at constant temperature suggesting nonlinear material changes.

- Bond deterioration caused by increased cyclic fatiguing.

- Plate waves used on aluminum and composite stiffener joints showed correlation between good and weakened adhesive joint conditions with peel ply.

- Scaled nonlinear parameter shown to be sensitive to 3 known good bonds & 3 bonds containing peel ply material.

- Microwaves are feasible for moisture detection in adhesive bonded non-metals.
FY99
- Investigate higher order microstructural properties thru harmonic detection
- Examine adhesive & cohesive property effects on an adhesive bond using nonlinear ultrasonics. Begin correlation to strength and quality.
- Conclude university research grant work & utilize conclusions reached for future study
- Fabricate & measure adhesively bonded joints with moisture contamination using microwaves (non-metal adherends)
- Investigate electrical properties of a bondline adhesive
- Test microwave method on good & disjoined bonds (non-metal adherends)

FY00
- Establish database of nonlinear ultrasonics measurements for selected adh. bonds with conditions ranging from initial degradation to accumulated damage to failure
- Determine good, weak and bad quality conditions for selected bonds using database of known stored ultrasonic signals
- Correlate to expected strength and quality level for selected adhesive bonds
- Determine capabilities, if any, for microwaves in general bond analysis
FY01

- Start development of prototype system and specifications for bond strength measurement instrumentation.

- Test prototype on adhesively bonded joint
Future In House Steps

- Continue literature research as more new information becomes available
- Build on existing results provided by university grantees
- Be open to technologies not previously used which could provide scientific insight into bond strength solution
  - Microwave (Moisture contamination)
  - OSEE (Q & A issue)
- Consider numerical modeling techniques to look at effects of changes in cohesive (volume) and adhesive (surface) properties on bondline performance
- Microwave theoretical & experimental work for adhesively bonded nonmetallic joints
  - Joint material changes
  - Moisture / water detection
- Create database of ultrasonic information for various adhesive bonds to quantify strength and characterize quality levels
SM.TF.06: Structural Integrity, safety, and health monitoring for reliable aircraft, space transportation vehicles, and space habitat

- Fundamental Concepts

<table>
<thead>
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<th>FY 99</th>
<th>FY 00</th>
<th>FY 01</th>
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<td>6</td>
<td>7</td>
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**Milestones**

1. Assemble measurement system to test fatigue sensor concept on AI 2024
2. Data on inspection systems for NDE of bond strength
3. Test fatigue/load history probe with AI 2024
4. Down select bond strength approach
5. Optimize sensor for fatigue/integrated history
6. Test and optimize bond strength measurement system
7. Ready fatigue/integrated load history sensor for technology transfer
8. Ready bond strength NDE technique for technology transfer
Microwaves for Bondline NDE

Dr. Mark J. Roberts

Friday, 6 November 1998
Advantages

- Good penetration of nonconductive media
- Contact or noncontact choice
- Small probe size
- No coupling gels needed on material being measured

Benefits

- Detection of disbond and delamination in stratified structures such as sandwich composites
- Moisture / Water detection capability in nonconductors
- Electrical material characterization
- Possible crack detection in ceramics and composites
- Porosity measurement capability
Small Horn Antennas
## Microwave Spectrum

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<th>Band</th>
<th>Frequency (GHz)</th>
<th>Wavelength (cm)</th>
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<tr>
<td>P</td>
<td>0.23 - 1</td>
<td>130 - 30</td>
</tr>
<tr>
<td>L</td>
<td>1 - 2</td>
<td>30 - 15</td>
</tr>
<tr>
<td>S</td>
<td>2 - 4</td>
<td>15 - 7.5</td>
</tr>
<tr>
<td>C</td>
<td>4 - 8.2</td>
<td>7.5 - 3.66</td>
</tr>
<tr>
<td>X</td>
<td>8.2 - 12.4</td>
<td>3.66 - 2.42</td>
</tr>
<tr>
<td>Ku</td>
<td>12.4 - 18</td>
<td>2.42 - 1.67</td>
</tr>
<tr>
<td>K</td>
<td>18 - 26.5</td>
<td>1.67 - 1.13</td>
</tr>
<tr>
<td>Ka</td>
<td>26.5 - 40</td>
<td>1.13 - 0.74</td>
</tr>
<tr>
<td>mm-Waves</td>
<td>40 - 300</td>
<td>0.75 - 0.1</td>
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</table>
MICROWAVE IMAGING

- Arranging microwave detected signal/data (i.e. raster scan) to produce a visual impression of the presence of defects or structural geometry.
- The microwave data may include such information as:
  - Phase of reflection or transmission coefficient
  - Magnitude of reflection or transmission coefficient
  - Combination of both (i.e Synthetic Aperture Imaging)
  - Attenuation information
- This may be accomplished using the far-field (i.e radar imaging) or in the near-field approaches.
- When using near-field microwave imaging, need to understand the information conveyed by the image.
- Probe type, field characteristics everywhere, geometry of the material under test, defect properties, position and geometry must be taken into account.
• Defect characteristics may be determined if all other non-defect influences are taken out

• When using open-ended rectangular waveguides for near-field imaging, one must have an intuitive understanding of its near-field properties.

• Ultimately, one may use inverse models or approaches to obtain defect property information.
Skin delamination in Thick Sandwich Composite @ 24 GHz
LOCAL POROSITY

Epoxy Resin

Scan Area

Air-Filled Microballoons

44% 49% 56%
Conclusions

• Microwaves are totally reflective to moisture & water
• Both delamination and porosity detection will be much more sensitive when containing moisture
• Cracks / small resolution flaws will have increased probability of detection if containing moisture

Plans for FY99

• Perform measurements to look at moisture between two composite layers which are unjoined
• Determine electrical property of adhesives
• Fabricate and measure composite joints with the following conditions
  • Good joint - no moisture
  • Water spray in or on adhesive before joining
  • Joint containing water voids in adhesive
• Perform analytical and/or numerical approach on above conditions
• Correlate between cohesive & adhesive properties and expected electrical property changes
Computational Chemistry of Adhesive Bonds

Donald H. Phillips
Talk Outline

- Background, Goals
- Model Systems
- Methods
- Comparison of Results for a Simple Model
Background and Goals

• The macroscopic response of a bond layer to external stimuli is determined by the molecular level mechanical and electrical properties of the bond layer. Also, changes in these properties due to degradation stem from chemical changes in the bond layer.

• This investigation is intended to determine the electrical mechanical, and chemical properties of a adhesive bonds at the molecular level. The initial determinations will be followed by investigations of the effects of environmental effects on the chemistry and properties of the bond layer.
Background and Goals
(Continued)

- Possart and Unger (Adhesion 15, p148 (1991)) found by x-ray photoelectron spectroscopy that the interaction between Aluminum Oxide and Poly(methyl methacrylate) (PMMA) occurred between the oxygen atoms of the oxide and the polymer. They found (1s) orbital energy shifts varying from +0.3 Ev for specific carbon atoms to -1.2 Ev for PMMA oxygen atoms.

