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CORROSION-INDUCED ACOUSTIC EMISSIONS FROM URANIUM 4.5-WEIGHT PERCENT NIOBIUM BINARY ALLOY

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Research and Development
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SUBJECT DESCRIPTORS

Corrosion
Stress-Corrosion Cracking
Acoustic Emission
Binary

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ATOMICS INTERNATIONAL DIVISION
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CORROSION-INDUCED ACOUSTIC EMISSIONS FROM URANIUM 4.5-WEIGHT PERCENT NIOBIUM BINARY ALLOY

Richard Mah, Robert L. Kochen, and James M. Macki

Abstract. The ability to monitor corrosion and stress-corrosion cracking of uranium 4.5-weight percent niobium was investigated. The acoustic emissions from stress-corrosion cracking were monitored using smooth four-point bend specimens, which were immersed in oxygen-saturated water containing chloride ions. The acoustic emissions from corrosion were monitored from rectangular corrosion coupons. These latter specimens were exposed to nitrogen-saturated and oxygen-saturated solutions of distilled water and distilled water with chloride ions. Dilute solutions of hydrochloric acid were also used. Findings show that stress-corrosion crack monitoring of the alloy was successful. Corrosion monitoring of the alloy showed a direct correlation between corrosion rate (based on weight loss) and total acoustic emissions. For a specific range, one can calculate the corrosion rate of uranium alloys in a corrosive solution from the total acoustic emissions generated. Studies to determine the source of corrosion-induced acoustic emissions were inconclusive.

INTRODUCTION

The usefulness of applying acoustic emission monitoring for the early detection of stress-corrosion cracking (SCC) has recently been given more recognition.[1,2] The source of these emissions has been attributed to a sudden release of elastic energy associated with crack growth[3] and the emission of elastic waves accompanying plastic deformation.[4] Initially, the purpose of this investigation was to determine the applicability of using acoustic emission to detect the onset of stress corrosion cracking in uranium 4.5-weight percent (wt %) niobium (Nb).

Specimens mounted in a four-point bend device, stressed to 85 percent of their yield strength, and subjected to an environment of oxygen-saturated water containing 50 parts per million (ppm) chloride (Cl^-) ions, were monitored for acoustic emissions. Initial results showed that appreciable acoustic emissions were detected in less than one-tenth the time to failure. However, emissions continued after the specimen had broken as noted in Figure 1, (darkened lines represent specimen break). Another source of these acoustic emissions appeared to come from unstressed material. Testing continued on unstressed specimens exposed to varying concentrations of Cl^-, with and without oxide coatings, and nitrogen (N_2) or oxygen (O_2) bubbled through the solution. The corrosion rates of several of the specimens were continually monitored.

The objective of the investigation was to (1) determine the source of these acoustic emissions, (2) determine what factors will affect these acoustic emissions, and (3) find if any correlation existed between these acoustic emissions and corrosion rates.

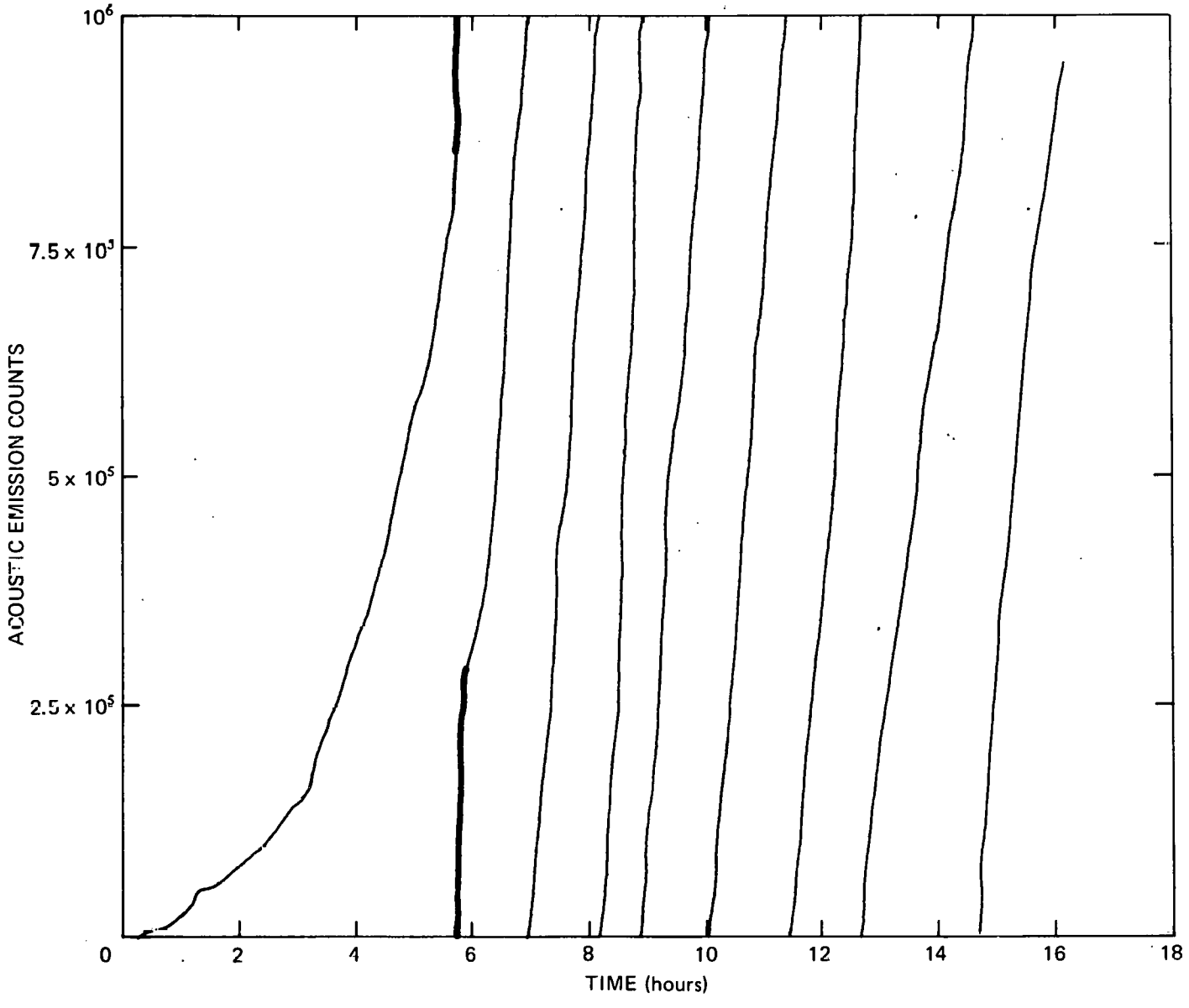
[1] W. W. Gerberich and C. E. Hartbower. "Monitoring Crack Growth of Hydrogen Embrittlement and Stress Corrosion Cracking by Acoustic Emission." *Conference Proceedings of Fundamental Aspects of Stress Corrosion Cracking.* Ohio State University, Columbus, Ohio. Published by National Association of Corrosion Engineering, Houston, Texas. 1969.

