Irradiation of Sensors and Adhesive Couplants for Application in LWR Primary Loop Piping and Components

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NSUF 1.1 Sensor Irradiation and Post Irradiation Examination (PIE) of Acoustic Sensors and Adhesive Couplants

NCSU PULSTAR Reactor and Intense Positron Beam Facility
ORNL LAMDA Laboratory

J. J. Wall et al., Nuclear Engineering & Design, 414, 112594 (2023)

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- Motivation: The industry would like to develop capabilities to monitor flaws online in primary loop piping and components using semi-permanently mounted acoustic sensors.
- For some locations, neutron irradiation effects on sensors and adhesive couplants need to be understood.

NSUF Contributions:

- R&D funding directly to EPRI.

- NCSU: Sample assembly irradiation at PULSTAR reactor, in-situ ultrasonic data collection and positron annihilation spectroscopy.

- ORNL: Electron microscopy - imaging and EBSD, x-ray microanalysis.

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Project Team

- **Joe Wall** – Principal Investigator.
- **Luke Breon** - EPRI – Irradiation experiment execution / acoustics SME.
- **Pradeep Ramuhalli** – ORNL – Consultant on experiment design and PIE
- **Josh Daw** – INL – Consultant on experiment design.
- **Chad Parish**, **J. Travis Dixon** and **Josh Schmidlin** – ORNL/LAMDA – Electron microscopy and x-ray microanalysis.
- **Scott Lassell**, **Ming Liu** and **Ayman Hawari** – NCSU/PULSTAR – Irradiation and positron annihilation spectroscopy.

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- 16 sample assemblies were created for potential irradiation in the NCSU test reactor.
- The assemblies consisted of piezoelectric sensors adhered to aluminum pucks using various adhesive couplants.
  - Sensors were lithium niobate and bismuth titanate.
  - Adhesive couplants were alumina based, aluminosilicate, zirconia based and epoxy.
  - Acoustic measurements were made prior to irradiation at EPRI.
    - Based on acoustic response the 16 samples were pared down to 8.
- Acoustic data was collected in-situ during sample irradiation.

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Table showing the sensor / couplant combinations for irradiation testing. The samples selected showed the best acoustic response during pre-irradiation testing of 16 assemblies at EPRI

As Recieved	PULSTAR			
	Adhesive Name	Sample ID	Adhesive Composition	Sensor Type
2	Zircar HITAC-4	EPRI-1	Aluminosilicate	Bismuth Titanate
4	Zircar HITAC-4	EPRI-2	Aluminosilicate	Lithium Niobate
8	Saureiusien Cement # 2	EPRI-3	Alumina Based	Lithium Niobate
5	Saureiusien Cement # 2	EPRI-4	Alumina Based	Bismuth Titanate
11	Contronics 904	EPRI-5	Zirconia Based	Bismuth Titanate
14	Masterbond EP17HT	EPRI-6	Epoxy	Bismuth Titanate
15	Masterbond EP17HT	EPRI-7	Epoxy	Lithium Niobate
16	Masterbond EP17HT	EPRI-8	Epoxy	Lithium Niobate