- This system was chosen for initial investigation.
Models

- Cluster Model Issues
  - Surface Structure and Composition
  - Surface to Volume Ratio
  - Dangling Bonds
  - Cluster Size Convergence
al2o3 cluster model w/ 3 O layers and 2 al layers
al2o3 cluster model w/ 3 O layers and 2 Al layers - with H atoms on dangling O
PMMA-AL-Oxide (hydrogenated) Complex
AL-Red, O-Blue, C-Brown, H-Pink
PMMA DZP optimization with ch3 group rotated close to carbonyl 349.0 CM**.1
AL-O2-H Aluminum Hydroxide molecule optimization
Methods

- **First Principles** - all electron-electron interactions
  - Basis Set Quality (Minimum, Good, Accurate)
  - Correlation?

- **Approximate, Semi-Empirical**
  - Parameterization Appropriate for Models?
  - Model Capable of Treating Hydrogen Bonds?
  - Information on Core Energy Levels?

- **Molecular Mechanics**
  - Parameterization Adequate?
  - Electronic, Mechanical Properties?
## Results for Monomers

<table>
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<tr>
<th>Mol</th>
<th>Method</th>
<th>Struct.</th>
<th>Dipole</th>
<th>Vib</th>
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<td>scf-dzp</td>
<td>standard</td>
<td>2.04 D</td>
<td>349/cm</td>
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<tr>
<td></td>
<td>scf-min</td>
<td></td>
<td>1.98</td>
<td></td>
</tr>
<tr>
<td></td>
<td>PM3</td>
<td></td>
<td>1.89</td>
<td>346 *</td>
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<tr>
<td></td>
<td>AM1</td>
<td></td>
<td>2.02</td>
<td>345</td>
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<tr>
<td></td>
<td>MNDO</td>
<td></td>
<td>2.35</td>
<td>363 *</td>
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<td>scf-dzp</td>
<td></td>
<td>6.19</td>
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<td>scf-min</td>
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<td></td>
<td>233</td>
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<tr>
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<tr>
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<td>AM1</td>
<td></td>
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<tr>
<td></td>
<td>MNDO</td>
<td></td>
<td>3.39</td>
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</table>
### Results for PMMA-AlO₂H Complex

<table>
<thead>
<tr>
<th>Method</th>
<th>$D_0^c$ (KJ/mol)</th>
<th>Dipole M Debye</th>
<th>R = o---h Angstroms</th>
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<tbody>
<tr>
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<td>1.89</td>
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<tr>
<td>SCF-Min</td>
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<td></td>
<td></td>
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<tr>
<td>AM1</td>
<td>20.5</td>
<td>5.81</td>
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<tr>
<td>PM3</td>
<td>7.8</td>
<td>4.28</td>
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<tr>
<td>MNDO</td>
<td>17.5</td>
<td>5.24</td>
<td>3.33</td>
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alo2h-pmma complex (carbonyl to h bond) show newpath grestore gsave 15 15 moveto 0 rota
alo2h-pmma complex (carbonyl to h bond c ) show newpath grestore gsave 15 15 moveto 0 rota
alo2h-am1 complex (carbonyl to h bond c ) show newpath grestore gsave 15 15 moveto 0 rotat
Core Electron Energy Shifts

• For this configuration of the complex, increases of approximately 0.8 eV were computed for both PMMA oxygens and the Carbon 1s attached to the carbonyl O atom.

• This in qualitative agreement with the experiment (ie binding energy increases) but the agreement is not quantitative.
A Portable Surface Contamination Monitor Based on the Principle of Optically Stimulated Electron Emission (OSEE)

Daniel F. Perey

NESB Group Leader for Instrumentation Development

NASA Langley Research Center
Hampton, VA
OSEE - How It Works
Probe Cross Section

- Bargraph Display
- Line Driver Ckt., etc.
- Isolation Amplifier
- Low Pressure Mercury Vapor Lamp
- Parabolic Reflector
- Manual Trigger (Momentary)
- Shutter Assy.
- Electrometer Assy.
- Skirt
- Pick-up Grid
Ultrasonic Nondestructive Characterization of Adhesive Bonds

Jianmin Qu
School of Mechanical Engineering

Larry Jacobs
School of Civil and Environmental Engineering

Georgia Institute of Technology
Atlanta, GA 30332
Objectives

This project is concerned with the qualification of reliability and integrity of metal/polymer bond joints. The objectives are

* To establish the correlation between the microstructural changes and ultrasound propagation characteristics.

* To develop ultrasonic nondestructive methods to measure the microstructural changes caused by the degradation of bond strength.

* To predict remaining bond strength from ultrasonic measurement based on the fundamental structure-property-performance relationships of the constituents and their interfaces.
Degradation, Damage and Failure (durability)

 Degradation  | Damage Accumulation  | Failure
---|---|---
 Adhesive  | creep and fatigue  | fracture  
 crosslink density  | microvoids  | crazing  
moisture absorption  | microcracks
 plasticization
 Interphase  | microvoids  | debonding  
corrosion  | microcracks  | delamination
 oxidation  
 Adherend  | deformation  | fatigue and fracture
 oxidation
Through Transmission Test

High Voltage Amplifier
ENT, DC ~ 10 MHz, 50dB

Transducer

Sample

Function Generator
Wavetek, 50 MHz

Receiver

Oscilloscope
Techtronix, 150 MHz

Computer

AF-163-2K made by 3M Corp.

Generation Transducer
* PZT (2.5 MHz, Narrow-band) (Ultra, KC50 -2, 1.25MHz at -6dB)
* PZT (5.5 MHz, Narrow-band) (Ultra, KC50 -5, 3.5MHz at -6dB)
* Single Crystals (Quartz, lithium niobium)

Detection Receiver
* PZT (5.5 MHz, Narrow-band) (Ultra, KC50 -5, 3.5MHz at -6dB)
* PZT (7.5 MHz, Narrow-band) (Ultra, KC50 -10, 8.5MHz at -6dB)
* Laser Interferometer
Fourier Transforms

\[
F(\omega) = \int_0^\infty f(t) \exp(i\omega t) dt \\
A_n = |F(n\omega_0)| \\
n = 1, 2, 3 \ldots
\]

Amplifier Output

\[\omega_0 = 2\text{MHz} \quad |F(\omega)|^2\]

Receiver Output

\[\omega_0 = 2\text{MHz} \quad |F(\omega)|^2\]
Samples with Different Curing States

![Diagram showing Al2024 material]

The Material Properties

<table>
<thead>
<tr>
<th>Material</th>
<th>ρ (kg/cm³)</th>
<th>E (GPa)</th>
<th>v</th>
</tr>
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<tbody>
<tr>
<td>Al2024</td>
<td>2.78</td>
<td>73.0</td>
<td>0.35</td>
</tr>
<tr>
<td>AF-163-2K</td>
<td>1.21</td>
<td>1.11</td>
<td>0.34</td>
</tr>
</tbody>
</table>

Different Curing Conditions

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>B1</th>
<th>B2</th>
<th>B3</th>
<th>B4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Curing Temperature (°C)</td>
<td>121</td>
<td>82</td>
<td>82</td>
<td>90</td>
</tr>
<tr>
<td>Curing Time (min.)</td>
<td>90</td>
<td>60</td>
<td>120</td>
<td>60</td>
</tr>
<tr>
<td>Curing Pressure (MPa)</td>
<td>0.34</td>
<td>0.34</td>
<td>0.34</td>
<td>0.34</td>
</tr>
<tr>
<td>Total Thickness (mm)</td>
<td>3.54</td>
<td>3.51</td>
<td>3.55</td>
<td>3.55</td>
</tr>
<tr>
<td>Shear Strength (MPa)</td>
<td>35</td>
<td>4.0</td>
<td>3.3</td>
<td>4.1</td>
</tr>
</tbody>
</table>
Nonlinear Parameter of Different Samples