[2] D. O. Harris and H. L. Dunegan. "Preliminary Investigation of Acoustic Emission Monitoring of Stress Corrosion Cracking of Aluminum Alloy 7004," Internal report of the Dunegan Research Corporation, San Juan Capistrano, California, February 1971.

[3] C. E. Hartbower, W. W. Gerberich, and H. Liebowitz. "Investigation of Crack Growth Stress-Wave Relationships." Pages 291-308. *Engineering Fracture Mechanics,* Volume 1. Pergamon Press, New York. 1968.

[4] H. L. Dunegan and A. S. Tetelman. "Nondestructive Characterization of Hydrogen-Embrittlement Cracking by Acoustic Emission Techniques." Pages 387-402. *Engineering Fracture Mechanics,* Volume 2. Pergamon Press, New York. 1971.

FIGURE 1. Stressed Uranium 4.5-Weight Percent Niobium Specimen Immersed in Oxygenated Water with 50 Parts per Million Chloride.



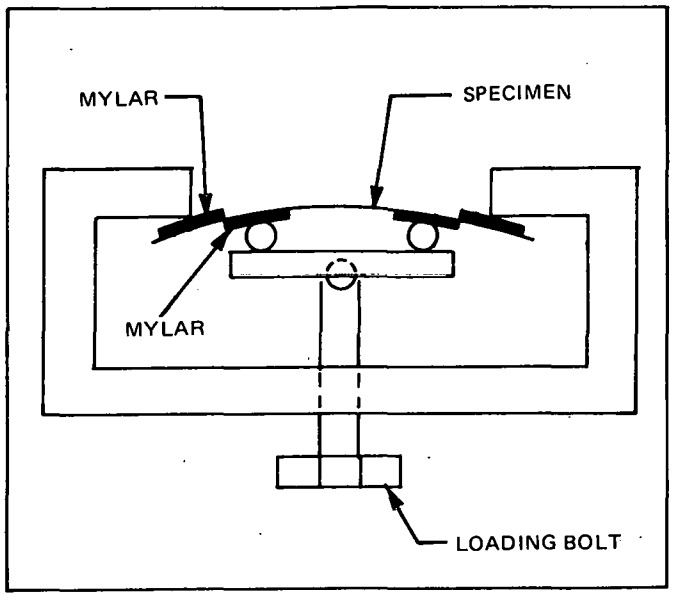


FIGURE 2. Four-Point Bend Stressing Device.

DISCUSSION

Experimental Procedure:

The specimens were uranium 4.5-wt % Nb with a two-year oxide coating. These specimens were solution-quenched and aged for 80 hours at 260 °C.

Test Monitoring for Stress-Corrosion Cracks:

Using Type-316 stainless steel, four-point bending fixtures (Figure 2), the specimens were then loaded to a stress-level equivalent to 85 percent of the yield strength. The load frame with specimen was then immersed into an oxygen-saturated water solution containing 50 ppm Cl^-. Two single-ended Dunegan transducers were used; mounting one on the specimen and one on the loading fixture to monitor for acoustic emissions. Amplifier gains of 93 and 85 decibels (dbs) with a high-pass filtering of 100 kilohertz (kHz) were used. Failure was expected to occur within 3 to 5 hours.[5, 6]

Corrosion Test Monitoring:

Unstressed uranium specimens with an oxide (built-up over a 2-year storage) and without an oxide (specimens belt-sanded on 220 grit paper) were immersed in water containing 5, 25, 50, and 100 ppm Cl^-. Both oxygen and nitrogen were alternately bubbled through the solution. These specimens (0.1 X 0.75 X 6-inches) were each monitored with two Dunegan single-ended transducers set at 93-db gain and a high-pass filtering of 100 kilohertz.

[5] J. M. Macki and R. L. Kochen. "*The Stress-Corrosion Cracking Behavior of the U-4.2 wt % Nb Alloy Aged 80 Hours at 260 °C.* USAEC RFP-1824, Rocky Flats Division, Dow Chemical U.S.A., Golden, Colorado. March 28, 1972.

[6] James M. Macki and Robert L. Kochen. "Technical Note: Stress Corrosion Cracking of Aged U-4.5 wt % Nb." *Corrosion* 29:153-156. April 1974.

A second series of tests was conducted with belt-sanded specimens to determine whether tensile residual stresses in the alloy could cause micro-cracking on the surface and thus acoustic emissions. These specimens were monitored before and after shot peening in both distilled water and water containing 100 ppm Cl^-. The shot-peening process should induce compressive residual stresses. These compressive stresses should either eliminate or greatly reduce the possibility of microcracking on the surface and acoustic emissions produced.

A third series of tests was conducted with unstressed specimens without oxide coatings immersed in 0.1, 0.5, and 1N HCl (hydrochloric) solutions. Oxygen was bubbled through the solution and the gravimetric corrosion rates of the specimens were recorded along with the total number of acoustic emissions.

Results:

Initial tests with unstressed specimens revealed that specimens immersed in chloride solutions definitely produced more acoustic emissions than those in distilled water (Figure 3). Theorizing that the microcracking of the oxide could be the source of these emissions, tests conducted with and without the oxide gave the results shown in Figures 4 through 7. The alternate bubbling of nitrogen and oxygen through the solution possibly had a decreasing or increasing effect (or both) on the growth of the oxide. The variation in chloride concentrations was to determine whether a chloride threshold level existed that would cause these acoustic emissions. Initial results reveal that the oxide has no effect on the acoustic emissions. Long-term testing also showed that even concentrations as low as 5 ppm chloride caused corrosion-induced acoustic emissions.

