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#4-99 Investigation of AISI 316L oxide film formation under PWR PW conditions using Electrochemical Noise (EN)

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Oxide film formation is a critical factor in the corrosion resistance of steel components in Nuclear Power Plants (NPPs), providing a protective barrier against aggressive environments. The stability and integrity of this oxide layer are key to maintaining the durability of steels over time. Electrochemical Noise (EN) has emerged as a promising in-situ monitoring tool for investigating oxide film behavior under operational conditions. It has the ability to capture the naturally occurring (stochastic) fluctuations in current/potential that reflect oxidation dynamics, without the need for external perturbation, as required in techniques like Electrochemical Impedance Spectroscopy (EIS). In stainless steels exposed to Pressurized Water Reactor (PWR) primary water (PW) conditions, the oxide layer typically develops a duplex morphology: an outer layer made of micrometric, individual magnetite crystallites, and a thin inner layer composed of a mixed Fe-Ni-Cr spinel oxide. According to the Available Space Model [*L. Martinelli et al., Corrosion Science 100, 253 (2015)*], the outer layer is developed through the time-continuous (noiseless) cationic release from the base metal, generating metal vacancies. These vacancies can cluster into nano-voids [*B. Eyre, Journal of Physics F: Metal Physics 3.2, 422 (1973)*] and allow O^{2-} anions to diffuse inward and drive the time-discrete growth of the inner oxide layer, resulting in anodic current transients (current noise). The primary objective of this study was to check the occurrence of such current transients and to analyze them in the frequency domain. Achieving this objective provides a foundation for using EN as a tool to monitor and better understand oxide layer dynamics in real-time. The experimental configuration consisted in a 3-electrode setup with two identical 316L AISI samples acting as the Working Electrode (WE) and pseudo-Reference Electrode (pseudo-RE), while a 304 AISI tube of much larger surface served as Counter Electrode (CE). All electrodes were positioned within a static stainless steel autoclave under simulated PW conditions at 320°C and connected via electrically insulated terminals to a BioLogic SP-200 potentiostat. A series of anodic potentiostatic polarizations of the WE (100 to 400mV above the reference) is conducted to promote oxide film formation. Once the stationary anodic current was reached, recording of the current was performed. Using the BioLogic SP-200, the lowest available cutoff low-pass filter is 5 Hz. Respecting the Nyquist-Shannon theorem, the sampling frequency for the current signal was established at 10 Hz (0.1 s). The current recordings consist of 10 individual samples, each purposely lasting 2^{17} s (i.e., 2^{18} data points per sample), to accommodate the upcoming *Fast Fourier Transformation (FFT)* calculations over a frequency domain ranging from 5Hz down to mHz scale. The EN data analysis was performed in the frequency domain via the Welch's algorithm. The original signal is divided to a number of segments (N) of length (L), overlapping by (D) points (i.e., avoiding any loss of information at the intervals'edges). Each of these overlapping segments was detrended to remove the signal's DC component & then windowed with selected window functions (e.g., Hann, Hamming) removing amplitude inconsistencies at the extremities of time records [*S. Ritter et al., Materials and Corrosion 63.4, 297 (2012)*]. The resulting *Power Spectral Density (PSD)* spectra describe the signal's power density per frequency "*bin*" in (A^2/Hz). Slight increase of stationary current values was observed between experiments, indicating that the rate of metal vacancy generation weakly increases with overpotential. The spectral representations of current noise primarily exhibited flicker-type noise, characterized by a single high-frequency roll-off slope of non-integer exponents. However, large amplitude transients followed by an exponential-like decay introduced lobes & oscillations in the corresponding PSD spectra. The occurrence rate of these strong anodic events increases with the anodic overpotential,

suggesting that the annihilation of large vacancy clusters is enhanced. In summary, passivation actually develops an EN signature which still needs to be fully analysed. The results obtained confirm the feasibility of developing an in-situ measurement technique to investigate the oxidation behavior of 316L AISI under PW conditions, initially in the absence of neutron flux.

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