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>B1</th>
<th>B2</th>
<th>B3</th>
<th>B4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Curing Temp. (°C)</td>
<td>121</td>
<td>90</td>
<td>82</td>
<td>82</td>
</tr>
<tr>
<td>Curing Time (min.)</td>
<td>90</td>
<td>60</td>
<td>60</td>
<td>120</td>
</tr>
<tr>
<td>Bond Strength (MPa)</td>
<td>35</td>
<td>4.1</td>
<td>4.0</td>
<td>3.3</td>
</tr>
</tbody>
</table>
Buehler Epoxide Resin (room temp)

$70^\circ C$

$(X \times 10^{-8})$

- 0H
- 44H
- 88H
- 135H

Jianmin Ou
Generation of Higher Order Harmonics

Wave Equation
\[ \frac{1}{\rho} \frac{\partial \sigma}{\partial \varepsilon} = \frac{\partial^2 u}{\partial t^2} \]

Stress-Strain Relation
\[ \sigma = E f(\varepsilon) \]
\[ \varepsilon = \frac{\partial u}{\partial x} \]

(material nonlinearity)

\[ \frac{1}{c^2} \frac{\partial^2 u}{\partial t^2} - \frac{\partial^2 u}{\partial x^2} = \left[ f'(\varepsilon) - 1 \right] \frac{\partial^2 u}{\partial x^2} \]
Example

\[ f(\varepsilon) = \varepsilon(1 - 0.5\gamma\varepsilon) \]

\[
\begin{array}{c|c|c}
\text{(X10^3)} & \frac{\sigma}{E} \\
\hline
-8 & 0 \\
-4 & 0 \\
0 & 0 \\
4 & 0 \\
8 & 0 \\
\end{array}
\]

- \( \gamma = 0.0 \)
- \( \gamma = 1.0 \)
- \( \gamma = 10.0 \)
- \( \gamma = 30.0 \)

\[ \sigma = Ef(\varepsilon) \]

\[ A_1 \sin(kx - \omega t) \]

\[ A_2 \cos(2kx - 2\omega t) \]

For

\[ h = 320\,\mu\text{m} \quad c = 6410\,\text{m/s} \quad \omega = 2\pi \times 2\text{Hz} \]

\[ \beta = \frac{8A_2}{k^2hA_1^2} = \gamma \quad \text{Nonlinear parameter} \]

\[ \frac{A_2}{A_1} \approx 1.5 \times 10^{-4} \gamma \frac{A_1}{\mu\text{m}} \]
Generation of Harmonics in Metals

Lattice Anharmonicity

\[ F(a_0) = 0 \]

\[ F(a) > 0 \]

\[ V(a) \]

\[ -2E_{ad} \]

\[ F(a) \]

\[ F_{max} \]

Dislocation Motion

\[ y(s) \]

\[ x \]

Fig. 8-10. Displacement of a dislocation under stress (schematic). (a) Curve 1: dislocation displacement amplitude at low damping (small phonon density); curve 2: dislocation displacement amplitude for high damping. (b) Out-of-phase and in-phase component of dislocation displacement for high damping. (c) Change in Fig. 3-10b when pinning point is added at center of dislocation loop. (Pinning increases area of in-phase component, thereby decreasing elastic modulus and decreasing velocity of ultrasonic wave) (after Truell and Granato [188]).
A Simplistic Model

\[ f(x - ct) \quad g(x - ct) \]

\[ E_s = \begin{cases} E_s' & \text{when } \sigma(t) > 0 \\ E_s^c & \text{when } \sigma(t) < 0 \end{cases} \]

\[ \eta_t = \frac{E_s'}{E} \quad \eta_c = \frac{E_s^c}{E} \]

\[ E_s = \text{Adhesive stiffness} \]

\[ E = \text{Adherend stiffness} \]

Interface Conditions

\[ \sigma(t) = \sigma(0^+, t) = \sigma(0^-, t) \]

\[ u(0^+, t) - u(0^-, t) = \frac{h}{E_s} \sigma(t) \]

Solution

\[ g(x - ct) = f(x - ct) + \frac{1}{2} V(t - \frac{x}{c}) \]

\[ V(t) = u(0^+, t) - u(0^-, t) \]
Example

\[
f(x-c-t) = A \cos[k(x-c-t)]
\]

\[
g(x-c-t) = f(x-c-t) + \frac{1}{2} V(t-c)
\]

where

\[
V(t) = \begin{cases} 
2A \sin(\omega t_n + \varphi_c) e^{-\xi \frac{t}{h}} & \text{if } n = 0, 2, 4, \ldots \\
2A \sin(\omega t_n + \varphi) e^{-\xi \frac{t}{h}} & \text{if } n = 1, 3, 5, \ldots 
\end{cases}
\]

\[
\xi = \frac{c}{h} \\
\varphi = \sin^{-1} \left( \frac{\omega}{\sqrt{\xi^2 + \omega^2}} \right) \\
t_n = 0
\]
$h = 100 \mu m \quad c = 6410 \, m/s \quad \omega = 2 \text{Hz}$

Graph showing $f(x - ct)$ and $g(x - ct)$, with a line indicating transmitted and a dotted line indicating incident waves. The graph spans from $t = 0$ to $10$ (µm) with values from $-1.0$ to $1.0$. The graph demonstrates the wave behavior over time and distance.
\[ h = 100 \mu m \quad c = 6410 \text{ m/s} \quad \omega = 2 \text{Hz} \quad \eta_c = 0.05 \]

\[
\frac{A_2}{A_1} \quad \frac{\eta_c - \eta_l}{\eta_c}
\]
Setup for Guided Wave Measurement

transducer

\[ \theta_i \]

wedge

Receiver

\[ \theta_r \]
ASSESSMENT OF ADHESIVE BOND DETERIORATION BY DETECTION OF NONLINEAR EFFECTS

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PRINCIPAL INVESTIGATOR

ZHENZENG TANG
RESEARCH ASSISTANT

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NORTHWESTERN UNIVERSITY
EVANSTON, IL 60208-3020

GENERAL OBJECTIVE

To develop an ultrasonic nondestructive technique to assess deterioration of adhesive bonds by the detection of nonlinear effects. The work on this project is both analytical and experimental in nature.
The adhesive bond behavior can be represented by a relation between tractions, $\tau$, and gross displacements, $\Delta$, across the adhesive layer. Figure 1 shows four typical $\tau$-$\Delta$ curves with their associated failure points. Figure 1a represents a brittle bond with a linear relation between $\tau$ and $\Delta$. When $\tau$ reaches a critical value $\tau_{cr}$ the bond breaks in a brittle fashion. Deterioration of the bond gives rise to a lower value of $\tau_{cr}$. Figure 1b shows a bond with nonlinear elastic behavior typical of rubbery adhesives. The failure point is reached for $d\tau/d\Delta=0$. Deterioration of this bond may be described by the curves shown in Fig. 1c or Fig. 1d. Note that in Fig. 1c the slope remains the same at $\tau=0$, while in Fig. 1d this slope changes. For the case of 1d the slope at $\tau=0$, which can be determined by ultrasonic methods, can be correlated with residual strength. This has been done by many investigators. Here we will address the more difficult case represented by Fig. 1c.