One specimen exposed to 10 ppm Cl^- in distilled water emitted many acoustic emissions until silver nitrate was added to precipitate the chloride content. After the precipitation was completed, the emissions ceased (Figure 8). Following Figures 9 and 10, Figures 11 and 12 show that microcracking due to tensile residual stresses had no direct correlation with the total acoustic emission output. In one case, the specimen emitted more acoustic emissions after shot peening and the opposite occurred in the other case.

Several specimens immersed only in hydrochloric acid solutions were monitored for acoustic emissions and corrosion rates. An increase in corrosion rate appeared to produce an increase in acoustic emissions (Figure 9).

One result, however, did not agree and it was theorized that the cause could only be attributed to a different mechanism of corrosion (i.e., pitting, general corrosion, etc.).

Using a stress-corrosion cracking frame, stressed specimens were monitored for acoustic emissions with the gain set of 85 db. This gain was the level found where no corrosion induced emissions had been detected. The results showed that emissions were still detected within 6 percent of failure with zero emissions after failure (Figure 10).

CONCLUSIONS

From the test results, the conclusions are:

1. Corroding uranium specimens emit some form of acoustic emission.
2. Oxide coatings appear to have no effect on acoustic emissions.
3. No correlation appears to exist between nitrogen (N_2) and oxygen (O_2) in the reaction causing acoustic emissions.
4. Residual stresses on the surface which may cause microcracking in corrosive environments have no effect on the acoustic emission output.
5. For certain corrosion rates, a correlation appears evident between acoustic emissions and corrosion rates.
6. The monitoring of stress-corrosion cracking specimens for acoustic emissions appears to be of value even with the amplifier gain set below the threshold of corrosion-induced emissions.

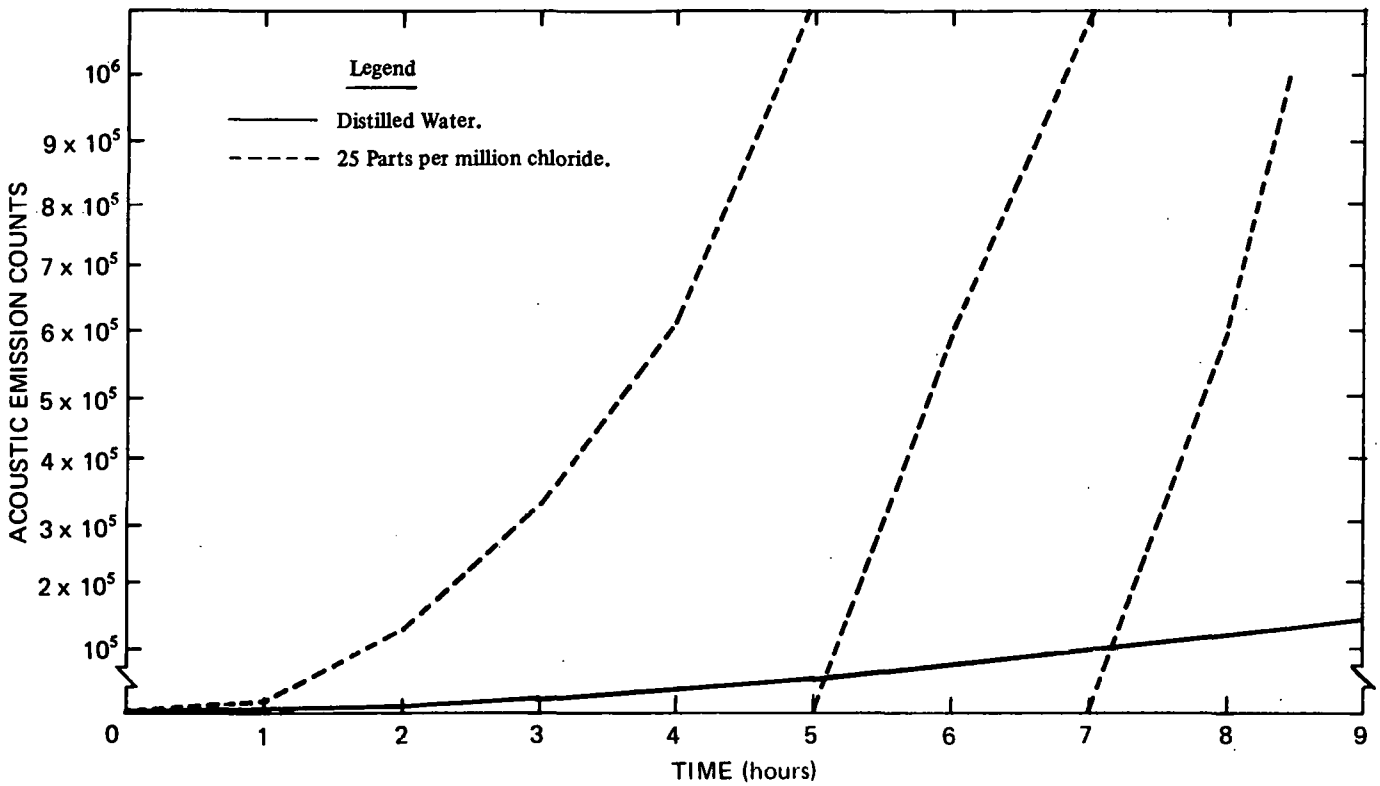
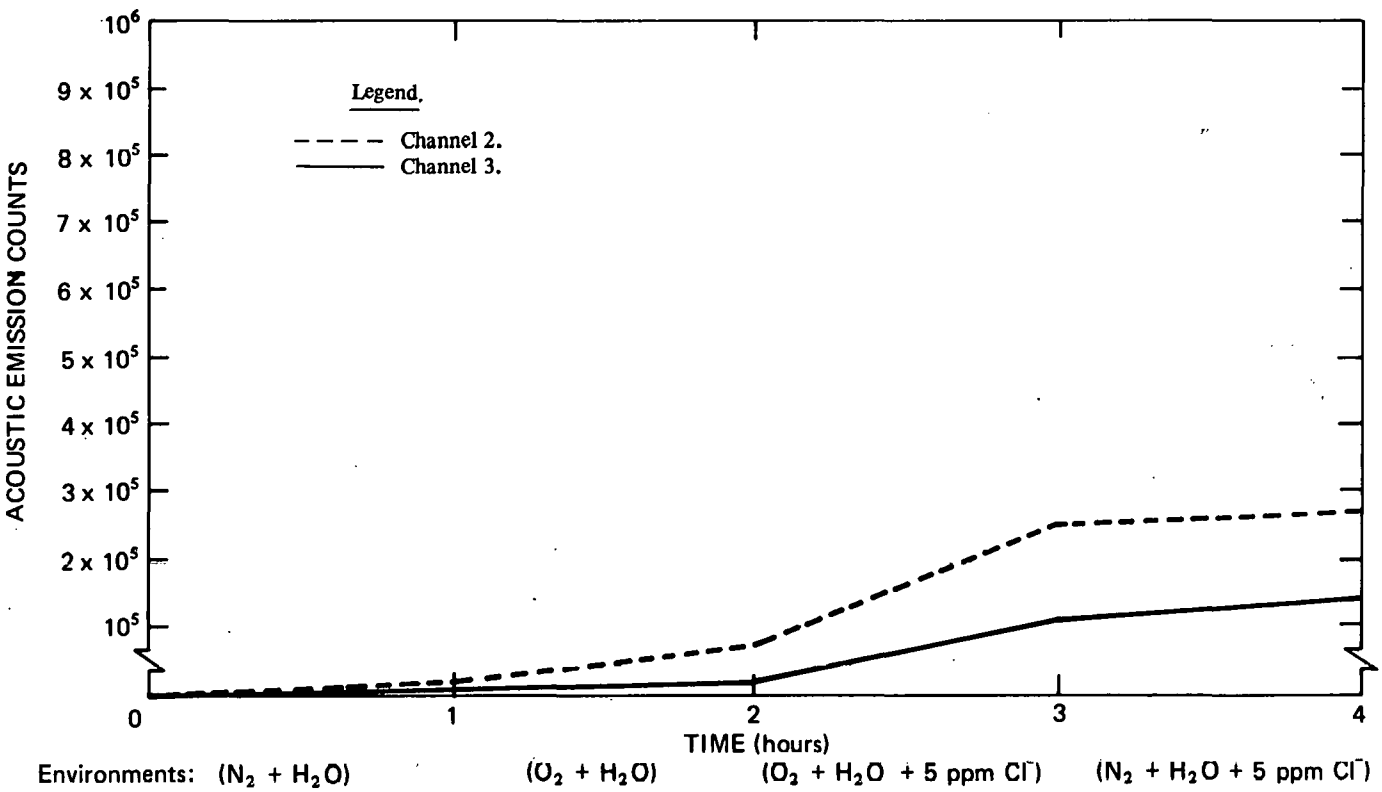


