



Protecting People and the Environment

NDE of Novel Materials (CFRP)

Muthu Elen, Nicholas Conway, Aaron Diaz Pacific Northwest National Laboratory

Summary Deliverable through FY24

Prepared for the U.S. Nuclear Regulatory Commission Office of Nuclear Regulatory Research Under Contract DE-AC05-76RL01830 Master Interagency Agreement: A2307-031-089-048662 Task Order Number: 31310024S0062

NRC COR: Carol Nove



PNNL is operated by Battelle for the U.S. Department of Energy



PNNL-36932

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor Battelle Memorial Institute, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or Battelle Memorial Institute. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

PACIFIC NORTHWEST NATIONAL LABORATORY operated by BATTELLE for the UNITED STATES DEPARTMENT OF ENERGY under Contract DE-AC05-76RL01830

Printed in the United States of America

Available to DOE and DOE contractors from the Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831-0062 <u>www.osti.gov</u> ph: (865) 576-8401 fox: (865) 576-5728 email: reports@osti.gov

Available to the public from the National Technical Information Service 5301 Shawnee Rd., Alexandria, VA 22312 ph: (800) 553-NTIS (6847) or (703) 605-6000 email: info@ntis.gov Online ordering: http://www.ntis.gov



Abbreviations

- ASME American Society of Mechanical Engineers
- ATT automated tap testing
- AWWA American Water Works Association
- CC code case
- CFRP carbon fiber reinforced polymer
- DMA dynamic mechanical analysis
- DRS dynamic response spectroscopy
- DSC differential scanning calorimetry
- EPRI Electric Power Research Institute
- FAA Federal Aviation Administration
- FEA finite element analysis

- GFRP glass fiber reinforced polymer
- MTT manual tap testing
- NDE nondestructive evaluation
- NPP nuclear power plant
- NRC Nuclear Regulatory Commission
- PCC Post Construction Committee
- PCCP prestressed concrete cylinder pipe
- PEC pulsed eddy current
- PNNL Pacific Northwest National Laboratory
- Tg glass transition temperature
- TIE technical information exchange
- UT ultrasonic testing



- Substrate The original metallic component to be repaired/modified (typically the base pipe)
- Fiber One or more filaments in an ordered assemblage; acts as a primary load-carrying member in a composite
- Matrix / Resin / Epoxy Material in which reinforcing fiber of a composite is embedded. A thermosetting polymer containing one or more epoxide or oxirane groups, curable by reaction with amines or alcohols. Does not include fillers or thickening agents. It is a two-part mixture
- Fabric A material constructed of interlaced yarns, fibers, or filaments. May be unidirectional or bi-directional or other ٠ forms depending on the fiber orientation
- Ply or Lamina or Laminae Fabric (CFRP or GFRP) when saturated with epoxy .
- Composite Thermoset plastic (polymer) that is reinforced by fibers, matrix, also known as composite laminate
- CFRP Repair or Composite Repair The complete composite system, consisting of primers, lamina, epoxy, putty, and top coatings installed in accordance with defined laminate architecture
- Delamination A flaw caused by failure of adhesion between layers of composite or between the composite and substrate •
- Putty or Thickened Epoxy A mixture of epoxy and prescribed thickening agent (e.g., fume silica) in the appropriate ratio ٠ that provides a smooth surface for the application of the CFRP laminate
- NDE NDE stands for nondestructive examination or nondestructive evaluation, a process that examines the condition of • a material or component without damaging it. NDE is also known as nondestructive testing (NDT) or nondestructive inspection (NDI). The process is also referred to as the NDE method or NDE technique



- Introduction
- Summary of Literature
- Lessons Learned from EPRI CFRP TIE
- Survey from Industry and Its Results
- CFRP Mockup Design and Fabrication
- NDE of CFRP Mockups
- Conclusion
- Potential Follow-on Work

4



NDE of CFRP Materials

Background

CFRP composites have not been used for nuclear **safety-related applications** until recently. In 2019, the ASME Boiler and Pressure Vessel Code Committee approved a new CC N-871 for internal repairs of Class 2 and 3 safety-related piping using CFRP for Service Levels A, B, C, and D for a service life of 50 years. The NRC did not review N-871 for inclusion in the Code Case Regulatory Guides.

Objective

To evaluate the capabilities and limitations of NDE methods for examining the CFRP repairs in commercial NPPs.



PNNL Task Plan for Mockup Fabrication

Identify guidance and best practices for qualification mockup fabrication as per ASME CC N-871-2 / PCC-2

Fabricate mockups that include representative flaws, varying substrate and laminate thicknesses, curvature, using multiple vendors



Assure mockups are representative of actual field applications



PNNL Task Plan for NDE Analysis

Assess various commercially available NDE methods to evaluate capabilities and limitations for detecting and characterizing flaws Assess whether tap testing may be used as a screening tool to find flaws of interest so that more sophisticated techniques can be used to characterize flaws and provide a permanent record



CFRP Repairs in Nuclear Power Plants

- CFRP repairs have been performed in NPPs for several non-safety-related piping systems
- Examples:
 - A) Repair and strengthening of 700 linear feet of 10-ft-diameter PCCP concrete pipe with CFRP at San Juan Generating Station in Farmington, NM, performed in 2007 by QuakeWrap Inc.
 - B) 57 linear feet of 144-in.-diameter PCCP pipeline repairs at Hope Creek Nuclear Generating Station, NJ, by Structural Technologies
 - C) 84-in.-diameter cement mortar lined steel piping at Brunswick NPP, Southport, NC, by Fyfe Company and Fibrwrap[®] Construction







*A) Quakewrap, "Repair & Strengthening of PCCP Concrete Pipe with Carbon FRP." https://quakewrap.com/project_sheets/Repair%20and%20Strengthening%20of%20PCCP%20Concrete%20Pipe%20with%20Carbon%20FRP.pdf *B) Structural Technologies, "Pipeline Repairs at Hope Creek Nuclear Generating Station." "https://www.structural.net/case-studies/pipeline-repairs-at-hope-creek-nuclear-generating-station/ *C) Gerard, Trevor J., and Tomas T. Jimenez. "Brunswick Nuclear Plant Circulating Water Piping Discharge Headers Pressure Barrier Replacement Using Advanced Fiber Reinforcement Polymer (FRP) Systems." (2015). 23rd Conference on Structural Mechanics in Reactor Technology Manchester, United Kingdom - August 10-14, 2015.