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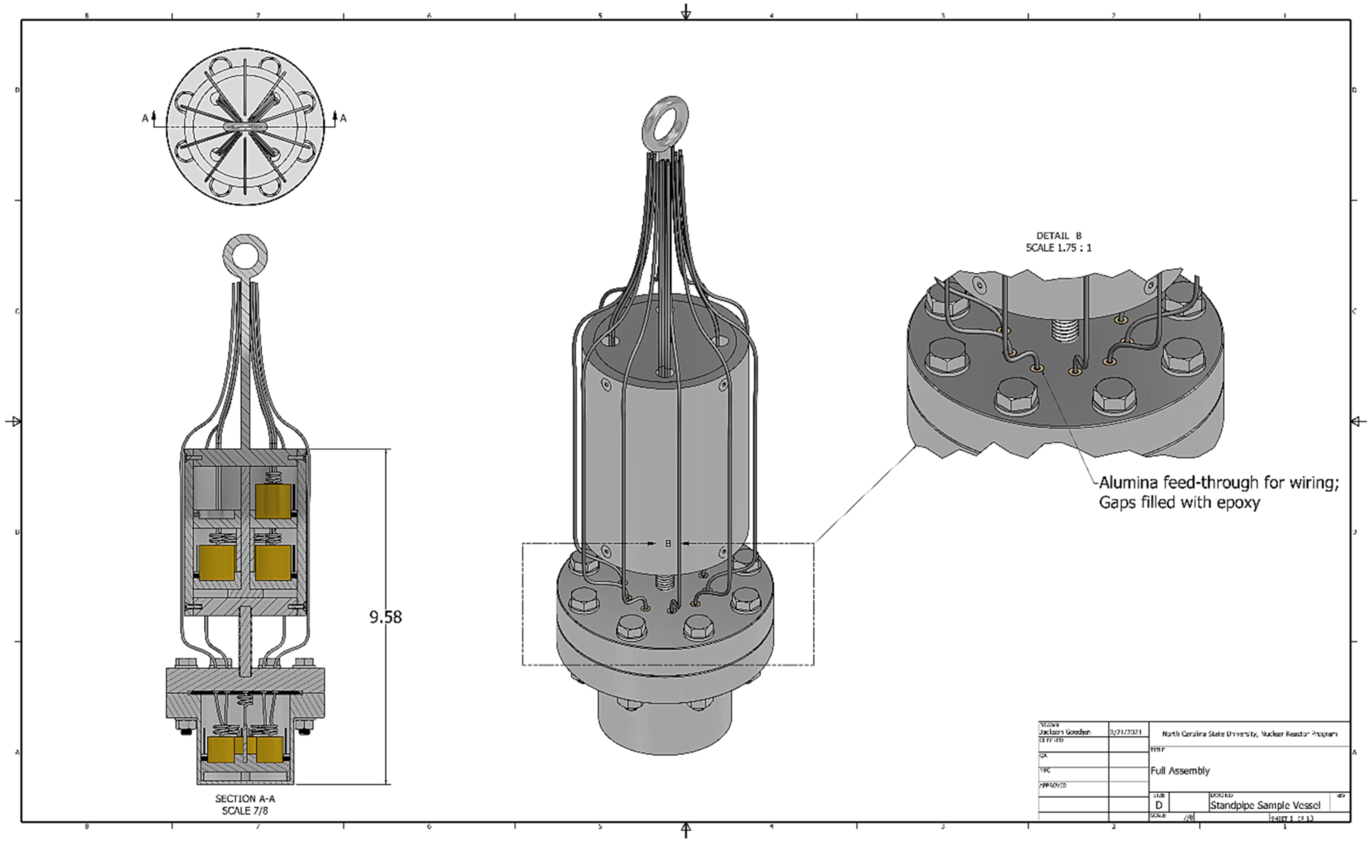
- The bismuth titanate samples were arranged in an upper chamber open to air while the lithium niobate samples were arranged in a hermetically sealed lower chamber.