FIGURE 3. Unstressed Uranium 4.5-Weight Percent Niobium Immersed in Distilled Water and Oxygenated Distilled Water with 25 Parts per Million Chloride.

FIGURE 4. Unstressed Uranium 4.5-Weight Percent Niobium with an Oxide Coating.



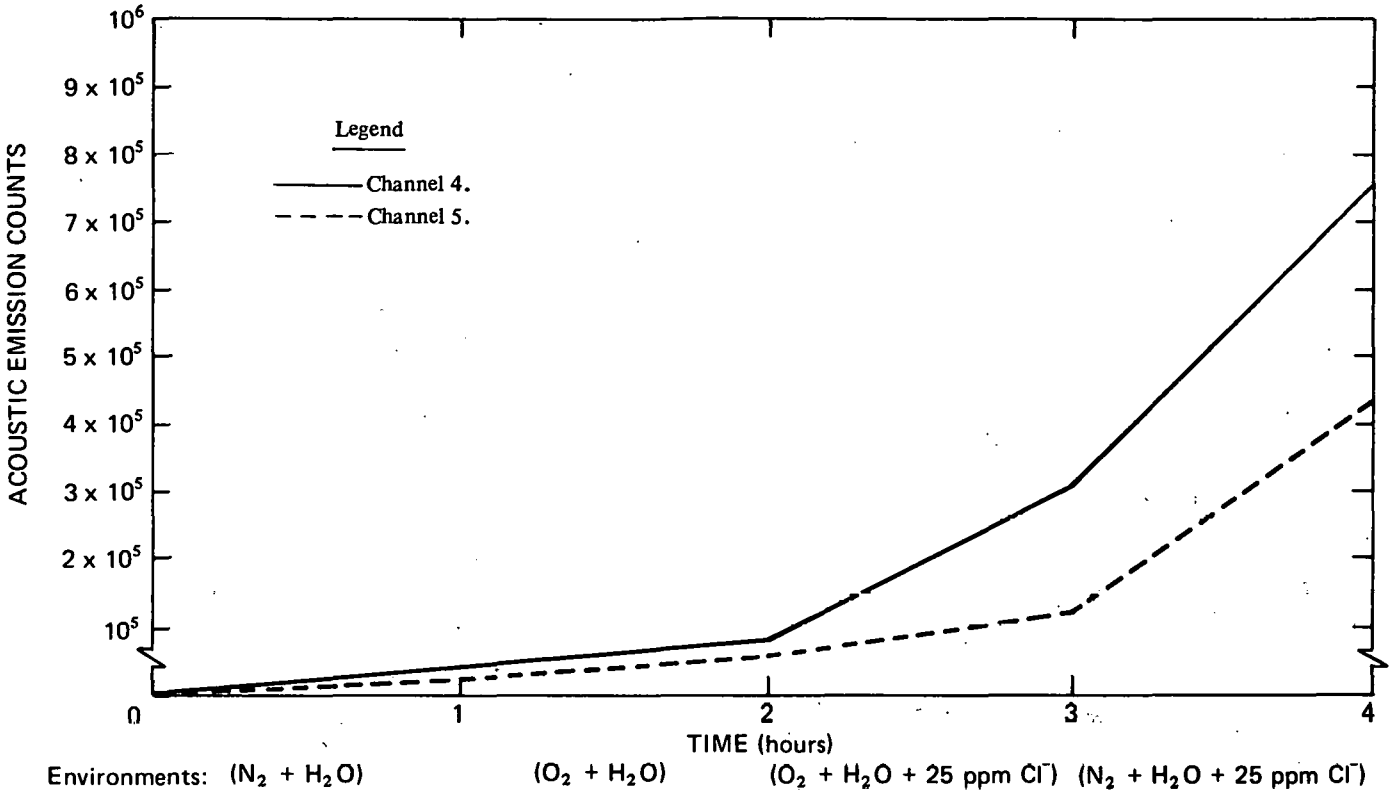
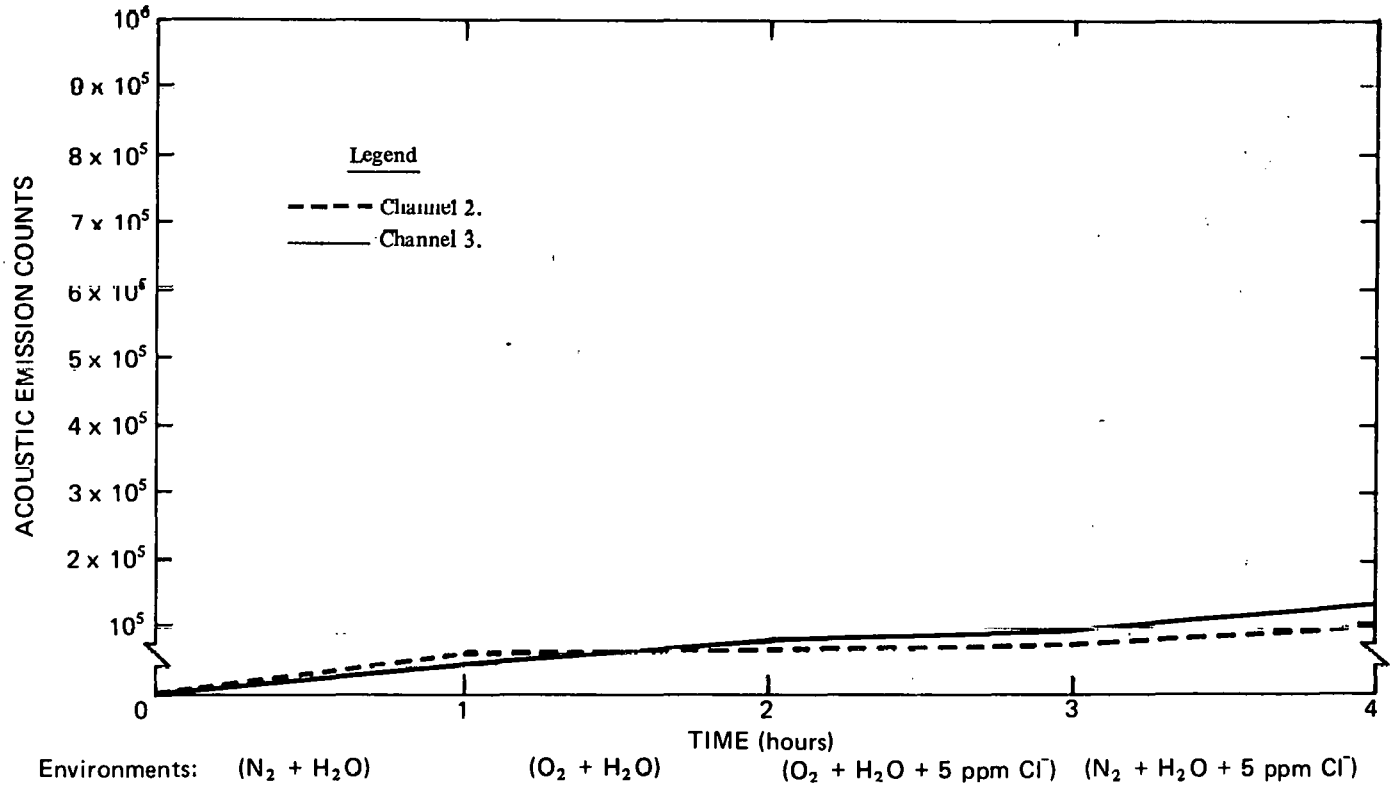


FIGURE 5. Unstressed Uranium 4.5-Weight Percent Niobium with an Oxide Coating.

FIGURE 6. Unstressed Uranium 4.5-Weight Percent Niobium after Oxide Removal.



