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Comparison Of Cleaning Methods For Analysis Of Underground Beryllium Corrosion

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COMPARISON OF CLEANING METHODS FOR ANALYSIS OF UNDERGROUND BERYLLIUM CORROSION

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ABSTRACT

The subsurface radioactive disposal site located at the Idaho National Laboratory contains neutron-activated beryllium metals from non-fuel nuclear-reactor-core components. A long-term underground corrosion test is being conducted to obtain site-specific corrosion rates of the disposed beryllium to support efforts to more accurately estimate the transfer of activated elements in the surrounding arid vadose zone environment. During the corrosion analysis, two cleaning methods were used. This paper describes the cleaning methods and presents a comparison of the results.

Keywords: beryllium, vadose zone, neutron-activated metals, nuclear reactor components, underground corrosion.

INTRODUCTION

The long-term corrosion test is designed to assist in the determination of site-specific corrosion rates of neutron-irradiated beryllium buried in an arid vadose zone environment at the radioactive disposal site at Idaho National Laboratory. Corrosion rates are based on mass loss from nonradioactive beryllium coupons exposed to underground site conditions.[1] The corrosion rates, once determined, reduce the uncertainty of the site-specific transfer of radioactive isotopes to the environment (radiological release rates). Of interest are the disposed beryllium reflector blocks and outer shim control cylinders. These components, when exposed to high neutron fluxes in a reactor environment, become activated with long-lived radioactive isotopes. After disposal, corrosion processes can cause these radioactive isotopes to be released from the irradiated beryllium waste to the environment.

The long-term corrosion testing implemented direct corrosion testing (i.e., burying beryllium coupons in the soil).[2,3,4,5] Previous beryllium corrosion studies do not include underground corrosion testing.[6] For this study, the corrosion analysis applies Test Method ASTM G 1-03[7] for cleaning and mass loss determination, but beryllium is a metal excluded from the ASTM G 1 procedures. During the corrosion analysis, two cleaning methods were used: ASTM G 1-C.5.1 *Magnesium and Magnesium Alloys* and ASTM G 1-C.1.1 *Aluminum and Aluminum Alloys*. This paper presents a comparison of the results.

EXPERIMENTAL PROCEDURE

The long-term corrosion testing implements direct corrosion testing (i.e., burying beryllium coupons in the soil, ASTM G 4). The beryllium corrosion coupons are retrieved after exposure to the underground environment. Following the procedures from Test Method ASTM G 1, the beryllium corrosion product and adjacent soils tightly adheres to the metal base requiring chemical cleaning after the simple brush-wash and rinse cycle. Figures 1 and 2 show the corroded beryllium coupon after exposure to soil environment and after cleaning.

Based on the beryllium manufacturer's recommendations, two chemical cleaning procedures are applicable for cleaning the beryllium corrosion coupons: ASTM G 1-C.5.1 *Magnesium and Magnesium Alloys* and ASTM G 1-C.1.1 *Aluminum and Aluminum Alloys*. Each of these procedures was used during the study. Following the procedures, multiple cleaning cycles were performed on corroded coupons and blank (archived) coupons until mass loss results indicated the successful removal of the corrosion products. Microscopic examination followed the cleaning process to visually verify the results. In addition, archived coupons (in an as-received state from the manufacturer) and cleaned coupons (from each of the two chemical procedures) were then further examined using the scanning electron microscope and compared.

RESULTS

The exposed beryllium coupons were cleaned following ASTM G 1. First cycle cleaning, water wash and brush, did not remove all of the adhering corrosion products. Chemical cleaning was then applied to all exposed coupons following ASTM G 1-C.5.1 *Magnesium and Magnesium Alloys* or ASTM G 1-C.1.1 *Aluminum and Aluminum Alloys*. Figure 3 shows the typical cleaning process for the beryllium coupons. The cleaning curves do not indicate a difference in method effectiveness.

To establish another basis of comparison, archived beryllium coupons were examined using the scanning electron microscope. The archived beryllium coupons provide the baseline. Baseline scans are shown in Figures 4 and 5 and the baseline spectrum is shown in Figure 6. The spectrum clearly shows peaks of the elemental impurities of aluminum, silicon, and iron.

The first set of beryllium corrosion coupons were chemically cleaned following the ASTM G 1-C.5.1 *Magnesium and Magnesium Alloys* procedure. Based on cleaning curves and visual examination with a microscope, corrosion products were successfully removed using this procedure, Figure 7. ASTM G 1-C.5.1 uses silver chromate to precipitate chloride as a silver salt. Upon closer examination using the scanning electron microscope, residual, crystalline precipitates remain in the corroded areas after thorough cleaning; Figures 8 and 9. Comparing the baseline spectrum (Figure 6) to the coupon spectrum (Figure 10), verifies the precipitates are identifiable as silver chloride. This precipitate strongly adheres to the beryllium base metal even after multiple cleaning cycles.