Figure 1. Traction-displacement curves and associated failure points.
Outline

- Ultrasonic Evaluation of Adhesive Bond Degradation By Detection of the Onset of Nonlinear Behavior Induced by Static Load
  - Superimposed longitudinal wave
  - Superimposed shear wave

- Summary

- New Approach — A Strain-Temperature Correspondence Principle

- Digitized Waveform Decomposition Technique

- Preliminary Results

- Conclusions and Future Work
EXPERIMENT SETUP

Oscilloscope
Tek TDS 520

Pulser Receiver
Model 5055PR

Computer

GPIB

Specimen

MTS Machine

Loading Direction
1. Adhesive (connection)
2. adhesive (testing layer)
3. Al block (adherend 2)
4. Al Tube (water tank)
5. Screw
6. Al block (adherend 1)
7. Transducer
Methodology of Nonlinear Behavior Study

Use different fatigue cycles to generate different severities of degradation.

By varying the static load, ultrasonic measurements allow us to get the slope of the $\tau - \Delta$ curve at several points.

$$\frac{d\tau}{d\Delta} \approx \frac{\sigma}{\delta} = \beta$$
Load vs. Effective Modulus for 50-50 Epoxy Layer

![Graph showing effective modulus vs. applied load for 50-50 epoxy layer with load cycles marked: 0 cycle, 50K cycles, and 100K cycles.]
Reconstructed Stress-Strain Relation (50-50)

Reconstructed Stress–Strain Relation for 50–50 Epoxy Layer

- X: 0 cycle
- O: 50K cycles
- *: 100K cycles
Load vs. Effective Modulus for 70-30 Epoxy Layer

![Graph showing the relationship between actual load applied (lbs) and effective modulus (GPa) for 70-30 Epoxy Layer. The graph includes data points for 0 cycle, 50K cycles, and 100K cycles.]
Reconstructed Stress-Strain Relation (70-30)

Reconstructed Stress–Strain Relation for 70–30 Epoxy Layer

- X: 0 cycle
- O: 50K cycles
- *: 100K cycles
A Superimposed Shear Wave

Shear deformation illustration

Experiment set-up

\[ \alpha = \sin^{-1}\left(\frac{C_w}{C_t}\sin\beta\right) \]  

\[ \theta = \frac{\pi}{2} - \alpha \]
Superimposed Shear Wave—Results

Load vs. Shear Modulus

Stress-Strain Curve

Simultaneous Measurements

Load vs. Modulus
Summary

The onset of nonlinear behavior of adhesive bonds can be detected ultrasonically. The results show that the degradation due to cyclic fatigue can be detected by the reduction of the linear portion of the stress-strain curve without any change of slope in the linear range.

Shear waves can be used to detect the onset of nonlinear behavior of adhesive bond degradation generated by cyclic fatigue while the specimen is under shear loading. Longitudinal waves can also be used for this purpose.

The nonlinear behavior of a cyclically fatigued specimen is initiated at a lower stress level of the shear loading than the tensile loading. For practical reasons, it is preferable to subject the specimen to shear loading while the detection uses longitudinal waves.
A New Approach — A Strain-Temperature Correspondence Principle

The initial slopes at temperature $T_1$ and $T_2$ and slopes at strain $\epsilon_1$ and $\epsilon_2$ are the same.

\[
\sigma = C_0 [\epsilon + f(\epsilon)]; \quad \frac{d\sigma}{d\epsilon} = C_0 [1 + f'(\epsilon)] \quad (7)
\]

\[
C(T) = C_0 [1 + h(T)] \quad (8)
\]

Strain-Temperature Correspondence Relation

\[
h(T) = f''(\epsilon) \quad (9)
\]
Preliminary Results

Temperature Dependence of velocity for several materials

Temperature dependence of velocity for various materials. Water (solid), FM73 (dashed), DER Epoxy (dashdot), AB Epoxy(solid)
Complete Recovery of Ultrasonic Signal

Complete recovery of ultrasonic signal after one cycle of heating of sample #3. 220°C before heating (solid line), 220°C after cooling (dashed line)
A Simple Model

Quadratic Nonlinear Term

\[ \sigma = C_0 \left[ \epsilon - \frac{\epsilon^2}{2\epsilon_0} \right] \]  
(11)

Definition of Temperature-Velocity Coefficient \( \alpha_c \)

\[ \frac{dc(T)}{c_0 dT} = -\alpha_c \]  
(12)

Strain-Temperature Correspondence Relation

\[ \frac{\epsilon}{\epsilon_0} = 2\alpha_c \Delta T - (\alpha_c^2 + 6\alpha_c \beta)\Delta T^2 + 3\alpha_c^2 \beta \Delta T^3 \]  
(13)
Reconstructed Results

(a) Temperature dependence of velocity in three samples and the bulk sample. Bulk sample (solid line), sample #1 (solid line, 'o'), sample #2 (dashed, 'x'), sample #3 (dash-dot, '*').

(b) Theoretical prediction of ultimate strain and ultimate stress for AB Epoxy specimens. Solid (sample #1), Dashed (sample #2), Dash-dot (sample #3).
Conclusions and Future Work

Conclusions

It has been shown that the new approach has potential. The application of the strain-temperature correspondence nondestructively yields nonlinear parameters which can only be obtained destructively otherwise. These nonlinear parameters can define the residual strength of an adhesive bond.

Future Work

(1) Improve the temperature controlling system.

(2) Measure the correspondence function.

(3) Apply this principle to study adhesive bond degradation.
Investigation of Adhesive Bond Cure Conditions using Nonlinear Ultrasonic Methods

Tobias P. Berndt and Robert E. Green, Jr.

CNDE
Center for Nondestructive Evaluation
The Johns Hopkins University
Department of Materials Science and Engineering

Acknowledgments:
Funding by NASA Langley and CNDE
Samples by BOEING

November 6, 1998
Objective

Investigate various Cure Conditions of Adhesive Bonds using Nonlinear Ultrasonic Methods with Water Coupling

- Normal Incidence
- Oblique Incidence
- Wave Mixing
Samples

- 250 °F for 90 min at 50 psi (Normal)
- 180 °F for 60 min at 50 psi
- 180 °F for 120 min at 50 psi
- 195 °F for 60 min at 50 psi

- No significant differences in Bond Thickness across Samples
- Thicknesses: ~360μm (center) to ~150μm (edge)
- Longitudinal Velocity through Normal Bond (~2.33 mm/μs) up to 7% lower than in all other cases
- Shear Velocity through Normal Bond (~0.96 mm/μs) up to 6% lower than in all other cases
Adhesive Bond Sample

2024 T3 Aluminum Sheet

~ 1.6 mm

BONDLINE (~ 0.15-0.35 mm)
Normal Incidence
(Longitudinal Transmission)
Bond Sample versus Single Aluminum Plate

