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Nanometer-precise optical length measurement using near-field scanning optical microscopy with sharpened single carbon nanotube probe

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ABSTRACT

We have developed and characterized a plasmon-excitation scattering-type near-field scanning optical microscope with sharpened single carbon nanotube probe. The developed microscope can optically capture differences in the refractive index of single-nanometer surface structures. Statistical analysis enabled us to estimate the precision of the optical length measurement to 1.8 nm.

1. Introduction

In the fields of nanotechnology and biochemistry, there is a latent need for nondestructive and noninvasive measurements and spectroscopic analyses at resolutions of a few nanometers, which cannot be achieved by electron microscopy and existing scanning probe microscopy. Optical microscopy is a powerful technique for studying the physical and chemical properties of materials although the diffraction limit of light restricts the lateral spatial resolution of far-field optical microscopy to approximately half the wavelength of the light. Near-field scanning optical microscopy (NSOM) has overcome this diffraction limit [1,2], enabling optical measurements with lateral spatial resolutions down to a few nanometers [3–11].

NSOM is widely utilized for practical science; for example, surface plasmon imaging [3,4], near-field Faraday-effect observation [5,6], nanoscopic spectroscopy, and individual quantum dot inspection [7]. Recent advances in NSOM using apertureless probes with nanometer-sized tips [3,8,9] or tip-on-aperture [10,11] have enhanced lateral spatial resolutions up to a few nanometers; however, the precision of the optical length measurement with a spatial resolution of a few nanometers has not been extensively discussed. NSOMs require further technological advances to be extensively applicable to laboratory research and industrial development. High lateral spatial resolution of a few nanometer and high precision of length measurement, together with removal of the restrictions of sample type must all be achieved. In this article, we present a precise optical length measurement apparatus that was developed with lateral spatial resolution up to a few nanometers using a sharpened multiwall carbon nanotube (CNT) in a scanning optical probe. The technique enables measurement of the nanoscopic spatial pattern of the refractive index at the sample surface.

To improve the precision of the optical length measurement, we enhanced the endurance of the optical probe using CNTs. The Young's modulus of CNTs can be up to 1 TPa [12], which is much higher than that of silica or other materials that are conventionally used for probes in NSOMs [1,2,4–11]. In atomic force microscopy, several previous studies have shown the superiority of CNT probes in terms of spatial resolution and endurance, which directly enhanced the precision of the images [12,13]. In scattering type NSOM (s-NSOM), bundled CNTs have been applied to the optical probe and imaging experimentally demonstrated [3]. A previous study achieved ∼10 nm resolution in optical imaging, although there was a problem with the reproducibility of the probe because of the low controllability of the physical dimensions at the apex of the bundled CNTs. As opposed to previous studies, we used a sharpened single multiwall carbon nanotube for the optical probe. Our CNT probe was fabricated using the nano-manipulation technique. This enhances the reproducibility of the physical dimensions

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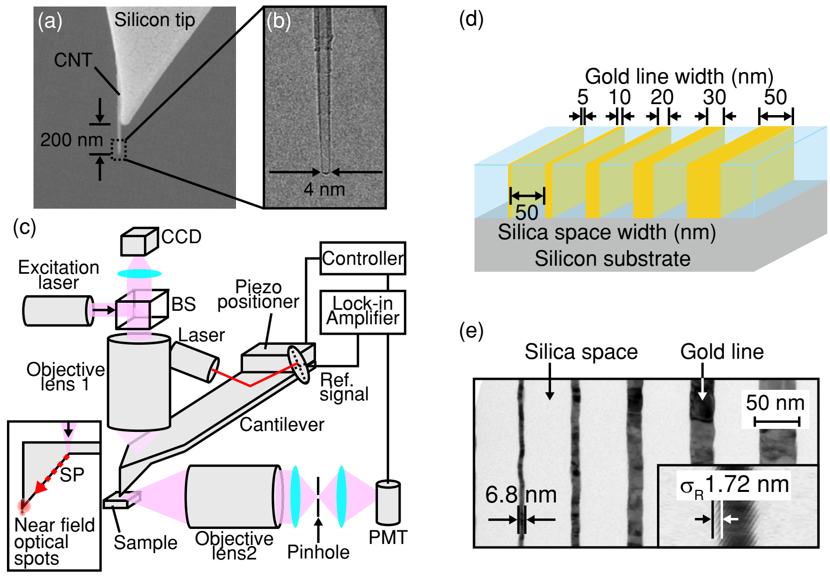

