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Micromachined submicrometer photodiode for scanning probe microscopy

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A submicrometer photodiode probe with a sub-50 nanometer tip radius has been developed for optical surface characterization on a nanometer scale. The nanoprobe is built to detect subwavelength optical intensity variations in the near field of an illuminated surface. The probe consists of an Al–Si Schottky diode constructed near the end of a micromachined pyramidal silicon tip. The process for batch fabrication of the nanoprobes is described. Electrical and optical characterization measurements of the nanoprobe are presented. The diode has a submicrometer optically sensitive area with a 150 fW sensitivity. © *1995 American Institute of Physics.*

In recent years there has been a multitude of developments in scanning probe microscopy that allow imaging on a nanometer scale, yet chemical and molecular identification with these probes remains a great challenge. Standard optical spectroscopic techniques provide a powerful tool for chemical identification but lack spatial resolution on this scale. Near-field optical microscopy offers the possibility of routinely extending the spatial resolution of optical spectroscopy into the nanometer scale. Near-field scanning optical microscopy (NSOM) is an established technique based on the collection or transmission of light through a subwavelength aperture scanned near a surface.[1] Near-field photodetection optical microscopy[2,3] (NPOM) utilizes a photodetector of subwavelength dimensions. The localized photodetector probe is brought near an illuminated surface where it can directly absorb optical power. As it is raster scanned across the surface the photocurrent signal is recorded to create a two-dimensional image of the optical intensity distribution. The small detector is necessary to achieve a high spatial resolution.

Line photodetectors and light sources that are small (submicrometer) in two dimensions have previously been constructed.[2–6] Here we describe the development of a photodiode probe for NPOM, that is submicrometer in all dimensions. This nanoprobe is an Al–Si Schottky photodiode with a sub-50 nm tip radius.

There are several criteria for optimal NPOM probe performance. First, the photodiode must be constructed on a sharp protruding structure so that it can be used in a scanning probe arrangement. It should be noted that the sharpness of the tip is a determining factor of the probes' ultimate resolution. Next, the leakage current of the diode should be minimized. In our design which utilizes an Al–Si Schottky contact for the diode this corresponds to minimizing the Al contact area. Finally, crystalline silicon has a diffusion length on the order of several hundred micrometers. All of the light that falls within a diffusion length from the Al–Si junction is collected. So, to make a submicrometer photodetector all silicon surrounding the detection area must be optically masked.

The nanoprobes are batch fabricated on a silicon wafer. The fabrication of sharp, vertical silicon tips follows processes developed for producing silicon field emission arrays.[7] A sandwich of 100 nm SiO_2 and 100 nm Si_3N_4 is grown on the Si wafer. A lithography step is performed and the exposed Si_3N_4 and SiO_2 layers are etched through leaving a 10×10 μm pad of the sandwich layers on the substrate. The pads are in a rectangular array with a spacing of 1×1 mm. The wafer is then placed in a quasi-isotropic silicon etch consisting of nitric acid (70% HNO_3), H_2O, ammonium fluoride (40% NH_4F) and hydrofluoric acid (50% HF) in the following ratios 280:140:6:1 (by volume) HNO_3:H_2O:NH_4F:HF. The silicon is etched until a broad tip is formed [see Fig. 1(a)]. The silicon is thermally oxidized (100 nm thickness) and the oxide is then removed with buffered HF (BHF). The resulting structure is again oxidized [see Fig. 1(b)]. This reoxidation process further sharpens the tip.[8] This procedure results in an array of oxidized silicon pyramids 10 μm square on the base and approximately 5 μm high.

The next step is to construct a photodiode near the end of the pyramidal tip. First the oxide is removed from a small

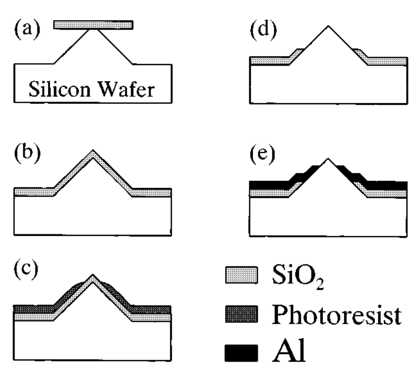

FIG. 1. Probe fabrication. (a) A pyramid is micromachined on a silicon wafer. (b) The tip is sharpened through oxidation. (c) A special application of photoresist exposes the oxide near the end of the tip. (d) The oxide is etched off the exposed region. (e) Al is deposited over the entire structure and then etched off the end of the tip.