CFRP Repairs in Nuclear Power Plants (cont.)

- D) Advanced FRP systems repaired a 54-in. cooling water gate valve at the nuclear facility on PCCP piping
- E) QuakeWrap repaired 9-ft-diameter PCCP piping, corroded by prestressing cables at a major NPP in September 1999
- F) 144-in. circulating water PCCP pipeline at Fermi 2, Monroe, MI, was repaired by Simpson Gumpertz & Heger (SGH)
- Several other CFRP installers have repaired piping in various NPPs for nonsafety-related applications
- Recently, NRC received several relief requests for safety-related applications (ML16355A346, ML16320A523, ML22013A394, ML20218A672, ML20114E275, and ML19274C393)
- Hence, it is vital to study the reliability of NDE methods for inspecting these **CFRP** repairs in NPPs





Defects in Composites

The most common defects during the manufacturing process and service life of fiber reinforced composites fabricated by hand-layup process are delamination, debonding, matrix cracks, wrinkles, resin-rich areas, voids, porosity, and fiber breakage.







NDE Mockups in Literature

- Mockups are primarily needed for performance demonstration/qualification purposes; however, prior to being able to qualify NDE techniques and personnel, mockups will be used to identify and develop best practices for NDE
- Sandia National Laboratories (Sandia) developed a report identifying recommendations for mockups (from FAA Study, Report: DOT/FAA/TC-15/63):
 - Vary substrate and laminate thickness
 - Include disbonds between substrate and composite layers and introduce interply delaminations
 - Use multiple suppliers to create mockups according to identical specifications
 - Ensure laminates reflect real-world repair scenarios

Criteria for FAA Test Specimens (Relevant to Nuclear CFRP Repair):

- Maintain at least 2-in. separation between flaws to avoid signal interference
- Introduce closely clustered flaws to test the ability of NDE methods to define flaw boundaries
- Place flaws at least 1.5 in. from specimen edges
- Use minimum flaw size at half the detection target •
- Distribute flaws in five discrete sizes across specimen sets •
- Add one large flaw for reliable detection across all NDE methods



NDE Mockups in Literature (cont.)

- Realistic flaw fabrication methods identified by Sandia:
 - 1. Pillow insert: Kapton tape around four layers of tissue paper
 - 2. Brass shims: Coated with silicone mold release
 - 3. Teflon disk inserts: Various thicknesses (3 mil, 5 mil, 8 mil), or stacked 3-mil inserts
- Disbonds can be simulated by machining honeycomb core areas and inserting pull tabs at specimen edges, and delaminations can be simulated using pillow inserts or stacked Teflon inserts
- A study on marine composites NDE revealed that multiple techniques are often required to assess damage, as no single method can detect all defects
- The recommended approach begins with simple, low-cost methods like tap testing and progresses to more advanced, time-intensive methods
- The Pipeline Research Council International noted that while inspection of substrate beneath repaired composites is possible, interfacial delaminations remain a challenge

Roach, D. (2016). A Quantitative Assessment of Conventional and Advanced Nondestructive Inspection Techniques for Detecting Flaws in Composite Honeycomb Aircraft Structures, DOT/FAA/TC-15/63. Albuquerque: Sandia National Nove, C. (2021) "NDE of Carbon Fiber Reinforced Polymer Composites - Some Thoughts", NDE Public Meeting, NRC ADAMS ML21012A002, pages 103-122 Battley, M., Skeates, A., Simpkin, R., & Holmavist, A. (2002), Non-destructive Inspection of Marine Composite Structures, High Performance Yacht Design Conference, Auckland



NDE on CFRP Repairs in NPPs

- The following reports have been published by EPRI on NDE of CFRP repairs:
 - "Non-contact Nondestructive Evaluation Technology: Dynamic Response Spectroscopy and Pulsed Eddy Current," EPRI Report 3002013174, 2018
 - "Nondestructive Evaluation of Metallic Substrates through Carbon Fiber Reinforced Polymer (CFRP) Composite Repair Systems," EPRI Report 3002020823, 2022
- Key findings:
 - Two NDE techniques, DRS and PEC, were successful in measuring steel substrate thickness through CFRP
 - Phased array ultrasonic techniques could not penetrate the CFRP material due to its high attenuation and were ineffective in measuring carbon steel thickness
 - DRS and PEC produced corrosion maps of steel substrates that closely resembled those from the baseline conventional single-element pulsed echo ultrasonic measurements obtained prior to the fabrication of CFRP repair
 - DRS measured aluminum bronze thickness through CFRP, but only when the remaining wall thickness was greater than 0.18-0.20 in. It struggled to detect thicknesses below this range
 - DRS was capable of measuring steel substrate thicknesses of 0.25 in. (6.4 mm) or more through 0.30-0.65 in. (7.6-16.5 mm) of CFRP, including areas with general and pitting corrosion larger than 0.40 in. (10 mm) in diameter
 - PEC is most accurate when the substrate thickness is uniform within the measurement area, but it may underestimate thickness in localized corrosion spots smaller than the probe's averaging area
 - Advanced algorithms can improve accuracy by compensating for these localized discontinuities

"Non-contact Nondestructive Evaluation Technology: Dynamic Response Spectroscopy and Pulsed Eddy Current" EPRI-Report 3002013174, 2018 "Nondestructive Evaluation of Metallic Substrates through carbon Fiber Reinforced Polymer (CFRP) Composite Repair Systems "EPRI-3002020823, 2022



