doi:10.1017/S1431927622008029
Microsc. Microanal. 28 (Suppl 1), 2022

Transmission EBSD of Aluminide Coatings on Stainless Steel in a Scanning Electron Microscope

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Aluminide coatings can provide an effective hydrogen permeation barrier on 316 stainless steel claddings in nuclear environments. A quantitative characterization of the complex microstructures in both pre-irradiated claddings will provide allow for identifying key microstructural transformations in these materials in nuclear environments. Previous work on the complex microstructure in the pre-irradiated aluminide coating identified four distinct regions; the interface, columnar, transition and functional layer (Figure 1; 1). Prior characterization efforts utilized EDS, WDS, EPMA and EBSD to understand the complex microstructures and chemistries of the micron to submicron features (Figure 1;1,2). While EBSD is beneficial for rapid characterization of crystallographic structures over large areas, there are resolution limitations for features on the submicron scale. Transmission Kikuchi diffraction (TKD) in SEM allows for a quantitative characterization of complex submicron features. The purpose of this study was to establish optimal beam parameters for TKD in SEM using a Bruker OPTIMUS on-axis TKD detector head to expand on the baseline characterization previously conducted on pre-irradiated aluminide coatings.

The OPTIMUS is a unique detector head that can be inserted underneath an electron transparent sample. This configuration allows for an increase in the diffracted electron signal while minimizing the distortions typically exhibited with a tilted sample orientation used by standard TKD geometry. This approach effectively transforms an SEM into a low energy TEM. TKD samples were prepared from the four different regions of the aluminide coating using a focused ion beam (FIB) sample preparation procedure similarly used for TEM specimens (<100 nm). Analytical conditions for TKD in SEM analysis were evaluated using various accelerating voltages, beam currents, and detector distances to determine the optimum conditions.

Our results demonstrate how the technique of TKD, with the advantage of the Bruker OPTIMUS on-axis detector geometry, can be used to produce orientation maps of microstructures, can discriminate between complex crystallographic phases, and identify key textural relationships within the grains and the grain boundaries, provide information about inherent localized strain at the nanoscale in pre-irradiated coated cladding (Figure 2).

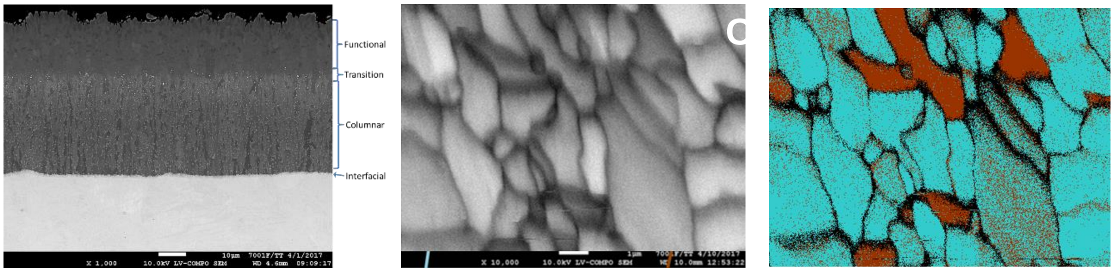

Figure 1. Micrographs of aluminide coating on 316 stainless steel and EBSD of functional layer. (A) BSE micrograph showing the full length of the coating; EBSD analysis of functional layer of aluminide coating. (A) EBSD Pattern quality map of outer layer; (B) EBSD phase distribution map showing the presence of 2 different phases: Brown = $Al_{182}Cr_{52}Ni_4$; Light blue = $Al_{3.2}Fe_1$

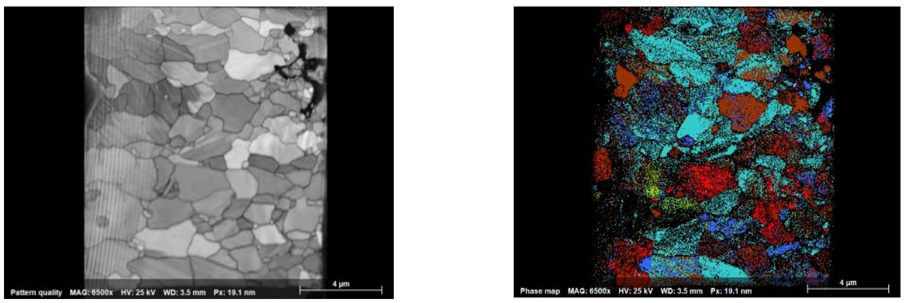

Figure 2. Transmission Kikuchi Diffraction analysis of aluminide coated at the functional layer. (A) TKD Pattern quality map; (B) TKD phase map showing the presence of five different phases: red = $Fe_{2.65}Ni_{1.45}Al_{9.9}$; green = $Al_{5.6}Fe_2$; Dark blue = Al_8Cr_5 ; Light blue = $Al_{3.2}Fe_1$; Brown = $Al_{182}Cr_{52}Ni_4$

Reference:

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