

## Compatibility of Thermally-Grown Alumina with Pb-Li at 500 °C

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The dual coolant Pb-Li (DCLL) blanket concept is the leading candidate for a U.S. test blanket module (TBM) for ITER [1], the large tokamak-type fusion reactor being built in France. Two of the key issues for the use of Pb-Li as a coolant are compatibility with the ferritic-martensitic structural materials of the blanket and tritium inventory control. Alumina and especially alumina-forming coatings have been suggested as a strategy for corrosion protection and alumina is well known to have a low tritium permeability [2]. However, experiments with thermally-grown alumina in contact with Pb-Li did not show a significant permeation reduction. The hypothesis that Pb-Li transformed the alumina barrier layer to  $\text{LiAlO}_2$  was recently confirmed for a Pb-Li exposure at 800 °C [3]. The goal of the present work was to study the compatibility of thermally-grown  $\alpha\text{-Al}_2\text{O}_3$  in Pb-17Li closer to the TBM operating temperature of 500 °C.

In this experiment, an alumina layer was formed on commercial ODS FeCrAl (Plansee PM2000, Fe-20Cr-10.6Al-0.4Ti-0.2Y-0.7O) by oxidizing for 2 hours at 1000 °C in dry  $\text{O}_2$ . The pre-oxidized coupon ( $\sim 1.5 \times 11 \times 19$  mm) was then isothermally exposed to Pb-Li in a sealed Mo capsule for 1000 hours at 500 °C. Experimental details can be found elsewhere [3]. After exposure, the specimen was characterized by scanning electron microscopy, X-ray diffraction (XRD) and metallographic sectioning. Transmission electron microscopy (TEM) samples were prepared by focus ion beam (FIB) lift-out in a Hitachi NB5000 dual-beam FIB. Microstructural characterization was performed using a Philips CM200 transmission/scanning-transmission electron microscope (TEM/STEM) equipped with a Schottky field emission gun (FEG) and operated at 200 kV. This instrument was also equipped with an EDAX energy dispersive spectrometer (EDS) interfaced with an EmiSpec data collection system.

After the 500 °C exposure, the specimen mass change was  $-0.11 \text{ mg/cm}^2$ , which was less than the  $0.2 \text{ mg/cm}^2$  mass loss observed after exposure at 700 °C. The XRD analysis did not show the presence of  $\text{LiAlO}_2$ , only  $\alpha\text{-Al}_2\text{O}_3$ . The TEM analysis showed a uniform adherent oxide layer on the ODS FeCrAl surface with a thickness of  $\sim 320$  nm (Fig. 1a). Like the initially grown alumina, the oxide scale after exposure contained two layers. An inner layer that contained columnar grains normal to the metal/oxide interface and an outer layer ( $\sim 130$  nm), which was equiaxed. Diffraction analysis confirmed the presence of  $\alpha\text{-Al}_2\text{O}_3$  and no evidence for  $\text{LiAlO}_2$  formation, suggesting that the transformation observed at 700° and 800°C does not occur after only 1000 hours at 500 °C. The initial EDS elemental maps did not show Y or Ti segregation to the alumina grain boundaries, Fig. 1b, as was observed in the pre-formed  $\alpha\text{-Al}_2\text{O}_3$  [3]. However, there was some indication of Ti enrichment near the metal-scale interface. Additional elemental maps are needed to study the segregation in the alumina scale and the outer oxide layer. Analytical techniques, such as Auger spectroscopy and electron energy loss spectroscopy (EELS), will be conducted to look for the presence of Li, since Li can not be detected by EDS.