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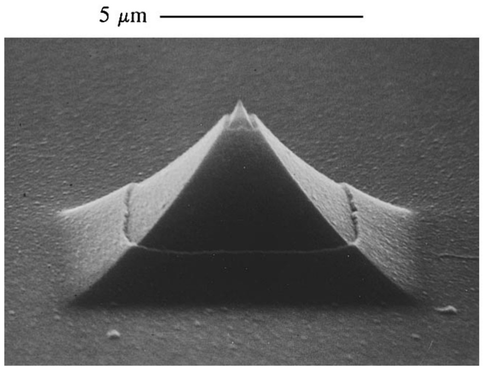

FIG. 2. A SEM image of the nanoprobe detector.

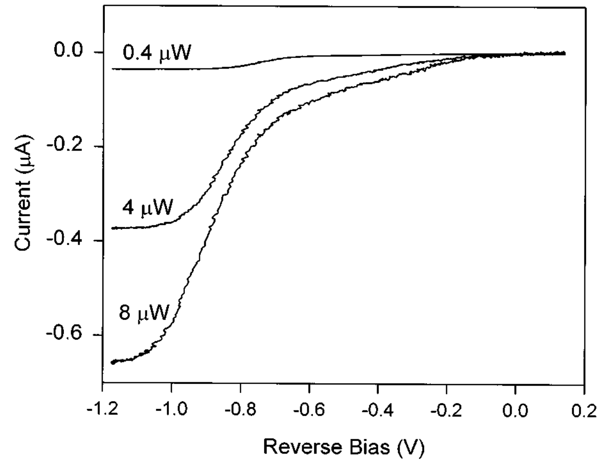

FIG. 3. The reverse bias $I-V$ curve of the nanoprobe for several illumination intensities.

region on the tip. When photoresist is spun on the wafer by the conventional method the pyramidal tip is not completely covered. This incomplete coverage by the photoresist is used to advantage. In our method the photoresist step is modified to minimize the opening at the end of the tip. The photoresist (Shipley 1400) is placed on the wafer and spun for only 0.2 s, resulting in a very thick layer of photoresist. The xylene in the photoresist is allowed to evaporate for 5 s. The wafer is then spun for 30 s at 3000 rpm. This gives the desired result, i.e., only the end of the tip protrudes through the photoresist [see Fig. 1(c)]. If the photoresist is spun on conventionally, omitting the intermediate evaporation step, a large nonsymmetrical portion of the tip will not be covered with photoresist. Following the photoresist step, the oxide is etched from the exposed tip region with BHF and the resist is removed [see Fig. 1(d)]. The amount of exposed silicon can be controlled by the oxide etch time. Al is then deposited over the entire structure. Photoresist is again applied in the manner described above and the Al in the tip region is removed with a standard Al etchant solution consisting of 80% phosphoric acid (H_3PO_4), 5% nitric acid (HNO_3), 5% acetic acid (CH_3COOH) and 10% water. The Al etching is done at room temperature to achieve a low etch rate. The photoresist is removed and the Al–Si contacts are sintered at 420 °C for 20 min. [see Fig. 1(e)]. A SEM image shows the resultant nanoprobe (see Fig. 2). The opening in the aluminum is 0.7×0.7 μm and the tip radius is less than 50 nm. These are typical dimensions for the opening size which ranges between 0.5 and 1.0 μm. In some cases the tip radius may be considerably smaller than 50 nm. Note that the NPOM probe requirements have been met, i.e., an ultrasharp tip (nanometer radius), a small Schottky contact area, and a very small (submicrometer) optically sensitive region. The silicon surrounding the junction has been optically masked by the Al layer.

Several measurements have been made to characterize the diode both electrically and optically. $I-V$, photo-$I-V$, and noise measurements were performed to characterize the sensitivity of the diode.

Photo-$I-V$ measurements with various illumination intensities were recorded. The reverse bias $I-V$ curves for three different illumination intensities are shown in Fig. 3. Note the strange shape of the photo-$I-V$ curve in reverse bias. With a standard photodiode the reverse bias photocurrent is independent of the applied voltage, whereas the reverse bias photocurrent of the nanoprobe is strongly bias dependent. It is largest with a reverse bias of -1.0 V or greater. At these reverse bias voltages, the photocurrent depends linearly on optical power, as is shown by the three curves in Fig. 3. The optical power levels indicated in Fig. 3 correspond to a HeNe laser beam focused through a microscope objective, with a focal spot size of approximately 3 μm.

