Thin Films and Coatings

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TEM investigations of alumina coatings after corrosion experiments

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Alumina coatings are demonstrated to protect metallic substrates against corrosion in order to increase their application area and lifetime. Thin coatings with thicknesses of around 400 nm were produced by a sol-gel-process on INCONEL 718 Ni-base superalloy substrates. For this coating system different corrosion experiments were performed to investigate the quality of the coating, the warranty of fitness for a particular corrosion type and the modification of the coating system during the tests. So, high- temperature corrosion experiments were done in different atmospheres like dry air or steam [1, 2, 3] at 800°C for 800 hours. Adhesion tests to evaluate the thermo mechanical properties of the coating-substrate system were performed by cyclic loading with a Nd:YAG Laser beam [4].

An established method to investigate corrosion processes during electro chemical influences to material is the electrical impedance spectroscopy. For this, samples were prepared by a spin-coating process and heat treated at 800°C for 30 minutes. Then they were brought in contact with a NaCl solution at room temperature for up to 180 hours. During the test time of few minutes an AC voltage was applied. As result of these investigations the polarization resistance of the samples increased with dwell time. This leads to the assumption, that material transport took place and the coating became denser by formation of an interlayer between substrate and coating or by deposition of corrosion products inside the coating. The aim of the TEM investigations was the comparison of the loaded and the unloaded samples to identify morphological and/or chemical differences between them and so to conclude on protection possibilities of alumina coatings on INCONEL 718 against corrosion in NaCl containing media. The TEM samples were prepared as cross-sections perpendicular to the coating surface by the focused ion beam method (FIB) by means of the in-situ lift out technique in a Quanta 3D instrument. To avoid surface damages, in front of the ion-beam-assisted deposition of a Pt bar an electron-beam-assisted Pt deposition was performed. The TEM investigations were carried out in an analytical STEM JEM 2200FS at 200 keV acceleration voltage. Beside images in TEM and STEM mode, HREM investigations, electron diffraction, energy filtered TEM (EFTEM) and EDX investigations were done. These results show small but visible morphological differences between the loaded and the unloaded samples. The superficial zone of the loaded sample shows a compacted region of 15 nm thickness, the superficial region of the unloaded sample does not show any compacting. Differences in the elemental distribution are also detected. So, the Cr-diffusion zone at the interface of the loaded sample shows a higher Cr content than this region of the unloaded sample. After the Cr-diffusion zone of the coating near the interface, an Al-rich region with a relatively sharp gradient is detected. In the unloaded sample the Al enrichment in this region is low and the gradient is flat. These features are demonstrated in the TEM images and EDX linescans in Figures 1 and 2. Thus, the assumption of material transport during exposure is supported. In the result the coating becomes more protective against corrosion by NaCl solution.

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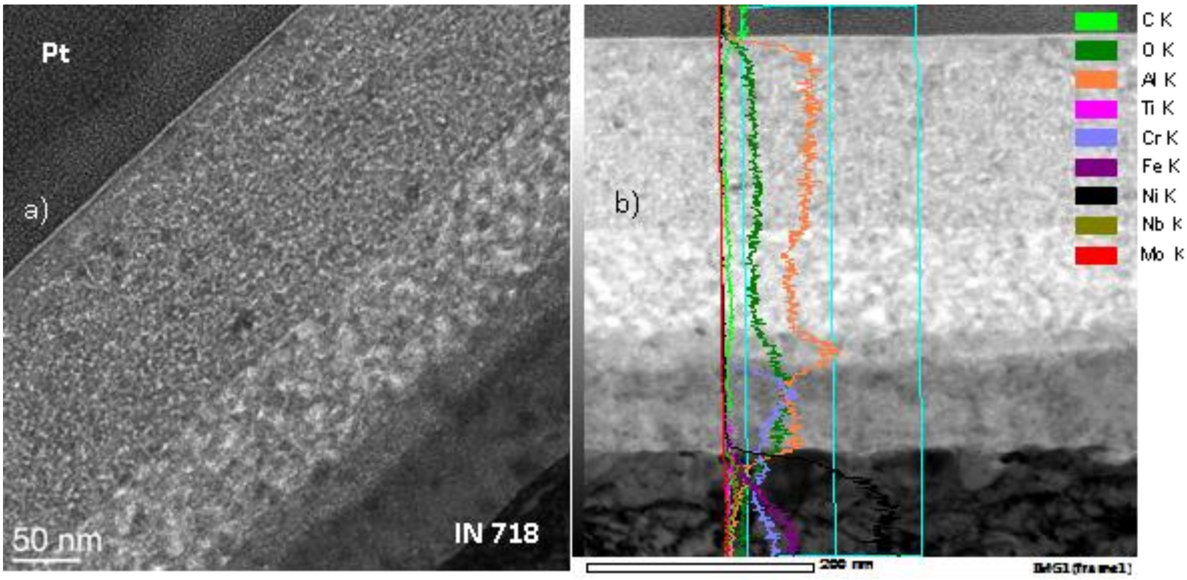

Figure 1. Loaded sample, a: TEM image, b: STEM bright field image with EDX linescan across the coating

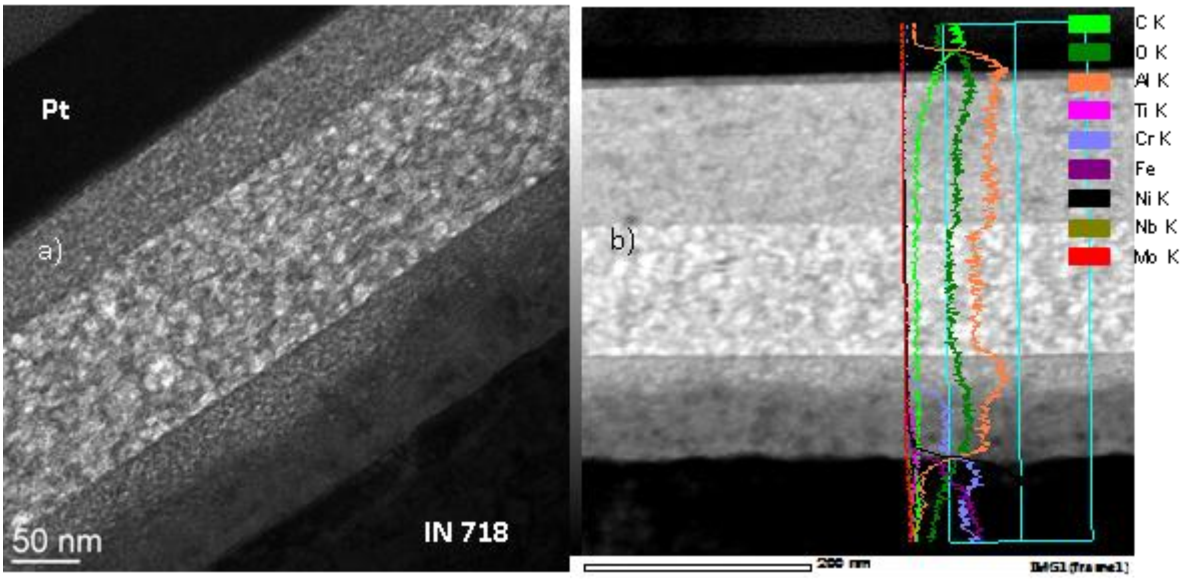

Figure 2. Unloaded sample, a: TEM image, b: STEM bright field image with EDX linescan across the coating