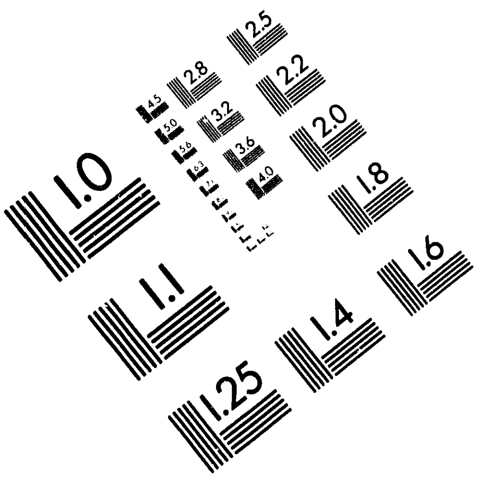

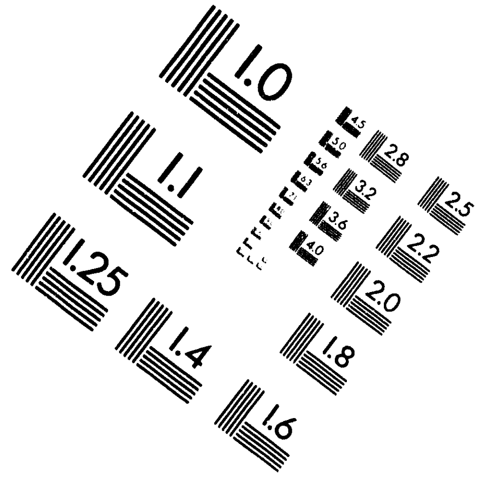

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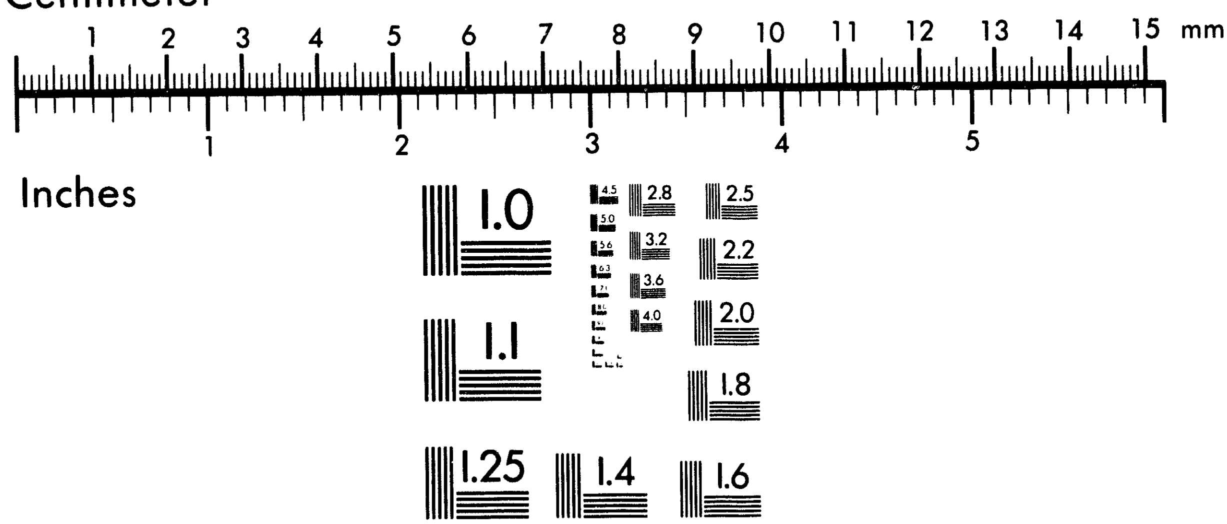