- **Amplitude of Fundamental (V)**
- **Sample Position along Transmitter-Receiver Axis (in)**

<table>
<thead>
<tr>
<th>Amplitude (V)</th>
<th>Sample Position (in)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.0</td>
<td>-0.6</td>
</tr>
<tr>
<td>2.5</td>
<td>-0.4</td>
</tr>
<tr>
<td>2.0</td>
<td>-0.2</td>
</tr>
<tr>
<td>1.5</td>
<td>0.0</td>
</tr>
<tr>
<td>1.0</td>
<td>0.2</td>
</tr>
<tr>
<td>0.5</td>
<td>0.4</td>
</tr>
</tbody>
</table>

- **Al-Plate**
- **Bond Sample**
Measuring between Resonances, but at optimum Transmitter Frequency (5.46 MHz)

Amplitude of 2nd Harmonic (V)

[Amplitude of Fundamental]² (V²)

C-series

180-60
180-120
195-60
250-90 (norm)

Normal Incidence Immersion
First Through Transmission Resonance of Adhesive

H-series

Amplitude of 2nd Harmonic (V)

[Amplitude of Fundamental]² (V²)

Undercured

Normal Bond

Undercured

Normal Incidence Immersion
Measuring between Resonances, but at optimum Transmitter Frequency (5.46 MHz)
Contact Measurements

• To verify existence of and differences in both 2\textsuperscript{nd} and 3\textsuperscript{rd} Harmonics in given Bond system
• Transmitter: 5 MHz LiNbO\textsubscript{3} bonded with SALOL
• Receiver: 10 MHz broadband (commercial contact)
• Samples: 0.25" Aluminum Substrates
180 °F 60 min (Undercured) versus 250 °F 90 min (Normal)

Transmitter Drive Voltage (Vpp)

Received Amplitude of Fundamental (V)

Contact Measurements

Undercured

Normal Bond
180 °F 60 min (Undercured) versus 250 °F 90 min (Normal)

Amplitude of 2nd Harmonic (V)

[Amplitude of Fundamental]² (V²)

Undercured

Normal Bond

Contact Measurements
Aluminum Screw Sample

Amplitude of Sum-Frequency (V)

Position from Transmitters (in)

- Frontsurface of 1st Plate
- Frontsurface of 2nd Plate
- Backsurface of 1st Plate
- Backsurface of 2nd Plate
180 °F 60 min (Undercured) versus 250 °F 90 min (Normal)

Amplitude of Sum-Frequency (V)

Position from Transmitters (in)
Adhesive Sum-Signal
normalized by Backsurface Sum-Signal

Graph showing the relationship between Normalized Signal and Transmitter Separation (mm) for H-Normal and H-1801.
ABUS System for Quantitative Inspection of Adhesive Bonds

Laszlo Adler, Stanislav I. Rokhlin
Christophe Mattei, Gabor Blaho

Adler Consultants, Inc.
Columbus, Ohio.

Supported by S.B.I.R. Program, Contract #N68335-97-C-0328
Outlines

• Our Approach to Quantitative Bond Inspection
  • Statement of the problem
  • Ultrasonic Spectroscopy
  • Bond Quality Scanning

• The ABUS Scanning Inspection System
  • System Hardware description
  • System Software Description

• Validation results
  • Inspection results
  • ABUS Data Correlation to Strength
- Adhesive bond integrity depends on:
  - Adhesive bulk properties
  - Adhesion between adhesive and the substrates

- Usually, weak bonds are associated with poor adhesion or effect of kissing bonds.

- Weak bonds remain hidden from conventional inspection methods
Conventional Ultrasonic Inspection

Amplitude Measurement

Transit Time Measurement
Angle Beam Ultrasonic Spectroscopy for Adhesive Joint Inspection: Approach

The novelty of this approach is:

- It combines the application of obliquely and normally incident ultrasonic beam on the bond line
- It measures the frequency response of the bond line
- The oblique wave introduces shear stress on the bond line, allowing discrimination of kissing or poor bond from good bonds
- The normally incident wave is used to decouple the effect of the bond line thickness
Angle Beam Ultrasonic Spectroscopy

Simultaneous Normal and Oblique Incidence Reflection Coefficient

Time signal

FFT

Reflection coefficient

Layer resonance
Reflection Coefficients Calculation

Conclusion:

Adhesive joint degradation effect:
  • Little effect on the normal incidence reflection spectrum
  • Oblique incidence reflection spectrum exhibits strong shift of the spectrum minima
Effect of Imperfect Layer/Substrate Interface on Ultrasonic Signature

- Oblique incident ultrasonic waves are sensitive to adhesive/adherent interface quality

- Interface modeling allows us to select incidence angle which is sensitive to interface quality

Simulation of frequency shift as function of interface spring constant for an oblique incident angle on composite
The ABUS concept: Layer Properties Reconstruction

Step 1
- Normal Incidence Reflection Coefficient

Step 2
- Oblique Incidence Reflection Coefficient

Parameters reconstruction

Effective moduli: longitudinal, transverse
Layer thickness

Bond quality factor: \[
\frac{\text{effective moduli}}{\text{adhesive moduli}}
\]
The Angle Beam Ultrasonic Spectroscopy (ABUS) Scanner Concept

y-axis motor

x-axis motor

Scanning Head

Scan Area

Composite plate

Adhesive Joint
The ABUS System
The ABUS System
ABUS System Hardware
Overall Design

XYZ Mechanical System

Motor X
Motor Y
Motor Z

X,Y,Z Motion Controller

A/D Converter

Ultrasonic Head

Pulser Receiver Module

PC Computer
- Three axis Parker Deadal mechanical system
X axis: Open Frame Square Rail Positioning Table
38"x24" opening in base plate
40" travel, Max. travel speed: 1'/s
Precision Ground Ball Screw
Optical home and end of travel
Y axis: Square Rail Positioning Table
23.6" travel, Max. travel speed: 1'/s
Precision Ground Ball Screw
Optical home and end of travel
Z axis: Square Rail Positioning Table
11.8" travel, Max. travel speed: 1'/s
Standard Ball Screw
Optical home and end of travel

- American Precision Industries Microstep Motors: 300 oz-in torque, 45000 step per revolution
- American Precision Industries Low EMI microstepping drive and power supply
- Parker Compumotor PC Based Step Motor Controller
ABUS System: Software Overall Design

Hardware control
- Motion Control
- Pulser/Receiver Control
- A/D Converter Control
- System control & operation

Processing and Algorithm
- Data Processing
- Reconstruction
- Display
- Storage

User Interface
ABUS System: Software User Interface Design

System operation control

A/D Control

2 Channels (Normal and Oblique)
- Sampling rate
- Trigger control
- Time domain signal display

3 gates per channel:
- Gates position control (start and width)
- Gates display

Scan Control

- Scanning size
- Scanning step
- Scanning speed
- Reference points control
Sample Preparation and Mechanical Testing

Aluminum 2024 T3 or Unidirectional composite coupon dimensions: 1” x 4”

FM73 Adhesive, layer dimensions: 1” x 0.5”

