

Wet and Dry Hydrogen-Firing of Stainless Steel†

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‡Abstract

The topography and surface chemistry of wet-only and wet- followed- by- dry H₂ firing of stainless steel were investigated by SEM and EDX.

1 Introduction

Wet hydrogen-firing of OFE copper is a method of reducing the carbonaceous surface layer from the material, via a chemical reaction involving the production of volatile hydrocarbon compounds. The wet-firing process oxidizes the copper surface as well and, so, subsequent dry hydrogen-firing of the copper is necessary to remove the oxide. When an assembly contains both copper and stainless steel components, the wet-firing process produces a thick surface chromium oxide layer which “greens” the stainless. Subsequent firing in dry hydrogen reduces this oxide and, hopefully, restores a much thinner chromium oxide surface passivation layer. The flange is protected from rust (iron oxide) by this very thin layer of transparent chromium oxide. Conversely, if the flange is improperly under-passivated, the chromium oxide layer may not completely cover the steel grains and the part is vulnerable to rust. The purpose of this study is to compare a standard “cleaned for UHV and passivated” stainless steel surface, as prepared in the SLAC Plating Shop with a wet-followed-by-dry hydrogen-fired surface, in order to determine the state of the passivation layer after the wet/dry firing process.

2 Experimental Details

The three samples used were standard Conflat mini-flanges, type 304 stainless steel. The Plating Shop recipe for the cleaning/passivating process is designated C02 (so the reference flange is referred to as the “C02 flange”). The “fired flanges” were processed in Klystron Dept. hydrogen furnaces, one wet-only and one wet-followed-by-dry-firing. Wet-firing was done at 980 °C (dew point, +5 °C) for two hours. Dry-firing was done at 1000 °C

(-60 °C dew point) for one-half hour. Fired flanges were not C02-processed prior to firing.

The data was taken in the form of secondary electron microscope (SEM) topographic images with elemental identification done via energy-dispersive x-ray spectroscopy (EDX). Both techniques are described in the Appendix.

3. Results

Figures 1 and 2 are images of the top of the knife edge and gasket land of the C02-cleaned unfired reference flange. The EDX spectrum underneath each image is the standard against which the elemental composition of the fired flanges will be compared. The EDX spectral peak heights of the elements are a measure of the flange composition.

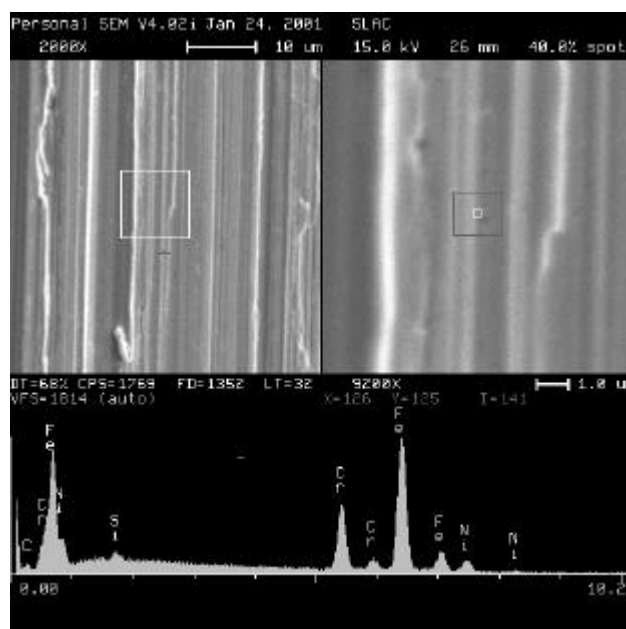


Figure 1. Knife edge, 304 stainless steel Conflat flange, cleaned and passivated in SLAC Plating Shop.

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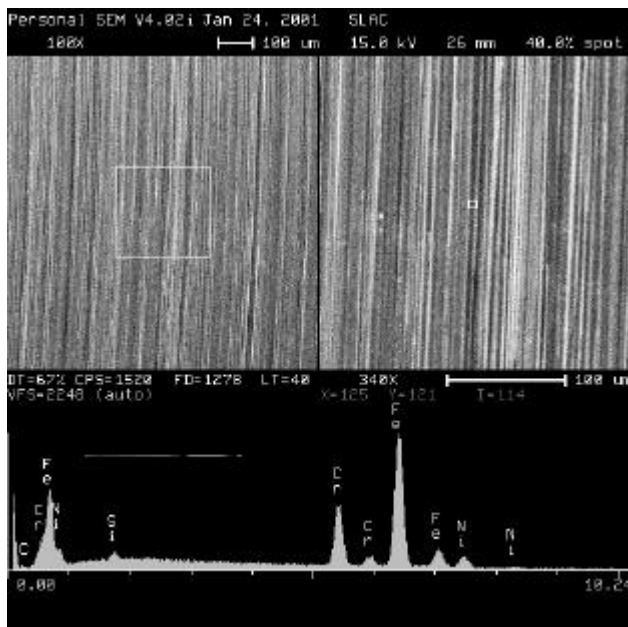


Figure 2. Same flange as Figure 1, gasket supporting surface (land) just inside knife edge ID.