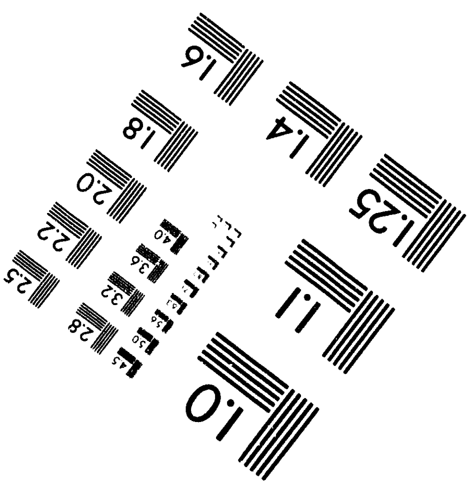

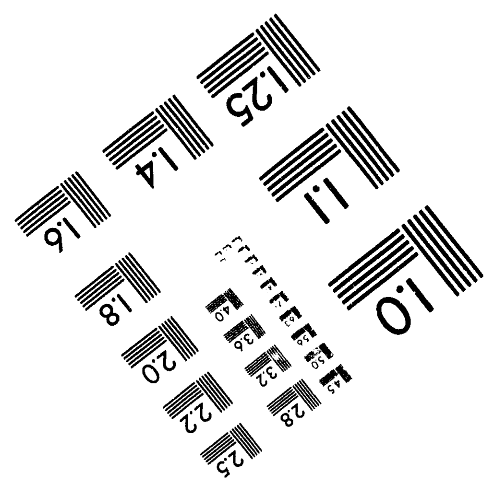

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ACOUSTIC EMISSION SENSOR RADIATION DAMAGE THRESHOLD EXPERIMENT

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DISCLAIMER

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ABSTRACT

Determination of the threshold for damage to acoustic emission sensors exposed to radiation is important in their application to leak detection in radioactive waste transport and storage. Proper response to system leaks is necessary to ensure the safe operation of these systems. A radiation impaired sensor could provide "false negative or false positive" indication of acoustic signals from leaks within the system. Research was carried out in the Radiochemical Technology Division at Oak Ridge National Laboratory to determine the beta/gamma radiation damage threshold for acoustic emission sensor systems. The individual system consisted of an acoustic sensor mounted with a two part epoxy onto a stainless steel waveguide. The systems were placed in an irradiation fixture and exposed to a Cobalt-60 source. After each irradiation, the sensors were recalibrated by Physical Acoustics Corporation. The results were compared to the initial calibrations performed prior to irradiation and a control group, not exposed to radiation, was used to validate the results. This experiment determines the radiation damage threshold of each acoustic sensor system and verifies its life expectancy, usefulness and reliability for many applications in radioactive environments.

INTRODUCTION

Acoustic emission sensors operate using the piezoelectric effect which transforms particle motion produced by an elastic wave into an electric signal. The piezoelectric element consists of a crystal with a unit cell that displays no center of symmetry. Work was done by Argonne National Lab to determine the effect of radiation on a piezoelectric element, but it answered the question of usefulness of a specific type of piezoelectric material at a specific exposure level[1]. The primary interest of this experiment was the effects of radiation upon the entire sensor system and its many interfaces (the piezoelectric element, the casing, the internal electronics and preamplifier, and the epoxy bond). Previous studies examined specific parts of the system independently. This experiment determines the radiation threshold for the whole system beyond which measurable damage occurs, and acoustic results are not predictable.

Determining the radiation damage threshold for an acoustic emission sensor system is necessary for validating the reliability of that system for use in a radiation field. Acoustic emission sensors can be used to detect and locate leaks in waste storage, and transport systems. Accurate detection of leaks is critical, especially in a radioactive environment.

The experimental system consisted of acoustic emission sensors mounted with a two-part epoxy onto stainless steel waveguides. Two models of sensors were used in this experiment. In order to provide reliable and accurate results, eight of each model were exposed to a Cobalt-60 source until the radiation threshold was reached, and four of each model were placed in a control group, not exposed to radiation, to validate the results. Both groups were kept together, except

during the irradiation process, handled and recalibrated in the exact same manner. The experimental sensors were placed inside a doubly contained irradiation fixture prior to irradiation with a Cobalt-60 source. After each exposure the sensors were recalibrated by Physical Acoustics Corporation in Princeton, New Jersey. The results were compared to the initial calibrations performed prior to irradiation.

