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TEM Study of Supercritical Water Corrosion in 310S and 800H Alloys

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Corrosion resistance is one of the key factors in materials selection for Gen IV supercritical water-cooled reactor (SCWR) concept; especially when it comes to selection for fuel cladding. Amongst the candidate materials, 310S austenitic stainless steel and INCOLOY® 800H are deemed very promising. A summary of the previous corrosion studies on candidate alloys can be found elsewhere [1,2]. In this work TEM study of FIB sectioned test coupons is presented in our effort to understand the microstructural evolution of these alloys upon SCW exposure. Characterization of the sigma phase as a high temperature Cr-Fe intermetallic that is brittle in nature and potentially detrimental to the mechanical properties [3] is discussed.

Coupons (20x10x2 mm) were cut from commercial austenitic steel 310S and 800H plates. The coupons were placed in a static autoclave containing deionized water and exposed to SCW condition (625°C, 25MPa) for 250 hours. Cross sectional transmission electron microscopy (TEM) samples were prepared using the FEI Helios NanoLab™ DualBeam™ FIB microscope from the top surface of the coupons [4]. A protective layer of Pt was first deposited on the surface to protect the corrosion layer. TEM samples were observed using the FEI Tecnai Osiris™ TEM equipped with Super X field emission gun (FIG) and ChemiSTEM™ X-ray detection technology operating at 200kV. EDX analyses were done in STEM High angle annular dark field (HAADF) imaging mode. The Esprit software was used for qualitative and quantitative elemental mapping. Conventional bright field/ dark field imaging and electron diffraction techniques were used to characterize different corrosion regions and for phase identification.

Fig 1 shows EDX elemental maps from the top region of a TEM specimen prepared from the 310S coupon. The top layer contains very fine grains of CrO2 with average grain size of ~50nm. A layer featuring recrystallized γ grains below the oxide layer is highlighted in Figure 2 and is also visible in the 800H sample in Fig 3. The average thickness of top chromium oxide layer for 310S and 800H samples were 270nm and 240nm respectively; and the thickness of the sublayer Cr-depleted recrystallized γ regions were 760nm and 800nm respectively. A selected area diffraction pattern (SAD) taken from this region showed superlattice spots coming from a σ grain grown coherently on the γ (Fig 2). An orientation relationship between the γ and σ was evident in both alloys. EDX mapping along with SAD analysis revealed that in the base metal in vicinity of the recrystallized region, the γ grains contained a network of very fine coherent σ nuclei. An example is shown in Fig 4 where an SAD from a large γ grains (region 1) below the recrystallized region 2 shows superlattice spots from the σ. The dark field image in Figure 4 acquired from, (0 0 2) reflection of σ reveals a network of σ within the γ matrix in the 800H. Similar phenomenon was observed in 310S. In summary, FIB/TEM provided a method for studying the microstructural changes after SCW exposure. This study sheds light on the nucleation and the evolution of σ phase during SCW exposure that may play an important role in corrosion resistance of SCWR candidate alloys.

References

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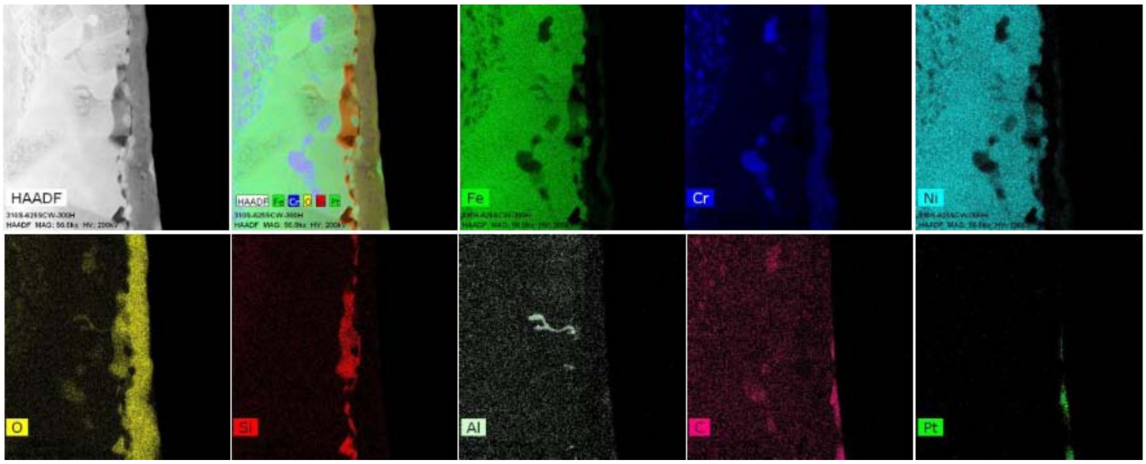

