Nano-Rough Gold for Enhanced Raman Scattering

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Conventional Raman scattering is a workhorse technique for detecting and identifying complex molecular samples. The presence of a nano-rough metallic surface in close proximity to the sample has been shown to enhance the scattered Raman light signal enormously.[1] This paper reports a technique for obtaining nano-rough gold surfaces using simple apparatus and no lithographic steps.

A (100) silicon wafer was first etched in a 3:7 solution of HF in ethanol under an applied electric field (Fig. 1).[2] In our apparatus the etched area was defined by an O-ring and Teflon block and the current density was typically 13mA/cm^2. The etching starts as closely spaced narrow holes which are undercut as the holes deepen. Figure 2a shows an SEM of the surface of an etched wafer, and Figure 2b shows a similar area after approximately 30 nm were removed by reactive ion etching in SF_6.

Approximately 20 nm of gold was sputtered on the etched surface of the silicon wafer, and covered with a thin layer of UV curing epoxy under a flexible plastic foil. The epoxy chosen is ordinarily used as a “lens bond” to cement optical components, and is transparent at visible wavelengths. When the epoxy was hardened the foil and then the epoxy were peeled off the silicon wafer. The gold had relatively poor adherence to the silicon, and acted as a mold release, remaining partially on the epoxy in a non-continuous layer. Additional gold was sputtered on the epoxy to enhance the Raman scattering signal.

A Raman test sample was prepared consisting of 1 mM of Rhodamine 6G dissolved in DI water. A drop of this sample was placed on a glass microscope slide and covered with a cover slip. Its spectrum was obtained with a Horiba LabRAM Raman spectrometer operating at 633 nm (Fig. 3). Enhanced spectra were obtained by placing a drop of the sample over the peeled epoxy covered with different thicknesses of additional nano-rough gold. “Smooth” gold samples, formed on epoxy over un-etched portions of the silicon wafer, had cracks visible in an optical microscope. These samples also showed enhancement, although not as much as the samples over the etched areas (Fig. 4). The degree of enhancement depends critically on the surface topography of the gold. Results will be presented showing enhancements as a function of the details of wafer preparation and gold deposition.

This work was supported in part by the National Institutes of Health.

1) G.L. Liu and LP Lee, Appl. Phys. Lett., 87, 074101, 2005.
2) P.R. Malempati, Master thesis, Louisiana State University, 2011.

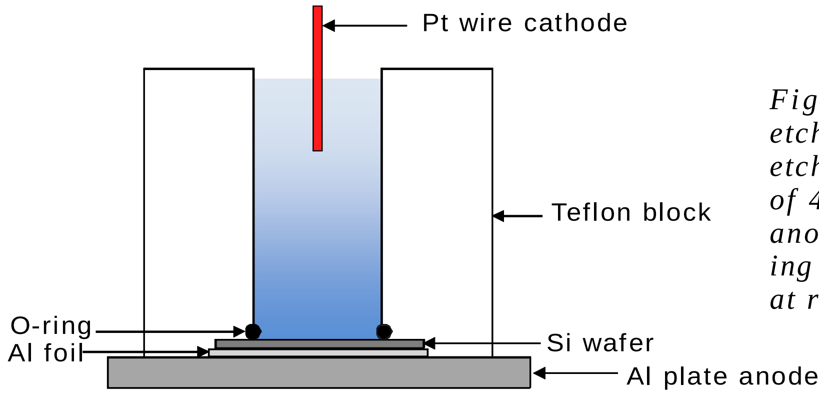

Fig. 1. Apparatus for etching the silicon. The etch was a 3:7 mixture of 48% HF acid in ethanol. The typical etching time was 5 minutes at room temperature.