EQUIPMENT

Acoustic Emission Sensor System

Acoustic sensors models R15 and R15I, manufactured by Physical Acoustic Corporation (PAC), were used in this experiment. The models are basically the same, however, the larger R15I incorporates a low noise 40 dB preamplifier within a rugged housing to provide immunity to interference, and the capability of driving longer cables (up to 1000 meters) without signal loss. In addition, the integral preamplifier dramatically increases the signal to noise ratio, and eliminates the need for cumbersome preamplifiers which reduces equipment cost and allows for permanent installation of the sensors.

The sensors were attached to stainless steel waveguides with a bonding agent, BIPAX TRA-BOND BA-2106T, manufactured by TRA-CON. Waveguides are devices that couple acoustic energy from a structure to a remotely mounted sensor and are often necessary when attaching sensors to systems with curved surfaces. The bonding agent, which physically attaches the sensor to the waveguide, and the sensor system to the structure, acts as a couplant. Couplants are used at the structure-to-sensor interface to improve the transfer of acoustic energy across the interface between the sensor and waveguide. Sensors and waveguides were mounted according to ASTM standards[2]. After assembly, the sensor systems were calibrated at PAC prior to exposure to radiation. This calibration served as a baseline for later comparisons.

Model 109 Cobalt-60 Irradiator

A model 109 Cobalt-60 irradiator, manufactured by J.L. Shepherd and Associates, was used to irradiate the sensor systems. The irradiator uses Cobalt-60 sources that are doubly encapsulated in 300 series stainless steel and meet "special form" criteria of the United States Nuclear Regulatory Commission. The Cobalt-60 sources completely surround the irradiation chamber and provide a dose rate of approximately 2.5×10^5 R/hr.

The irradiation chamber size is 6 inches in diameter by 8 inches high. During the experiment the chamber was loaded with specimens, closed and lowered into the source for a specific time to simulate the performance of the sensors in an anticipated radiation field.

Irradiation Fixture

The irradiation fixture consisted of polypropylene inner trays mounted on an aluminum threaded rod. Two trays held four sensors each. The trays were machined so the waveguides fit snugly in position to insure the sensors remained upright. Each tray was permanently marked with position numbers to insure the sensors were placed in the same positions for each irradiation.

Loaded trays were placed inside an outer aluminum container. The fixture was then placed inside the irradiation chamber. Care was taken to align the fixture to the same position for each irradiation to more accurately determine the dose that individual sensor systems received.

Calibration Equipment

Calibration equipment is used to determine any deterioration of acoustic emission sensor sensitivity. Calibrations were done in accordance with ASTM standards[3]. The equipment consisted of:

Hewlett Packard, model 3325A Synthesizer/Function Generator - Signal source pulsar and channel "A" of phase/gain meter.

Panametrics, model V103 Broad Band Pulsar - Converts electrical to mechanical energy. The device under test is coupled face to face with this device.

Physical Acoustics, model 1220A-BPSYS Preamplifier - Provides 40 dB gain to output of the device under test. Output to channel "B" of gain/phase meter.

Physical Acoustics, model AE1A-BPSYS Post Amplifier/Power Supply - Provides 28 V to the above amplifier.

Hewlett Packard, model 3573A Gain/Phase Meter - Compares amplified output of the device under test to output of Synthesizer/Function Generator, outputs difference (analog).

Dunegan Corporation, model 935 A/D Converter - Converts analog signal from gain/phase meter to digital format, interfaces with desktop computer.

Hewlett Packard, model 9825A Desktop Computer - System control and data processing.

Hewlett Packard, model 7225A graphics Plotter - Generates hard copy presentation of the device under test's performance data.

Physical Acoustics, R15 Standard Transducer - Reference standards to periodically gauge performance of calibration system.

DOSIMETRY

For this experiment it was necessary to determine a dose rate for the individual positions in the irradiator. The dose rate for each position within the irradiation fixture yielded a dose for the individual sensor systems after each exposure to the source. This allowed the radiation damage threshold to be more accurately determined. After considering several methods, an aqueous chemical dosimetry solution, the Fricke dosimeter, proved best for this application. Fricke dosimetry is widely accepted as a standard, readily reproducible, has a range of 4000 to 40,000 Rads, will show if the dose is linear, and can measure a dose to an accuracy of 1-2% [4].