The surfaces of the wet-fired flange (Figures 3 and 4) show that the principal material on the surface is made up of chromium and oxygen, the thick green chromium oxide layer. Only very small iron peaks are evident in the spectra, indicating that the steel grains are covered by several microns of chromium oxide.

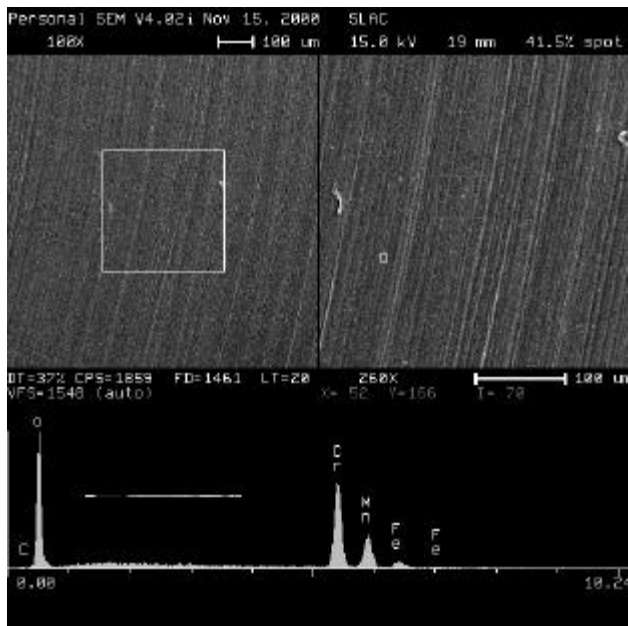


Figure 3. Test flange 1, wet-H₂ fired, land surface.

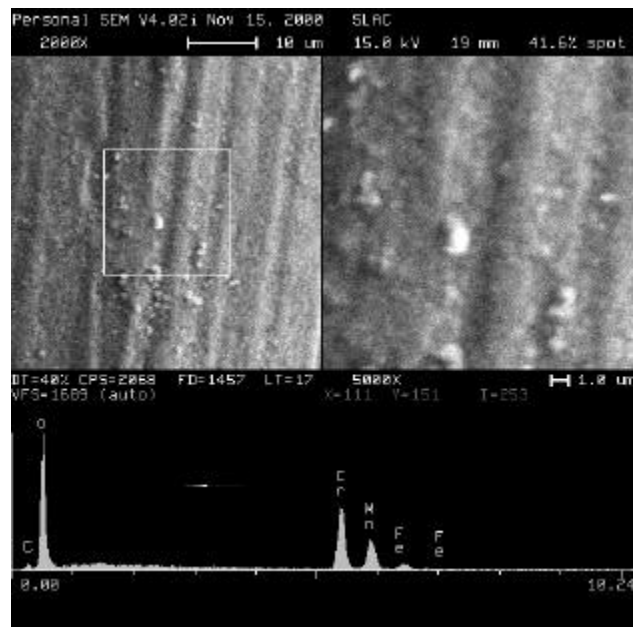


Figure 4. Test flange 1, wet-H₂ fired, knife edge. EDX spectrum is from the large white feature in the center of the right hand image.

Figure 5 shows that the oxide layer, away from the feature in Figure 4 is also thick.

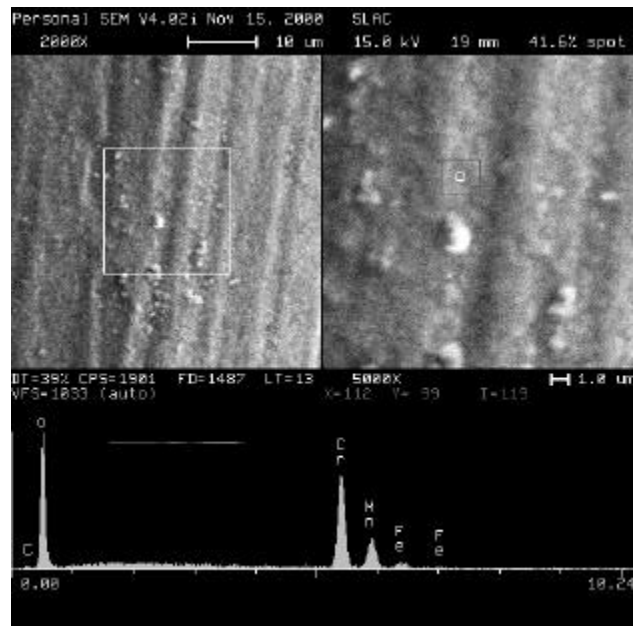


Figure 5. Test flange 1, wet-H₂ fired, knife edge. EDX spectrum of clear area near the center of the right-hand image.