The second set of beryllium corrosion coupons were chemically cleaned following the ASTM G 1-C.1.1 *Aluminum and Aluminum Alloys* procedure. Based on cleaning curves and visual examination with a microscope, corrosion products were successfully removed using this procedure, Figure 11. The coupons were more closely examined using the scanning electron microscope, Figures 12 and 13. By comparing the baseline spectrum (Figure 6) to the coupon spectrum (Figure 14), residual phosphorous does remain on the coupon from the cleaning solution. However, the coupon is free from any residual precipitates.

CONCLUSIONS

Two procedures were used to clean beryllium corrosion coupons after underground exposure: ASTM G 1-C.5.1 *Magnesium and Magnesium Alloys* and ASTM G 1-C.1.1 *Aluminum and Aluminum Alloys*. Both procedures resulted in coupons clean of adhering soils and corrosion products. The coupons were then further examination with scanning electron microscopy and compared with an archived coupon. Both cleaning procedures left residuals from the cleaning solutions, but the coupons cleaned using ASTM G 1-C.5.1 *Magnesium and Magnesium Alloys* left silver chloride precipitates predominately in the corroded areas of the beryllium. Based on the results of this study, ASTM G 1-C.1.1 *Aluminum and Aluminum Alloys* is the preferred procedure for chemically cleaning underground beryllium corrosion coupons.

ACKNOWLEDGEMENTS

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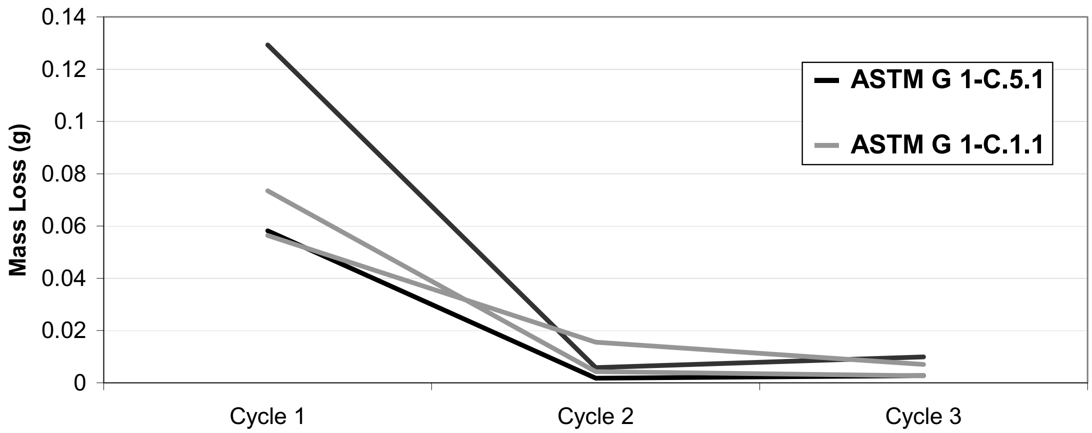
FIGURE 1 - Corroded Beryllium Coupon after Underground Exposure

FIGURE 2 - Corroded Beryllium Coupon after Cleaning



Method	Solution Chemistry	Cleaning Time	Temperature
C.5.1	150 g chromium trioxide 10 g silver chromate Reagent water to make 1L	1 minute	boiling
C.1.1	50 ml phosphoric acid 20 g chromium trioxide Reagent water to make 1L	10 minutes	90 C