Since two sizes of sensors were to be irradiated, and the sensor array could perturb the gamma flux during irradiation, the dosimeters needed to emulate the sensor systems in order to determine an accurate dose rate for the individual positions within the irradiation fixture. This was achieved by mounting the outer sensor casings, devoid of their internal components, onto waveguides in the same manner as the sensor systems used in the experiment. Small glass vials and caps were fabricated to fit inside the sensor casings to hold the dosimetry solution.

The dosimetry solution for this experiment consisted of 0.28 g $FeSO_4$, 0.06 g NaCl, 22 ml H_2SO_4, and H_2O (to make 1 liter of solution). Ferrous sulfate and sodium chloride were mixed with sulfuric acid. Water from a Milli-Q water purification unit was placed inside a 1 liter volumetric flask. The acid mixture was added to the volumetric flask and stirred. Additional water from the purification unit was added until a 1 liter volume was achieved. The dosimetry solution was mixed in batches as needed. Each batch was numbered, and the exact amount of chemicals used in each batch was recorded. The solution was used immediately after mixing to insure consistent results.

The glassware used for mixing and containing the dosimetry solution required special preparation prior to irradiation. The glassware was baked at 550 °C for 1 hour, allowed to cool, and kept inside covered glass containers used to avoid contamination before, during, and after the baking process. After baking, the dosimeter vials were filled with dosimetry solution, capped, and allowed to soak in the solution overnight. Prior to irradiation, all vials were filled with fresh dosimetry solution, capped, and labeled with a unique three digit number indicating dosimeter solution batch number, exposure number, and position number. The vials were placed in the hollow sensor casings and arranged in the same array within the irradiation fixture as the experimental sensor systems. This process closely reproduced the perturbation of the flux and shielding by the sensor housings and waveguides.

Three exposures were performed in each of the lower, mid, and upper ranges of the Fricke dosimetry for both sensor models. Each exposure was timed and recorded. After every exposure the dosimetry solution of each position within the irradiation fixture was read by spectrophotometer at 304 mμ to find the absorbance, or the change of the ferrous ion to ferric. Unirradiated dosimetry solution from the same batch was used as a control for comparison during spectrophotometry. This data, as well as the room temperature during use of the spectrophotometer, was used to determine the dose rate. The room temperature was important in determining the extinction coefficient of the solution which is temperature dependent. The dose the individual positions received from the irradiator was calculated using the following equation[5]:

$$D_{rads} = \frac{N\,[\Delta(OD)]100}{(\Delta\epsilon)\ 10^3\ G(Fe^{3+})\ f\rho l} \quad \text{(Eq. 1)}$$

where:

D_{rads}	=	dose in rads
N	=	6.022 X 1023 molecules/mole
Δ(OD)	=	difference between the optical densities of irradiated and control samples

$\Delta\epsilon$	=	difference in molar extinction coefficients ($M^{-1}cm^{-1}$) between ferric and ferrous ions, at the wavelength used for the optical density measurement
$G(Fe^{3+})$	=	15.6, radiation-chemical reaction yield under given conditions
f	=	6.245 X 10^{13}eV/rad, conversion factor for transition from electron volts per milliliter units into rad
ρ	=	1.024, density of irradiated solution
l	=	1.0 cm, optical path length

The difference in molar extinction coefficients, $\Delta\epsilon$, is dependent upon the temperature. The following equation was used to calculate $\Delta\epsilon$[6]:

$$\Delta\epsilon \text{ @ } T_{meas} = (1 - .0069\ \Delta T)\epsilon_{25°C} \quad \text{(eq. 2)}$$

where:

T_{meas}	=	Room temperature during spectrophotometer use
ΔT	=	(25 °C - T_{meas})
$\epsilon_{25°C}$	=	2197 $M^{-1}cm^{-1}$

Dose versus Time was graphed for each of the eight positions within the irradiation fixture using the data from all nine exposures. The slope of the graph represents the dose rate. The results of the graph showed a strong linear relationship for all eight positions.