ASME PCC-2 and ASME CC N-871

The ASME standards related to the evaluation of CFRP repair are sections in:

- □ ASME PCC-2, "Repair of Pressure Equipment and Piping" PART 4 NONMETALLIC AND BONDED REPAIRS – Article 401 – Nonmetallic Composite Repair Systems: **High-Risk Applications**
- □ ASME CC N-871 "Repair of Buried Class 2 and 3 Piping Using Carbon Fiber-**Reinforced Polymer Composite**"
- Table 401-5.2-1 of ASME PCC-2 lists the defect type and allowable limits for the • composite wrap
- ASME PCC-2 identifies visual inspection, hardness test, tap test, and other methods as identified by the repair system supplier as the examination methods for the composite repair



Pacific

Northwest NATIONAL LABORATO



Figure on "CFRP Repair" from ML20014E606, Jim O Sullivan, "Code Case N-871-1 Status Update," NRC Technical Exchange meeting, 2020



ASME CC N-871 Requirements for Examination (as per 05-09-2023 Markup)

Terminal End Only

Prior to Installation of Repair

Inspect the thickness of metal substrate to ensure structural integrity Ultrasonic thickness mapping

After Each layer of Installation

In-process examination of CFRP layers Acoustic tap examination

After Installation of the Final Layer

Volumetric examination of the accessible surface of laminate repair

Metal substrate examination beneath the laminate Acoustic tap or ultrasonic or other volumetric examination

In-Service Inspection (ISI)

Once between 4 to 6 years after repair, and once per 10-year ISI interval Volumetric methods similar or equivalent to previous step

Entire Composite Repair

Prior to Installation of Repair Visual examination of the prepared pipe

surface

During Installation

Layer-by-layer visual and acoustic tap examination of CFRP

Preservice Exam

Visual examination after the laminate has achieved tack-free surface

In-Service Inspection (ISI)

Visual examination once between 4 to 6 years after repair, and once per 10-year ISI interval



Critical NDE Examinations for CFRP Repair

- The most critical examinations are:
 - The substrate at the terminal end under the composite
 - The bond between the substrate and composite
 - The integrity of the underlying substrate at the terminal end and the composite to metal bond is critical for load transfer
 - If terminal end fails to retain structural integrity or the bond fails, it is reasonable to expect that the repair will fail to function as intended
- The second-most critical examinations are for laminar flaws (voids or delaminations) within the composite laminate itself







Variables in Fabrication of CFRP Mockups

Based on the in-depth literature review, the following variables were identified for the fabrication of CFRP repair mockups:

- Substrate and laminate thickness
- Curvature of the piping or flat-plate substrates
- Weight of the fabrics
- Use of thickened epoxy
- Flaw types and how they affect structural integrity
 - Substrate to laminate disbond
 - Interply delaminations
 - Others: Air bubbles, blisters, matrix cracking, moisture ingress, ply waviness, resin rich and starved areas, voids, porosity, foreign object debris, orientational variations, barely visible impact damage



18



Broader Concerns Related to the NDE of CFRP Repairs

Based on the literature review, the following NDE concerns were identified:

- What critical defects are expected to be observed during inspections? Specifically, are substrate-tolaminate disbond, interply delamination, porosity, and water intrusion among them?
- Among the mentioned defects, which are the most challenging to detect using the current inspection methods? Are there other available systems that could potentially detect these difficultto-identify defects?
- Are there any defects that are considered undetectable with the currently available NDE systems? What are the critical flaw sizes and locations of concern during inspections? For example, what would be the impact of finding a 2 in. \times 2 in. disbond or delamination along the terminal end compared to the "body" of the repair?
- Additionally, ASME N-871-2 defines a critical flaw as a laminar flaw larger than 25 in². However, the technical basis for this specific size is not provided
- Have models been developed to ascertain critical flaw types, sizes, and locations for the CFRP repair process?
- What inspection procedures are in place for the areas immediately forward and aft of the compression rings, where non-uniform flow-wear mechanisms may be introduced? Why is only a visual examination performed for the NDE of the entire composite repair system? Are
- there specific reasons for not employing other NDE methods in this context?



EPRI TIE 2023

- EPRI organized a CFRP TIE workshop, held in Charlotte, NC, on July 25-26, 2023
- A month before this workshop, a virtual meeting was held with several CFRP domain experts, and EPRI and PNNL presented on the need and focus of the workshop
- The participants of this workshop included:
 - EPRI, PNNL, and NRC
 - Utility vendors such as Framatome, PSEG Nuclear, Ameren, STP Nuclear, Constellation
 - CFRP fabricators and material suppliers such as Carbon Fiber Engineered Solutions, Kinectrics, Composite Technology and Infrastructure, Sargent & Lundy, Engineering Mech Corp of Columbus, Structural Technologies, A&G Industrial Services, Thin Film Technology, Inc.
 - NDE companies such as Sonomatic
 - Others including faculty from Michigan State University, Korea University
- Several presentations focused on CFRP fabrication, operator experiences, and NDE during this workshop



Lessons Learned from EPRI TIE 2023

Thoughts on Fabrication of CFRP Repairs

- Key steps in fabric installation include dewatering, surface preparation, adhesion testing, fabric saturation, weight ratio testing, fabric installation, inspection, installation of expansion ring, final inspection
- Surface preparation of the substrate is important for the bond strength between the first layer of fabric and the metallic piping
- The recommended surface preparation for the substrate is a white metal blast of 3-mil profile
- The Tg of the resin is an important metric for fabrication, to achieve desired properties
- Safety margins on the material quality and installation are required additional strength and Tg properties indicating a high safety margin
- Predictive models and cure monitoring techniques are required to monitor Tg, predict saturation time, and determine cure quality
- Defects during installation are primarily caused by poor workmanship
- The most common location for defects in the piping is at the12:00 o'clock position (i.e., directly overhead)
- Defect reduction can be achieved by on-the-job training, modifications to procedures, and modifications to systems installed. Approximately 90% of defects are under 2 in²
- Defects in composites may occur due to material quality (both fabric and epoxy), environmental conditions, excessively thickened epoxy, dry spots, wrinkles, air bubbles, fabric bridging, and delamination



Lessons Learned from EPRI TIE 2023 (cont.)