<table>
<thead>
<tr>
<th></th>
<th>Average failure load</th>
</tr>
</thead>
<tbody>
<tr>
<td>Treated samples</td>
<td>800 lb</td>
</tr>
<tr>
<td>Reference samples</td>
<td>1400 lb</td>
</tr>
</tbody>
</table>
ABUS System: Software User Interface

Data processing and display

Processing control
- FFT control parameters
- Frequency domain gate control
- Reconstruction
- Storage

Result display
- Bond quality display
- C-scan display
- Gray and/or color levels
- Storage
ABUS System: Software User Interface

Channel 1: Normal incidence

Channel 2: Oblique incidence

- Main gate (Storage)
- Front surface echo gate
- Interface echo gate
ABUS System Software Algorithms

Data processing and properties reconstruction

Signal processing → Properties reconstruction

Algorithm

Scattering reduction

Fast Fourier Transform

Algorithm

Propagation in multi-layered composites

Reconstruction

Separation of adhesive/adherent properties

Adler Consultant Inc.
ABUS System: Validation

Sample Matrix
- Preparation of Graphite/Epoxy and Al Joints

Samples Testing
- Testing of Graphite/Epoxy and Al/Al Joints

Boeing Sample Testing
- Testing of Al/Al and GrE/GrE Boeing Samples
**ABUS System: Validation Sample Matrix**

### Sample Matrix Description

<table>
<thead>
<tr>
<th>Type of Sample</th>
<th>Number of treated samples</th>
<th>Number of non-treated samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al/FM73/Al</td>
<td>20</td>
<td>5</td>
</tr>
<tr>
<td>GrE/FM73/GrE (quasi-isotropic multilayered)</td>
<td>20</td>
<td>5</td>
</tr>
</tbody>
</table>
Weak (treated) Lap-shear Joint

Mechanical Testing:

Failure load: 720 lb

Abus Scan

C- Scan

- Low average value of the effective stiffness
- Inhomogeneous pattern over the bond area
Results: Reference Lap-shear Joint

Mechanical Testing:

Failure load: 1100 lb

- High average value of the effective stiffness
- Homogeneous pattern over the bond area
INSPECTION RESULTS

Reference bond: mixed mode failure

![Reference bond image](image1)

**Abus Scan**

**C-Scan**

Poor bond: Interfacial mode failure

![Poor bond image](image2)

**Abus Scan**

**C-Scan**

ABUS Data Correlation to Strength

![Data correlation graphs](image3)

1.2
1.1
1.0
0.9
0.8

Normalized longitudinal modulus

0 10 20 30 40 50 60 70 80 90

Bond strength reduction (%)

0 10 20 30 40 50

Normalized effective longitudinal modulus

0 0.6 0.7 0.8 0.9 1.0 1.1 1.2

Bond strength reduction (%)
ABUS System Validation
System Testing: Composites samples

Testing of GrE/GrE

- Testing of samples from the Samples Matrix
- Correlation with mechanical tests

GrE/FM73/GrE

Normalized effective longitudinal modulus vs. Bond strength reduction (%)
ABUS System: Validation Boeing Sample Testing

Boeing Sample Testing

• A series of six Al/Al joint have been sent by Boeing. Two process were used to joint the bonds (standard and with additional fabric).
• The Abus system has been able to discriminate the two process.

Non-dimensional parameter $\frac{\omega h}{V_t}$

Reconstructed thickness

Bond with inserted fabric

Regular bond
Conclusion

The Angle Beam Ultrasonic Spectroscopy (ABUS) Scanning System for evaluation of adhesive bond integrity has been developed.

Features:
- Separation of thickness effect from bond quality effect.
- Ability to give a bond line quality factor.
- Ability to scan bond line quality over the bond area

Correlation between ultrasonic signatures measured with the ABUS method and the strength of the adhesive bond was obtained.

The ABUS System is capable of detecting weak bonds which are not detectable by C-Scan.
Characterization of Adhesive Bonds Integrity
Using Angle Beam Ultrasonic Spectroscopy

S. I. Rokhlin\textsuperscript{1,2}, L. Adler\textsuperscript{2}

1) The Ohio State University
2) Adler Consultants, Inc.
Outline

- Angle beam ultrasonic spectroscopy
- Assessment of environmental degradation of adhesive bonds
- Manufacturing of adhesive bonds with weak interfaces
- Characterization of adhesive bond integrity after manufacturing
**APPROACH:**

**Theoretical model:**

- An adhesive joint is considered having multilayered interphases including anisotropic porous aluminum oxide and weak boundary layers.

- A special ultrasonic goniometer is developed for experimental investigation.

- Spectrum of the obliquely reflected ultrasonic signals is basis for interphase property reconstruction.
Ultrasonic technique for evaluation of adhesive joints
Transfer Matrix Algorithm

Unknown transfer matrix $B_j$
(Layer properties)

$Z_N, h_l, h_{0l}, h_{0t}, \alpha_l, \alpha_t$

six nondimensional parameters

$(\lambda+2\mu), \mu, \rho, h, \alpha_l, \alpha_t$

six dimensional parameters

$R, T(\prod_{i=1}^{n} B_i)$ is function of the product of transfer matrices

$B_i$ ($i=1$ to $n$) is $[6 \times 6]$ transfer matrix for off plane orthotropic layer
Nondimensional Parameters

\[ Z_N = \frac{\rho V_\ell}{Z_1} \]
\[ \bar{h}_\ell = \omega_0 \frac{h}{V_\ell} \]
\[ \bar{h}_{\theta \ell} = \omega_0 \frac{h \cos \theta_\ell}{V_\ell} \]
\[ \bar{h}_{\theta t} = \omega_0 \frac{h \cos \theta_t}{V_t} \]
\[ \alpha_\ell \]
\[ \alpha_t \]

where:
\[ V_\ell = \sqrt{\frac{\lambda + 2\mu}{\rho}}, \]
\[ V_t = \sqrt{\frac{\mu}{\rho}}, \]
\[ \theta_\ell = \arcsin \left( \frac{V_0}{V_\ell} \sin \theta_0 \right), \]
\[ \theta_t = \arcsin \left( \frac{V_0}{V_t} \sin \theta_0 \right) \]
Objective

- Determine six parameters of the layer:

  \[(\lambda + 2\mu), \mu, h, \rho, \alpha_l \text{ and } \alpha_t\]

  using normal and oblique incident ultrasonic wave
Relation between Parameters

\[(\lambda + 2\mu) = \frac{Z_N Z_1}{\xi_0} \frac{\sqrt{\bar{h}_e^2 - \bar{h}_{\theta e}^2}}{\bar{h}_e^2},\]

\[\mu = \frac{Z_N Z_1}{\xi_0} \frac{\sqrt{\bar{h}_e^2 - \bar{h}_{\theta e}^2}}{\bar{h}_e^2 + \bar{h}_{\theta e}^2 - \bar{h}_{\theta e}^2},\]

\[\rho = \frac{Z_N Z_1}{\xi_0} \frac{\sqrt{\bar{h}_e^2 - \bar{h}_{\theta e}^2}}{\xi_0},\]

\[h = \frac{\sqrt{\bar{h}_e^2 - \bar{h}_{\theta e}^2}}{\xi_0\omega_0},\quad \text{where} \quad \xi_0 = \frac{\sin \theta_0}{V_0} \]
Strategy for Reconstruction

