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Characterization of Corrosion Oxides on NiCrFe Alloys by Grazing Incidence X-ray Diffraction

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Introduction: Corrosion-resistant NiCrFe alloys are used in various locations throughout water-cooled nuclear reactor circuits. Despite their low corrosion rates, the corrosion oxide layers, commonly in the form of AB_2O_4 (spinel structure), that form on alloys have a tendency to incorporate radioactive cobalt circulating in the reactor coolant. To fully understand the corrosion mechanism and to evaluate the effectiveness of potential fixes, it is of importance to determine the chemistry of spinel phase as a function of water conditions. In this work, only laboratory prepared, non-radioactive alloy specimens were studied. Since the corrosion layer is typically a few hundreds Å thick, synchrotron grazing incidence X-ray diffraction was used to (1) determine the spinel chemistry through lattice parameter – the relationship between them has been reported in various literature; and (2) estimate the corrosion layer thickness by changing X-ray incidence angle.

Results: The experimental setup consists of an incidence beam Si double-crystal monochromator, and a Ge(1 1 1) diffracted beam analyzer. $\lambda = 1.15 \text{ \AA}$ was used. Diffraction patterns were measured with various incidence angles, starting from 0.1° , to monitor the spinel peak intensity change and the occurrence of substrate peak as a function of penetration depth. Figure 1 below show a pattern in which the spinel peaks are marked based on a refined cubic cell of 8.3524 \AA . Prior to lattice parameter refinement, all the peak positions were corrected for refractive index effect according to Lim et al [1]

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References:

[1] G. Lim, et al., "Grazing Incidence Synchrotron X-ray Diffraction Method for Analyzing Thin Films", J. Mater. Res. **2**(4), 471, Jul/Aug 1987

