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

Compiled by Mark J. Roberts U.S. Army Research Laboratory Vehicle Technology Directorate Langley Research Center, Hampton, Virginia

> 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

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PREFACE

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 Assessement 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 Perey, NASA, and "Computational Chemsitry 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.

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The statement of the second of the second

Bond Strength Program

Dr. Mark J. Roberts

Friday, 6 November 1998

TECHNOLOGY CE	

Schedule



NDE BRANCH WELCOME

NTER

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

Characterization of Adhesive Bonds Using Nonlinear Ultrasound 10:30-11:15 Georgia Technology Institute: Dr. Jianmin Qu -

Assessment of Adhesive Bond Deterioration by Detection of Nonlinear 11:15-11:45 Northwestern University: Dr. Jan Achenbach - An Effects

11:45-1:15 LUNCH



Schedule



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

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2:45-3:00 COFFEE BREAK

3:00-4:00 GENERAL DISCUSSION

4:00 ADJOURN

Sign-IN Sheet

Name	Phone	Org	email
Jan D. Achenha	ch (247)491-59	27 Northwe	Jen D. achenbach Comes
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NESB SUPPORT OF PROGRAMS

OFFICE OF SAFETY AND MISSION ASSURANCE (AGENCY LEAD)

SOLID ROCKET COMPOSITE NOZZLE INTEGRITY

SSME COMBUSTER 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

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HIGH SPEED RESEARCH

NDE FOR DURABILITY

IN-SITU MEASUREMENTS (DENSITY, STIFFNESS, AND FLAW LOACTION)

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

BASE: QUANTITATIVE NDE OF BOND STRENGTH, MORPHING STRUCTURAL HEALTH MONITORING & SHUTTLE UPGRADES

Structures & Materials Research Group NASA Langley Research Center





Program Issues



Results of Successful Program

- Could eliminate or reduce rivet / fastening technology 1
- Production of higher strength bonds
- Higher confidence in bonded structures
- Measurement technique to quantitatively measure strength and assign quality I
- Reduce operational downtime of aircraft significantly I
- Prototype instrument developed would have potential in any industry requiring "on the spot" bond analysis 1



Program Issues



LARVENING AS APPEND IN .

- Customer Needs & Requirements
- Aircraft operational safety
- General structural integrity (Composite Structures) I
 - 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 I

APPROACH

- Industry / NASA / University collaboration into third year of work
 - problem solution: Ultrasonics (Overall), Microwaves (Moisture) Selected NDE technologies best suited for the bond strength
 - Perform strength measurements using selected NDE methods 1
 - Begin system prototype design of bond strength instrument I



Bond Deterioration Mechanisms







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)





- ultrasonics best method for bond strength analysis: Johns University grantees unanimously conclude that nonlinear 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)

CENTER

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

	FY Goals & Milestones : FY99-FY01
	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
16	 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 stream.
	 Correlate to expected strength and quality level for selected 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)
- changes in cohesive (volume) and adhesive (surface) properties Consider numerical modeling techniques to look at effects of 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

	reliable	FY 01	ω)	t on Al 2024						insfer	
-ESTONES	th monitoring for ice habitat	FY 00	6 7		ique sensor concen	nd strength	4		ory	ient system	or for technology tra	chnology transfer
ROGRAM MIL	safety, and healt rehicles, and spa	FY 99	5		system to test fati	ems for NDE of bor	probe with AI 202	gth approach	ue/integrated histo	strength measurem	load history senso	E technique for tec
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Microwaves for Bondline NDE

Dr. Mark J. Roberts

Friday, 6 November 1998



Microwave NDE Technology



VEHICLE TECHNOLOGY CENTER

Advantages

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

Benefits

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- 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



Band	Frequency (GHz)	Wavelength (cm)
۵.	0.23 - 1	130 - 30
	1-2	30 - 15
S	2 - 4	15 - 7.5
ပ	4 - 8.2	7.5 - 3.66
×	8.2 - 12.4	3.66 - 2.42
Ku	12.4 - 18	2.42 - 1.67
×	18 - 26.5	1.67 - 1.13
Ka	26.5 - 40	1.13 - 0.74
ım-Waves	40 - 300	0.75 - 0.1





Apparatus



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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) 1
 - 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.



