Pb-Induced Transgranular SCC of Alloy 690 in a PbO + 10% NaOH Solution

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Abstract – Focused ion beam (FIB) and analytical electron microscopy (AEM) techniques have been applied to study transgranular SCC of Alloy 690. Detailed characterization of the cracks formed in SCC samples has provided evidence for corrosion slot/tunnel formation as the SCC mechanism in Pb-caustic environments.

Stress corrosion cracking (SCC) of Alloys 600 and 690 in caustic and Pb-caustic environments was first identified in the early 1960's, and continues to be the subject of numerous studies. It is known that Pb added to high temperature water environments can dramatically increase the propensity of SCC in these materials. Alloy 690 generally outperforms Alloy 600, except under highly alkaline conditions containing Pb, at which point the Alloy 690 performance decreases precipitously. [1] The Alloy 690 evaluated in this study received a mill-anneal at ~1100˚C and thermal treatment at ~700˚C for 10 h and a final treatment at ~600˚C for 7 h. Specimens were evaluated using standard light optical metallography prior to detailed AEM characterization.

The focused ion beam (FIB) technique was used to section SCC specimens to obtain site-specific AEM specimens from stress corrosion crack tips. A Micrion 2500 FIB instrument was employed for all specimen preparation and imaging. Initial sample milling was performed at 50 kV, with final thinning performed at low voltage to minimize ion damage and Ga implantation.

Using the FIB and AEM, it was possible to obtain a unique perspective on the SCC crack tip by examining the tip from a plane normal to the crack growth direction. In this way, subtle features associated with the tip itself were assessed. The apparent "slotted" or "tunnel-like" features visible in the FIB secondary electron image obtained from a cross-section of an SCC crack in a C-ring specimen are consistent dimensionally with the transgranular fractographic features observed on the SCC fracture surface (Figure 1). Furthermore, these "slot"-like features have been detected at the tip of the main transgranular SCC crack in C-ring as well as in split-tube U-bend (STUB) specimens. These "slots" were filled with Pb-containing NiO. The diffraction analysis showed that these features were not formed along {111} slip traces, but exhibited a morphology consistent with the {110} slot faces reported for austenitic stainless steels. Data obtained from these Alloy 690 SCC crack tip samples indicates that these features are similar to the corrosion "slots" reported by Swann and co-workers in their study of transgranular SCC of austenitic stainless steels. [2-3]

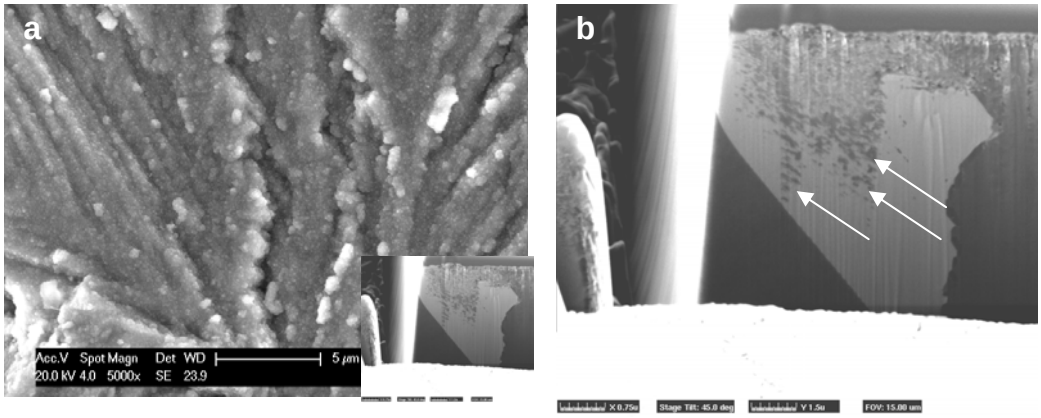

Figure 1. (a) Secondary electron image of the transgranular SCC fracture with inset FIB secondary electron image at comparable magnification to demonstrate the similarity of feature dimensions with the transgranular fracture detail; (b) higher magnification view of the crack tip "slot-like" features (arrowed) beneath the cross-section surface.

References

[1] G.P. Airey, Corrosion, 35 (1979) 129.
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[3] J.M. Silcock and P.R. Swann, in Mechanisms of Environment-Sensitive Cracking of Materials (The Metals Soc., London, 1977) 66.