Thoughts on NDE of CFRP Repairs

- Proper quality control measures should be established during the installation, along with monitoring procedures to track the degree of cure.
- Computational FEA modeling of defects and NDE analysis can be performed to study various composite laminates
- Air gap, soil contamination, dry sand, epoxy saturation, mold release are possible defects
- DRS measurements are sensitive to small variations in thickness
- Corrosion pits smaller than 0.4-in. diameter cannot be detected by DRS
- DRS cannot predict the quality of the bond or determine the bond strength ٠

Thoughts on Code Case (ASME N-871-2 Concerns)

- Apart from the guidelines proposed in the ASME CC N-871, there are various alternative measures that the fabricators are allowed to use, as the code case mentions "CFRP supplier recommended practices"
- The uncertainties involved in the installation process need to be defined and proper remedies ٠ should be identified





Lessons Learned from EPRI TIE 2023 (cont.)

Other Thoughts

- There is no "one-size-fits-all" approach ullet
- Independent research on best practices is required to implement policies to be followed ٠
- CFRP undergoes aging through thermo-oxidation, radiation degradation, UV and ionizing radiation, water ingression, contact with other chemical substances
- Destructive testing of pipes at higher temperatures may be required ٠





Action items from EPRI TIE 2023

Based on the discussion in TIE, the following action plan was developed:

- As several variables are involved in CFRP repair process, it is important to prioritize the variables for CFRP mockup fabrication process
- Further, to identify critical variables, it was decided to survey TIE participants and other stakeholders
- NDE research efforts should be carried out by evaluating the following NDE methods such as tap testing, UT, DRS, laser UT, microwave, thermography, and shearography
- Other research and development needs were also identified, such as
 - Tg test of through-pipe chip samples
 - Bond testing at terminal ends
 - Structural testing of CFRP to identify critical flaw size

Variable

Thickne: Substrat

Weight of Layers

Thicken Epoxy

Cure Tempera

Flaw Siz

Flaw Typ

Humidit

•	Matrix
ss of te	0.5", 0.75", 1"
of	20 oz, 40 oz
ed	Yes, No
ature	Study on Tg
ze	2", 3", 4", 5", 10"
pes	Wrinkles, air gaps, substrate defect, delamination
y	High, Low



NDE of CFRP Survey

- To address the concerns discussed from the EPRI TIE 2023 on CFRP, PNNL and EPRI created a survey (https://forms.office.com/g/N3UF5mNWsa) on the "NDE of CFRP" to collect useful information from the CFRP repair community, which consists of designers, asset owners, fabricators/installers, NDE researchers, and certified technicians
- The survey questions were framed to collect information on
 - the design of internal and external CFRP repairs
 - \checkmark Weight of fabrics, orientation of fabrics, number of layers, configurations of installation, usage of thickened epoxy (putty), usage of topcoat, thickness of repair
 - typical flaw types
 - \checkmark Design related such as loss of adhesion to the substrate, delamination between layers, air voids, cracking of epoxy, under saturation, air bubbles, foreign object defects

Pacific

NDE of CFRP Survey (Cont.)

- The survey also requested respondents to:
 - Rank the degree of criticality of the flaws identified from 1-5, with 1 being very critical and 5 being relatively in-consequential
 - Identify additional flaw types that would be of concern
 - List common NDE techniques used in the field
 - Describe limitations of current NDE techniques used in the field
- The survey was created in MS Office Forms and sent to 40 people during October 2023. A total of eight responses were received. A few people declined as it was out of their expertise or the information was proprietary
- Results were analyzed to make critical decisions on mockup fabrications and NDE methods to be used



Survey Responses – Design

- The weight of fabrics varies between about 10 to 40 oz/yd^2 (0.3 to 1.4 g/m²) depending on the fabricator
- For internal and external repairs:
 - 40% responded "Yes" to using thickened epoxy between layers
 - 72% responded "Yes" to using thickened epoxy as a "top coat" and at the "interface of the substrate to CFRP"
 - Few responses mentioned "thickened epoxy" thickness varies around the circumference



Survey Responses – Critical Flaws

The following flaws were ranked as most critical for CFRP repairs:

- Loss of adhesion to the substrate (terminal end regions)
- Delamination between layers
- Under-saturation of fabric
- Cracking of epoxy matrix



Survey Responses – Other Flaw Types

- Apart from the critical flaws identified by PNNL, the survey respondents mentioned other flaw types that may arise during fabrication or in-service
 - High-velocity fluid systems may "peel" the CFRP repair up from the substrate, causing failure
 - Excess thickened epoxy may affect the desired performance of CFRP repair
 - Fabric misalignment during fabrication
 - Having overlaps, lifts the edges of CFRP layers
 - Applying too much top coat, pinholes in top coat
 - "Water ingress" into the CFRP system will have a significant impact on strength
 - Cracks promulgating into the CFRP repair system
 - Any new bubbles or delamination, identified flaws that were left unrepaired during the original install that have grown
 - In service wear (erosion) or damage due to debris within the system



Survey Responses – NDE Techniques and Concerns

NDE Techniques

Visual

Pacific

Northwest

- Tap testing
- Laser UT
- DRS
- Microwave
- Thermography
- Shearography

Concerns

- Tough to effectively measure degradation
- Bond integrity can't be measured \bullet
- Difficult to interpret tap testing \bullet
- Choice of technique depends on specific ulletflaws and specific inspection requirements
- DRS needs a smooth surface
- Laser needs a darker surface •