## References

- [1] M. Abdou, et al., *Fus. Sci. Tech.* 47 (2005) 475.  
 [2] J. Konys, A. Aiello, G. Benamati and L. Giancarli, *Fus. Sci. Tech.* 47 (2005) 844.  
 [3] B. A. Pint and K. L. More, *J. Nucl. Mater.* 376 (2008) 108.  
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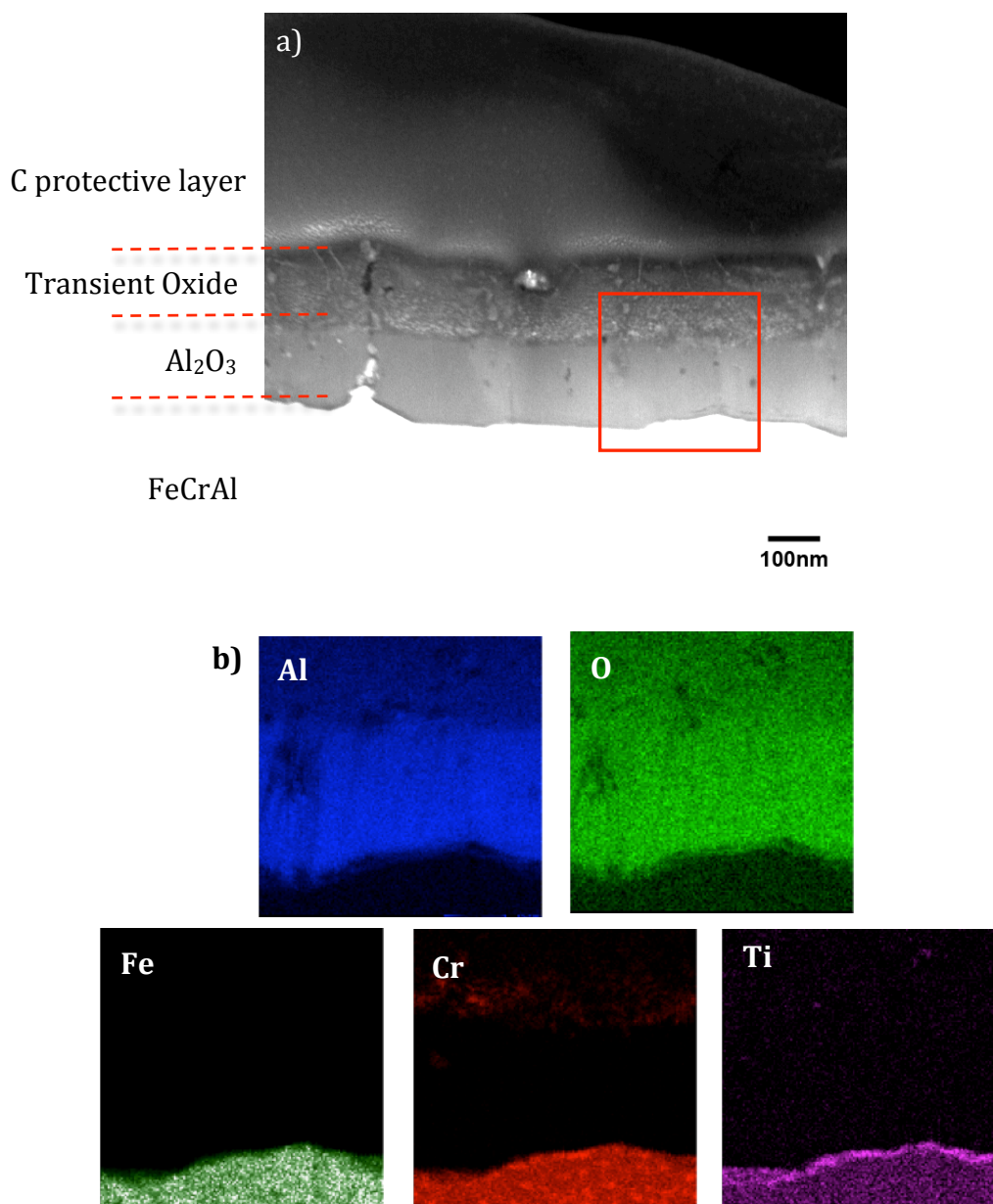


FIG. 1. a) High angle annular dark field (HAADF) STEM image showing the oxide scale formed on FeCrAl alloy following pre-oxidation for 2h at 1000°C and Pb-Li exposure for 1000h at 500°C. b) Elemental maps for Al, O, Fe, Cr, and Ti acquired from the area marked on the DF HAADF image.