



XPS Analysis of Stainless Steel Surfaces

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The Thermo Scientific™ K-Alpha™ X-ray Photoelectron Spectrometer (XPS) System was used to analyze the surface and layer composition of stainless steel surfaces to identify the successfulness of the passivation process.

Introduction

The surface chemistry of stainless steel is strongly influenced by the chemical or mechanical processing of that surface. Preventing corrosion is a major ambition of any steel manufacturing process and this is often achieved through passivation. This typically involves the formation of a chromium-rich 'passive' layer on the top surface (Figure 1) and there is a number of different methods employed to encourage the growth of such a layer. The key metrics which can be used to identify properly passivated steel are the total Cr/Fe atomic ratio, the Cr oxide/Fe oxide ratio, and the total oxide thickness.



Figure 1: Passivated stainless steel samples and schematic of layer composition

Experimental and Results

Passivated and unpassivated steel samples were analyzed using Thermo Scientific K-Alpha XPS. The iron chemistry of surfaces was investigated using high energy resolution XPS. Metallic and oxide states were observed in both cases, with significantly reduced oxidation on the passivated sample. Figure 2 shows the chemistry of the top 5 nm of the surface. Peak fitting of the two spectra allows us to quantify the different chemical states. The broader oxide peak shape of the passivated sample is due to a mixture of iron (III) and iron (II) states. These two oxide states are present in almost equal quantities. In contrast, the unpassivated sample only has iron (III).

Passivation significantly changes the ratio of iron oxide and metallic states. Chromium data from the unpassivated and passivated samples, in contrast, showed only a minor difference in the oxide-to-metal ratio between the two samples (Table 1). Combining the iron and chromium datasets, we can derive the useful parameters which are then used to assess the quality of the passivation (Figure 3). The goal of passivation is to maximize the chromium-containing layer on the surface.

Non-destructive Surface Measurement		
	Passivated	Unpassivated
CrTotal/FeTotal	1.56	0.77
Cr-O/Fe-O	2.85	0.76
Fe-O/Femetal	0.77	4.26
Cr-O/Crmatal	5.33	5.38
Oxide Thickness	1.2 nm	3 nm

Table 1: Useful steel parameters measured non-destructively with K-Alpha



Thermo Scientific K-Alpha XPS

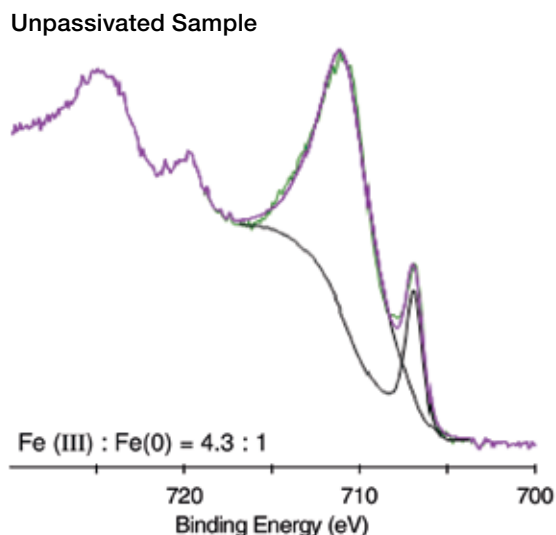
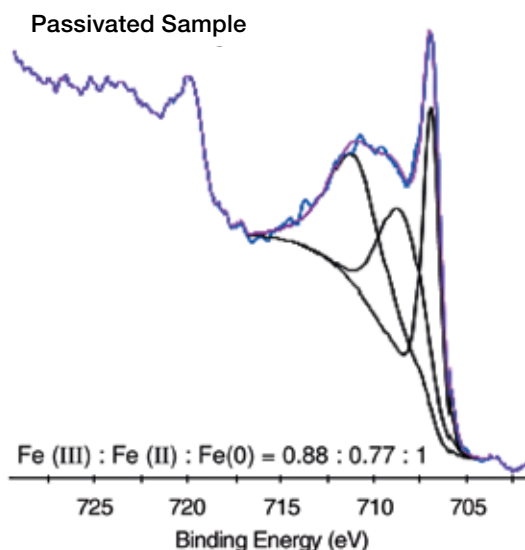


Figure 2: High resolution Fe spectra from passivated and unpassivated stainless steel surfaces