CFRP Mockup Fabrication Parameters

- A CFRP vendor was identified to fabricate the mockups, and a contract (#740856) was established with the vendor
- Each mockup consisted of 7 plies, which included 6 plies of CFRP and 1 ply GFRP as the first layer
- The CFRP layers were placed in 0/90 orientation (horizontal/vertical) on alternate layers
- Carbon Fiber 2339 (Toray T700S) Unidirectional carbon fiber, 19.5 oz/yd², nominal thickness of 0.036 in.
- Glass Fiber 1210 (Hybon 2026) Bidirectional fiberglass, 25.8 oz/yd², nominal thickness of 0.031 in.
- 212N Saturant Used as a primer and saturant for fiberglass and carbon fiber
- 130N Tack Coat Thickened epoxy used between layers of fiber
- Fumed Silica Used to thicken the 130N tack coat
- Substrate 24 in. × 24 in. × 1/2 in. thick carbon steel plates





CFRP Mockup: Test Matrix

	I

	1	2	3	4	5	6	7	8
Amount of Fume Silica – 6.3%	Х			Х				
Amount of Fume Silica – 8.3%		Х			Х		Х	Х
Amount of Fume Silica – 10.3%			Х			Х		
Thickness of Putty (in.)	0.031	0.031	0.031	0.125	0.125	0.125	0.031	0.125
Undersaturated (Dry) Spots of Epoxy (9 in ²) – 3 in. × 3 in. Square	P2- Q1 P4- Q3							
Undersaturated (Dry) Spot (3 in ² circle)	P5-Q4	P5-Q4	P5-Q4	P5-Q4	P5-Q4	P5-Q4		
Wrinkles (2 - 3 in.)	P1-Q1 P3-Q3	P1-Q1 P3-Q3	P1-Q1 P3-Q3	P1-Q1 P3-Q3	P1-Q1 P3-Q3	P1-Q1 P3-Q3		
Gaps (3 in. long × 1 in. thick)	P3-Q4	P3-Q4	P3-Q4	P3-Q4	P3-Q4	P3-Q4		
Overlaps (3 in. long × 1 in. thick)	P3-Q2	P3-Q2	P3-Q2	P3-Q2	P3-Q2	P3-Q2		
Undersaturated GF Ply							P0-Q1	P0-Q1
Primer Partial Cure before Ply							P0-Q2	P0-Q2
Lack of Adhesion (Improper Surface Prep)							P0-Q3	P0-Q3

P – Ply, Q – Quadrant

CFRP Mockup: Generic Design



Pacific

CFRP-6 CFRP-5 CFRP-4 EPOXY CFRP-3 1 CFRP-2 CFRP-1

*Putty and Topcoat are not shown



Undersaturation of Epoxy (9 sq. in) – 1st quadrant of 2nd CFRP Ply, 3rd quadrant of 4th CFRP Ply Undersaturation of Epoxy (3 sq. in) - 4th quadrant of 5th CFRP Ply Wrinkles – 1st quadrant of 1st CFRP, 2nd quadrant of 3rd CFRP, 3rd quadrant of 5th CFRP Gaps – 4th quadrant of 3rd CFRP Ply Overlap - 2nd quadrant of 3rd CFRP Ply

Gaps (3" long x 1" wide)

Overlap (3" long x 1" wide)

33

Side View

	EPOXY
	EPOXY
	\bigwedge
F	RP

Wrinkles ($\sim 2-3$ ")



CFRP Mockup Fabrication

24 in. × 24 in. plates #8, #3, and #6

- #8 with a different surface preparation, as described in the figure
- #3 with 1 layer of glass fiber fabric, with 10.3% thickened epoxy underneath, applied at 0.031-in. thickness
- #6 with 1 layer of glass fiber fabric, with 10.3% thickened epoxy underneath it, applied at 0.125-in. thickness



.031-in. thickness 0.125-in. thickness





Step 1 : Surface Preparation







Step 2 : Preparation and Application of Putty







CFRP Mockup Fabrication (cont.)

Step 3 : Saturation of Fabric









Step 4 : Application of Fabric Layer



Steps 2, 3, 4 are repeated for each CFRP or GFRP layer as per the design









CFRP Mockup Fabrication (cont.)

Placement of Defects





CFRP Mockup Fabrication (cont.)

Step 5: Final coating



38



CFRP Mockup Fabrication: Fabric Saturation



The fabrics were saturated manually (by hand). The resin was mixed in appropriate ratios as per the manufacturer's recommendations, weighed, and applied to the fabrics. The resin-impregnated fabrics were weighed again to ensure and achieve the correct fiber-volume ratio required as per the installer's specifications.







Mixed putty

Pacific



Applied putty at 0.031 in. and 0.125 in. thickness on CF layer







Trowels used to maintain the thickness of putty





CFRP Mockup Fabrication: Planned vs. Actual Defect Size



The above figures show the undersaturated epoxy spots. These dry spots were planned to be 3 in. × 3 in. squares, but due to the resin saturation the dry spot size decreased to be between ~2 in. to 2.5 in. in both dimensions



CFRP Mockup Fabrication: Other Defects



Pacific

3rd CFRP ply



This figure shows an overlap in the 2nd quadrant, a wrinkle in the 3rd quadrant, and a gap in the 4th quadrant on the



Observations during CFRP Mockup Fabrications

- The CFRP mockups were fabricated by the vendor over a period of 8 days
- PNNL team visited the CFRP vendor for 2 days during the fabrication of these mockups
- The objective of the visit was to observe and learn the fabrication process of CFRP mockups and the challenges faced during the mockup fabrication and placing defects
- As per the test plan, 3 in. × 3 in. square flaw was planned; however, due to resin saturation from the edge of the planned defect location, the size of the dry spot was reduced to ~ 2 in. $\times 2$ in. square flaw
- The preparation (mixing fumed silica with epoxy), application, and assurance of a consistent putty thickness requires skill and needs to be performed with the utmost care
- If the putty is not smoothed to maintain the consistent quantity and thickness or left over time before the application of the next layer, the putty cures, which makes it difficult to spread and be smoothed
 - This may cause a lot of airgaps between the layers •



CFRP Panels (No Metal Substrate)



