In-situ real-time and ex-situ spectroscopic analysis of Al_2O_3 films prepared by plasma enhanced atomic layer deposition

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In-situ techniques provide a very powerful means to further improve atomic layer deposition (ALD) processes and their preparation equipment. Ellipsometry has widely been used to investigate thin films prepared by ALD. Consequently, in-situ ellipsometry has been developed to monitor ALD growth in operando.[1]
In this work in-situ real-time ellipsometry (irtE) with a very high time resolution of 24 ms was used to investigate the growth of inductively coupled plasma enhanced (ICPE) ALD of Al_2O_3. Utilizing this technique, it is possible to resolve each step of the ALD cycle in detail and in real-time. The combination of in-situ measurements with ex-situ ellipsometry (UV-VIS-NIR-SE and IR-SE) and X-ray photoelectron spectroscopy (XPS) allows correlating surface effects observed by in-situ ellipsometry with the bulk properties of the ALD layers. For benchmarking, an Al_2O_3 film deposited by thermal ALD (T-ALD) with a very similar equipment was used.
The ICPEALD films were deposited at substrate temperatures ranging from 80 to 250 °C, while the reference T-ALD layer was prepared at 200 °C. The influence of the plasma exposure step was studied by varying plasma parameters such as plasma power and pulse duration.
The Al_2O_3 ICPEALD process exhibits a higher growth rate than its thermal counterpart. However, XPS measurements revealed an increase in the amount of incorporated carbon compared to layer(s) prepared by T-ALD. Simultaneously, the refractive index decreased. In-situ measurements indicated an adsorption

process, which is not typical for an ALD process. Additionally, IR measurements pointed to the presence of CH_x species, in agreement with the XPS results. A correlation was found between the duration and power of the plasma pulse and the non-stoichiometric composition of the ICPEALD aluminum oxide films. The irtE technique was successfully used to minimize the incorporated carbon and improve the film quality by optimizing the plasma parameters, resulting in high-quality Al_2O_3 layers. [2]

Reference

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