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Resolving Nanostructures in Complex Penetrative Oxidation for Ni-30Cr Alloys Exposed to High-Temperature Water using APT and TEM

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The use of three dimensional atom probe tomography (3DAPT) has become an indispensible tool in the analysis of corrosion and oxidation phenomena in both Fe- and Ni-base stainless alloys. A unique penetrative oxidation phenomenon has been observed during stress corrosion crack (SCC) growth testing of Ni-30Cr alloys (Figure 1). While 3DAPT has provided insights into intergranular SCC mechanisms through the detection of light element segregation along grain boundaries, its application to understanding this issue of penetrative oxidation has proved challenging. This research highlights a complementary approach at tackling this complex material science problem through the use of APT and transmission electron microscopy (TEM).

Figure 2 illustrates a typical data set from atomic reconstructions of the filamentary penetrative oxidation and the remaining metallic alloy. Near the crack wall surface, the metal has been nearly completely oxidized (Fig. 2a). Deeper in the sample, the oxide penetrations can be observed as discrete filaments in the metal matrix. The morphology of these penetrations can more easily be observed through the isoconcentration surfaces of atomic Cr, Ni and O at various levels. The filaments have a complex structure with a nearly continuous Cr/Ni oxide (Figure 2b, orange, 80%) penetrating from the crack surface with individual Cr_2O_3 platelets in the middle of the filaments (Fig. 2b,c). Measurements of these platelets from the isoconcentration surfaces illustrate that they are 2-5 nm wide and are 20-25 nm in length, and appear to be at very distinct crystallographic orientations.

The large discrepancy in field strength between the oxide filaments and surrounding metal, as well as the nanodimensions of the platelets, makes determining the true shape and size difficult in APT. In addition, APT provides no crystallographic information for these oxides. The application of TEM Fresnel contrast and darkfield imaging provide a complementary observation into the nanostructural characteristics (Figure 3) and provides critical information to resolve aspects of the penetrative oxidation. TEM reveals that these penetrations (Fig. 3a) consist of two nanometer-scale, oriented crystalline phases, MO-structure oxide particles (Fig. 3b) and Cr_2O_3 platelets (Figure 3b). The size and shape of the Cr_2O_3 platelets are in close agreement with the APT reconstructions.

These results illustrate the benefits of complementary assessments of nanostructural characteristics by APT and TEM. In the current example, projection issues confound composition analyses of the penetrative oxidation in TEM but APT is able to resolve this limitation. Conversely APT cannot identify phase crystallography and small particle morphologies, TEM darkfield imaging provides this information. Future work will concentrate on analyzing APT tips in the TEM in order to obtain direct, complementary measurements of structure, composition and morphology.

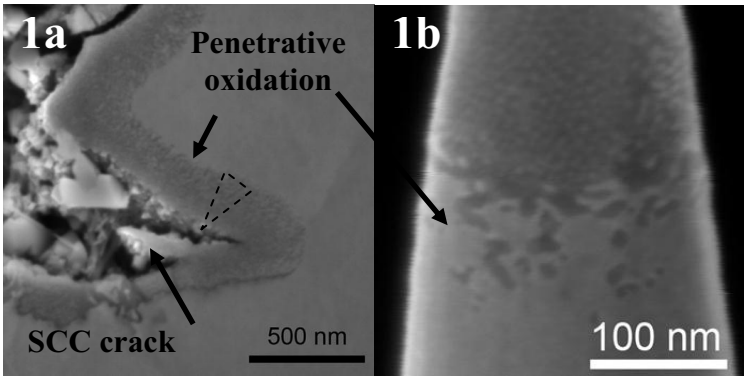

FIG. 1. Scanning electron micrographs (LABE) of oxide filled SCC crack illustrating penetrative oxidation along the either side of the crack wall. a) The location and orientation of an APT tip is represented by a dashed triangle. b) Final APT tip with shallow penetrations.

