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(G*) Formation of titanium oxide in radiolytically decomposed water

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Performing in-situ ion beam analysis to determine metal oxide growth mechanisms poses challenges due to the incompatibility of a electrolyte solution with ultra-high vacuum (UHV). To circumvent this problem, a specialized in-situ cell was developed which isolates the liquid electrolyte from the UHV using a silicon wafer, preventing any contact between vacuum and liquid. This wafer is equipped with a 50-200nm thin Si3N4 window, then coated in the metal under investigation and inserted with the metal in contact with the electrolyte, isolating the electrolyte from the vacuum. As a result, the ion beam can pass through the Si3N4 window, interacting with the material on the opposite side. This technique allows electrochemical methods such as anodization and impedance measurements to be taken under UHV and in-situ with Rutherford backscattering spectroscopy (RBS).
Upon preliminary testing of magnetron sputter deposited thin film Ti, higher oxide growth during anodization was reported compared to literature and ex-situ anodization studies. Exposure of Ti/Si3N4 sample to a 1 MeV He+ ion beam for 30 minutes with no applied potential showed a spontaneous formation of a continuous oxide layer. Next, the study focused on radiolysis product generation after ruling out considerations such as charging from the ion beam on the Si3N4 surface facing vacuum. Radiolysis was done using two separate alpha sources; 241Am at 0.525 Bq, 5.7 MeV, and 241Am/247Cm/244Pu at 0.525 Bq, 5.7 MeV. Ti samples with known oxide thickness were submerged in 0.27 M NaCl solution for various times, with a maximum of 193 hours, with the nuclide source facing the solution. Additional covered and uncovered samples were created with similar setups for control. Channeling RBS experiments were performed on the samples, and the resultant spectra were analyzed using SIMNRA to determine oxide growth as a function of incident alpha particles. Using linear regression analysis, the growth of titanium oxide as a result of alpha radiolysis in a conductive analyte was quantified. From these experiments, it was found that the formation of radiolysis products in an electrolyte solution contributed to the spontaneous oxide growth of Ti.

Keyword-1

Titanium

Keyword-2

Radiolysis

Keyword-3

Oxide Growth

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