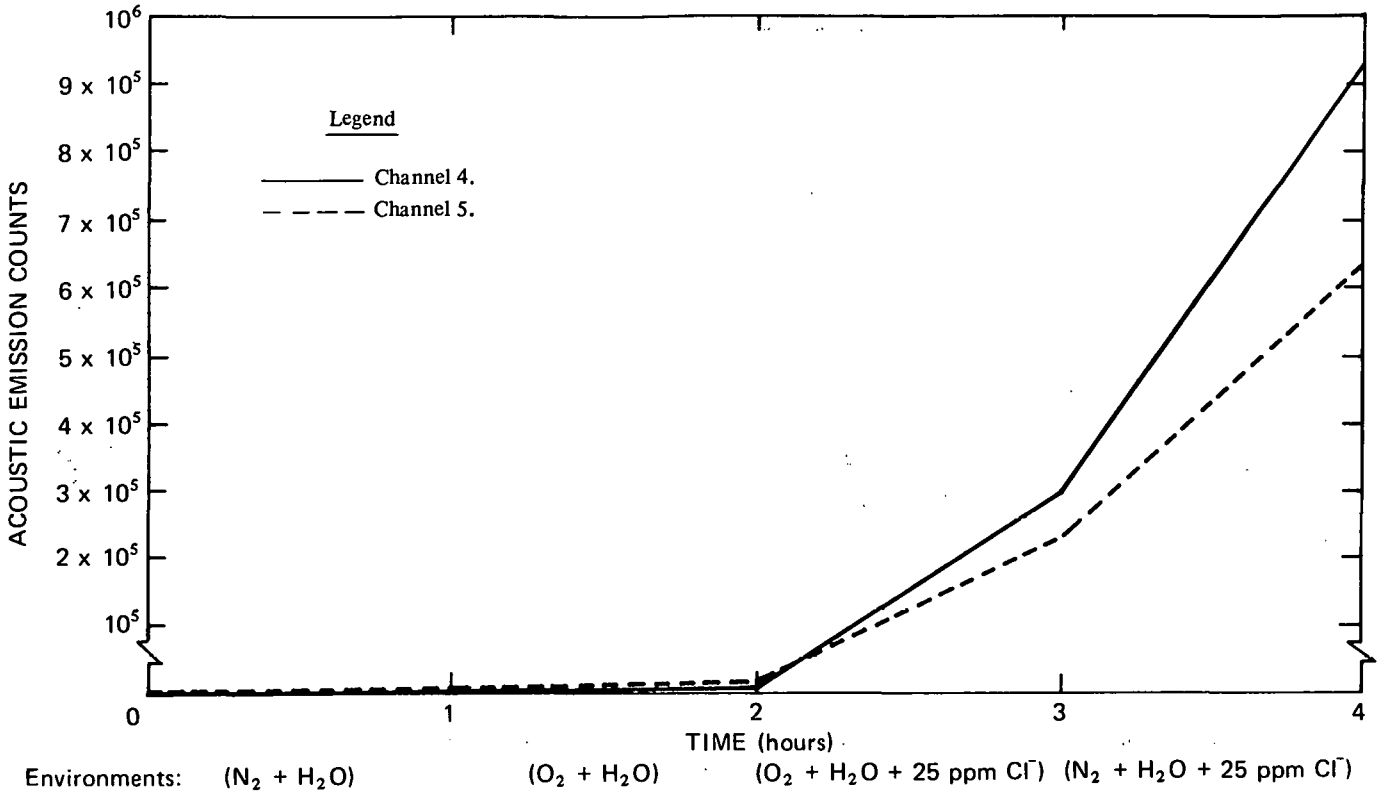
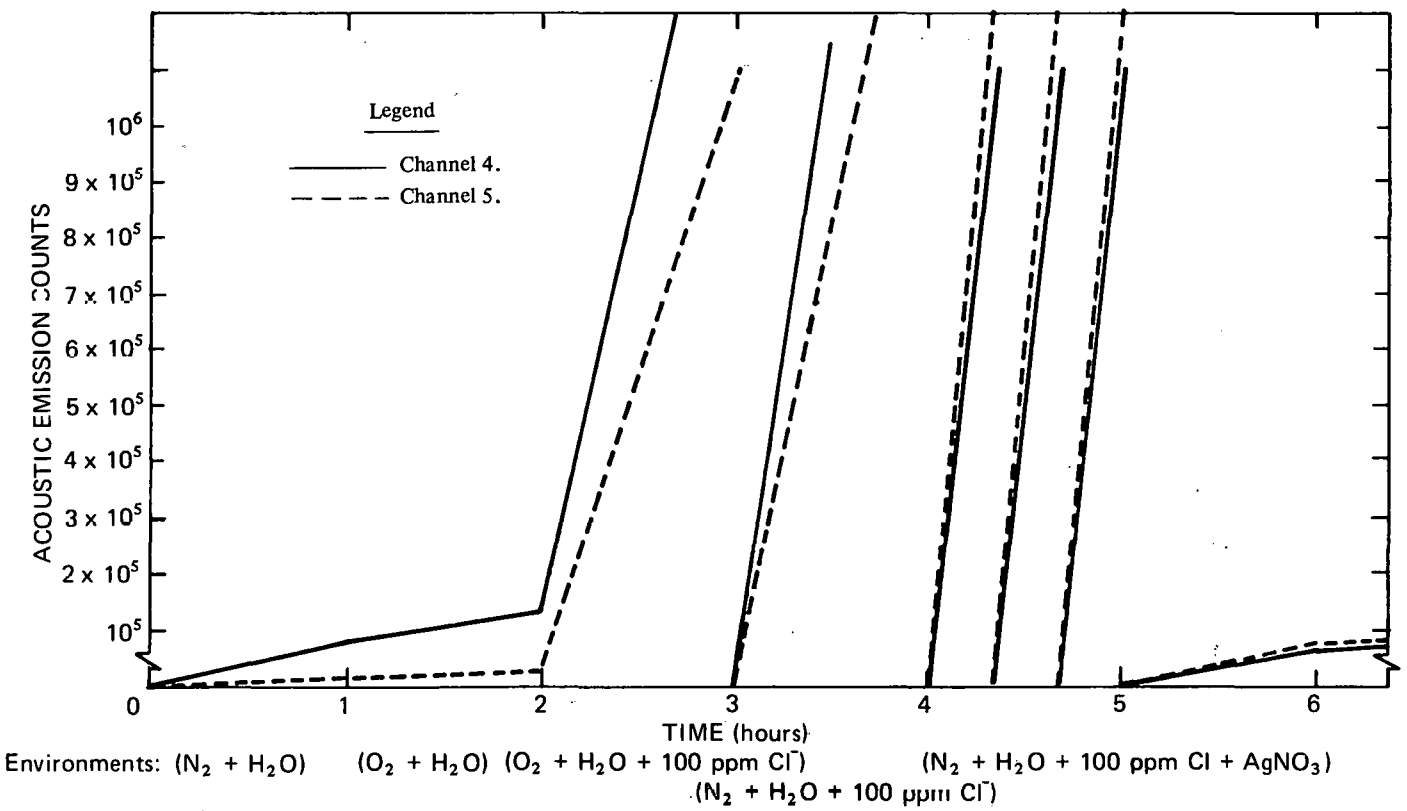


FIGURE 7. Unstressed Uranium 4.5-Weight Percent Niobium after Oxide Removal.

FIGURE 8. Unstressed Uranium 4.5-Weight Percent Niobium after Oxide Removal.



