Spectroelectrochemical Detection in Bio-Electrochemical Systems with Nanocrystalline, Boron-Doped Diamond

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Diamond has exceptional material properties, including chemical inertness, extreme hardness, a broad range of optical transparency in the IR and UV-visible ranges, and advantageous electrochemical properties when boron-doped. Very few materials are both optically transparent and electrically conductive; this combination makes conductive diamond an attractive electrode material for spectroelectrochemical application. Spectroelectrochemistry is a powerful detection technique coupling electroanalytical and spectroscopic measurement, to quantitatively study *in situ* chemical concentrations "near" and at an electrode/electrolyte interface. We present here our progress in fabrication and application of conductive, nanocrystalline diamond films as optically transparent electrodes, with specific focus on internal reflection, infrared spectroscopic detection.

One major challenge for using diamond in optical applications is interfacing it to other optical materials; single-crystal diamond prisms are prohibitively expensive to permit widespread use, or the device involves bonding diamond to other materials, the bonding compromised by temperature or chemical attack. Alternatively, another simple optical geometry would involve deposition of polycrystalline diamond (micro/nano grain size) directly onto an internal reflection element. This compromise results in observation of the composite response from different diamond crystal planes and the grain boundaries. Yet, diamond-coated prisms should still provide much advantage for sensing applications.

Nanocrystalline, boron-doped diamond was deposited by standard, chemical vapor deposition onto sapphire and fused silica substrates (temperature ~ 800 C). These two materials were selected for their wide UV and IR transparency and high melting temperature. Fused silica has advantage over sapphire because its thermal expansion coefficient is closer to diamond, resulting in less film stress and thus better film adhesion. Sapphire, on the other hand, has a larger optical window than fused silica, allowing for more spectral versatility. For either substrate, the diamond film must remain sufficiently thin to maximize transparency and adhesion.

On sapphire, 1.5 hours of growth yielded a nanocrystalline, relatively smooth film that adhered well to the wafer, and maintained nominally constant transparency. The analogous growth period for fused silica was over 3 hours before the film delaminated; the film's transparency dropped from 80% down to 25% on extending growth time from 2 to 3 hours. The loss in transparency correlates with increasing grain size during continued growth, the larger grains scattering more light.

The loss in transparency with increasing growth time is also related to the increasing film conductivity, due to incorporation of (IR absorbing) boron; the increase in growth time from 2 to 3 hours on fused silica, increased the conductivity by a factor of 3. Therefore, sensor design will need to balance these factors. Currently, we are testing these diamonds, optically transparent electrodes with standard, electrochemical analytes, and will present example application in biochemical sensing.