MICROWAVE IMAGING



- 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.



BISBORD RESULTS

amntl





30	Microwaves are totally reflective to moisture & water "Ronce area 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 Cracks / small resolution flaws will have increased probability of detection if containing moisture Plans for FY99 - erform measurements to look at moisture between two composite layers which are unjoined - efform measure composite joints with the following conditions - water of a solution flaws with the following conditions - water of the since before joint of a solution for a bound on a solution of the since - erform analytical and/or numerical approach on abour conditions - Perform analytical and/or numerical approach on abour 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

- in these properties due to degradation stem from chemical and electrical properties of the bond layer. Also, changes stimulii is determined by the molecular level mechanical The macroscopic response of a bond layer to external changes in the bond layer.
- mechanical, and chemical properties of a adhesive bonds at followed by investigations of the effects of environmental effects on the chemistry and properties of the bond layer. This investigation is intended to determine the electrical the molecular level. The initial determinations will be

Background and Goals (Continued)

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



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Methods

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

Approximate, Semi-Empirical

- Parameterization Appropriate for Models?

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- Model Capable of Treating Hydrogen Bonds?
 - Information on Core Energy Levels?

Molecular Mechanics

- Parameterization Adequate?
- Electronic, Mechanical Properties?

Results for Monomers

Vib 349/cm		346 * 345	363*	233				
Dipole 2.04 D	1.98	1.89 2.02	2.35	6.19		2.62	3.22	3.39
Struct. standard								
Method scf-dzp	scf-min PM3	AMI	MNDO	scf-dzp	scf-min	PM3	AM1	MNDO
Mol PMMA				Alo2H				

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Result	s for PMN	[A-A]O ₂ H (Complex
Method	$D_0^{\mathfrak{c}}$	Dipole M	R =0h
SCF-DZP (uncvgd) SCF-Min	(KJ/mol) 24.7	9.40 9.40	Angstroms 1.89
AM1	20.5	5.81	2.11
PM3	7.8	4.28	2.93
MNDO	17.5	5.24	3.33







Core Electron Energy Shifts

- computed for both PMMA oxygens and the Carbon 1s attached to the carbonyl O atom. increases of approximately 0.8 eV were For this configuration of the complex,
 - experiment (ie binding energy increases) This in qualitative agreement with the but the agreement is not quantitative.

دیکہ ہو۔ اوری مسلس







Gr GWW School of Mechanical Engineering
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

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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.
- microstructural changes caused by the degradation of bond strength. * To develop ultrasonic nondestructive methods to measure the
- on the fundamental structure-property-performance relationships of the * To predict remaining bond strength from ultrasonic measurement based constituents and their interfaces.



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PZT/PZT

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Samples with Different Curing States

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The Material Properties

Material	p(kg/cm ³)	E(GPa)	>
Al2024	2.78	73.0	0.35
AF-163-2K	1.21	1.11	0.34

Differenct Curing Conditions

B4	06	60	0.34	3.55	4.1
B3	82	120	0.34	3.55	3.3
B 2	82	60	0.34	3.51	4.0
B1	121	06	0.34	3.54	35
Sample Number	Curing Temperature (°C)	Curing Time (min.)	Curing Pressure (MPa)	Total Thickness (mm)	Shear Strength (MPa)





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Generation of Higher Order Harmonics

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Wave Equation

 $\frac{1}{\rho} \frac{\partial \sigma}{\partial \varepsilon} = \frac{\partial^2 u}{\partial t^2}$

Stress-Strain Relation (material nonlinearity)