FIGURE 3 – Beryllium Coupon Cleaning Curves and Procedures

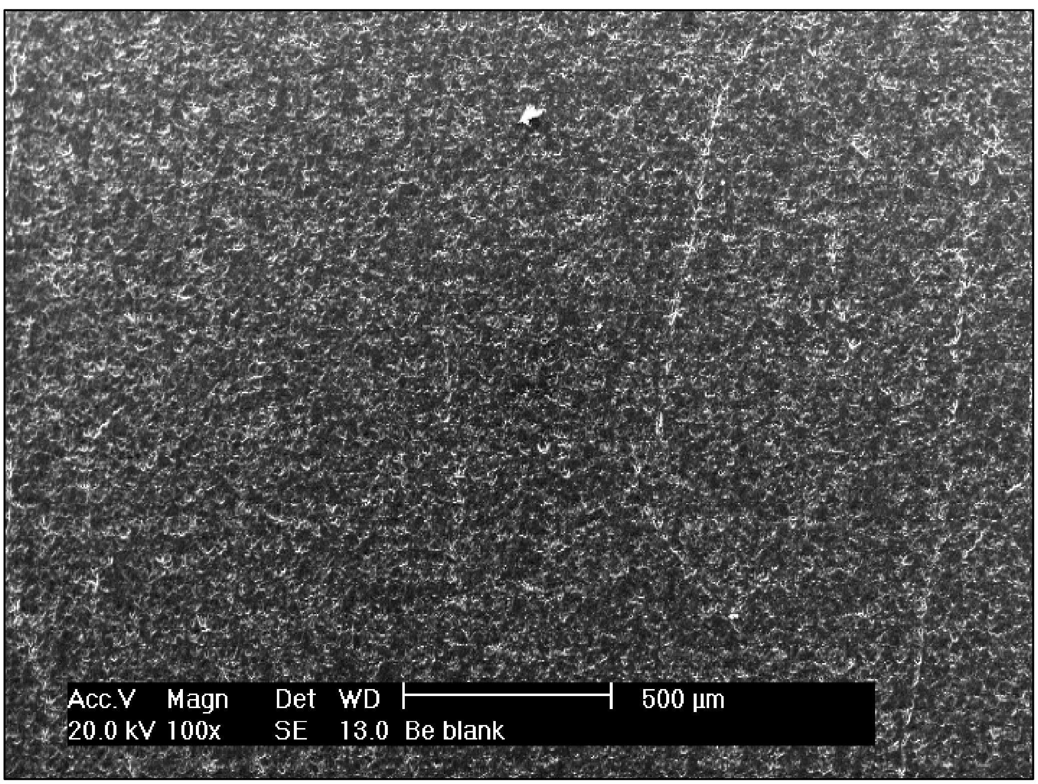


FIGURE 4 - Archived Beryllium Coupon (As-Received Condition), Scanning Electron Microscope Image 500 µm

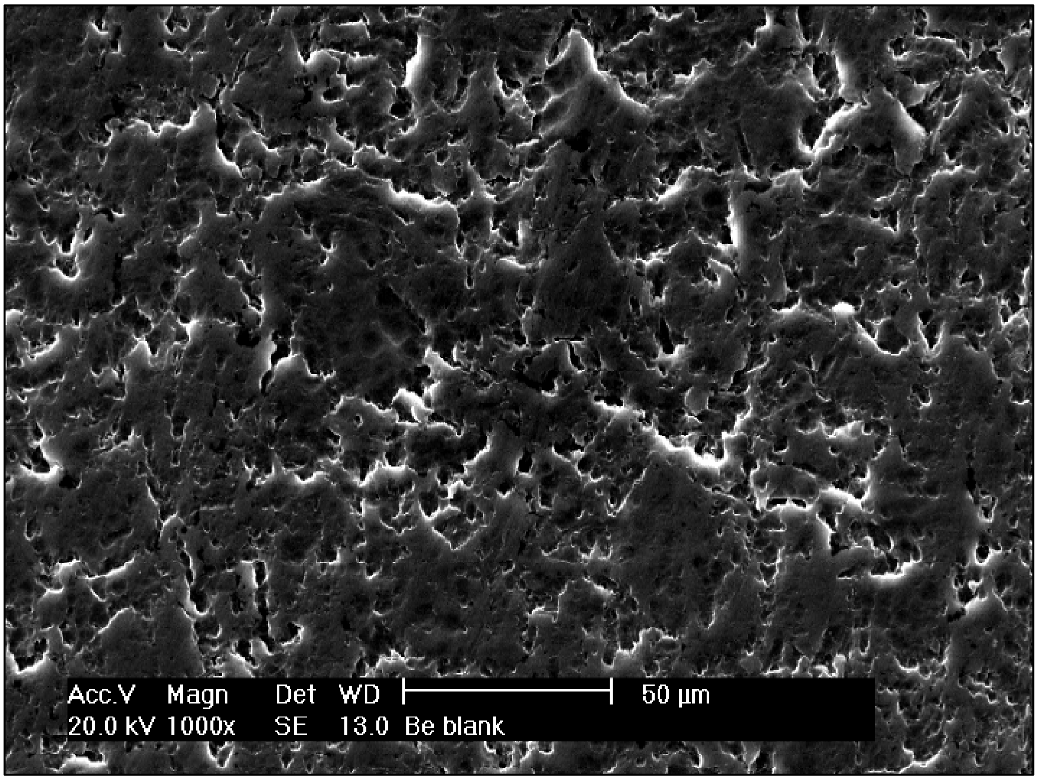


FIGURE 5 - Archived Beryllium Coupon (As-Received Condition), Scanning Electron Microscope Image 50 µm

