Interferometrically Defined 3D Pyrolyzed-Carbon Sensors

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Interferometric lithography is capable of creating 3D structures with sub-micron length scales not possible using conventional lithography. Recently, we have demonstrated that we can convert these 2D and 3D submicron photoresist structures into amorphous carbon via pyrolysis in a reducing atmosphere ¹. Electrodes formed from these pyrolyzed structures show qualitatively similar electrochemical performance to standard planar glassy carbon electrodes, but possess the periodic patterns characteristic of interferometric lithography. This paper details the use of these structures for two separate sensing applications: 1) A non-enzymatic glucose sensor; 2) A SERS substrate with large average SERS enhancement factor uniform over macroscopic regions of the sample.

Figure 1A shows a scanning electron micrograph (SEM) of lithographically defined 3D carbon matrix decorated with palladium nanoparticles deposited from an organic solvent solution. The Pd particles are distributed homogenously through the matrix. Figure 1B shows the amperometric response of the Pd-decorated electrode to successive additions of glucose in a 0.1M NaOH solution. The sensor demonstrates a fast response (achieving 95% of the steady state current in less than 5 seconds) and a detection limit of 10 μ M glucose concentration.

Figure 2A contains SEM images of pyrolyzed carbon scaffolds with thin (left) and thick (right) sputtered Ag coatings. These Ag coated scaffolds provide robust enhancement factors ranging from 7 to 9 orders of magnitude. Figure 2B contains a plot of Raman scattering of three different adsorbed analytes, all showing significant enhancement. For comparison, the Raman signal from a rhodamine coated planar silver film is shown, magnified by 100 to fit on the scale. Figure 2C shows a 100x optical microscope image and Figure 2D shows the a spatial image of the Raman signal over the 5 x 5 micron region from Figure 2C, covering several lattice periods of the structure in 50nm steps. The variation of the Raman signal over the entire scanned area is only a factor of 2.5, demonstrating excellent uniformity for such large enhancement factors. This paper will present detailed fabrication and characterization data of these materials.

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Figure 1. (A) 3D carbon scaffold decorated with Pd NPs from an aqueous solution (2mM Pd/0.5 M H_2SO_4). (B) Typical amperometric response of Pd-modified porous carbon electrode towards successive additions of glucose in 0.1 M NaOH.



Figure 2. (A) SEM images of carbon scaffold with thin (left) and thick (right) sputtered Ag coatings. (B) Graph of Raman shift for 3 different target analytes. Bottom curve (x 100) is the Raman signal from rhodamine on a planar silver film with equivalent sputtering conditions. (C) 100 X mag optical image (D) Raman signal from region in (C). (inset) Graph of maximum and minimum Raman signal over area. The max and min signals differ by a factor of 2, showing the large average Raman signal from these platforms.