- Two composite panels of 12 in. × 12 in. with no substrate were also fabricated with 6.3% and 8.3% amounts of putty
- Same layup sequence as CFRP plate mockups (1 layer GFRP + 6 layer CFRP)
- On the third CFRP ply, a dry spot defect was induced on one-half of the panel
- Putty was smoothed to a uniform thickness on these plates (0.031-in. thickness)



Observation of CFRP Panels Cross-Section

The CFRP panels were cut into smaller coupons to examine the dry spot region and application of putty



CFRP panel with 6.3 % putty



CFRP panel with 8.3 % putty

Optical microscope scans of cross-sections of 8.3% panel – without and with dry spot





Horizontal and Vertical CFRP Layers







Data Collection: Tap Testing Hardware

- Manual tap testing (MTT)
 - Aerospace tap hammer
 - Low-cost
 - Easily deployable
 - Requires trained inspector to interpret audible responses for flaw identification
- Automated tap testing (ATT)
 - Woodpecker WP-632AM-R
 - Uses a ratio of the response to a reference "good" location the operator collects to identify flawed regions
 - \checkmark Not dependent on an individual's hearing abilities
 - Records measurements (but not position)
 - Designed for inspection of thin laminates







Data Collection: UT Hardware

- Encoded/Automated pulse-echo ultrasonic testing (PE-UT) method
 - Zetec DYNARAY w/ ZMC² (UV3, 2.5-mm scan/step increment)
 - Olympus V101 (0.5 MHz, 1.0-in. diameter) and Krautkramer 389-057-070 BMC (1 MHz, 0.5-in. diameter
 - w/1-in. Rexolite delay line)
 - Frequencies are chosen based on preliminary testing
 - Normal beam (i.e., 0-degree incident angle)
 - Contact probe
 - Pulse-echo
 - Volumetric inspection
 - Widely accepted
 - Couplant: Ultragel







Data Collection: Flaw Matrix

- 8 plates with a variety of fumed silica percentage, trowel sizes, and defect types were examined
- The quadrants with large square dry spots were scanned with UT, and they will be the focus of this comparison
 - Primarily quadrants 1 and 3 on each plate
 - Areas not scanned are blacked out in the matrix
- Detections over the flawed region for each detection method are marked with a check in the table
 - UT detection: A region that was identifiable as different from the surrounding areas in amplitude and consistency
 - Tap hammer detections: An indication within the flaw zone
- Sizing of defects was not performed
 - Qualitative comparison across different NDE techniques
 - Detected regions were marked and documented for future consideration

Diato #	Fumed	Trowel	Quadrant	Plv #	Flaw Type	Planned	PE-UT		Manual	Woodpecker
i tate #	Silica (%)	Notches	Quadrant	1 (y #	T tuw Type	Size	500 kHz	1MHz	Hammer	632AM-R
1 6.3			1	CF1	Wrinkle	4.5"L				
			1	CF2	Dry Spot	3"x3"	√	√	√	√
			2	CF3	Overlap	3"x1"				
	6.3	1/16"	3	CF3	Wrinkle	4"L				
			4	CF3	Gap	3"x1"				
			3	CF4	Dry Spot	3"x3"	√	√	√	
			4	CF5	Dry Spot	2"dia.			\checkmark	
			1	CF1	Wrinkle	2.5"L				
			1	CF2	Dry Spot	3"x3"	√	\checkmark	√	√
			2	CF3	Overlap	3"x1"				
2	8.3	1/16"	3	CF3	Wrinkle	2.5"				
			4	CF3	Gap	3"x1.25"				√
			3	CF4	Dry Spot	3"x3"	√	\checkmark		√
			4	CF5	Dry Spot	2"Dia.			√	√
			1	CF1	Wrinkle	2.5"L				
			1	CF2	Dry Spot	3"x3"	\checkmark	√	√	√
			2	CF3	Overlap	2-7/8"x1"				
3	10.3	1/16"	3	CF3	Wrinkle	3"L				
			4	CF3	Gap	3"x1"				
			3	CF4	Dry Spot	3"x3"	√	√	√	√
			4	CF5	Dry Spot	2"Dia.		-		
			1	CF1	Wrinkle	4.5"L				
			1	CF2	Dry Spot	3"x3"			√	√
			2	CF3	Overlap	1-3/8"x3"				
4	6.3	1/4"	3	CF3	Wrinkle	4.25"L				
			4	CF3	Gap	3"x1"				
			3	CF4	Dry Spot	3"x3"				
			4	CF5	Dry Spot	2"dia.		•		1
		1/4"	1	CF1	Wrinkle	3"L				
			1	CF2	Dry Spot	3"x3"	1	J	1	1
			2	CF3	Overlap	1.25"x3"		•	•	<u>√</u>
5	8.3		3	CF3	Wrinkle	3.5"L				
	0.0		4	CF3	Gap	3"x1"				1
			3	CF4	Dry Spot	3"x3"	_√	J	1	1
			4	CE5	Dry Spot	2"dia			•	1
			1	CF1	Wrinkle	3"				
		3 1/4"	1	CF2	Dry Spot	3"x3"		J		1
	10.3		2	CE3	Overlan	3"x1"				
6			3	CF3	Wrinkle	2"1				
•			4	CE3	Gan	2-5/8"x1"				
			3	CF4	Dry Spot	3"x3"	J	J	1	1
			4	CF5	Dry Spot	2"dia				V
	8.3	1/16"	1	FG	Dry Spot	2"dia				
			1	FG	Dry Spot	3"x3"				1
7			2	FG	Partial Cure	2'x2'				V
			2	FG	Poor Surface	2 ×2				
			1	FG	Dry Spot	2"dia				1
		1/4"	1	FG	Dry Spot	2 uia. 3"v2"				
8	8.3		2	FG	Partial Cure	2'v2'				V
			2	FG	Poor Surface	2 ^2				
[J J	10		L 77				1



UT Observations

- Due to the nature of alternating layers of fabric, epoxy, and putty, layers visible in the UT are not flat/planar responses
 - Changes in layer thicknesses can compound distortion seen in UT images
 - Gating individual regions or layers is difficult due to time variation
 - Examples of layer responses seen in the UT data are highlighted







UT Observations (cont.)

