

# Electron Microscopy Analyses of Oxide Scales Formed on Aluminide Diffusion Coatings and NiAl Bulk Model Alloys

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Aluminide diffusion coatings have long been used as systems increasing the oxidation resistance of nickel base superalloys. Over the last years addition of Pt to the coatings became an interesting method used to improve the performance of the coatings. However the mechanisms behind this improvement and oxidation behaviour of such coatings, are not yet fully understood. One of the reasons for this is that present knowledge about nucleation and growth of  $\text{Al}_2\text{O}_3$  scale and the microstructure and microchemistry of the oxide-metal interface on the nanometer-scale is still very limited.

The present work deals with microstructural investigation of both commercial aluminide diffusion coatings and NiAl model alloys after short-time oxidation in air. The commercial coatings (one simple and three Pt-modified aluminide coatings with different Al- and Pt-content) were applied on the same Ni-based superalloy (CMSX-4). To exclude possible influence from the other elements than Al and Ni on the oxide growth two  $\beta$ -NiAl model materials containing Al and Ni in different proportions were also investigated.

The materials were polished to the same surface finish prior to oxidation. The oxidation treatment was performed in laboratory air at 1050 °C for 1 h. The microstructure of all the materials was studied using Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM). To study the oxide scale cross sectional samples for TEM were prepared by Focused Ion Beam (FIB) method.

Studies of the commercial coatings reveal two clear trends: 1) faster formation of the stable  $\alpha$ - $\text{Al}_2\text{O}_3$  scale on coatings containing high Al content at their surface 2) faster formation  $\alpha$ - $\text{Al}_2\text{O}_3$  on coatings with high amount of Pt at their surface (Fig1 a, b).

Investigations of the oxide scales formed on the two model NiAl alloys show that the scales have very few contact areas with the underlying metal, Fig 2. The oxide scale thickness at contact areas and above the interfacial voids differs slightly, being thinnest at the contact areas. This is a consequence of the contact areas consisting of the slowly growing stable  $\alpha$ - $\text{Al}_2\text{O}_3$  phase, while the remaining oxide contains mainly fast growing meta-stable alumina phases Fig 3. The observed origin of large interfacial voids seems to be best explained by fast outward transport of Al through lattice defects within the meta-stable alumina. This will result in an Al vacancy flux toward the interface, vacancy coalescence and formation of voids. The  $\alpha$ - $\text{Al}_2\text{O}_3$  grows mainly by inward oxygen transport through the grain boundaries. Thereby,

no vacancies are injected to the oxide-metal interface and this part of the oxide scale can remain in contact with the alloy. The fast grain-boundary transport of oxygen through the interface between  $\alpha$ - $\text{Al}_2\text{O}_3$  grains in contact with the metal and the surrounding detached oxide accounts for the fast growth of the stable alumina phase.

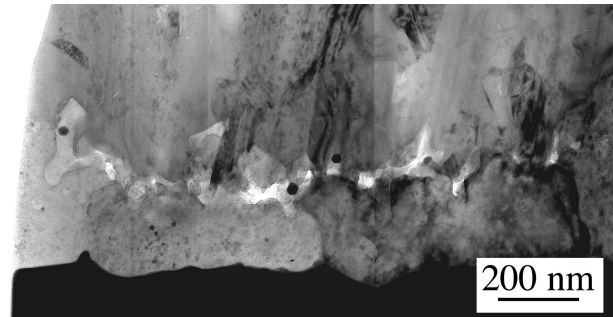


Fig. 1a TEM image of the oxide grown on the coating with high Al content (55%) at the surface. A continuous  $\alpha$ - $\text{Al}_2\text{O}_3$  layer at the oxide/metal interface is clearly visible. A layer containing  $\gamma$ - $\text{Al}_2\text{O}_3$  is situated on top of the  $\alpha$ - $\text{Al}_2\text{O}_3$  layer.

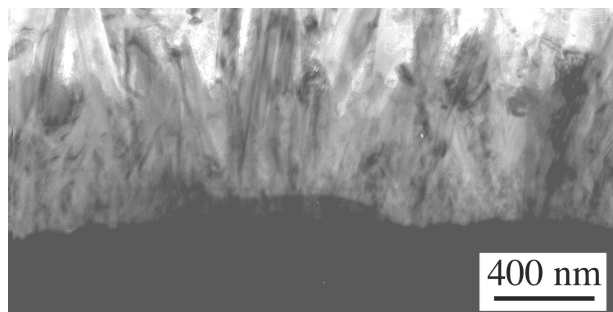


Fig. 1b TEM image of the oxide grown on the coating with low Al content (43 at%) at the surface. The oxide consists mainly of  $\gamma$ - $\text{Al}_2\text{O}_3$ .

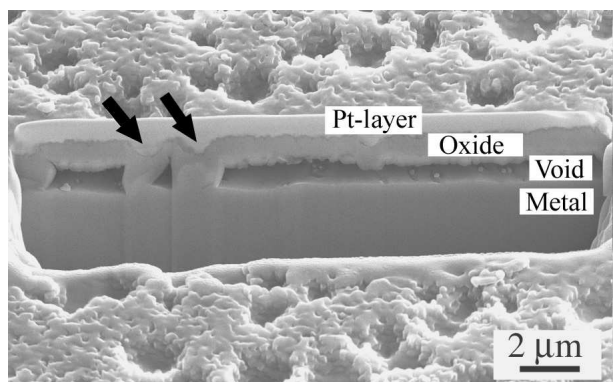


Fig. 2 FIB-SEM cross section image. The interfacial voids and the oxide-metal contact areas (arrows) are clearly visible

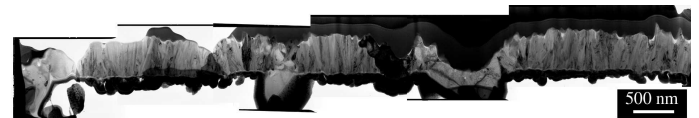


Fig. 3. TEM image of oxide scale on  $\beta$ -NiAl (39 at% Al) alloy. Transient  $\text{Al}_2\text{O}_3$  phases have grown on top of the voids, while  $\alpha$ - $\text{Al}_2\text{O}_3$  is the only phase at the oxide-metal contact areas.