Step 1
Normal Incidence Data

\[ Z_N, \bar{h}_\ell, \alpha_\ell \]

Step 2
Oblique Incidence Data

\[ \bar{h}_\theta \ell, \bar{h}_\theta, \alpha_\ell \]

\[ \lambda + 2\mu, \mu, \rho, h, \alpha_\ell, \alpha_\ell \]
## Results of Inversion

<table>
<thead>
<tr>
<th></th>
<th>$\lambda + 2\mu$, GPa</th>
<th>$\mu$, GPa</th>
<th>$\rho$, g/cc</th>
<th>$h$, mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polystyrene film inside joint</td>
<td>4.46</td>
<td>0.844</td>
<td>1.07</td>
<td>0.141</td>
</tr>
<tr>
<td>Polystyrene film extracted</td>
<td>4.48</td>
<td>1.072</td>
<td>0.144</td>
<td></td>
</tr>
<tr>
<td>Heat treated polystyrene</td>
<td>4.69</td>
<td>0.828</td>
<td>1.072</td>
<td></td>
</tr>
</tbody>
</table>
Multi-ply Laminated Composite Sample

There is an excess of epoxy forming a thin bonding layer between the plies.

Ply thickness: app. 0.2 mm
Number of plies: 16
Plate thickness: 3.1 mm

Symmetry plane

[0/-45/90/45/-45/90/45]s laminate lay-up
Transfer Matrix Algorithm

\[ H_p = \sum_{i=1}^{n} k_i h_i \sim 0 / 1000 \]

\[ R, T(\prod_{i=1}^{n} B_i) \text{ is function of the product of transfer matrices} \]

\[ B_i (i=1 \text{ to } n) \text{ is } [6 \times 6] \text{ transfer matrix for off plane orthotropic layer} \]
Model for the composite joints
Multiply Composites Ultrasonic Measurements

\[ \alpha: \text{Incident angle} \]
\[ \theta: \text{Orientation angle (Angle between incident plane and plate axis)} \]

Investigate reflection and transmission characteristics vs. incident angle \( \alpha \) and orientation angle \( \theta \)
Normal Reflection Time Domain Signal from the Laminate

5 MHz

10 MHz

Reverberation signal can be used to find parameters of thin bonding layer between different plies.
Experimental Apparatus for Double Transmission Amplitude Measurement

DIGITAL OSCILLOSCOPE

PULSE/RECEIVER

FUNCTION GENERATOR

COMPUTER

DC MOTOR CONTROLLER

SAMPLE

T/R

BACK REFLECTOR

CYLINDER REFLECTOR
Double Through Transmission Characteristics at Orientation Angle $\theta = 25^\circ$ (Experiment)

2.25 MHz center frequency

![Graph showing amplitude vs. incident angle for different orientations.](image)

- **Orientation $\theta = 25^\circ$**
- **Amplitude vs. Incident Angle $\alpha$ (degree)**
  - $\alpha = 0^\circ$
  - $\alpha = 14^\circ$
  - $\alpha = 30^\circ$
  - $\alpha = 50^\circ$
  - $\alpha = 60^\circ$

![Waveform at different incident angles.](image)

- Waveform for $\alpha = 0^\circ$
- Waveform for $\alpha = 14^\circ$
- Waveform for $\alpha = 30^\circ$
- Waveform for $\alpha = 50^\circ$
- Waveform for $\alpha = 60^\circ$

**Amplitude**

**Waveform at different incident angle**
Amplitude of Double Transmission vs. Incidence Angle $\alpha$

Experiment

2.25 MHz center frequency pulse:

transducer \hspace{1cm} reflector

sample
Amplitude of Double Through Transmission Signals at Different Incidence and Orientation Angles

Double through transmission amplitude
- has 180° rotation symmetry
- has no reflection symmetry (θ and −θ)
Amplitude of Double Transmission vs. Incidence Angle $\alpha$

Experiment (5 MHz tone-burst)

Transmission amplitude depends strongly on frequency
OUTLINE

• STATEMENT OF THE PROBLEM

• SUMMARY OF THE EXPERIMENTAL RESULTS

• MODELS OF INTERFACE IN ADHESIVE JOINT:
  - "COMPOSITE" WEAK BOUNDARY LAYER
  - INTERFACIAL SPRINGS

• CONCLUSIONS
Layered structure of adhesive joints

Al oxide (0.1 μm)

Primer (5 μm)

Adhesive (100 μm)
Texture of Porous Anodic Oxide
EFFECT OF SURFACE PRETREATMENT ON THE PERFORMANCE OF ALUMINUM-ALLOY EPOXY JOINTS SUBJECTED TO ACCELERATED AGING IN WATER AT 50°C

Phosphoric-acid anodised pretreatment
Chromic-acid anodised
Chromic-acid etched
Grit-blasted
Degreased

(Epoxy/aluminium-alloy joints)

Time exposed to water at 50°C(h)
PROCEDURE FOR JOINT PREPARATION

• Surface cleaning in Alconox detergent solution

• Surface deoxidation
  (sodium dichromate + sulfuric acid + distilled water)

• Phosphoric acid anodization

• Priming of surface by BR-127 primer

• Bonding by FM-73 adhesive film

CONDITIONS FOR ACCELERATED AGING

• EXPOSURE MEDIUM:
  saturated solution of NaCl at 68°C

• LOAD:  800 or 1000 lb

Under these conditions the joints broke in 1-3 weeks
INITIAL FAILURE LOADS OF ALUMINUM SINGLE LAP ADHESIVE JOINT SAMPLES BONDED WITH FM-73 FILM ADHESIVE AND BR-127 PRIMER

![Graph showing failure loads for 1" and 3/4" wide samples.](image-url)
LOADING ARRANGEMENT FOR A SINGLE LAP ADHESIVE JOINT SAMPLE USING STRESS FIXTURE

APPLIED LOAD IS MEASURED AS DEFLECTION OF THE FIXTURE BY DIAL INDICATOR
LIFETIME OF SINGLE LAP SHEAR ADHESIVE JOINTS
AGED IN SATURATED NaCl SOLUTION
UNDER 1000 lb LOAD AT 70°C

Number of Failed Samples

Exposure Time, hours

12
10
8
6
4
2
0

0 ... 18 ... 24 ... 48 ... 48 - 96
Problem Statement:

- Adhesive joints can fail catastrophically in severe environments
- Residual strength and lifetime of joints is independent of initial joint strength

This shows the necessity of nondestructive evaluation of adhesive joints in service
Modes of failure in adhesive joints

Before degradation

After degradation

Interfacial mode of failure

Cohesive mode of failure

Frequency, MHz

Amplitude, dB

Before degradation

After degradation

Before degradation

After degradation
Two types of experiments:

Reflection minima shift vs. interfacial failure area

Reflection minima shift vs. time of degradation
Measurement of water layer thickness at the coating/Ge interface

Diagram:
- Organic coating
- GE IRE element
- Water
- Reflector
- ATR element holder
- Incoming IR beam

Graph:
- Thickness of water layer, nm vs. Time, min
- Alkyd
- Epoxy

Graph data:
- Thickness values range from 0 to 50 nm
- Time range from 0 to 6000 min
EXAMPLE OF THICKNESS MEASUREMENT FOR THE SAMPLE BEFORE DEGRADATION