To assure that the nanoprobe detector has the same spectral response as a conventional Si photodiode, it is compared with a commercial Si photodetector (Spectra Physics 404 Power Meter) as shown in Fig. 4. The rising signal level with increased wavelength is due to the variation in the spectral power coming from an incandescent source through a monochromator. The two detectors show a similar spectral response.

The signal and noise were measured with the diode at a -1.0 V reverse bias. The measurement frequency was 10 KHz. The laser beam was focused to an estimated 1.6 μm spot. With a 50 nW illumination the photocurrent was 6 nA and the noise was 30 fA/$\sqrt{\text{Hz}}$. This corresponds to a conversion efficiency of 0.2 A/W. The signal-to-noise ratio was

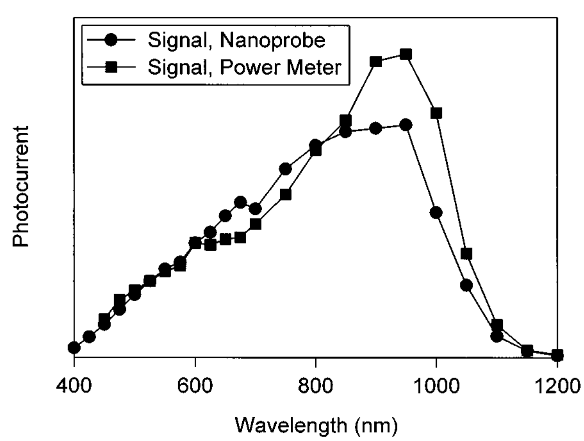

FIG. 4. The spectral response of the nanoprobe compared with a commercial photodetector. Both show a similar response.

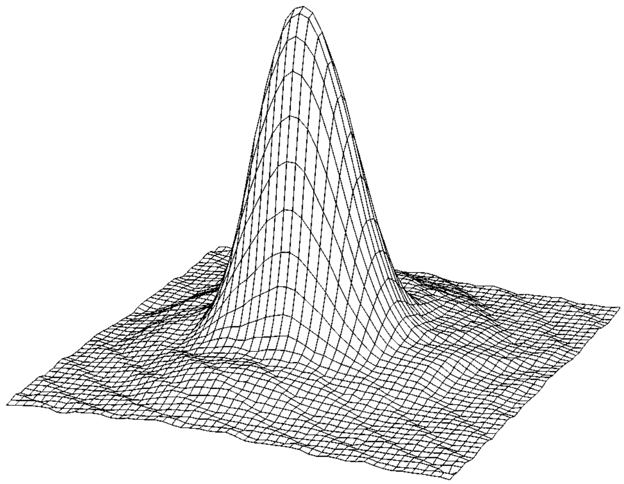

FIG. 5. A focused HeNe laser beam, imaged by the photodiode nanoprobe. This is a two-dimensional image (11×11 μm) of the optical intensity in the focal plane.

measured to be 2×10^5 in a 1 Hz bandwidth. From SEM images, we estimate the size of the light sensitive area on this particular photodetector to be near 1 μm^2. Thus the power falling on the active detector region is 0.37 of the total incident power (50 nW). This indicates that the minimum detectible optical power ($S/N=1$) is approximately 150 fW. This sensitivity is compatible with a 50×50 nm spatial resolution at this illumination intensity (5 W/cm^2).

A focused laser beam profile has been imaged by placing the nanoprobe on a piezoelectric tube and scanning it across the focused beam while measuring its photoresponse. An image of the HeNe laser beam profile is shown in Fig. 5. This is a two-dimensional image (11×11 μm) of the optical intensity in the focal plane. This demonstrates that the nanoprobe is sensitive only in a region at the top of the pyramid. The FWHM focused spot size, in this case, is about 2 μm. With further improvements, the nanoprobe could be employed as an optical beam profiler for tightly focused beams.

In summary, a submicrometer photodiode has been realized. It has been fabricated by a process that can be scaled for mass production. The nanoprobe appears to meet the requirements for NPOM; it has a submicrometer optically sensitive region with an ultrasharp tip. The diode has a maximum optical sensitivity at a reverse bias of -1.0 V where it can detect optical variations as small as 150 fW. It has a spectral response similar to bulk silicon detectors. Future work will include the measurement of optical power variations near an illuminated surface on a nanometer scale.

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