 $\sigma = Ef(\varepsilon)$

 $\frac{\partial u}{\partial x} = \frac{\partial u}{\partial x}$

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

GWW School of Mechanical Engineering	umple	$\sigma = Ef(\varepsilon)$ $A_1 \sin(kx - \omega t)$	A sin($kx - \omega t$) A sin($kx - \omega t$) A sin($kx - 2\omega t$) A sin($kx - 2\omega t$) A sin($kx - 2\omega t$)	For $h = 320 \mu m$ $c = 6410 m/s$ $\omega = 2\pi \times 2 Hz$	$\frac{A_2}{A_1} \approx 1.5 \times 10^{-4} \gamma \frac{A_1}{\mu m}$
G. T	Exa	$f(\varepsilon) = \varepsilon(1 - 0.5\gamma\varepsilon)$ $(\times 10^{-3}) \frac{\sigma}{E}$ $4 + \frac{1}{2}$	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$A_2 = \frac{\gamma}{8}k^2 x A_1^2 = \frac{\gamma}{8c^2}\omega^2 x A_1^2$	$\beta = \frac{8A_2}{k^2hA_1^2} = \gamma$ Nonlinear parameter



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### **A Simplistic Model**

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$$f(x-ct) \qquad g(x-ct)$$

**Interface Conditions** 

$$E_{s} = \begin{cases} E_{s}^{t} & \text{when } \sigma(t) > 0 \\ \\ E_{s}^{c} & \text{when } \sigma(t) < 0 \end{cases}$$

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

 $\sigma(t) = \sigma(0^+, t) = \sigma(0^-, t)$ 

$$\eta_t = \frac{E_s^t}{E} \qquad \eta_c = \frac{E_s^c}{E}$$

$$s_s = Adhesive stiffness$$

E = Adherend stiffness

$$f_s = Adhesive stiffness$$

$$s_{s}^{2}$$
 = Adnesive stittness

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

 $V(t) = u(0^+, t) - u(0^-, t)$ 

**Solution** 

Ъ Т	<b>Example</b> $f(x-ct) = f(x-ct) = \frac{1}{2}$	g(x - ct) GWW School of Mechanical Eng
f(x-ct)=Ac	os[k(x-ct)]	$g(x-ct) = f(x-ct) + \frac{1}{2}V($
where	·	
$\left[ 2Asi \right]$	$\ln \varphi_c [\sin(\omega t_n + \varphi_c) e^{-\xi_c(t-t_n)}]$	$(n^{1} - \sin(\omega t + \varphi_{c})), n = 0, 2, 4$
$V(0) = \begin{cases} 2As \end{cases}$	$\sin \varphi_t [\sin(\omega t_n + \varphi_t) e^{-\xi_t(t-t_n)}]$	$(n)^{n} - \sin(\omega t + \varphi_{t})], \ n = 1, 3, 5$
$\xi = \eta \frac{c}{h}$	$\varphi = \sin^{-1}\left(\frac{\omega}{\sqrt{\xi^2 + \omega^2}}\right)$	$V(t_n)=0 \qquad t_0=0$

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$$h = 100 \,\mu \text{m}$$
  $c = 6410 \,m/s$   $\omega = 2 \,\text{Hz}$ 




# Setup for Guided Wave Measurement

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### ASSESSMENT OF ADHESIVE BOND DETERIORATION BY DETECTION OF NONLINEAR EFFECTS

### J. D. ACHENBACH PRINCIPAL INVESTIGATOR

### ZHENZENG TANG RESEARCH ASSISTANT

### CENTER FOR QUALITY ENGINEERING AND FAILURE PREVENTION 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.

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



# 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

.



# **SPECIMEN**



- Adhesive (connection)
   adhesive (testing layer)
   Al block (adherend 2)
   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.

$$\frac{d\tau}{d\Delta} \approx \frac{\sigma}{\delta} = \beta$$



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### 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.



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

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

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

Strain-Temperature Correspondence Relation

$$h(T) = f'(\epsilon) \tag{9}$$

# **Preliminary Results** Temperature Dependence of velocity for several materials 2.8 2.6 2.4 Velocity (kn/s) 1.8 1.6 1.4 25 30 35 40 Temperature (° C) 45 50 Temperature dependence of velocity for various materials. Water (solid), FM73 (dashed), DER Epoxy (dashdot), AB Epoxy(solid)



Complete recovery of ultrasonic signal after one cycle of heating of sample #3.  $22^{0}C$  before heating (solid line),  $22^{0}C$  after cooling (dashed line)

# A Simple Model

**Quadratic Nonlinear Term** 

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

Definition of Temperature-Velocity Coefficient  $\alpha_c$ 

$$\frac{dc(T)}{c_0 dT} = -\alpha_c \tag{12}$$

**Strain-Temperature Correspondence Relation** 

$$\frac{\epsilon}{\epsilon_0} = 2\alpha_c \Delta T - (\alpha_c^2 + 6\alpha_c\beta)\Delta T^2 + 3\alpha_c^2\beta\Delta T^3 \quad (13)$$



(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.