$V_1 = 2.11 \text{ km/sec}$

Thicknes along center line measured by:

- ○○○○○ 50MHz transducer (time delay)
- □□□□□ 15MHz transducer (spectrum)
- △△△△△ micrometer

Distance along glue line, mm
RELATIVE THICKNESS CHANGE OF ADHESIVE LAYER DUE TO EXPOSURE IN NaCl SATURATED SOLUTION AT 68°C UNDER 800 LB LOAD

Change of thickness, %

Position on the joint, mm

-12 -9 -6 -3 0 3 6 9

120 hours
19 hours
2.5 hours
0 hours
after release
CONCLUSIONS:

i) Three factors were found to affect position of frequency minimum for obliquely reflected signal:
   • adhesive layer thickness increase (including creep)
   • bulk adhesive properties change
   • interface degradation

ii) Measurements at normal incidence on the joint are sensitive only to thickness and bulk property changes
   • this is a basis for separation of interfacial degradation effects from thickness and bulk property change effects

iii) Frequency-minimum shift at the edges of the joint is larger than predicted from the adhesive thickness change.
    The excess is due to interface degradation

iv) Frequency-minimum shift at the center of the joint is less than predicted from the adhesive thickness change.
    This may be attributed to stress redistribution and stress change at the joint center
NON-LINEAR PROPAGATION OF GUIDED ELASTIC WAVES

I. THEORETICAL ASPECTS

D C Price

CSIRO Telecommunications & Industrial Physics

November 1998
Non-linear wave equation:

\[ p_{ou} = \sum C_{ijklm} \frac{\partial u_k}{\partial x_j \partial x_m} \]

\[ + \sum M_{ijklmp} \frac{\partial u_k}{\partial x_j \partial x_m} \frac{\partial u_p}{\partial x_o} \]

\[ + \ldots \]

\[ M_{ijklmp} = C_{ijklmp} + C_{ijmn}s_{kp} + C_{ijnp}s_{ik} + C_{iklm}s_{ip} \]

**OR**

\[ p_{ou} = C_{ijklm} \frac{\partial u_k}{\partial x_j \partial x_m} = F_i(u) \]

\[ F_i(u) \] is non-linear driving force
Successive approx. approach

Put

\[ U = \Sigma U^{(i)} + \Sigma^2 U^{(ii)} + \ldots \]

\[ U^{(i)} \gg U^{(ii)} \gg \ldots \]

\( \lambda \) an arbitrary expansion parameter

Equating terms of equal powers in \( \lambda \)

\[ \rho_0 \ddot{U}_i^{(0)} - C_{ijklm} \frac{\partial^2 U_k^{(0)}}{\partial x_k \partial x_m} = 0 \]  \( (1) \)

\[ \rho_0 \ddot{U}_i^{(ii)} - C_{ijklm} \frac{\partial^2 U_k^{(ii)}}{\partial x_k \partial x_m} = F_i(\dot{U}^{(0)}) \]  \( (2) \)

\[ F_i(\dot{U}^{(0)}) = M_{ijklpq} \frac{\partial^2 U_k^{(0)}}{\partial x_k \partial x_m} \frac{\partial U_p^{(0)}}{\partial x_q} \]

\( F \) is driving force that depends only on linear waves \( U^{(0)} \)
Boundary conditions

- must be satisfied at all points at all times

.: continuity conditions must be true at F and 2F separately.

At 2F, stress at boundary includes:

- stress due to free harmonics (to be determined)
- stress due to driven harmonics
- second-order stress terms from linear waves

\[ T_{ij} = C_{ijklm} \frac{\partial u_k}{\partial x_m} + B_{ijklmpq} \frac{\partial u_k}{\partial x_m} \frac{\partial u_p}{\partial x_q} + \ldots \]

Summary
Summary

incident wave

Boundary cond. at fundamental $f$

Linear waves, Driven harmonics

Boundary cond. at harmonic $2f$

Freely propagating harmonics
Polystyrene/Al Interface
LINEAR SOLUTION
TRANSVERSE INCIDENT WAVE

Reflected waves

Transmitted waves

\[ \frac{U}{U_0} \text{ (magnitude)} \]

\[ \theta_i \text{ (deg.)} \]

\[ \theta_i \text{ (deg.)} \]
Polystyrene / AL Interface - 2F Solution
Transverse Incident Wave

Reflected waves

Transmitted waves
Polystyrene / AL Interface

Reflected waves Nonlinearity in Polystyrene

\[
\frac{U}{U_{0}^2 \omega} \text{ (magnitude)}
\]

\[
0 \quad 10 \quad 20 \quad 30 \quad 40 \quad 50 \quad 60 \quad 70 \quad 80 \quad 90
\]

\[
\theta_i \text{ (deg.)}
\]

Transmitted waves

\[
\frac{U}{U_{0}^2 \omega} \text{ (magnitude)}
\]

\[
0 \quad 10 \quad 20 \quad 30 \quad 40 \quad 50 \quad 60 \quad 70 \quad 80 \quad 90
\]

\[
\theta_i \text{ (deg.)}
\]
Aluminium plate immersed in water, 2.5 MHz incident wave.

Reflected waves (in water above plate)

Transmitted waves (in water below plate)
Aluminium plate immersed in water, free harmonics in plate.

Internal plate waves, \( f = 2.5 \text{ MHz} \)

![Graph showing internal plate waves, \( f = 2.5 \text{ MHz} \)]

Internal plate waves, \( f = 5 \text{ MHz} \)

![Graph showing internal plate waves, \( f = 5 \text{ MHz} \)]
Aluminium plate immersed in water, 5 MHz incident wave.

Reflected waves (in water above plate)

Transmitted waves (in water below plate)
Al bonded plate immersed in water, 2.5 MHz incident wave.

Reflected waves (in water above plate)

Transmitted waves (in water below plate)
Bonded Al plate immersed in water, 5 MHz incident wave.

Reflected waves (in water above plate)

Transmitted waves (in water below plate)
AI bonded plate immersed in water, 2.5 MHz incident wave.
Effect of increased (x2) non-linearity of adhesive
Reflected waves (in water above plate)

Transmitted waves (in water below plate)
Al bonded plate immersed in water, 5 MHz incident wave.
Effect of increased (x2) non-linearity of adhesive

Reflected waves (in water above plate)

Transmitted waves (in water below plate)
Ultrasonics, microwaves, optically stimulated electron emission (OSEE), and computational chemistry approaches have shown relevance to bond strength determination. Nonlinear ultrasonic nondestructive evaluation methods, however, have shown the most effectiveness over other methods on adhesive bond analysis. Correlation to changes in higher order material properties due to microstructural changes using nonlinear ultrasonics has been shown related to bond strength. Nonlinear ultrasonic energy is an order of magnitude more sensitive than linear ultrasonic to these material parameter changes and to acoustic velocity changes caused by the acoustoelectric effect when a bond is prestressed. Signal correlations between non-linear ultrasonic measurements and initialization of bond failures have been measured. This paper reviews bond strength research efforts presented by university and industry experts at the Second Annual Symposium for Nondestructive Evaluation of Bond Strength organized by the NDE Sciences Branch at NASA Langley in November 1998.