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In situ FTIR-ATR monitoring plasma-activated hydroxylation of silica using dielectric barrier discharges, vapor-phase amino-silanization, and amino-group derivatization

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In situ monitoring of thin film growth and surface modification is helpful for the elucidation of physico-chemical process mechanisms and for studies of the nature and chemical reactions of surfaces and coatings before they come into contact with the environment. FTIR-ATR spectroscopic monitoring of the modification of polyethylene surfaces in the afterglow region of dielectric barrier discharges (DBDs) in N_2/H_2 mixtures, for example, has recently been used to provide indications of the non-selectivity of aldehyde derivatization for primary-amine detection.[1,2] Previous experiments performed at IOT and IST were limited to studies of the interaction of relatively stable active species in outflows from DBDs such as oxygen or nitrogen atoms, and nitrogen molecules in the $N_2(A)$ state, with polymer surfaces. Multi-ATR elements made from electrically conductive materials like Si or Ge may, however, be used as the ground electrode in asymmetric DBD arrangements. FTIR-ATR monitoring of a surface or a growing thin film and its interaction with reactive neutral and charged species as well as energetic photons from a DBD is possible.The present contribution reports about plasma-assisted surface hydroxylation of silicon oxide layers thermally grown on Si multi-ATR crystals. The discharge is generated within a fused quartz flat DBD reactor, enabling a gas flow parallel to the crystal surface and transverse to its long axis. Argon with admixtures of H_2, O_2, and H_2O, respectively, was applied as plasma gas to find the optimum DBD treatment conditions for a subsequent vapor-phase silanization process, using aminopropyl trimethoxysilane (APTMS). Information about process kinetics and chemical nature of the surfaces could be gained from the in-situ experiments.

[1] C.-P. Klages, A. Hinze, Z. Khosravi, Plasma Process. Polym. 2013, 10, 948–958.
[2] C.-P. Klages, S. Kotula, Plasma Process Polym. 2016, 13, 1213–1223.

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