

# STAINLESS STEEL

## Surface Analysis

*AES and XPS analysis of the passivation layer on stainless steel can help determine how well it will resist corrosion.*

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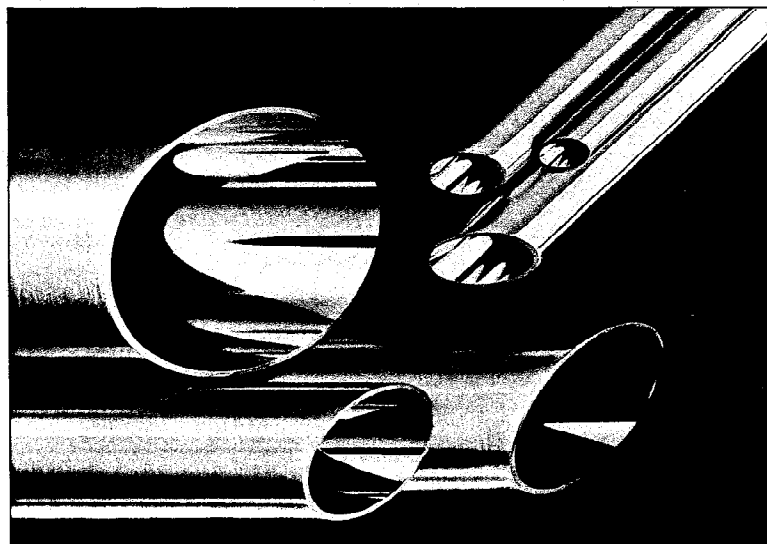
**A**uger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) are valuable techniques for analyzing the surface of stainless steels to establish their ability to resist corrosion. AES identifies the atoms in a layer by measuring the characteristic Auger electron energy, which is emitted in steps from an inner atomic shell. XPS (also known as ESCA, electron spectroscopy for chemical analysis) measures the energy spectra of electrons emitted when the surface is exposed to X-rays.

Both of these techniques have an analysis depth of approximately 2 to 4 nm (20 to 40 angstroms, or about ten atom layers); shallower measurements can be made under specially designed experimental conditions. AES can have a very small analysis diameter, down to 20 nm (200 angstroms), and its primary function is to provide elemental identification. XPS has a larger diameter of analysis (about ten microns), and is mainly used to determine the chemical state of near-surface elements. Both techniques can detect elements above atomic number two, with a limit of approximately 0.1 atomic percent. Standard AES and XPS instruments have the ability to sputter argon ions to depth-profile the surfaces of interest, yielding composition as a function of depth. For stainless steel analysis, the total depth of analysis is typically 15 nm.

AES and XPS data can be quantitatively analyzed by means of elemental sensitivity factors, normalization values that are based on yields for pure elements. Because of these factors, analysis of the same surface with AES and XPS can yield slightly different quantified compositions. When precise

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*Stainless steel is rendered resistant to corrosion because of the development of a chromium-rich chromium oxide passive layer on the surface.*

quantification is required, standards similar to the unknown should be the benchmarks.

This article explains how AES and XPS analysis of stainless steel surfaces treated by various chemicals can provide valuable information about passivation layer composition and depth, and its ability to protect against corrosion.

### **The surface of stainless steel**

Analysis of "clean" metallic samples exposed to the atmosphere typically shows levels of 10 to 20% carbon and 30 to 50% oxygen. The carbon (commonly called adventitious carbon) is present primarily because of adsorbed hydrocarbons and carbon oxides; the oxygen is from instantaneous oxide formation, as well as adsorbed water vapor and carbon dioxide. A clean metallic surface is very reactive and adsorbs more water vapor and atmospheric contaminants than a ceramic or a contaminated surface. Figures 1 and 2 show AES and XPS survey scans of mechanically polished 316 stainless steel that has been exposed to the atmosphere.

