Intergranular oxidation of nickel base alloys potentialities of focused ion beam tomography

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INTERGRANULAR OXIDATION OF NICKEL BASE ALLOYS: POTENTIALITIES OF FOCUSED ION BEAM TOMOGRAPHY

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Extended Abstract : Nickel base alloys such as Alloy 600 or Alloy 182 (chromium contents: 14-20 wt.%) are susceptible to intergranular stress corrosion cracking (SCC) in the primary circuit of pressurized water reactors for temperatures ranging between 280°C and 360°C. SCC of nickel base alloys in primary water decomposes into several stages: (i) incubation during which oxide penetrations develop in the grain boundaries, (ii) crack initiation that results from the fracture of the intergranular oxide for a critical intergranular oxide depth coupled with a critical normal stress and (iii) crack propagation [1]. In the as-described scenario, intergranular oxidation is supposed to play a key role in the initiation of stress corrosion cracks. Establishing the intergranular oxidation kinetics is thus required for further understanding of the mechanisms driving SCC initiation and for further modelling on physical bases. In addition, the intergranular oxide kinetics is likely to depend on several metallurgical and mechanical factors and the local interactions between oxidation and those parameters need to be studied. The aim of the present work is to show the potentialities of focused ion beam (FIB) tomography in a scanning electron microscope (SEM) to study intergranular oxidation. The FIB/SEM technique consists in acquiring a stack of SEM images throughout a volume of 10 to 100 μm^3 after successive abrasions using an ionic beam. FIB/SEM tomography allows following the intergranular oxidation depth and its scattering along a given grain boundary and revealing potential interactions between oxidation and metallurgical and mechanical factors such as precipitates or deformation bands [2]. It thus gives information complementary to those obtained by SEM observations in cross-section or transmission electron microscopy (TEM).

This paper focuses on the intergranular oxidation of Alloy 600 and its weld material Alloy 182. Oxidation tests were performed in primary water at 340°C and 360°C during 1000h to 1400h. The effects of intergranular chromium carbide precipitation, grain boundary character and pre-straining

are studied either on industrial heats or model microstructures. An example of the 3D reconstruction obtained on a model microstructure of Alloy 600 without any intergranular chromium carbide is shown in **Figure 1**.

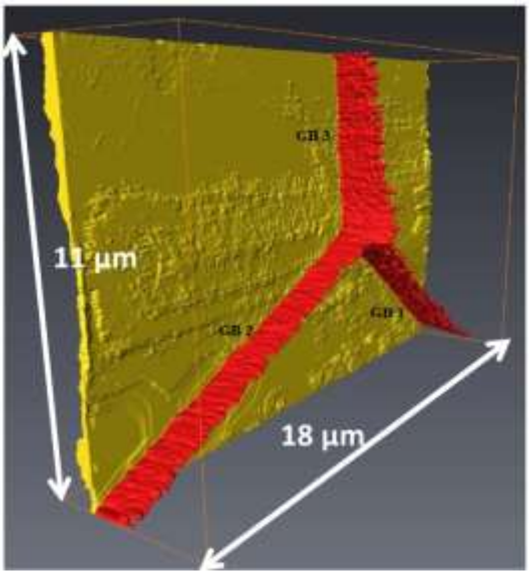

***Figure 1** :* 3D reconstruction of the intergranular oxide penetrations along three grain boundaries in a sample without any chromium carbides (intergranular oxide in red; surface oxide layer in yellow).

The results obtained from FIB/SEM tomography show that, in a model microstructure without any carbide or pre-straining, the oxide depth is homogeneous along a given grain boundary. On the contrary, a local strain heterogeneity (measured by digital image correlation) at the grain boundary significantly enhances grain boundary oxidation and generates scattering in the oxide depth along the grain boundary. By contrast, no significant effect of a homogeneous local strain is observed. The presence of intergranular chromium carbides strongly decreases the intergranular oxidation kinetics. The oxide depth is inversely proportional to the grain boundary coverage with carbides. Additional TEM analyses suggest that chromium carbides favor the formation of Cr_2O_3 instead of the chromium-rich spinel oxide usually observed. At last, twin boundaries are much less susceptible to intergranular oxidation than high angle grain boundaries. At last, a tensile test was performed in-situ in a SEM on a pre-oxidized specimen until fracture of the intergranular oxide occurs. FIB/SEM tomography was then used to study the crack path in the oxidized grain boundaries. The results show that cracking occurs at the alloy / oxide interface and stops at the intergranular oxide tip.

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