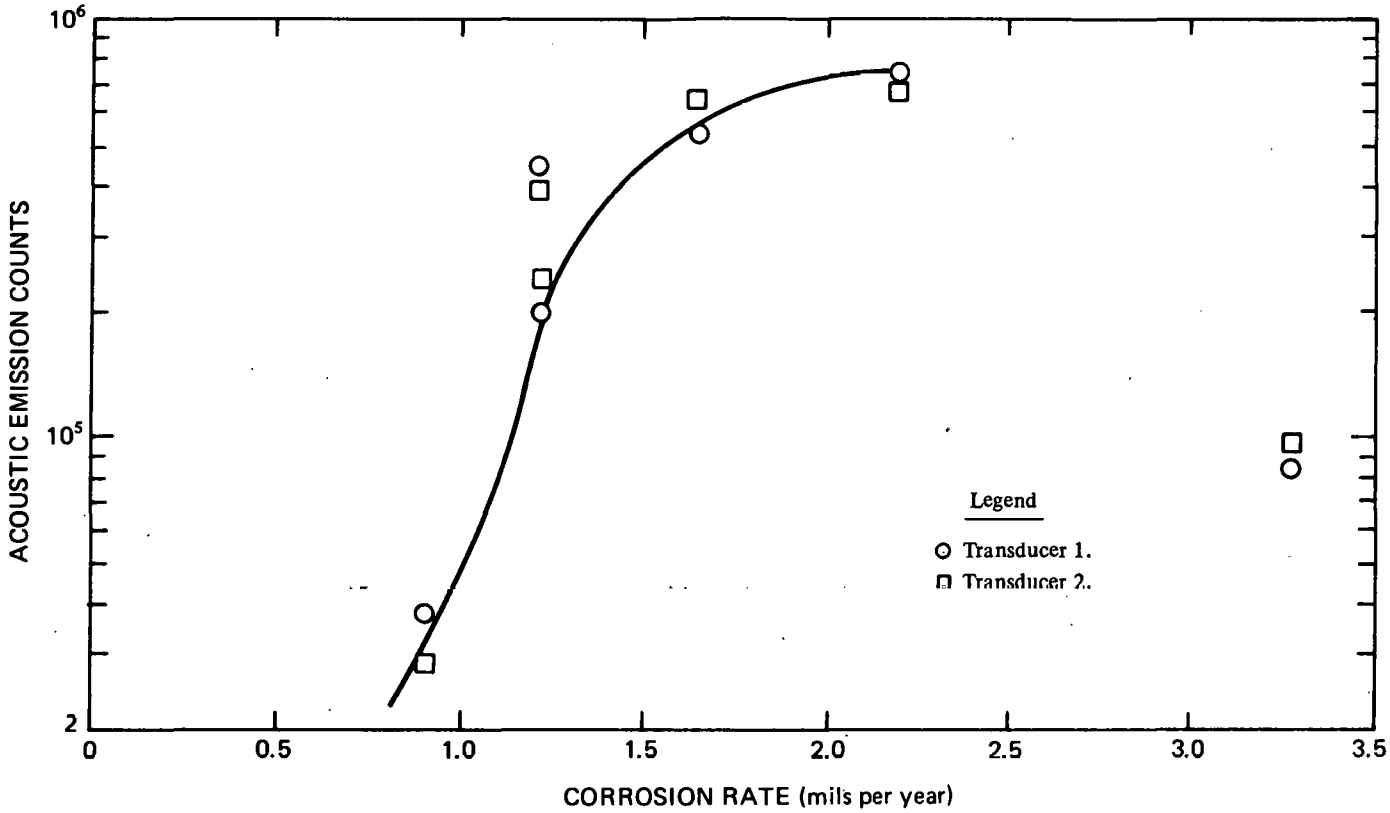
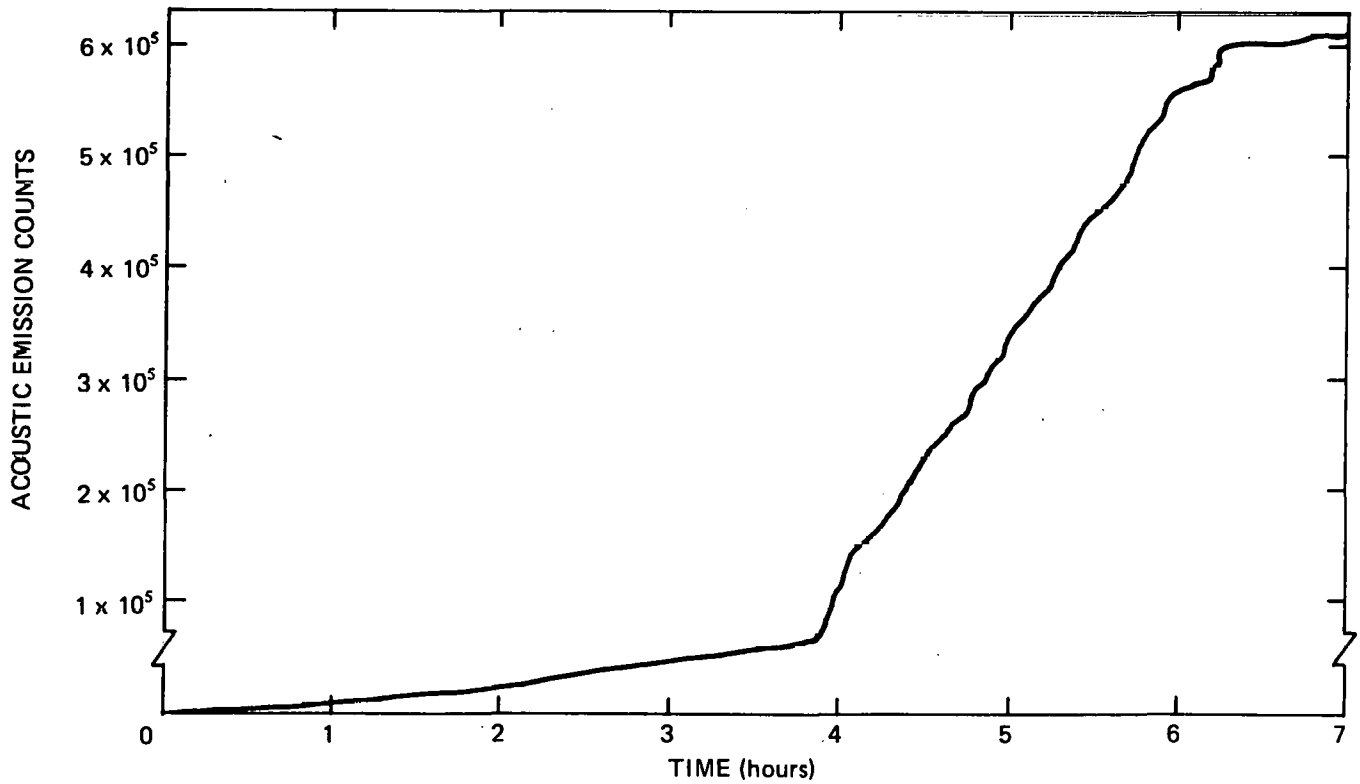


FIGURE 9. Gravimetric Corrosion Rates versus Acoustic Emissions for Uranium 4.5-Weight Percent Niobium.

FIGURE 10. Stressed Uranium 4.5-Weight Percent Niobium Undergoing Stress-Corrosion Cracking in Oxygenated Water with 50 Parts per Million Chloride.