- To capture the interface response (CFRP to base plate), the gain was increased substantially
 - Surface saturation drowns out some of the shallow layers
- Images show a dry spot two layers below the surface
 - Top image is gated based on the layer location (yellow lines)
 - \checkmark Response from the defect is higher amplitude than the surrounding signal
 - Lower image is based on gating on a deeper layer (black lines)
 - ✓ Lack of signal penetration due to dry spot
 - ✓ Shadow under the defect
- The lower image shows the potential for gating under shallow flaws for detection via the flaw shadow (blocked UT signal)



shadow-black)

Low amplitude due to blocked UT in layer above (shadow)



Tap Testing Observations

- The ATT system (632 AM-R) had difficulties providing consistent responses
 - The system is designed for thin (< 4 mm or 0.16 in.) laminates such as those used in</p> aerospace
 - Multiple single locations within a flawed region would be reported, but nothing coherent across the region of interest to indicate area defect
- MTT testing requires the inspector to clearly hear the sound response
 - Additional background noise (such as that present in industrial environments) can mask the audible defect noise
- All responses from the ATT are based on the user-defined reference location
 - No complete true state "good" region or calibration specimen to use for reference
 - \checkmark Choosing an appropriate area for a reference signal is critical for these units
 - \checkmark UT showed a broad variation in signal responses across each plate, suggesting that a "good" region is subjective and potentially difficult to find



NDE of Dry Spots

- For CFRP plates #1 to #6, Q1, where the dry spot is located at the second CFRP ply, and Q3, where the dry spot is located at the fourth CFRP ply, were analyzed in UT at 500 kHz and 1 MHz
- For the dry spot in Q1, ply 2
 - UT: 5 of 6 identified
 - MTT: 6 of 6 identified
 - ATT: 6 of 6 identified
- For the dry spot in Q3, ply 4
 - UT: 6 of 6 identified
 - MTT: 4 of 6 identified
 - ATT: 4 of 6 identified

Fumed Trowel Plate # Quadrant Ply # Flaw Type Silica (%) Notches CF2 Dry Spot 1 1 6.3 1/16" 3 CF4 Dry Spot 4 CF5 Dry Spot CF2 Dry Spot 1 2 CF4 8.3 1/16" 3 Dry Spot CF5 4 Dry Spot CF2 **Dry Spot** 1 3 10.3 1/16" 3 CF4 Dry Spot 4 CF5 Dry Spot CF2 Dry Spot 1 4 6.3 1/4" 3 CF4 Dry Spot 4 CF5 Dry Spot 1 CF2 Dry Spot 5 8.3 1/4" 3 CF4 Dry Spot 4 CF5 Dry Spot 1 CF2 Dry Spot 6 10.3 1/4" 3 CF4 Dry Spot 4 CF5 Dry Spot FG Dry Spot 1 7 8.3 1/16" 1 FG Dry Spot FG Dry Spot 1 8 1/4" 8.3 FG Dry Spot 1

None of the dry spots in the GFRP layer (bottommost layer next to the substrate) were identified by UT or MTT

Planned	PE-UT		Manual	Woodpecker		
Size	500 kHz	1MHz	Hammer	632AM-R		
3"x3"	\checkmark	√	√	√		
3"x3"	\checkmark	\checkmark	√			
2"dia.			\checkmark			
3"x3"	\checkmark	\checkmark	\checkmark	√		
3"x3"	\checkmark	\checkmark		\checkmark		
2"Dia.			\checkmark	√		
3"x3"	\checkmark	\checkmark	\checkmark	\checkmark		
3"x3"	\checkmark	\checkmark	\checkmark	√		
2"Dia.						
3"x3"			\checkmark	√		
3"x3"	\checkmark	\checkmark				
2"dia.				√		
3"x3"	\checkmark	\checkmark	\checkmark	√		
3"x3"	\checkmark	\checkmark	\checkmark	√		
2"dia.				√		
3"x3"		\checkmark		√		
3"x3"	\checkmark	\checkmark	\checkmark	\checkmark		
2"dia.						
2"dia.						
3"x3"				√		
2"dia.				\checkmark		
3"x3"				√		



Plate #2 – Q1 Dry Spot Indications

- Defect is located in the second layer of carbon fiber applied
 - ~2/3 of the way through the CFRP
- Dry spot detected by both MTT and UT inspection
 - Indications reported had similar locations and sizes for both techniques
 - UT indication is gated behind layer #2 to show a lack of signal penetration
- True-state dimensions are not necessarily accurate
 - Size and location were defined as the plates were being fabricated
 - Epoxy may have flowed and distorted the true size of the defect during curing







Plate #4 – Q1 Dry Spot Indications

- Defect is located in the second layer of carbon fiber applied •
- Recorded as "no detection" for UT •
 - UT response lacked continuity across a majority of the flawed region (solid red box highlight a few discrete indications)
 - Presence of other signals outside of planned flaw fabrication region with comparable magnitude nearby (highlighted in red dashed box)
- UT signal responses within the planned defect region were similar in location to MTT detections





54



Plate #5 - Q1 Dry Spot Indications

- Defect is located in the 2nd layer of carbon fiber applied
- Dry spot detected by both MTT and UT inspection
 - Indications reported were similar location and size for both techniques
 - Gated UT signal has low surrounding noise
- Small indication on the left edge of the UT detection may have just been missed by MTT
 - Small response which can easily be missed without an encoded data record