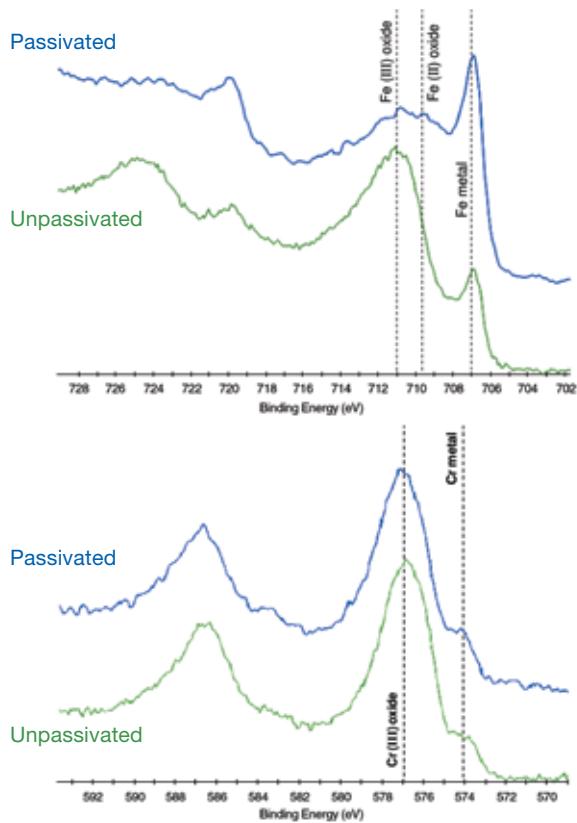


Figure 3: Comparison of Cr and Fe chemical states of passivated and unpassivated steel surfaces

These parameters describe the elemental and chemical properties of the steel between 0 and 5 nanometers. When the results of these measurements indicate a deviation from required passivation standards, a sputter profile experiment can be used to gain a better understanding of the problems involved. Profiles of the passivated and unpassivated samples (Figure 4) confirm the significant reduction in oxide thickness on the steel. The passivation prevents much of the iron oxidation observed on the unpassivated sample. However, the chromium oxide thickness is also reduced on the passivated sample. This reduction in total oxide thickness means that more of the iron-rich metallic substrate is seen by the surface XPS measurement.

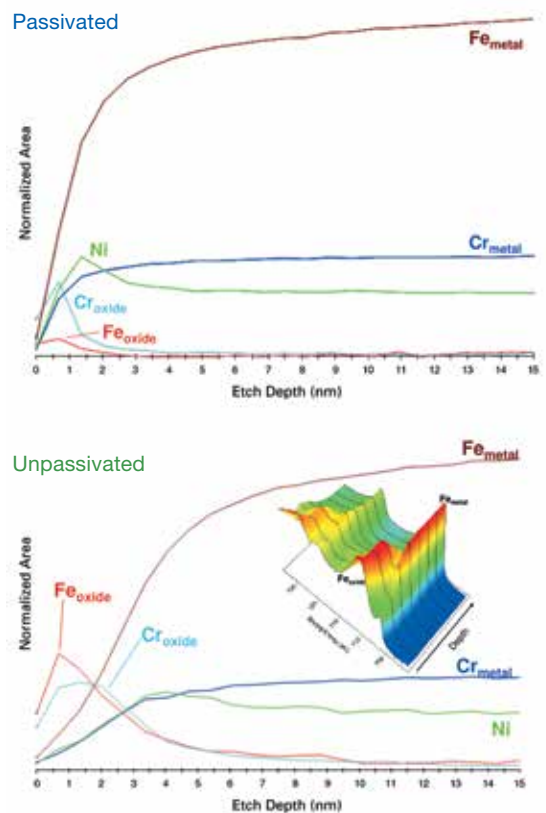


Figure 4: Sputter depth profiles of unpassivated and passivated steel surfaces

Summary

K-Alpha analysis indicated significant reduction in iron oxidation as a result of passivation. Quantification of elemental and chemical states using XPS showed that the passivation quality fell short of requirements. Sputter depth profiling revealed the cause of the passivation problem to be a significant reduction in the level of iron oxidation at the surface. Quantitative measurement of a number of useful parameters, however, demonstrated that the passivation method had not produced a protective layer of the required quality. Sputter profiling of the surface revealed that the passivating chromium oxide layer was too thin.