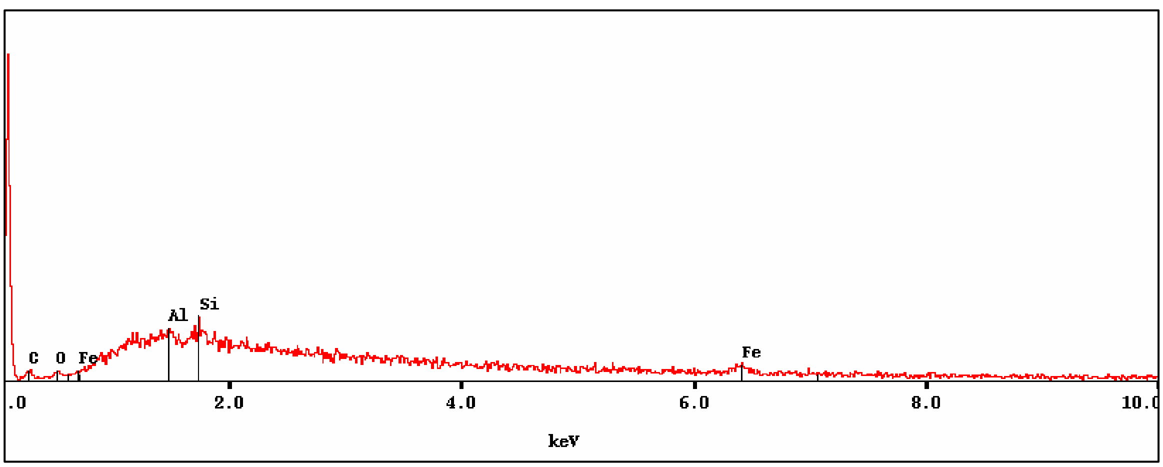


FIGURE 6 - Archived Beryllium Coupon (As Received Condition), Scanning Electron Microscope Spectrum

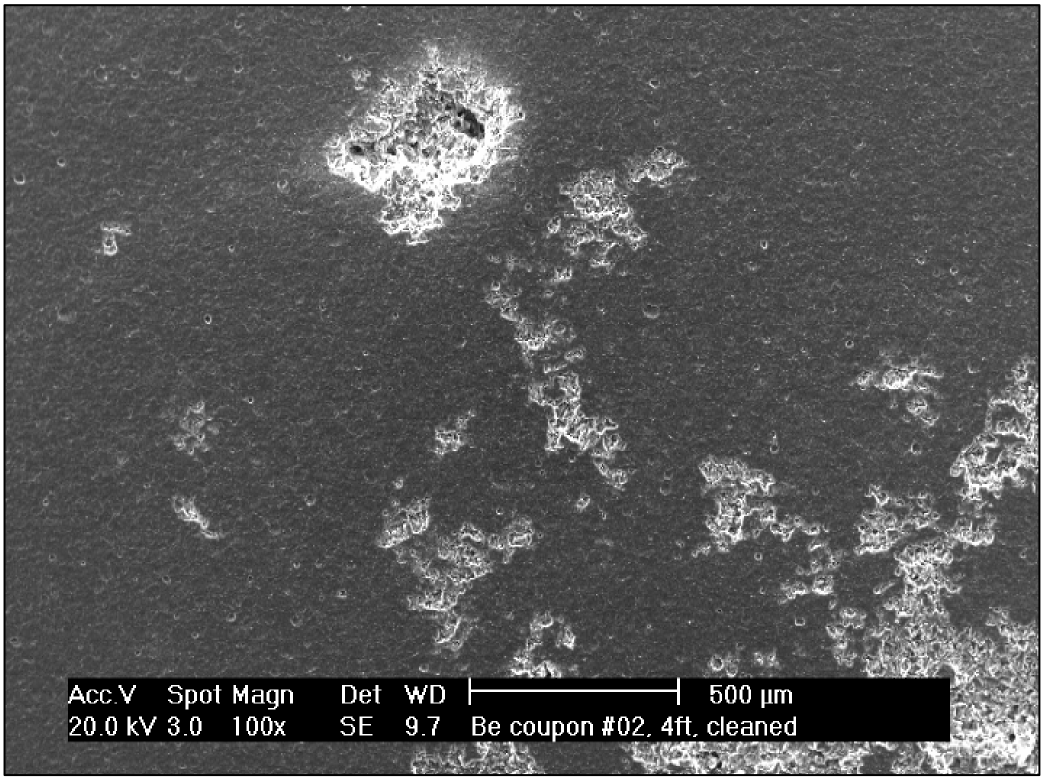


FIGURE 7 - Beryllium Corrosion Coupon Cleaned Using ASTM G 1-C.5.1, Scanning Electron Microscope Image 500 µm