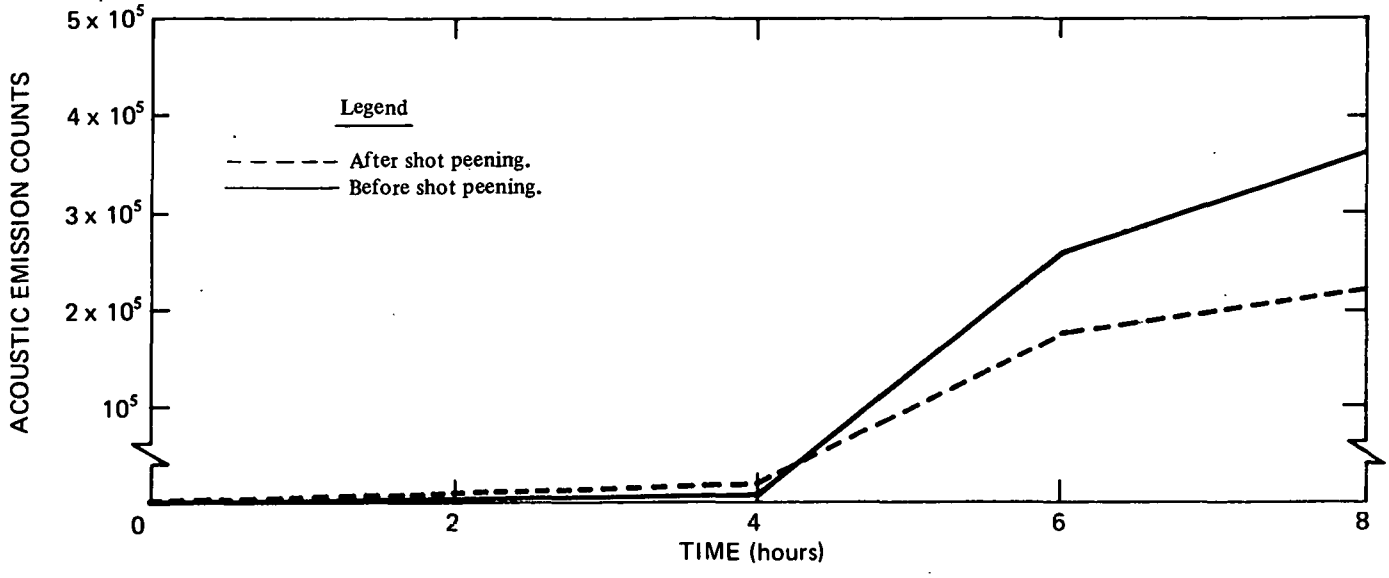
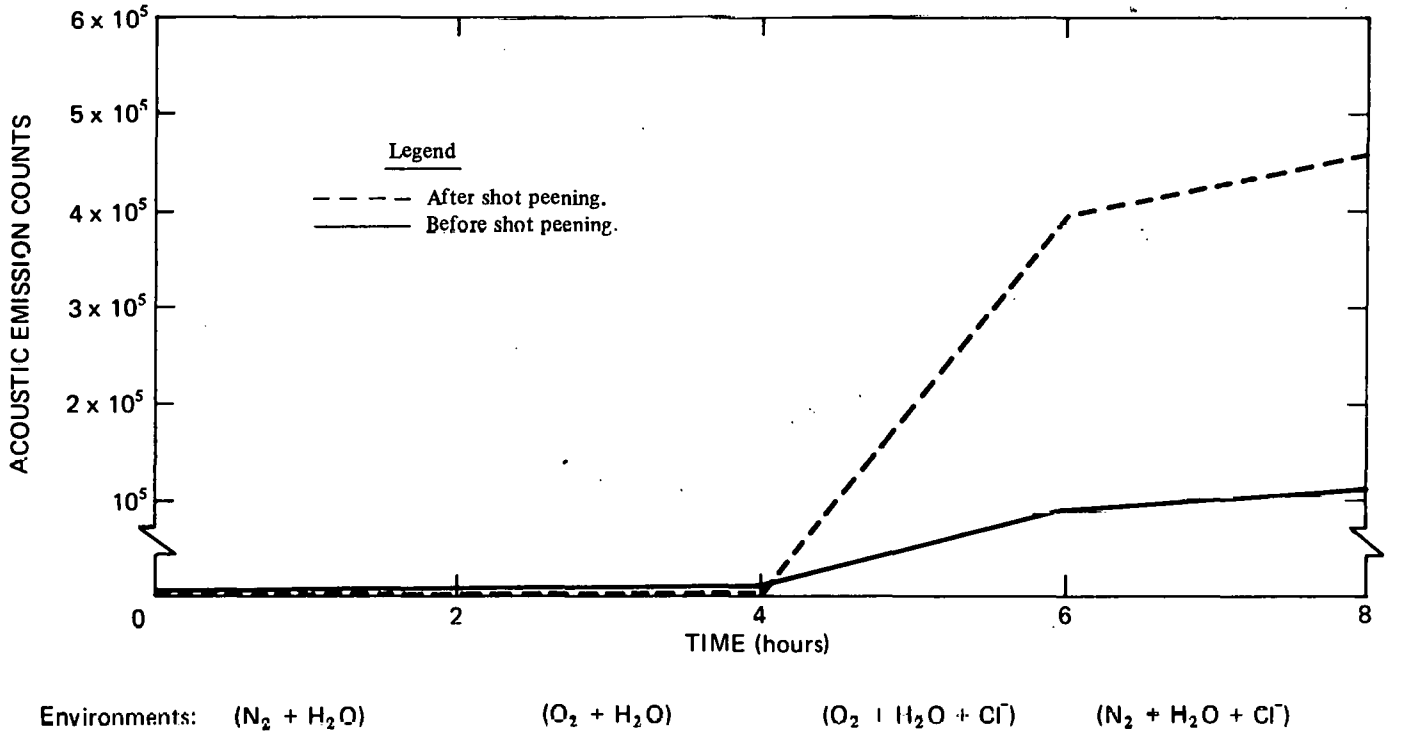


FIGURE 11. Corrosion-Induced Acoustic Emissions from Uranium 4.5-Weight Percent Niobium.

FIGURE 12. Corrosion-Induced Acoustic Emissions from Uranium 4.5-Weight Percent Niobium.



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