It was found that the larger R15I sensors indicated a lower dose rate. This corresponds to the larger volume, therefore greater self shielding, of the R15I sensors. The positions on the bottom tier of the irradiation fixture also display a larger dose rate. This coincides with the longer exposure time the bottom tier receives due to the raising and lowering of the irradiation chamber. Following are the dose rates for each sensor type and position within the irradiation fixture:

Position #	Model R15 Serial #	Dose Rate (R/min)	Model R15I Serial #	Dose Rate (R/min)
1	AL96	3300	AS26	3200
2	AL98	3300	AT01	3300
3	AL99	3400	AT02	3200
4	AM03	3300	AT04	3000
5	AM04	4300	AT05	4200
6	AM07	4300	AT07	4200
7	AM08	4200	AT09	4100
8	AM09	4200	AT10	4100

IRRADIATION

To ensure accurate results, the sensor systems were divided into two groups. The experimental group was exposed to radiation while the control group was not. Both groups were kept together, except during the actual irradiation process, to guarantee that any unavoidable background radiation received would be the same for each group.

An irradiation fixture map was maintained which recorded the serial number and position number for each sensor system to insure the sensor systems were placed in the same position during each irradiation. The cable connections on the outside of the sensors were aligned with permanent marks made on the irradiation fixture trays. This maintain the exact placement of each sensor system. Prior to each irradiation, the irradiation fixture was aligned to the same position within the irradiation chamber. The chamber door was closed and the chamber was lowered into the irradiate position. Each exposure was manually timed to irradiate the sensor systems to the desired dose range. After each irradiation, all sensor systems were returned to PAC for recalibration. The measured dose received was accumulated from all exposures.

CALIBRATION

The acoustic emission sensor calibrations were used to determine any degradation of sensor sensitivity. After each irradiation from the Cobalt-60 source the sensor systems were recalibrated . Both experimental and control groups were recalibrated and compared to the initial calibrations done after the sensor system assemblies and prior to exposure to radiation. In order to reduce sources of error, the calibration system was calibrated with a reference transducer prior to any test sensor calibrations to ascertain that the calibration system was in working order. Each calibration was done by the same individual.

The calibrations of the sensor systems proceeded as follows: The sensor system under test was placed in a holding fixture and a spring compression was adjusted to 50% of travel when the sensor systems was coupled to the pulsar. The pulsar converts electrical energy to mechanical energy and the sensor under test is placed face to face with the pulsar. The face of the sensor system and pulsar was gently wiped to remove any residue, and a thin film of mineral oil was applied to the pulsar. The sensor system was then placed on the pulsar and clamped into position. The calibration system began a sweep of the sensor systems' range. Graph paper was selected for the particular model under test, placed on the plotter with the paper reference points aligned and the results were plotted.

EXPERIMENTAL RESULTS AND ANALYSIS

The sensor systems have been exposed to twelve doses of radiation. The accumulated dose ranged from 2.23×10^6 to 2.9×10^6 Rads for the R15 systems. The R15I systems received an accumulated dose ranging from 2.03×10^6 to 2.84×10^6 Rads. The doses were given in increments that correspond to dose rates the systems would be subjected to during use on a Liquid Low Level Wasteline at Oak Ridge National Laboratory. By characterizing the waste to determine a dose rate in Rads per year, and using this information to calculate a time for each

exposure that represents a certain number of years of radiation exposure for the sensor systems, the systems were evaluated for their ability to perform for the thirty year life span of that particular wasteline.

All twelve calibrations of each sensor system have been compared to their initial calibrations. The sensitivity of each sensor was examined at 127 kHZ. This approaches the resonance frequency of the sensors. The only visible sign of change to the sensor systems exposed to radiation has been a color change, from clear to amber, of the epoxy bond. The epoxy bond on the control sensor systems have maintained the original color and show no signs of degradation.