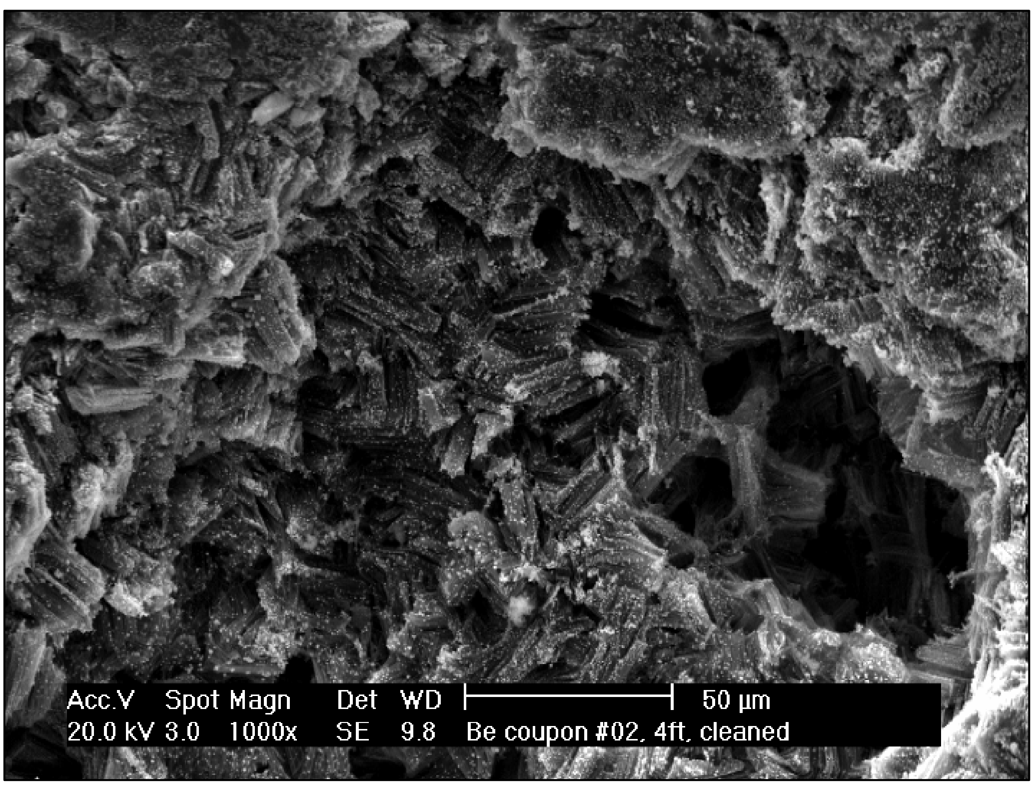


FIGURE 8 - Beryllium Corrosion Coupon Cleaned Using ASTM G 1-C.5.1, Scanning Electron Microscope Image 50 µm

