An XAS Study of the Surface of Low-Temperature Carburized Stainless Steel

D. F. Roeper[1], W. E. O'Grady[1], K.I. Pandya[2] and P. M. Natishan[3]

[1]EXCET, Inc.
Springfield, VA 22151

[2]SAIC, Inc.
Alexandria, VA

[3]Naval Research Laboratory, Code 6130
Washington, DC 20375

Introduction

Recent work by others (1-3) on stainless steels has demonstrated that the Swagelok low-temperature carburization process improves the SS alloy performance; i.e. hardness, wear resistance and corrosion resistance. The process creates a carbon-rich case by forming a layer of interstitially dissolved carbon that extends to approximately 20 um into the bulk metal. The high nickel content in austenitic stainless steels maintains the austenite phase on cooling, the face centered cubic (FCC) structure. The FCC structure has two types of interstitial sites to where carbon could potentially migrate, octahedral and tetrahedral. The purpose of this study is to determine where the carbon is and to ascertain the structure of the surface.

Experimental

Stainless steel (SS) samples of 316L were carburized with the Swagelok low-temperature process. A sample size of approximately 0.75" x 0.75" x 0.25" was used for both carburized and untreated SS and they were mechanically polished to a 1200 grit finish with SiC.

The X-ray absorption spectroscopy (XAS) data were measured for the Cr, Mn, Fe and Ni K-edges on beamline X11-A at the National Synchrotron Light Source at Brookhaven National Laboratory. The storage ring was running with a ring current between 150 and 300 mA with an energy of 2.5 GeV. The fluorescence X-rays and electron yield intensities were recorded using a Lytle X-ray fluorescence detector with an electron yield detector. The electron yield data was used because of its much greater signal to noise ratio and its enhanced surface sensitivity. The XAS data was analyzed using the standard XDAP (4) data analysis package. A smooth background was removed from each spectrum by fitting a linear function to the pre-edge region of the spectrum and then subtracting the function from the entire spectrum. The data was normalized by fitting a polynomial function to the post-edge region and normalizing the edge jump to 1 at 50 eV above the edge to put all of the data on a per atom basis.

Results and Discussion

XAS data of the metal edges in the untreated SS and the carburized SS were collected and compared to the XAS data of the respective pure metal edges. Chromium metal exists in the body centered cubic (BCC) structure at room temperature. The lattice structure of 316L SS is FCC and the difference in lattice structure with Cr foil is reflected in the change in the edge structure of the X-ray absorption near edge structure (XANES). The carburized SS spectrum has approximately the same step height as the untreated SS but the X-ray absorption fine structure (XAFS) oscillations are damped due antiphase components. These interference effects are likely due to carbon contributions which are out of phase with the transition metal and from lattice distortions introduced by the carbon interstitials. This antiphase behavior is seen in the Fe and Mn spectra as well but not in the Ni spectrum indicating that there is a negligible amount of carbon associated with the Ni.

Analysis of the step height data of the absorption spectra for the three primary elements, Cr, Fe, and Ni, in the carburized 316L SS sample indicate that the twelve nearest neighbors of the first metal shell will have an approximate composition of 6 chromium atoms, 4 iron atoms and 2 nickel atoms. It is not possible to discriminate between the interactions of the three main components with one another since the backscattering for these elements is very similar. The location of the carbon has been determined to be in the octahedral interstices of the FCC lattice and it is most closely associated with the chromium which has ~2 to 3 low-Z near neighbors and carbon is the predominate one. Nickel shows only a slight interaction with low-Z backscatterers which is not sufficiently strong to allow us to analyze it.

Acknowledgements

The authors would like to acknowledge the financial support of the DARPA and the U.S Department of Energy. This research was carried out at the National Synchrotron Light Source, Brookhaven National Laboratory, which is supported by the U.S. Department of Energy, Division of Materials Sciences and Division of Chemical Sciences, under Contract No. DE-AC02-98CH10886.

References

1. N. Agarwal, H. Kahn, A. Avishai, G. Michal, F. Ernst and A. H. Heuer, *Acta Materialia*, **55**, 5572 (2007).
2. F. J. Martin, E. J. Lemieux, T. M. Newbauer, R. A. Bayles, P. M. Natishan, H. Kahn, G. M. Michal, F. Ernst and A. H. Heuer, *Electrochemical and Solid-State Letters*, **10**, C76 (2007).
3. F. J. Martin, P. M. Natishan, E. J. Lemieux, T. M. Newbauer, R. J. Rayne, R. A. Bayles, H. Kahn, G. M. Michal, F. Ernst and A. H. Heuer, *Metallurgical and Materials Transactions A: Physical Metallurgy and Materials Science*, **40A**, 1805 (2009).
4. M. Vaarkamp, J. C. Linders and D. C. Koningsberger, *Physica B: Condensed Matter (Amsterdam)*, **208 & 209**, 159 (1995).