The R15I sensor systems, which have received an accumulated dose of up to 2.84×10^6 Rads, have performed well. Seven of the eight experimental sensor systems are still performing within the accepted ASTM guidelines[7] of plus or minus a 3 dB change in sensitivity. Serial # AT02 had a sensitivity drop of 3 dB which was noted during recalibration after the fourth exposure. The accumulated dose for this sensor system when the deterioration occurred was 4.31×10^5 Rads. To insure that the dose rates for the individual positions within the irradiation chamber were not altered by removing one of the experimental sensor systems, the deteriorated sensor system continued to be irradiated and recalibrated. It was noted during the next calibration that the sensitivity had come back within the acceptable range. The sensitivity of this particular sensor system continued to move in and out of the acceptable range during the following irradiations.

The R15 sensor systems have also performed well. Six of the eight experimental sensor systems are still operating within the acceptable range and five of the six have shown a gain in sensitivity. Deterioration of two sensor systems was observed during recalibration after the seventh exposure. Serial # AM03, after exposure to 1.11×10^6 Rads, showed a sensitivity drop of 9.6 dB and serial # AM09, after an exposure to 1.42×10^6 Rads, experienced a drop of 4.5 dB. The sensor systems continued to be irradiated after deterioration to insure the flux within the irradiation chamber remained the same. During the next calibration it was noted that AM09 gained back 2.5 dB of sensitivity putting it back within the acceptable range and has remained so through out the following calibrations. AM03 showed a 4.5 dB gain in sensitivity during the next calibration but still remained outside of the acceptable range where it has remain through out subsequent recalibrations.

Following the twelfth recalibration the epoxy bond on AM03 failed while it was being wiped clean of mineral oil used at the sensor-to-pulsar interface. The bond broke cleanly away from the waveguide and remained attached the ceramic plate located on the face of the sensor. The bonding material showed an amber, uniform discoloration. When the bonding material was touched gently it disintegrated leaving the ceramic plate free of bonding material. The ceramic plate was also observed to show the same amber discoloration, however, close inspection revealed no other visible signs of deterioration. The bonds on the remaining sensor systems were checked and appear to be stable.

After the bond failed on the R15 - AM03 sensor, the sensor alone was recalibrated. This calibration was compared to the original one done when the sensor was manufactured. The

calibration curves were identical and support a conclusion that the loss in sensitivity appeared to occur because of the deterioration of the bonding agent and not the sensor itself.

SUMMARY AND CONCLUSION

The experimental results demonstrate the acoustic emission sensor systems ability to withstand significant exposure to gamma radiation. Application of the sensor systems in radioactive environments can be advocated up to the limits established in this experiment. A significant number of sensors in the experimental group are still functioning within the acceptable range of sensitivity after an accumulated dose of up to 2.9×10^6 Rads. Characterizing the waste traveling through the doubly contained pipe lines to the Melton Valley Storage Area and calculating a dose over its thirty year life span using a point source, which would represent a worst case scenario, a thirty year dose is approximately 1.7×10^6 Rads. This is well within the range that a significant number of the sensor systems have withstood thus far without detectable deterioration.

Experimentation will continue until the ultimate radiation damage threshold is reached for a significant number of sensor systems. Extended use of acoustic sensors in high radiation fields should be investigated by dose and type of exposure to ensure predictable life expectancies and performance parameters are maintained.

ENDNOTES

1. S.L. HALVERSON, T.T. ANDERSON, A.P. GAVIN, AND T. GRATE, "Radiation Exposure Of A Lithium Niobate Crystal At High Temperatures", Argonne National Laboratory
2. ASTM Designation E650-85, "Standard Guide for Mounting Piezoelectric Acoustic Emission Sensors"
3. ASTM Designation E976-84, "Standard Guide for Determining the Reproducibility of Acoustic Emission Sensor Response"
4. HUGO FRICKE AND EDWIN J. HART, "Radiation Dosimetry, Chapter 12 - Chemical Dosimetry", Academic Press (1966)
5. HUGO FRICKE AND EDWIN J. HART, "Radiation Dosimetry, Chapter 12 - Chemical Dosimetry", Academic Press (1966)
6. KARL SCHARF AND RICHARD M. LEE, "Investigation of the Spectrophotometric Method of Measuring the Ferric Ion Yield in the Ferrous Sulfate Dosimeter", Radiation Research, vol. 16, 115-124 (1962)
7. ASTM Designation E976-84, "Standard Guide for Determining the Reproducibility of Acoustic Emission Sensor Response"

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