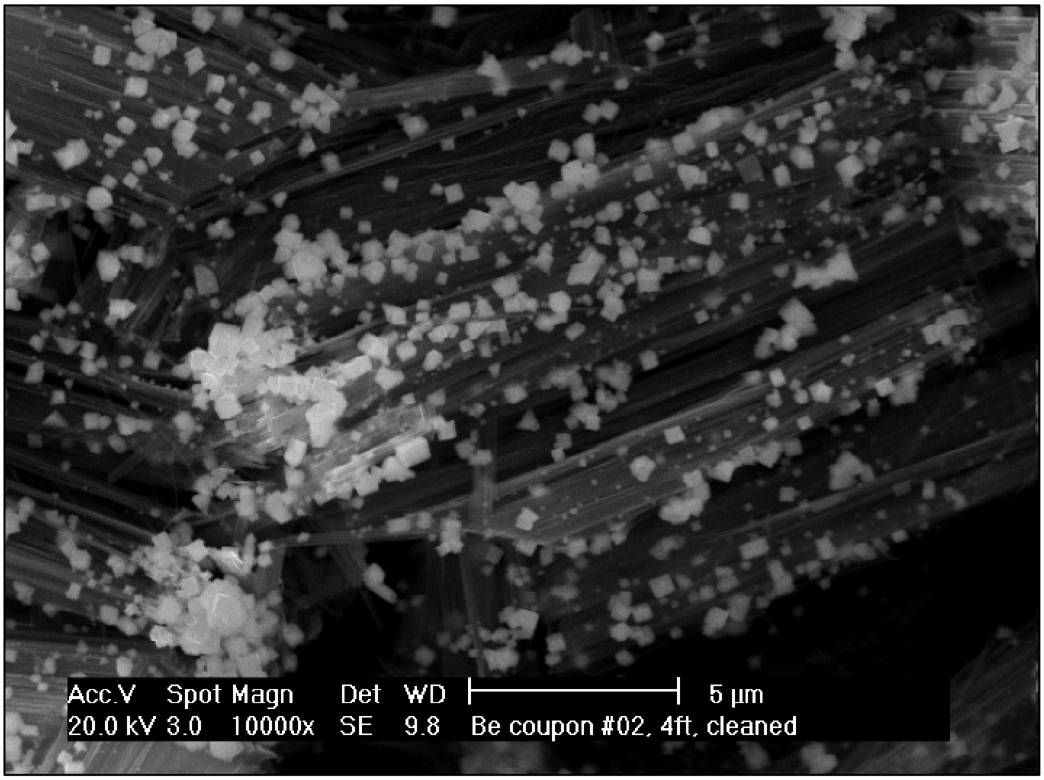


FIGURE 9 - Beryllium Corrosion Coupon Cleaned Using ASTM G 1-C.5.1, Scanning Electron Microscope Image 5 µm

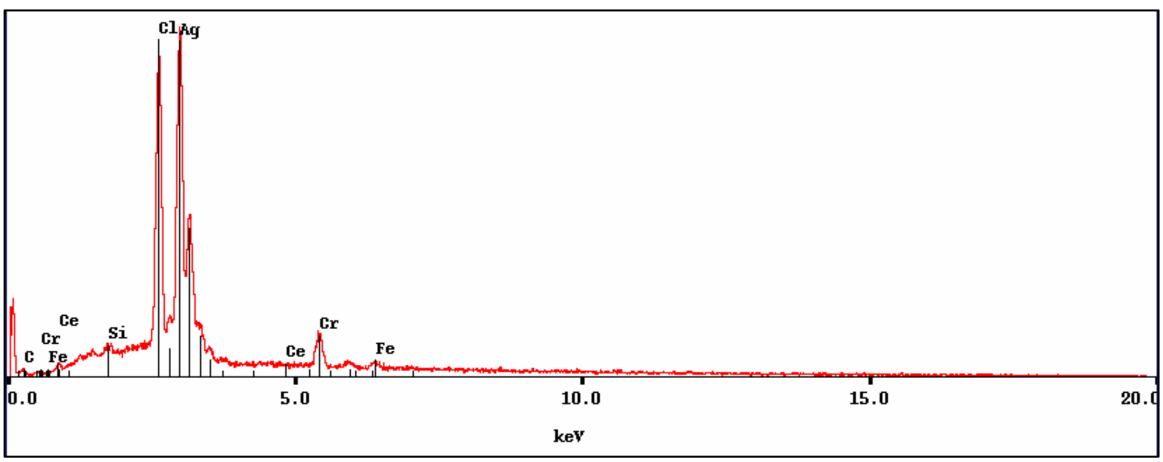


FIGURE 10 - Beryllium Corrosion Coupon Cleaned Using ASTM G 1-C.5.1, Scanning Electron Microscope Spectrum