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The Johns Hopkins University

**Department of Materials Science and Engineering** 

Acknowledgments:

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November 6, 1998



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Investigate various Cure Conditions of Adhesive Bonds using Nonlinear Ultrasonic Methods

with Water Coupling

► Normal Incidence

► Oblique Incidence

► Wave Mixing

# Samples

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- 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



A. A.



SFLTT_5.CDR



**Bond Sample versus Single Aluminum Plate** 







(V) sinomation of 2nd Harmonic (V)





# **Contact Measurements**

ي يبيد بدين محمد محمد

- To verify existence of and differences in both 2nd and 3rd
   Harmonics in given Bond system
- Transmitter: 5 MHz LiNbO₃ bonded with SALOL
- <u>Receiver:</u> 10 MHz broadband (commercial contact)
- Samples: 0.25" Aluminum Substrates

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









# 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

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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
- line, allowing discrimination of kissing or poor bond from The oblique wave introduces shear stress on the bond good bonds
- The normally incident wave is used to decouple the effect of the bond line thickness

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Layer/Substrate sonic Signature	<ul> <li>Oblique incident ultrasonic waves are sensitive to adhesive / adherent interface quality</li> <li>Interface modeling allows us to select incidence angle which is sensitive to interface quality</li> </ul>	Adler Consultants Inc.
Effect of Imperfec Interface on Ultra	Frequency shift as function of incident angle on composite	σ,





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ABUS System Hardware **Overall Design** 



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ABUS System Hardware Mechanics	- 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 Square Rail Positioning Table 23.6" travel, Max. travel speed: 1'/s Precision Ground Ball Screw	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 <b>Microstep Motors</b> : 300 oz-in torque, 45000 step per revolution - American Precision Industries Low EMI <b>microstepping drive and power supply</b> - Parker Compumotor PC Based <b>Step Motor Controller</b>
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ABUS System: Software Hardware Control

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· Reference points control Scan Control Scanning speed Scanning step Scanning size ABUS System: Software User Interface Design System operation control 2 Channels (Normal and Oblique) Time domain signal display Gates position control A/D Control 3 gates per channel: (start and width) Trigger control Sampling rate Gates display Adic Consultant Inc.





Adler Consultant Inc. Channel 2: Oblique incidence ABUS System: Software Main gate (Storage) Front surface echo gate User Interface. Interface echo gate <u>Channel 1: Normal incidence</u>



Adler Consultant Inc. ABUS System: Validation _____ Testing of Al/Al and GrE/GrE Boeing Samples Preparation of Graphite/Epoxy and Al Joints Testing of Graphite/Epoxy and Al/Al Joints **Boeing Sample Testing** Samples Testing Sample Matrix

<del>3</del>3

ABUS System: Validation Sample Matrix

	ated Number of non- treated samples	S	ß	
ion	Number of tre samples	20	20	
Sample Matrix Descript	Type of Sample	Al/FM73/ Al	GrE/FM73/GrE (quasi-isotropic multilayered)	

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Low average value of the effective stiffness
 Inhomogeneous pattern over the bond area



Weak (treated) Lap-shear Joint



### ANGLE BEAM ULTRASONIC SEEL TROSCOPY

### **INSPECTION RESULTS**

Reference bond: mixed mode failure



Abus Scan

C-Scan

Poor bond: Interfacial mode failure



Failure load, 1100





C-Scan

ABUS Data Correlation to Strength

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he Angle Beam Ultrasonic Spectroscopy (ABUS) Scanning System	<ul> <li>eatures:</li> <li>- Separation of thickness effect from bond quality effect.</li> <li>- Ability to give a bond line quality factor.</li> <li>- Ability to scan bond line quality over the bond area</li> </ul>	orrelation between ultrasonic signatures measured with the ABUS ethod and the strength of the adhesive bond was obtained.	he ABUS System is capable of detecting weak bonds which are not etectable by C-Scan.	
The Angle Beam Ultrasonic Spectroscopy (ABUS) for evaluation of adhesive bond integrity has been	Features: - Separation of thickness effect from bond quali - Ability to give a bond line quality factor. - Ability to scan bond line quality over the bond	Correlation between ultrasonic signatures measure method and the strength of the adhesive bond was	The ABUS System is capable of detecting weak bo detectable by C-Scan.	