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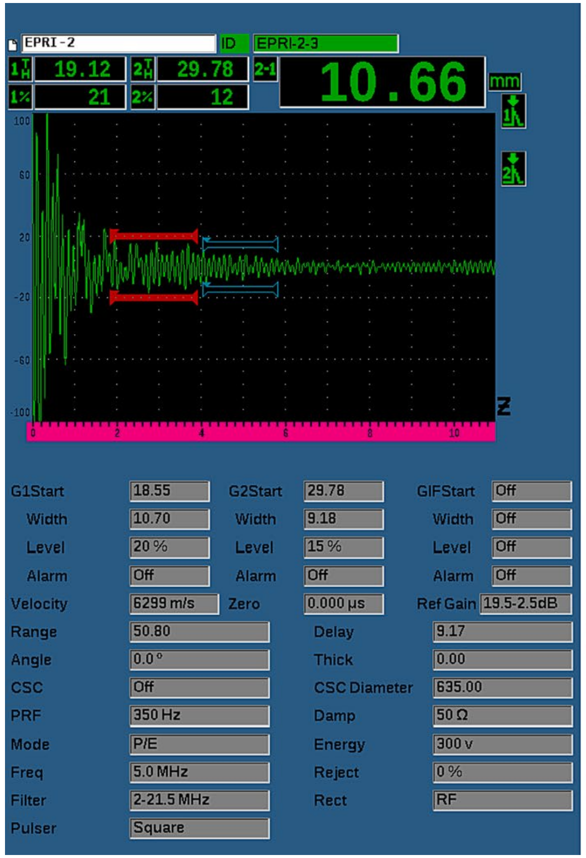
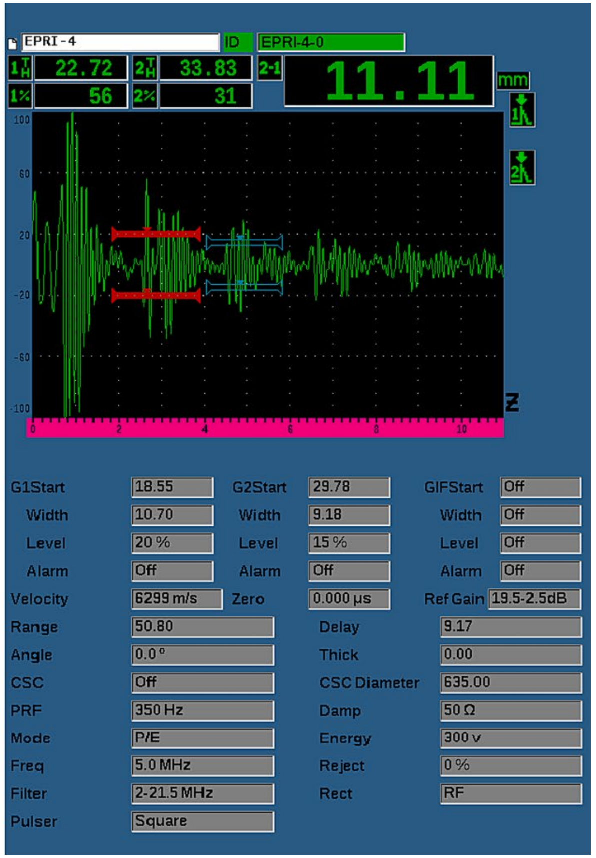
- The samples were wired and attached to a pulser-receiver for in-situ data collection during irradiation.





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- The target fluence was 5×10^{16} n/cm^2 (E > 1MeV) which was determined from neutron transport calculations in the vicinity of the hot and cold led dissimilar metal welds.
- Three of the eight samples were found to generate usable ultrasonic waveforms beyond the target neutron fluence.
  - Sample EPRI-4: Bismuth titanate sensor adhered with aluminosilicate couplant
  - Sample EPRI-7: Lithium niobate with high temperature epoxy couplant.
  - Sample EPRI-8: Lithium niobate with high temperature epoxy couplant.

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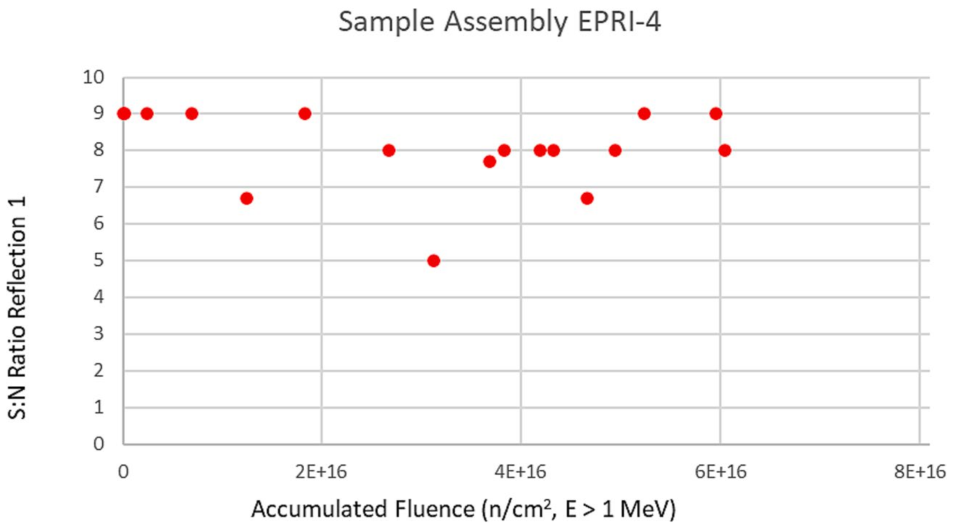