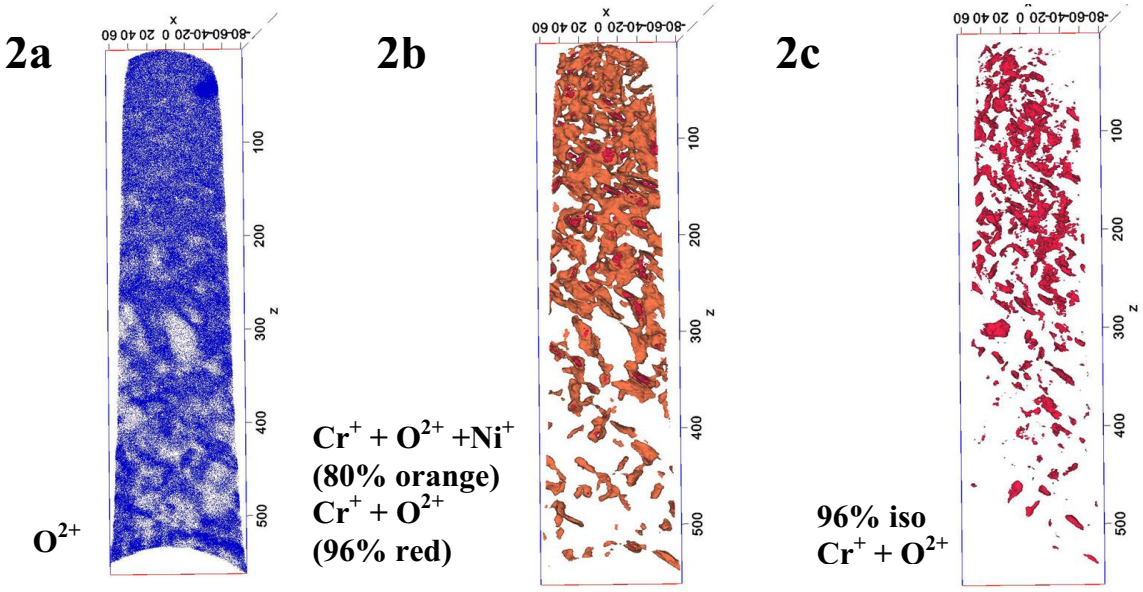

FIG. 2. Three dimensional atom probe reconstruction and isoconcentration surfaces of a penetrative oxidation. The penetrative oxidation is observed as continuous filaments of oxide with a Cr/Ni oxide surrounding platelets of Cr_2O_3.

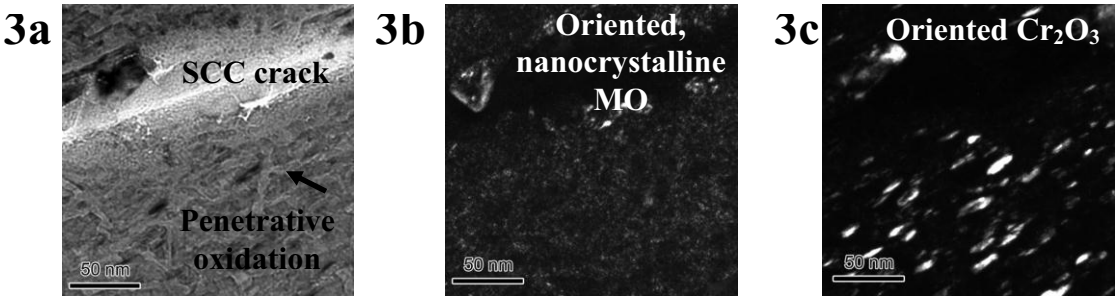

FIG. 3. TEM Fresnel contrast (a) and darkfield images (b,c) illustrating penetrative oxidation is comprised of nanocrystalline, oriented MO structure oxide as well as oriented Cr_2O_3 platelets.

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