- Summary
- CFRP repairs in NPPs are not typical in the nuclear industry compared to other industry applications (e.g., aerospace)
 - On-site fabrication process, use of putty, and other process parameters
 - NDE methods must be evaluated for inspecting these applications
- 8 CFRP repair mockups were fabricated with varying amounts and thicknesses of putty, under-saturated (dry) spots, wrinkles, ply-gaps, and overlaps, in various layers.
- Both UT and MTT were evaluated on 3 in. × 3 in. dry spot regions across plates
 - Only defect detection was compared
 - It of the 14 were detected by UT for at least one frequency
 - 9 of the 14 were detected by MTT
 - ATT was not consistent through this thickness of CFRP (instrument is designed for much thinner composites)



Key Takeaways: Fabrication of CFRP Mockups

- Based on EPRI CFRP TIE 2023 and the PNNL survey of CFRP vendors, important mockup parameters and critical defects to be considered were identified
- There is no technical basis available publicly for the relationship between critical defects and the performance of CFRP repair in nuclear applications
- Planned defects are difficult to maintain in size and shape during fabrication for evaluation
- Application of putty may cause additional defects and air gaps in-betweens layers
- Fabrication of the CFRP repairs, including mixing and applying resin, and putty requires trained professionals
- Strict adherence to protocols needs to be maintained during CFRP repair fabrication
- n-betweens layers resin, and putty



Key Takeaways: NDE of CFRP Mockups

- MTT was a useful screening tool for dry spots
 - Reliant on a trained inspector to detect and document responses
- Encoded UT can detect a majority of the larger dry spots scanned
 - Encoded data helps to see continuous flaw responses over an area
 - Variation in epoxy thickness can make gating specific layers difficult across a scan region
 - Near-surface defects can be masked by the probe's near-field (the region close to the transducer where the sound field fluctuates) and resulting saturation of the front surface response based on the current setup
- Destructive evaluation of planned defects (and bonus detections) may be necessary to verify true state for screening of inspection techniques





Potential Follow-on Work

- Quantitative Analysis of Flawed Regions
 - Evaluate the capabilities and limitations of different NDE techniques across a range of defect types
 - Perform comprehensive tap-testing of the plate's full surface area, focusing on false positive rate analysis, leveraging MTT and ATT systems (e.g., Woodpecker and Evotis)
 - Conduct UT scans to inspect the remaining flawed regions
 - Perform detailed sizing, depth analysis, and statistical evaluations of NDE results
 - Assessment of various tap hammers under different operational conditions
 - Investigate alternative NDE methods to fingerprint mockups without resorting to destructive testing, or validate the true state of mockups by destructive testing



Potential Follow-on Work (cont.)

- Confirmation of Other Field-Deployable NDE Inspection Techniques
 - Assess commercially available methods for identifying defects within materials, focusing on their capabilities and limitations
 - ✓ DRS
 - ✓ Laser UT
- Fabrication of New Mockups
 - Determine additional variables to address based on findings from this study
 - Develop new pipe mockups incorporating:
 - ✓ Varied amounts of thickened epoxy
 - \checkmark Different fabric weights for structural variations
 - ✓ Different flaw configurations in CFRP layers and substrate-to-bond interface region



- Battley, M., Skeates, A., Simpkin, R., & Holmgvist, A. (2002). Non-destructive Inspection of Marine Composite Structures. High Performance Yacht Design Conference, Auckland,
- Elen M., N.A. Conway, and A.A. Diaz. 07/26/2023. "NDE of CFRP and Concerns on Mockup Fabrications." Presented by M. Elen at CFRP Technical Information Exchange - July 2023, Charlotte, North Carolina. PNNL-SA-187967.
- Elen M., N.A. Conway, and A.A. Diaz. 01/24/2024. "Fabrication and NDE Analysis of CFRP Repair Mockups." Presented by M. Elen at NDE Technical Information Exchange meeting, Washington, District Of Columbia. PNNL-SA-194066.
- Gerard, Trevor J., and Tomas T. Jimenez. "Brunswick Nuclear Plant Circulating Water Piping Discharge Headers Pressure Barrier Replacement Using Advanced Fiber Reinforcement Polymer (FRP) Systems." (2015). 23rd Conference on Structural Mechanics in Reactor Technology, Manchester, United Kingdom - August 10-14, 2015.
- Lee, R., Burch, S., & Wall, M. (2012). PRCI Project NDE 2-3 NDE & Inspection Techniques Applied to Composite Wrap Repairs (PRCI Project No. PR-398-113705). 2nd Annual Composite Repair Users Group. Houston, TX.
- Nove, C. (2021) "NDE of Carbon Fiber Reinforced Polymer Composites Some Thoughts", NDE Public Meeting, NRC ADAMS ML21012A002, pages 103-122.
- "Non-contact Nondestructive Evaluation Technology: Dynamic Response Spectroscopy and Pulsed Eddy Current." EPRI-Report 3002013174, 2018.
- "Nondestructive Evaluation of Metallic Substrates through Carbon Fiber Reinforced Polymer (CFRP) Composite Repair Systems "EPRI-3002020823, 2022.
- Quakewrap, "Repair & Strengthening of PCCP Concrete Pipe with Carbon FRP." https://guakewrap.com/project_sheets/Repair%20and%20Strengthening%20of%20PCCP%20Concrete%20Pipe%20with%20Carbon%20FRP.pdf.
- Roach, D. (2016) "A Quantitative Assessment of Conventional Nand Advanced ondestructive Inspection Techniques for Detecting Flaws in Composite Honeycomb Aircraft Structures," DOT/FAA/TC-15/63. Albuquerque: Sandia National Laboratories for Federal Aviation Administration...
- Structural Technologies, "Pipeline Repairs at Hope Creek Nuclear Generating Station." https://www.structural.net/case-studies/pipeline-repairs-at-hope-creek-nucleargenerating-station.
- Talreja., R. (2015) 'Manufacturing defects in composites and their effects on performance", Polymer Composites in Aerospace Industry 2015.





Protecting People and the Environment

Thank you

Muthu Elen <u>Muthu.Elen@pnnl.gov</u> Nicholas Conway <u>Nicholas.Conway@pnnl.gov</u>

