THE FUNDAMENTAL ROLE OF MICROSTRUCTURAL AND MICROCHEMICAL ANALYSIS IN HIGH-TEMPERATURE OXIDATION SCIENCE

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Advanced microstructural characterization and specimen preparation techniques have become necessary tools for the analysis of oxidized Ni- and Fe-based alloys to understand the effects of alloy composition and segregation phenomena on oxide scale growth mechanisms, oxide stress formation, and scale adhesion. Atomic-level microchemical and structural evaluation of the corrosion products formed on model and commercially-available alloys is currently performed using techniques such as scanning transmission electron microscopy (STEM) on thin, cross-section specimens prepared from oxidized surfaces using a focused ion beam (FIB) system.[1] As an example of this, a STEM image of an oxidized surface (~1.75 μm α-Al_2O_3 scale) of a Hf-doped, β-Ni(Pt)Al alloy (1150°C for ~700 h in dry, flowing O_2) prepared using the FIB technique is shown in Figure 1 and the corresponding elemental Hf-map from an Al_2O_3 grain boundary (area within 0.25 X 0.25 μm^2 box shown in Figure 1) is shown in Figure 2. In most of the high-temperature oxidation studies conducted at ORNL, microstructural analysis plays an integral role in elucidating alloy oxidation mechanisms, ultimately relating oxide scale and alloy sub-scale microstructural development to oxidation performance at high temperatures.

In this presentation, several examples of extensive post-exposure microstructural characterization of oxidized alloys will be used to illustrate the benefit of using these analyses to interpret performance data, to relate scale/alloy microstructrual development/changes to oxidation behavior, and to compare different alloys exposed under similar high temperature conditions. These examples will include the use of high-resolution STEM/TEM imaging, energy dispersive spectrometry (EDS) spectrum imaging, and nm-scale EDS line profiling to understand

(1) the role of reactant elements on Ni- and Fe-based alloy oxidation performance (single-doped and co-doped combinations of Hf, Zr, and Y)
(2) the role of alumina-scale microstructures on the formation of oxide growth stresses
(3) the effects sub-surface alloy grain boundary degradation and surface Cr-depletion during long-term oxidation studies of commercial alloys being investigated to replace type 347 stainless steel for use as high-temperature recuperator alloys.

In each of these oxidation studies, microstructural and microchemical analyses have proved invaluable tools for the interpretation of performance data and in developing a fundamental understanding of oxidation mechanisms for different alloy compositions.

References:

[1] K.L. More, D.W. Coffey, B.A. Pint, K.S. Trent, and P.F. Tortorelli, "TEM Specimen Preparation of Oxidized Ni-Based Alloys Using The FIB Technique," in Microscopy and Microanalysis **6**(2) G.W. Bailey, S. McKernan, R.L. Price, S.D. Walck, P.M. Charest, and R. Gauvin, eds., Springer-Verlag, New York, NY (2000) p. 540.

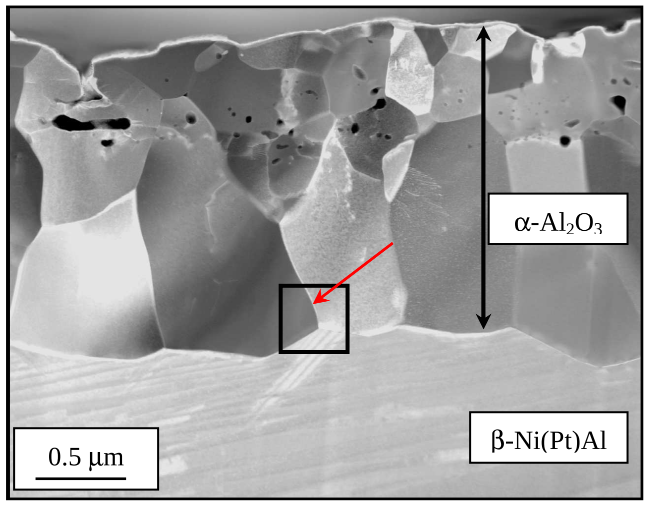

Figure 1. Cross-section STEM image from an oxidized surface of a Ni(Pt)Al alloy (1150°C for ~700 h in dry flowing O_2) prepared using the FIB technique.

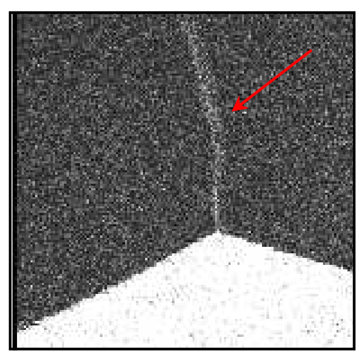

Figure 2. EDS elemental map (data accumulated from region shown by box in Figure 1) showing Hf-segregation at α-Al_2O_3 grain boundary.

Acknowledgements:

Research sponsored by the U.S. Department of Energy, Office of Power Technologies, Distributed Energy Program, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.