By definition, stainless steel contains a minimum of 50% iron and 10.5% chromium. Corrosion resistance is the result of protection conferred by a chromium-rich passive layer, which is typically on the order of 3 to 5 nm thick, or about 15 layers of

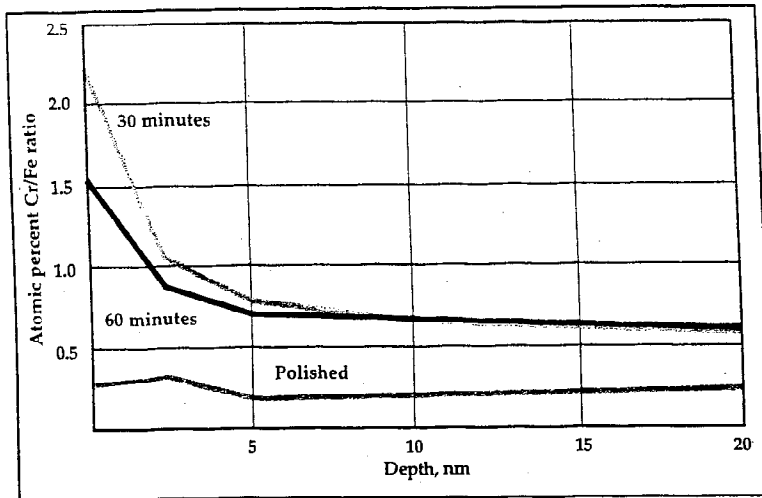


Fig. 6 — The Cr/Fe ratio as a function of depth for nitric acid passivating times of 0, 30, and 60 minutes.



lution mode, each of the peaks of interest in Fig. 2 is scanned individually to measure a more precise value of the energy of the peak. Comparison of this peak location to literature standards may indicate which compound is present. For the most part, oxides can be differentiated from metallic species, and identification of specific oxide compounds is usually possible. A high-energy resolution iron peak is shown in Fig. 7. Through this additional analysis, the ratio of metal-to-oxide for each of the elements can be determined by deconvoluting the peaks and measuring their area. Table 2 summarizes the ratio

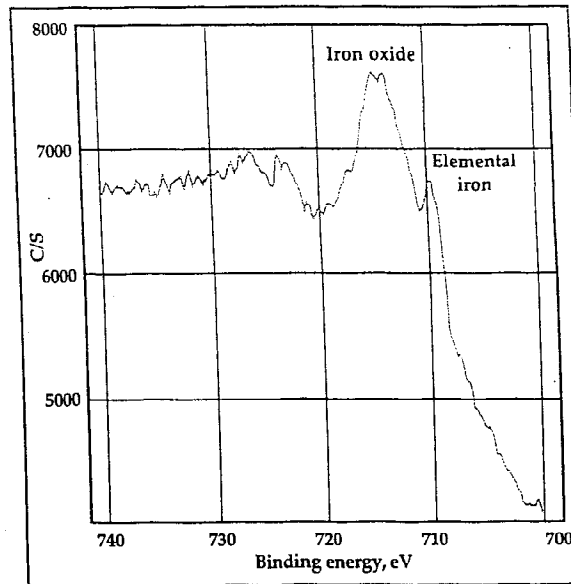


Fig. 7 — XPS high energy resolution scan of iron, showing the presence of iron and iron oxides.

of iron and chromium oxides to their metals for various passivation times.

The data in Table 2 confirm that the concentration of metallic species of both elements drops substantially after 30 minutes of treatment, showing that at least this much time is necessary for the successful oxidation of the surface.

#### Test results

Salt spray testing data indicate that for a nitric acid passivation procedure, 30 minutes of treatment gives the best corrosion resistance. The surfaces were analyzed by AES and XPS to investigate the cause for this behavior. Results showed that the maximum near-surface chromium/iron ratio develops with 30 minutes of treatment time, and the surfaces are almost fully oxidized shortly thereafter.

New passivation processes that are more environmentally friendly are currently being designed, and analysis with AES and XPS will be an integral part of direct research and routine quality control.

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