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The behavior of additive manufactured aluminum alloys upon anodization

Lydia Daum[1], Dr. Stefan Ostendorp[1], Dr. Martin Peterlechner[2], Prof. Dr. Gerhard Wilde[1]
[1]Institute of Materials Physics, University of Münster, Münster, Germany, [2]Laboratory of Electron Microscopy, Karlsruhe Institute of Technology, Karlsruhe, Germany

Background incl. aims

Additive manufacturing of aluminum alloys, i.e. laser powder bed fusion (3D printing), introduces significant versatility and innovation to various industrial applications. Al-Fe-Zr alloys, which are suitable for additive manufacturing, exhibit mechanical properties comparable to conventionally cast aluminum alloys that are alloyed with Mg, Mn and Si, such as the 60xx series Al-alloys. Anodization of aluminum alloys is performed to enhance their chemical and mechanical performance by the formation of a protective anodic aluminum oxide (AAO) layer. The absence of those 60xx series alloying elements in Al-Zr-Fe, which typically form voids in AAOs and alter the structural integrity, suggest a distinct anodization behavior. This might lead to improved chemical and mechanical properties of the grown passivation layer.

Additionally, the overall microstructure of 3D printed alloys inherently differs from conventionally casted, processed and artificially aged microstructures of the same alloy. Here, a variety of elongated and equiaxed grains are observable along the printing direction. This disparity underscores the necessity for a comprehensive comparison of differently treated microstructures prior to anodization. Afterwards, the anodization behavior of Zr- and Fe-enriched precipitates are investigated to ascertain their impact on the chemical resistivity of the resulting AAOs.

Methods

The microstructure of the Al-Fe-Zr alloy was modified through changes in the manufacturing method, varying degrees of severe plastic deformation and adjustments to the heat treatment before anodization. Polished sample surfaces are analyzed with EBSD and subsequently anodized under galvanostatic conditions in oxalic acid solutions following a standardized protocol which allows to determine the impact of different precipitates' sizes and electrochemical potentials. All AAO-coated samples are bisected using a wire saw, which each half treated independently thereafter. The first half is directly coated with Cr/Au to enhance electron conductivity for TEM lamella preparation in FIB whereas the other half was exposed to reactive ion etching to study the chemical resistivity of the AAO.
TEM samples are prepared by employing Ga-, Si/Au- or He/Ne-FIB on randomized surface positions. The AAOs were analyzed in a Gatan Double Tilt LN2 Cooling Holder 636 using a Titan Themis 60 – 300 operated at 300 kV and at a temperature of 96 K. EDX spectra are recorded with a quadrupole SuperXG2 detector. A CCD-GIF camera is used to record nano-beam diffraction patterns (NBDPs), also denoted as 4D-STEM. Beam damage is suppressed by using beam currents as low as 10 pA during NBDP acquisition.

Results

Given the difference of current densities during anodization of various types of microstructures while maintaining the standardized protocol, the analysis of various NBDPs of different precipitates – each with a stack size of at least 10000 NBDPs – suggests a size-dependent anodization behavior of Fe-enriched precipitates and underscores the influence of the underlying microstructure and the participating precipitates on the grown AAO thickness. Using 4D-STEM and fluctuation electron microscopy, various oxide types within the AAO were distinguished and information about the precipitates' structure and chemistry are correlated, which supports the hypothesis of size- and type dependent influence. Ga-contamination due to FIB preparation at grain and phase boundaries was diminished by changing to Si/Au or He/Ne FIB for the final polishing step. Finally, we attribute the microscopic differences in AAO growth to macroscopic properties such as chemical resistivity.

Conclusions

Based on the results, Fe-rich precipitates remain partially unaffected by anodization, depending on their size and type, while Zr-rich ones are anodized with the matrix. Unlike conventional alloys, Al-Zr-Fe alloys do not develop voids within the AAO, which leads to a distinct chemical resistivity. Moreover, the microstructure is affecting the properties of the AAO. This emphasizes the importance of selecting an advantageous microstructure tailored to the intended application of the protective layer.

Keywords:

Anodization, 4D-STEM, cryogenic measurements, precipitates