Conclusion

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Characterization of Adhesive Bonds Integrity Using Angle Beam Ultrasonic Spectroscopy

## S. I. Rokhlin^{1,2}, L. Adler²

The Ohio State University
 Adler Consultants, Inc.

57-33

### 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.  $4^2$ 



- A special ultrasonic goniometer is developed for experimental investigation.



- Spectrum of the obliquely reflected ultrasonic signals is basis

for interphase property reconstruction.






Ohio State University QNDE-96, July 31, 1996



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Ohio State University ONDE-96. July 31. 1996

Objective

where  $\xi_0 =$  $(\lambda + 2\mu) = \frac{Z_N Z_1}{N} \sqrt{h_\ell^2 - h_{\theta\ell}^2}$ **Relation between Parameters**  $= \frac{Z_N Z_1}{\xi_0} \frac{\sqrt{h_e^2 - h_{\theta e}^2}}{h_e^2 + h_{\theta}^2 - h_{\theta e}^2},$  $-\overline{\bar{h}_{ heta \ell}^2} \xi_0,$ ny  $\overline{h}_{\ell}^2 - \overline{h}_{\theta\ell}^2$ 5 1 0 0 1 1 1 0 0 <u>h</u>2 ヨゴ П О  $= \eta$ 

Ohio State University QNDE-96, July 31, 1996

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Ohio State University ONDE-96.-Julv 31. 1996





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Results of Inversion

-				
	$\lambda + 2\mu$ , GPa	μ, GPa	p, g/cc	h, mm
Polystyrene film	4.46	0.844	1.07	0.141
inside joint				
Polystyrene film	4.48		1.072	0.144
extracted			1 1 1 2 2 2 1 2 1 2	
Heat treated	4.69	0.828	1.072	
polystyrene				

Ohio State University QNDE-96, July 31, 1996

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Multi-ply Composites Ultrasonic Measurements



 $\theta$ : Orientation angle (Angle between incident plane and plate axis)

Investigate reflection and transmission characteristics

vs. incident angle  $\alpha$  and orientation angle  $\theta$ 







ncidence Angle $\alpha$	2.25 MHz center frequency pulse: $\Delta = \alpha$	transducer reflector
Transmission vs. Ir Experiment	Amplitude (mv) 1200 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1000 1	amplitude (mv) 1200 1200 1000 1200 1200 1000 1200 0 1200 0 0 0 0 0 0 0 0 0 0 0 0
Amplitude of Double	Amplitude (mv) 1200 1200 $200 + 000$ orientation $\theta = 0^{\circ}$ 200 + 200 + 000 Incident Angle $\alpha$ (degree)	1200 1000 600 600 200 0 200 0 0 0 200 0 0 0 0 0 0 0





Double through transmission amplitude has 180° rotation symmetry

Fiber direction of the first ply



1:

#### OUTLINE

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• STATEMENT OF THE PROBLEM

سيمتديد سيستعرضات المييين بالرديات وسنا

• SUMMARY OF THE EXPERIMENTAL RESULTS

• MODELS OF INTERFACE IN ADHESIVE JOINT:

### - "COMPOSITE" WEAK BOUNDARY LAYER

- INTERFACIAL SPRINGS

• CONCLUSIONS





Texture of Porous Anodic Oxide

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# EFFECT OF SURFACE PRETREATMENT ON THE PERFORMANCE OF ALUMINUM-ALLOY EPOXY JOINTS SUBJECTED TO ACCELERATED AGING IN WATER AT 50°C



