

TEM Analysis of Aluminum Anodization Layers – Cryo-EFTEM and Electron Tomography

C. Kübel* and S. Dieckhoff*

*Fraunhofer Institute for Production Technology and Applied Materials Science, Wiener Straße 12, 28359 Bremen, Germany

Corrosion protection of aluminum is essential for applications in the aircraft or automotive industry. Anodization of aluminum with chromic acid has been successfully used to generate thick, porous oxide layers, which exhibit good long-term stability with organic coatings such as paint systems or adhesives. However, due to environmental and health reasons, the chromium (VI) containing anodization processes traditionally used for corrosion protection have to be replaced by environmentally friendly alternatives. So far, these chromic acid free alternatives must still be optimized to become a good replacement for the chromium (VI) containing processes.

An important aspect of the anodization layer is its porosity and interaction with the organic coating. The organic coating must penetrate the pores of the oxide to enable a good mechanical interlock between the coating and the substrate. In addition, the organic coating also needs to fill the pores to prevent diffusion of water into the pores, which would weaken the oxide layer. We used a combination of cryo energy filtered imaging (EFTEM) and EDX analysis to image the distribution of the organic coating in porous anodization layers prepared by both a chromic acid and a sulfuric acid containing process (Fig. 1). In both cases, we could show that the organic coating penetrates the pores down to the interface with the aluminum substrate, thus fulfilling one basic requirement for a stable coating.

Furthermore, electron tomography was used to gain a better understanding of the pore structure itself. Due to the complex, irregular geometry of these oxides, direct 2D images are difficult to interpret accurately. However, electron tomography results in a complete 3D reconstruction of the sample structure (Fig. 2), which provides additional insight into the pore structure. In this talk, we will show the possibilities that these techniques bring to coating technology.

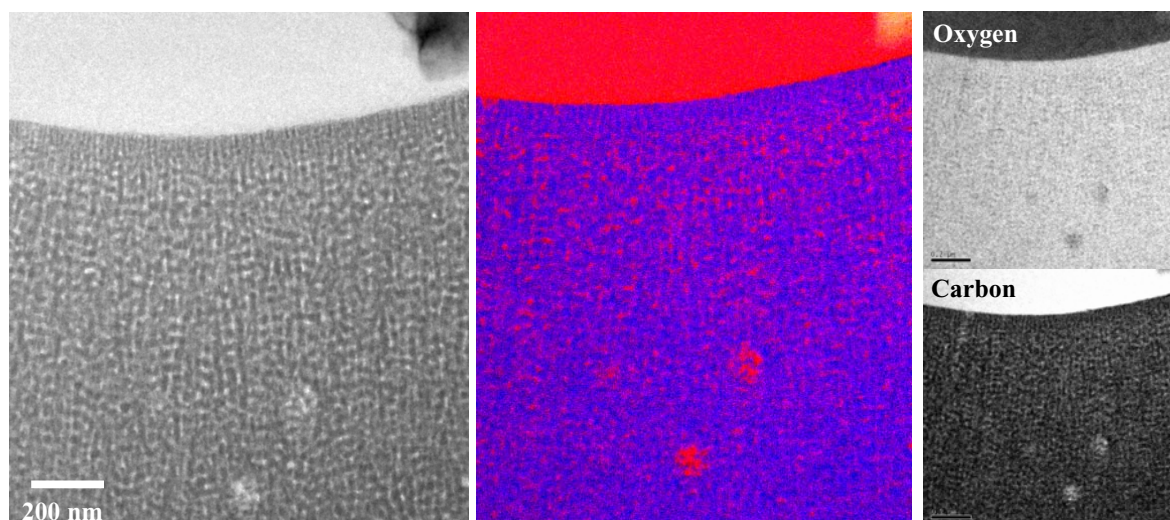


Figure 1: BF-TEM image of the interface between anodization layer and organic coating and the corresponding false color image showing the elemental distribution (carbon – red, oxygen – blue). The small images show the original carbon and oxygen elemental maps obtained under cryo conditions.

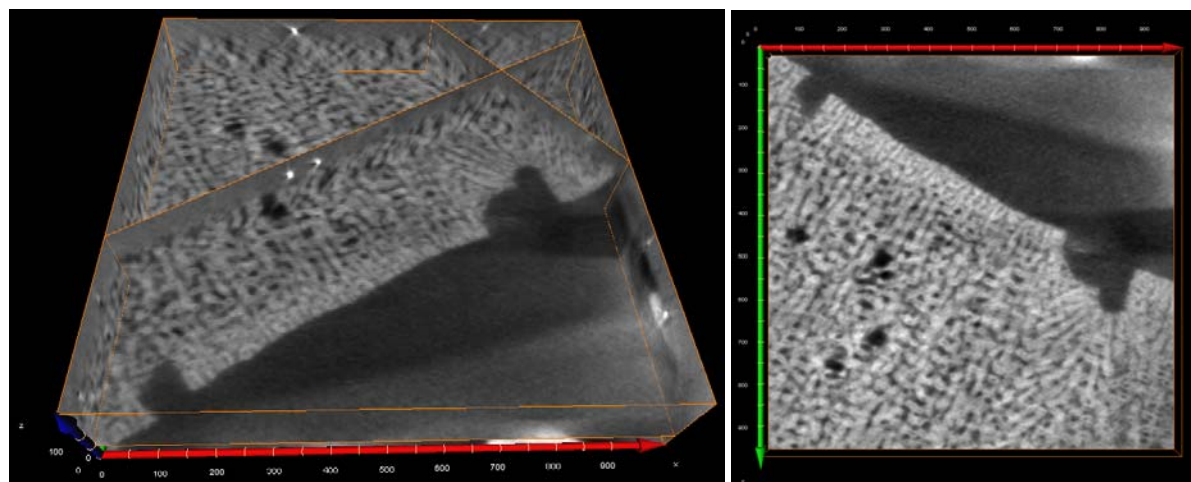


Figure 2: Two nanometer thick digital slices through the reconstructed 3D volume of the anodization layer showing the complex pore structure obtained by the sulfuric acid containing process. Several small voids and surface defects are clearly visible. Furthermore, significant changes in the local orientation of the pores can be observed underneath the bigger defect in the surface.