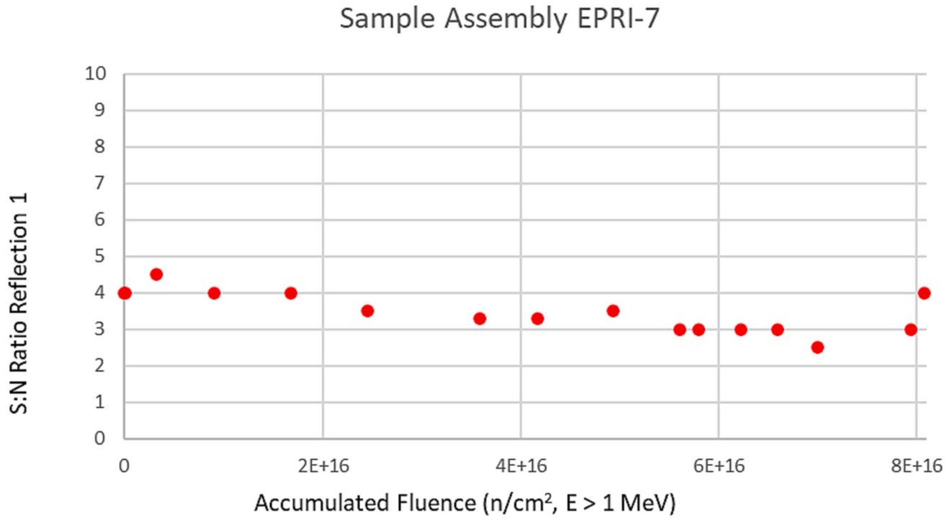

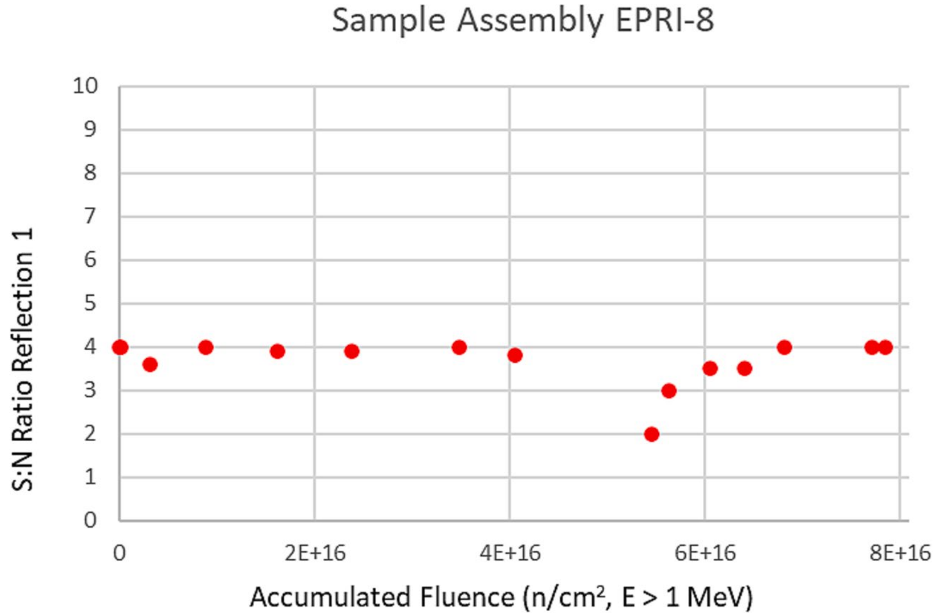

Signal to noise ratios of the first reflection in the acoustic waveforms as a function of fast neutron fluence for samples EPRI-4 (upper left), EPRI-7 (upper right) and EPRI-8 (lower right)