Fig. 1. (a) Scanning electron micrograph of the probe. A sharpened CNT was attached to the end of the silicon tip with 200 nm extrusion. (b) An example transmission electron micrograph of the apex of the CNT optical probe. This probe had a 4-nm diameter at the end point. (c) Schematic diagram of experimental setup. The laser beam for excitation of surface plasmons (SPs) was focused at the edge of the gold film through microscope objective 1. Scattered light was collected by microscope objective 2 and transmitted to the photomultiplier (PMT). The PMT output was connected to a lock-in amplifier and recorded by the probe controller. BS: beam splitter, CCD: charge coupled device for monitoring excitation beam position. (d) Perspective of sample structure, and (e) transmission electron micrograph. Inset of (e) schematically shows the interface roughness.

of the probe, which also directly enhances the precision of length measurement and the reproducibility of images.

2. Experimental evaluation

2.1. Fabrication of a sharpened single CNT probe

We adopted a near-field to near-field coupling technique for excitation of the optical spot at the apex of the CNT probe. This mechanism is analogized to the electric field concentration at the apex. An analytical study on the induced electrical field at the apex demonstrated that the strength of the electrical field is inversely proportional to the curvature of the apex [3,14]. We prepared the CNT probe using the following process. Multiwall CNTs (MWNTs) synthesized using the pulsed arc discharge method aligned on a knife-edge were positioned onto the silicon tip by a manipulator and fixed by carbon deposition using the electron beam in the electron microscope. The current injection method was then used to cut and sharpen the CNT [13,23]. Using pulsed current to break the outermost graphene layer of the MWNT, we obtained a sharpened MWNT by repeating pulsed current injection and pulled out the inner nanotubes. Further, using this method, we fine-tuned the physical dimensions of the apex curvature and the extrusion length of the CNT probe. We used Arrow ™ NC supplied by NanoWorld as a base cantilever with thickness 4.6 µm. The silicon tip of this cantilever was coated with a gold film of thickness 50 +/− 10 nm via thin film deposition in advance. The radius of curvature of the tip after gold film deposition was 25–35 nm. The ratio of the curvatures of the prepared sharpened CNT to the silicon tip was in the range 1:12.5–1:17.5. This means that an electric field is attracted more strongly to the apex of the CNT probe than to the top of the silicon tip. Fig. 1(a) and (b) show perspective views of the prepared probe taken using an electron microscope. The example in Fig. 1(b) has a radius of curvature of 2 nm, which was determined by the diameter of the innermost nanotube. We drove the probe in non-contact dynamic mode with a contact force of a few nano-Newton. To avoid buckling and vibration of the CNT probe, we used a multi-wall CNT of 20 nm diameter and adjusted the extrusion to 200 nm. Buckling of the cylinder or column occurs when force beyond the Euler buckling force F_E, expressed in Eq. (1), is loaded [12,21]:

$$F_E = \frac{\pi^2 Y I}{(kL)^2} \tag{1}$$

where Y is Young's modulus, I is the moment of inertia, k is the effective factor, and L is the extrusion length of the CNT. In the case where the column is fixed at one end and pinned at the other end, an effective factor of 0.7 is empirically derived [21]. Using typical dimensions and the Young's modulus of CNT [12], the Euler buckling force of our CNT probe for 200 nm extrusion was found to be ~10 nN. We can avoid the buckling and achieve the stabilized measurement using the CNT probe with extrusion less than 200 nm.

Measurements with s-NSOMs can sometimes suffer from low signal-to-noise ratio, because the scattering light of the near-field excitation beam generates a background light field that makes the noise [3,8,9]. In this study, the remote excitation method and spatial filtering were applied to suppress the background light field, whereas interferometric techniques are frequently used in other studies to eliminate the background light [9]. Interferometric methods achieve advanced measurement, but they require intricate apparatus such as a Michelson interferometer with piezoelectric actuator and heterodyne detection systems. Our excitation method, on the other hand, utilizes a simple optical system. In our excitation method, the excitation beam is focused on one end of the gold film through the silicon cantilever. The beam is partially reflected at the surface of the silicon cantilever and partially absorbed by silicon, 52% of the incident energy reaches the gold film [22]. The focused beam excites surface plasmons (SPs) on the gold film via the end-fire method [16]. Optical energy injected at one end of silicon tip was transferred to the opposite end, to which was attached the CNT probe, via SPs on the gold film (inset of Fig. 1(c)) [15]. Three-dimensional finite-difference time domain (3D-FDTD) simulation was used to estimate the energy transmissivity from the excitation point to the apex of the CNT as ~10^{-4}. A near-field optical spot at the end of the tip was generated by surface plasmon and another was generated at the apex of the CNT by electric field concentration. The distance between the excitation point and apex