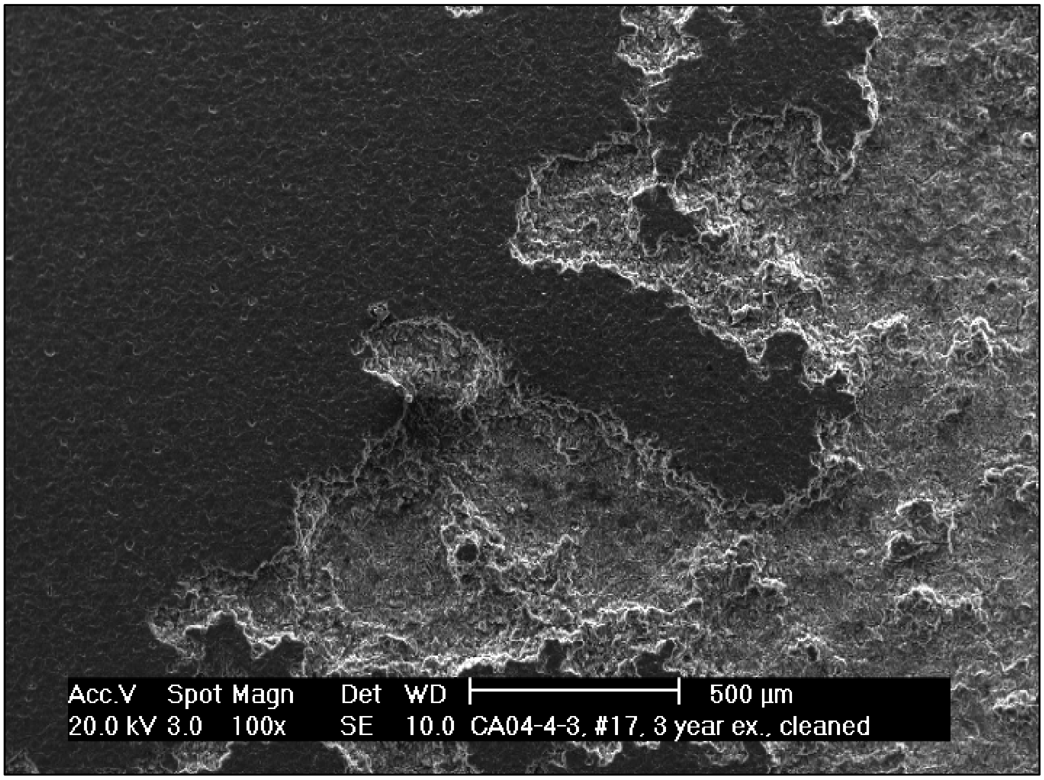


FIGURE 11 - Beryllium Corrosion Coupon Cleaned Using ASTM G 1-C.1.1, Scanning Electron Microscope Image 500 µm

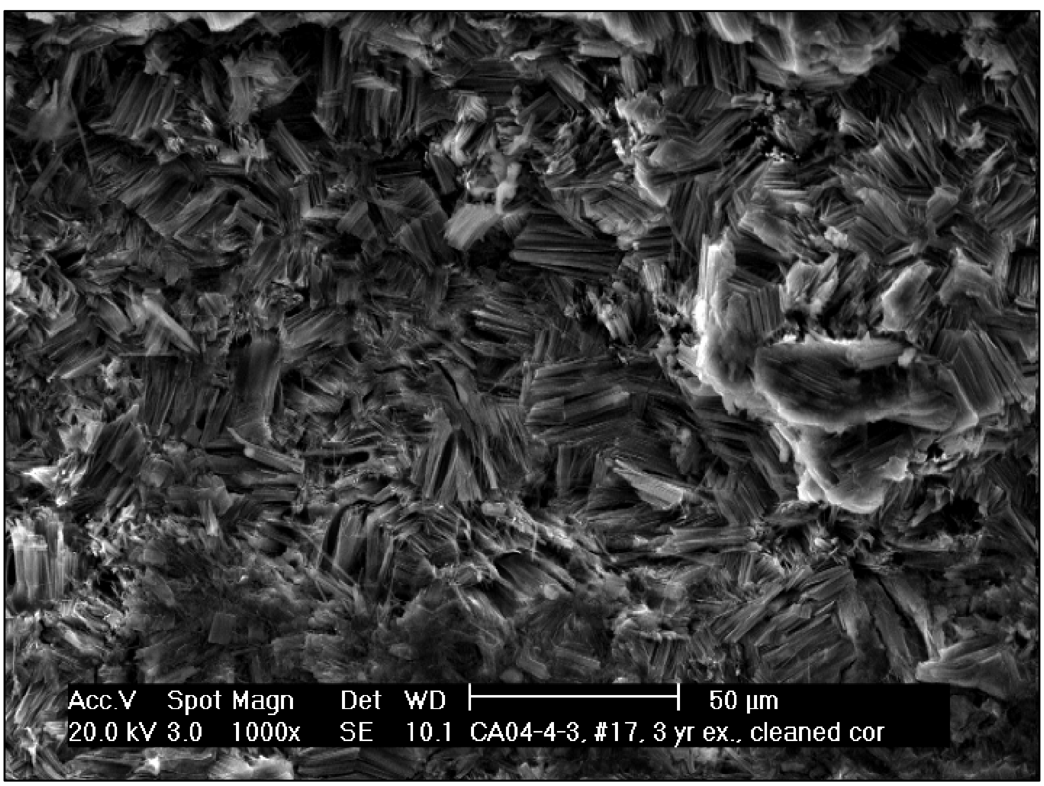


FIGURE 12 - Beryllium Corrosion Coupon Cleaned Using ASTM G 1-C.1.1, Scanning Electron Microscope Image 50 µm