After a wet-dry-hydrogen firing (Figures 6 and 7), test flange 2 shows that the dry-firing process restores the passivation layer to a thickness similar to that of the C02 flange.

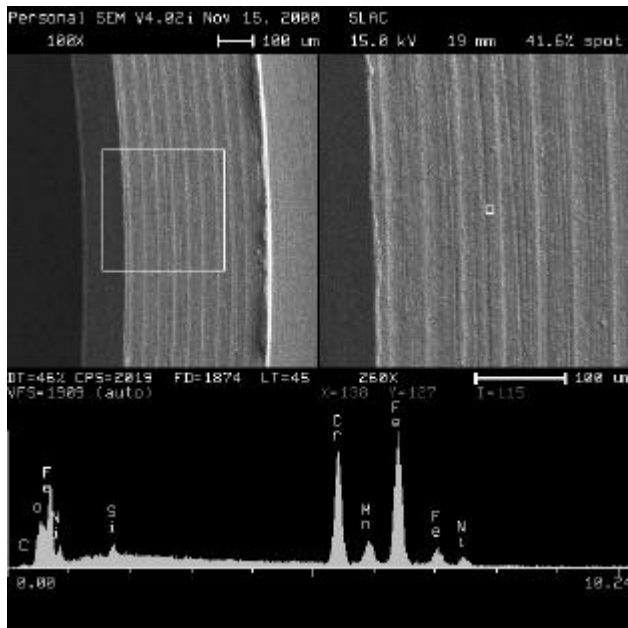


Figure 6. Test flange 2, wet/dry-H₂ fired, land area.

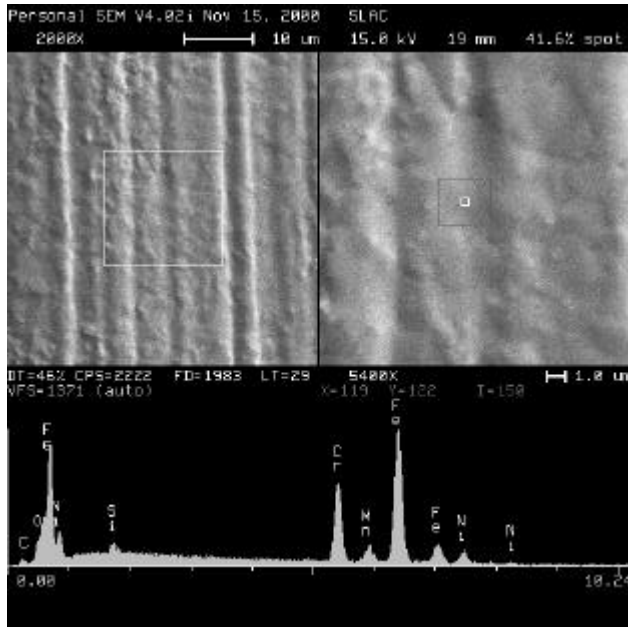


Figure 7. Test flange 2, wet/dry-H₂ fired, knife edge.

4. Discussion

The fired flange 2 surface, Figure 7, appears rougher than that of the reference flange, Figure 1. However, the significance of this observation is uncertain because the flanges were not selected for initial surface finish for this study. Thickness of the passivation layer after firing was the focus.

5. Conclusion

Wet/dry firing of stainless steel produces a surface with a suitable chromium oxide passivation layer, as shown by EDX.

6. Appendix

Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy images surfaces at very high resolution (10 nanometers, depending on atomic number and surface finish). Images are made by scanning a high-energy primary electron beam onto the sample and collecting the low-energy secondary electrons, which are very surface sensitive. Images can be made more sensitive to sub-surface stress by selectively collecting only elastically-scattered ("backscatter mode") primary electrons.

Energy-Dispersive X-Ray Analysis (EDX)

Energy-Dispersive X-Ray Analysis provides an elemental concentration analysis of the outer several microns of sample surface. High-energy primary electrons generate x-rays in the sample, whose energy is determined by the atomic shell from which they were ejected. The x-ray energy is measured by a silicon-crystal detector and displayed as a series of peaks whose heights are related to elemental concentration.