Pre and Post Irradiation Electron Microscopy (To be Completed in 2024 Under an NCE)

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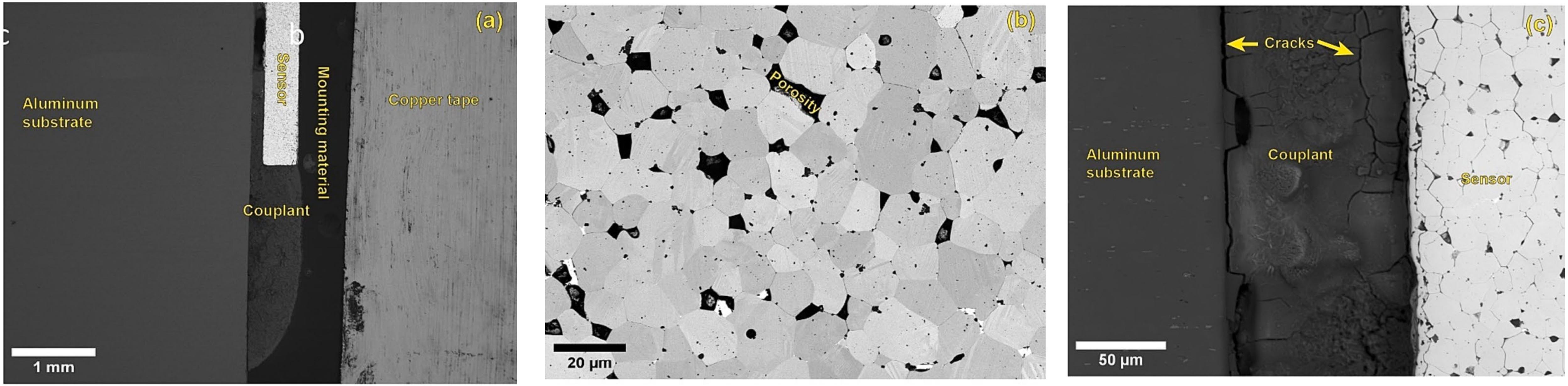

Unirradiated Sample: (a) Backscatter electron micrograph showing the bismuth titanate sensor (white) aluminosilicate couplant (dark gray) and aluminum substrate (lighter gray). (b) Backscatter electron micrograph of the bismuth titanate sensor microstructure. (c) Backscatter electron micrograph showing poor adhesion to substrate bonding. This sample showed poor acoustic behavior and was therefore not irradiated.