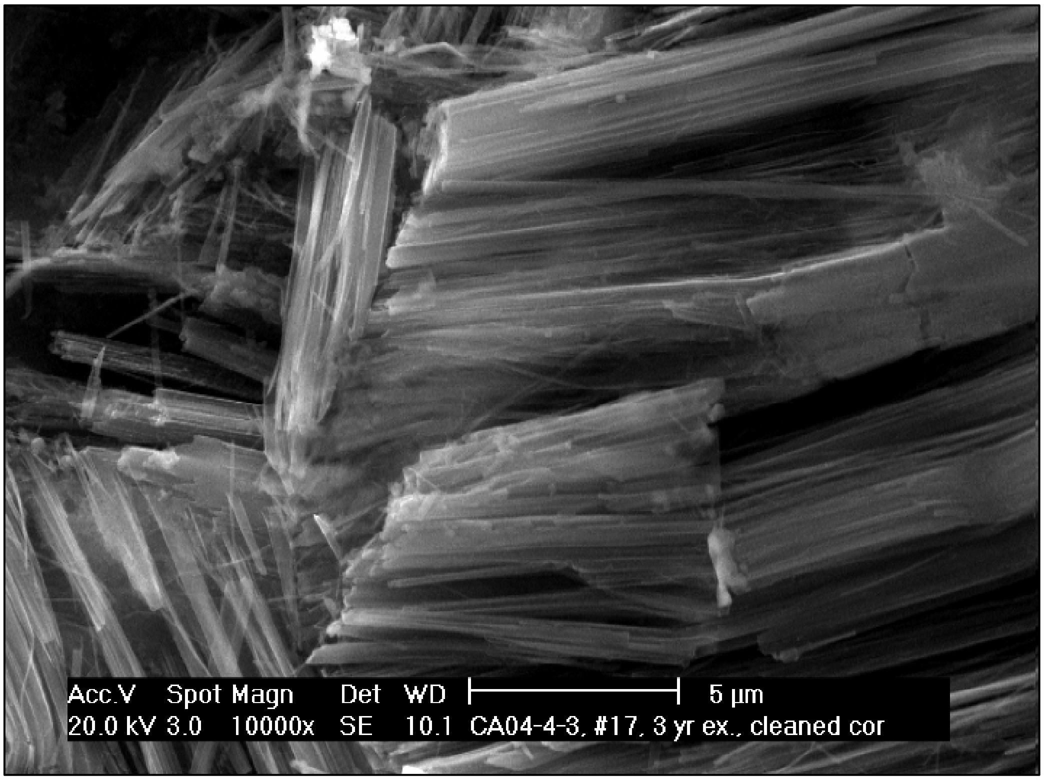


FIGURE 13 - Beryllium Corrosion Coupon Cleaned Using ASTM G 1-C.1.1, Scanning Electron Microscope Image 5 µm

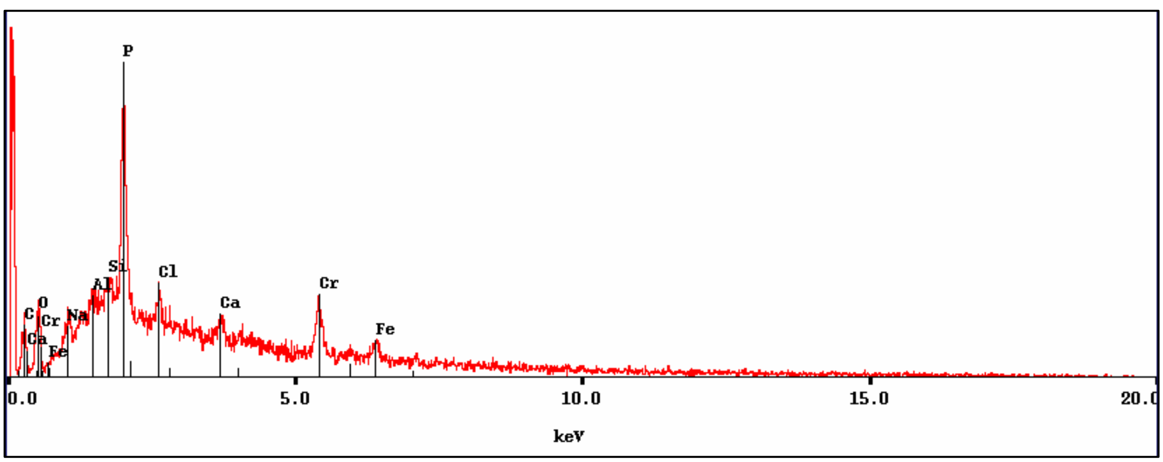


FIGURE 14 - Beryllium Corrosion Coupon Cleaned Using ASTM G 1-C.1.1, Scanning Electron Microscope Spectrum

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