Figure 1. EDX quantified elemental maps of the corroded top surface of the alloy 310S

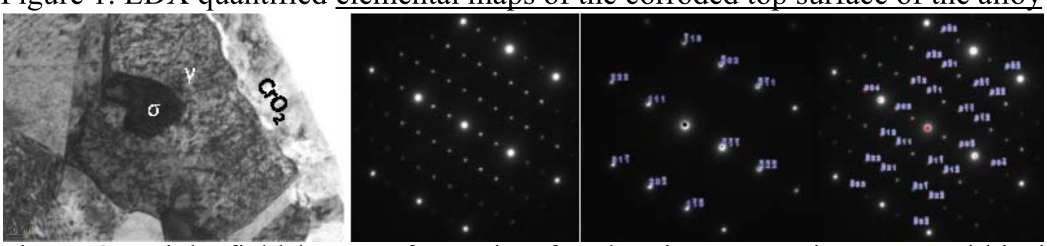

Figure 2. Bright field image of a grain of γ showing a σ grain grown within it; along with the SAD diffraction patterns taken along [-1 -2 1]γ and along [1 -3 0]σ in 310S.

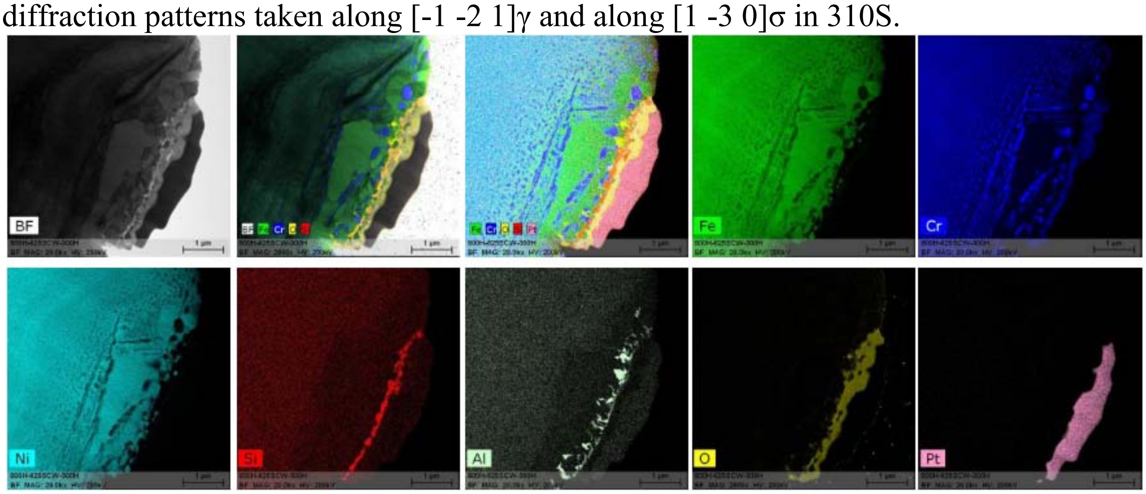

Figure 3. EDX quantified elemental maps of the corroded top surface of the alloy 800H

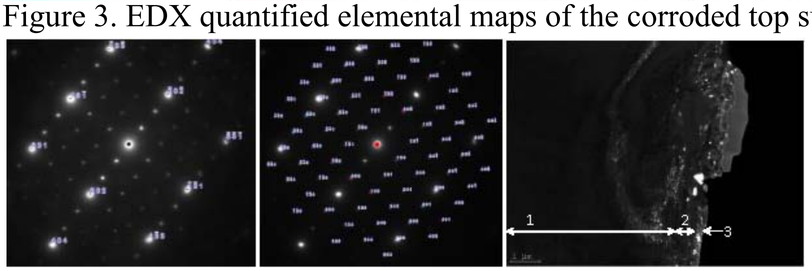

Figure 4. SAD from a γ grain in 800H showing spots from [-3 2 3] zone axis of γ (brighter spots) and [2 -1 0] zone axis of σ. The dark field image is taken using the (0 0 2)σ reflection.

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