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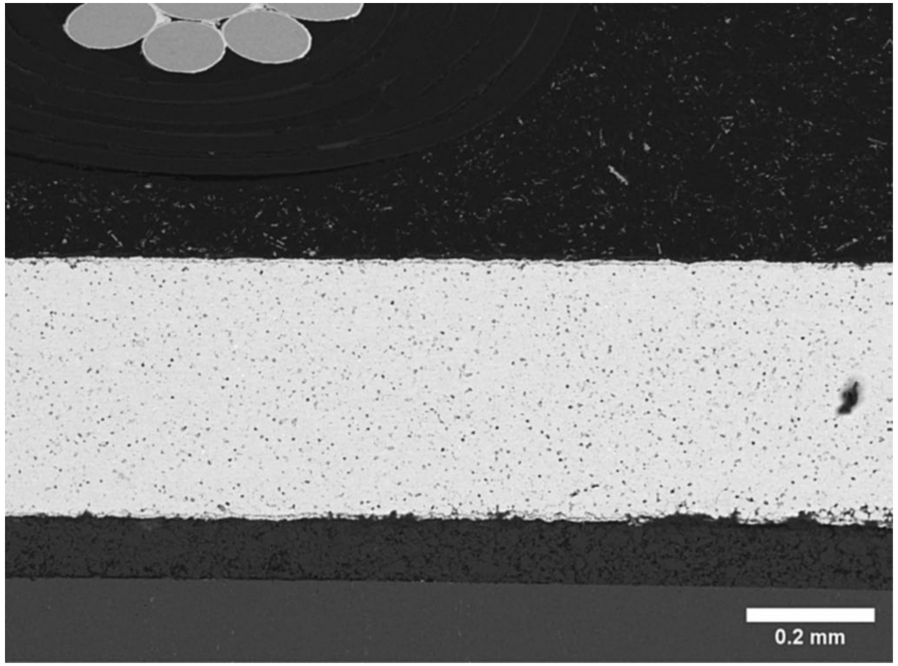
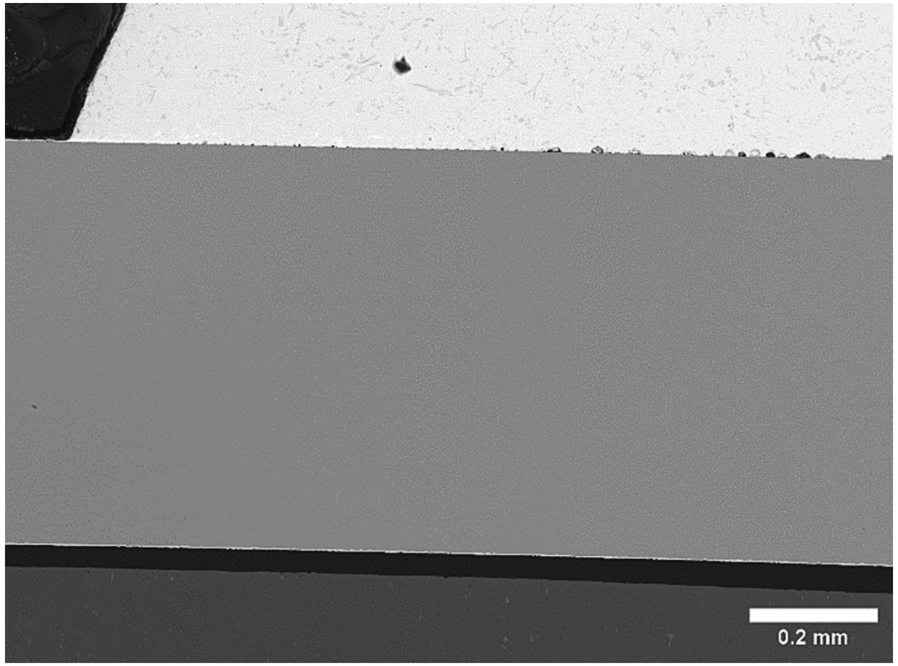


Backscatter electron micrograph of sample assembly EPRI-4 showing adhesion of the bismuth titanate sensor (white) to the couplant (dark gray) and the substrate (lighter gray) to the aluminosilicate couplant (dark gray). This sensor assembly gave usable ultrasonic data throughout the irradiation campaign. Note the artifact at the top is a wire soldered to the sensor that was cut by the plane of polish.



Backscatter electron micrograph of sample EPRI-8 showing – from top to bottom - the solder ball for the electrical connection (white), lithium niobate sensor (gray), epoxy couplant (black) and aluminum substrate (dark gray).