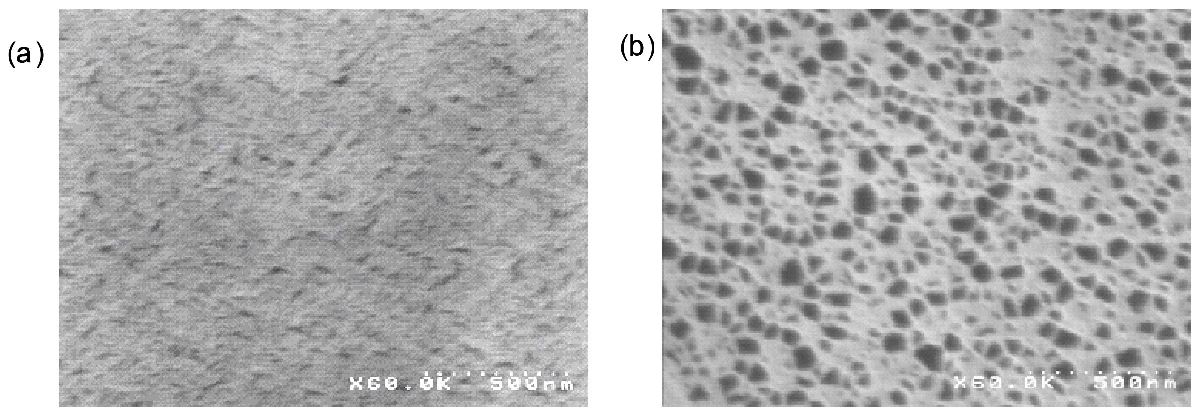

Fig. 2. (a) SEM of the surface of the etched silicon wafer. (b) similar area on the wafer after reactive ion etching in SF_6.

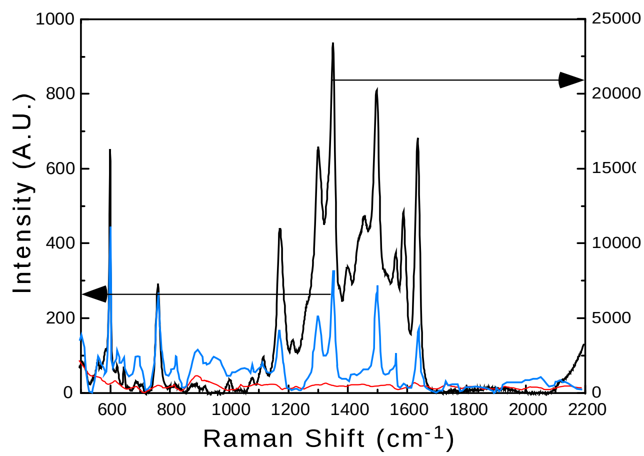

Fig. 3. Unenhanced Raman spectrum of Rhodamine 6G (bottom curve, left scale) and spectra obtained with 20 nm of additional nano-rough gold (middle curve, left scale) and 40 nm of additional nano-rough gold (top curve, right scale).

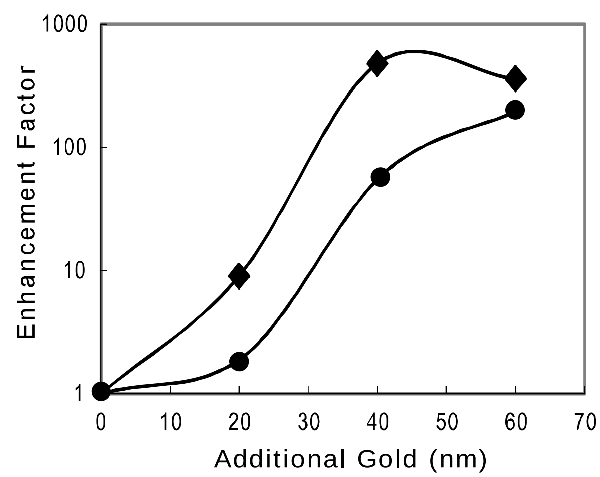

Fig. 4. Enhancement factor for nano-rough gold (top curve) and "smooth" gold (bottom curve) as a function of the additional gold deposited on the epoxy.