#### 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



### 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 Ib LOAD AT 70°C



# 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







### EXAMPLE OF THICKNESS MEASUREMENT FOR THE SAMPLE BEFORE DEGRADATION



### RELATIVE THICKNESS CHANGE OF ADHESIVE LAYER DUE TO EXPOSURE IN NaCI SATURATED SOLUTION AT 68°C UNDER 800 LB LOAD



#### 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

### BOEING / CSIRO JOINT RESEARCH PROJECT

#### NDT OF BONDED STRUCTURES

#### Report No. 16

### NON-LINEAR PROPAGATION OF GUIDED ELASTIC WAVES

### I. THEORETICAL ASPECTS

D C Price

CSIRO Telecommunications & Industrial Physics

November 1998

Non-thear wave equation:  $C_{o}\ddot{u}_{i} = \sum_{ijkm} \frac{\lambda^{2} u_{k}}{\lambda x_{i} \lambda x_{m}}$ + Z Mijkmpg Juk Juk Jup Mijkmpg = Cijkmpg + Cijmg Sky + Cjupg Sik + Cikny Sip OR  $P_{o}\ddot{u}_{i} = C_{ijkm} \frac{\delta \ddot{u}_{k}}{\partial c_{j} \partial x_{m}} = F_{i}(\underline{u})$ F(y) is non-linear driving force

Successive approx, approach

Put  

$$\underline{U} = \underline{\angle U}^{(1)} + \underline{\angle U}^{(2)} + \dots$$
  
 $\underline{U}^{(1)} \gg \underline{U}^{(2)} \gg \dots$   
 $\underline{U}^{(1)} \gg \underline{U}^{(2)} \gg \underline{U}^{(2)}$   
Equating terms of equal powers in  $d$   
 $\overline{P} = \overline{U}_{i}^{(1)} - C_{ijkm} \frac{\lambda^{2} U_{k}^{(1)}}{\lambda x_{k} \lambda x_{m}} = 0$  (1)  
 $P_{e} \overline{U}_{i}^{(2)} - C_{ijkm} \frac{\lambda^{2} U_{k}^{(2)}}{\lambda x_{k} \lambda x_{m}} = \overline{F}_{i} (\underline{U}^{(1)})$  (2)  
 $\overline{F}_{i} (\underline{U}^{(1)}) = M_{ijkmpq} \frac{\lambda^{2} U_{k}^{(1)}}{\lambda x_{j} \lambda x_{m}} \cdot \frac{\lambda U_{k}^{(2)}}{\lambda x_{j}}$   
 $\overline{F}$  is driving force that depends only on  
linear wayes  $U^{(1)}$ 

### 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 viewes

$$T_{ij} = C_{ijkm} \frac{\partial U_k}{\partial x_m} + B_{ijkmpq} \frac{\partial U_k}{\partial x_m} \frac{\partial U_p}{\partial x_q} + \cdots$$
Umma

incident wasne Boundary cond's at fundamental Linear woves, Driven harmonics Boundary coudit at harmonic 2F Freely propagating harmonics



















Aluminium plate immersed in water, 5 MHz incident wave.





Bonded AI plate immersed in water, 5 MHz incident wave.





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Ultrasonics, microwaves, o	ptically stimulated electron e	mission (OSEE), and	computational chemistry
approaches have shown rel	evance to bond strength deter	mination. Nonlinear	ultrasonic nondestructive
evaluation methods, howev	ver, have shown the most effe	ctiveness over other	methods on adhesive bond analysis.
Correlation to changes in h	igher order material propertie	es due to microstructu	iral changes using nonlinear
ultrasonics has been shown	related to bond strength. No	nlinear ultrasonic ene	rgy is an order of magnitude more
sensitive than linear ultrasc	ound to these material parame	eter changes and to ac	coustic velocity changes caused by
the acoustoeleastic effect v	when a bond is prestressed. Si	gnal correlations betw	ween non-linear ultrasonic
measurements and initializ	ation of bond failures have be	een measured. This p	aper reviews bond strength research
offorts presented by univer	sity and industry experts at the	ne Second Annual Sy	mposium for Nondestructive
Evaluation of Rond Streng	th organized by the NDE Sci	ences Branch at NAS	A Langley in November 1998.
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