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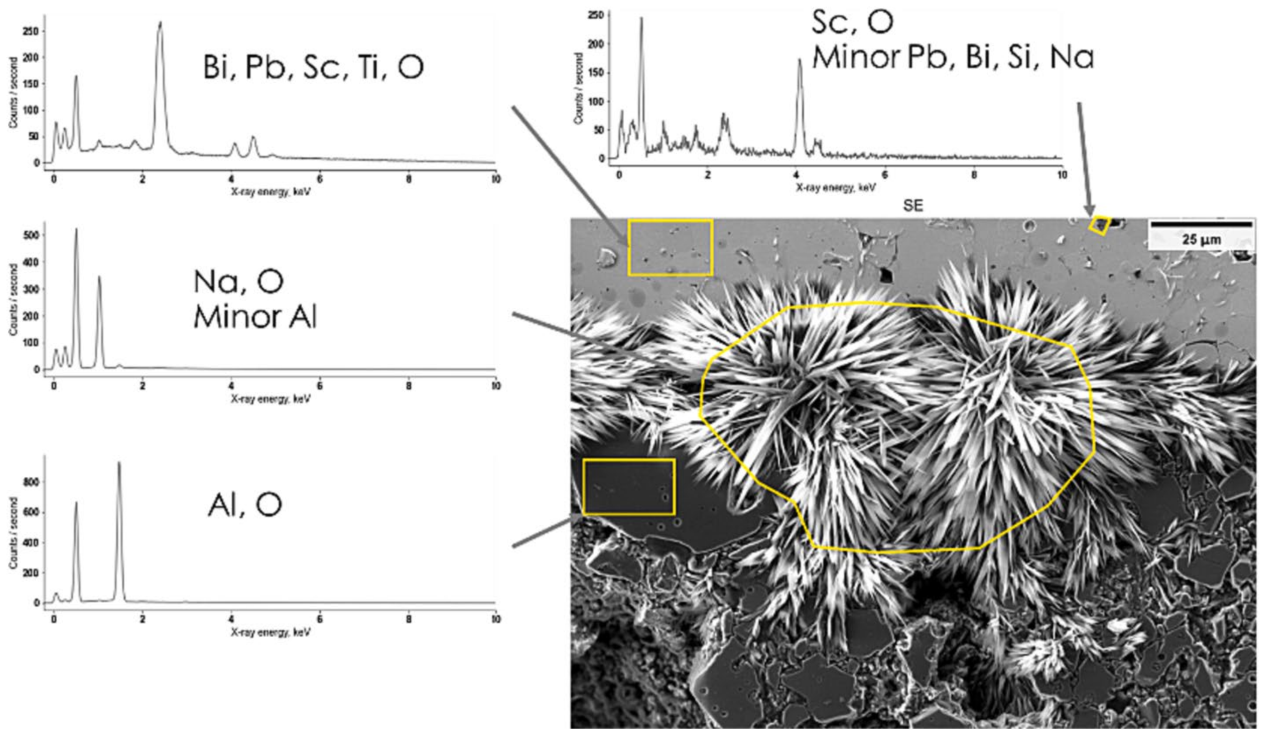

Backscattered electron micrograph and EDX spectra of an Na-O feature found in sample EPRI-4 at the aluminosilicate couplant – bismuth titanate sensor interface. These features appear to have formed in between polishing and examination and may be an artifact of the humidity in the lead storage vaults.

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Project Update

- A no-cost extension through FY 2024 has been granted to finish the PIE at the LAMDA laboratories at ORNL
- The cause for the delay is that the FFRDC contract with ORNL terminates at the end of FY 2024.
- The EPRI R&D contract and NCSU FFRDC contract terminated at the end of FY 2023.
- Lesson Learned – Check all contract paperwork to confirm all components of the research project schedule are consistent!

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Preliminary Conclusions:

- Three assemblies of the eight tested produced usable ultrasonic date up to to beyond the target fluence.
- PALS and DBS showed that ~0.1 ppma of vacancy defects were introduced into the microstructures of the sensors.
- Irradiation to the target fluence did not appreciably change the S:N ratio for lithium niobate or bismuth titanate sensors.
  - It is believed that the failures occurred due to failure of the couplants.
  - Of the couplants studied, the high temperature epoxy worked best.

For more details see *Nucl. Eng. Des. 414, 112594 (2023)*.

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