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Sensitivity maximized near-field scanning optical microscope with dithering sample stage

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We developed a new scheme for a higher sensitivity near-field scanning optical microscope (NSOM) by using a dithering sample stage rather than a dithering probe for the constant gap control between probe and sample. In a conventional NSOM, which use tip dithering feedback mechanism, the Q factor drastically decreases from 7783 to 1000 (13%) or even to 100 (1%) because harmonic oscillating characteristic is deteriorated owing to the large change of stiffness and mass of one prong of tuning fork when a probe is attached to it. In our proposed scheme, on the other hand, we use sample dithering feedback mechanism, where the probe is not attached to the tuning fork and the sample is loaded directly onto the surface of dithering tuning fork. Thus, the Q factor does not decrease significantly, from only 7783 to 7480 (96%), because the loaded sample hardly changes the stiffness and mass of tuning fork. Accordingly, gap control between the immobile fiber probe and the dithering sample is performed precisely by detecting the shear force with high sensitivity. Consequently, the extremely high Q factor enables clear observation of graphene sheets with sub-nanometer vertical resolution, which is not possible with a conventional NSOM setup. © 2012 American Institute of Physics. [http://dx.doi.org/10.1063/1.4754290]

I. INTRODUCTION

The near-field scanning optical microscope (NSOM) is a high resolution imaging tool used in measuring both the surface profile and the near-field optical signal of a sample, and thus has been widely used in such research fields as the measurement of micro/nanophotonic devices,1–7 plasmonic devices,8,9 semiconductor devices,10–12 and biomaterials,13–16 etc. In addition, it plays an important role in the field of nanolithography.17–19 Because of this utility, the performance improvement of the optical sensitivity20,21 and application techniques22,23 of NSOM have been actively studied.

Generally, an electrical tuning fork glued to a fiber probe is used as the gap control sensor between the probe tip and sample in NSOMs.24,25 However, using this tuning fork/fiber probe sensor is somewhat problematic because a highly skilled technique is required to precisely glue the fiber probe to the tuning fork. Furthermore, the Q factor of the sensor is severely deteriorated due to differences in the natural frequencies of the fiber probe and the tuning fork and the additional effective mass and stiffness imparted to the one prong.26 These problems cause dissipation of the vibration energy supplied to the tuning fork. For this reason, the construction of NSOM systems has been regarded as a difficult task compared to that of far-field microscopes.27–29 Therefore, a considerable amount of research has been devoted to enhance the Q factor of the tuning fork/fiber probe sensor by developing electrical or mechanical based methods in order to obtain higher sensitivity.26,30–34 Although several proposed mechanical vibration control methods26,34 were very effective in enhancing the Q factor, most failed to principally resolve the severe deterioration of the Q factor caused by gluing the fiber probe. To overcome this problem, we propose a simple NSOM scheme, in which the sample is directly loaded onto one prong surface of a dithering bare tuning fork. The gap control between immobile probe and dithering sample is performed by detecting the shear force. As a consequence, the intrinsically high Q factor of the bare tuning fork can be maintained.

In this paper, the details of the proposed method are presented and the schematic of an experimental setup is described, and its resonance characteristics are experimentally analyzed. The surface profiles of graphene sheets measured by both a conventional and the proposed method are compared to verify the improvement of the sensitivity in tip-sample distance control. Finally, topographic and near-field images of the soft sample observed by the proposed NSOM are presented.

II. METHODS AND EXPERIMENTS

Figures 1(a) and 1(b) show the conceptual illustration of the conventional and proposed NSOM system, respectively. Contrary to a conventional NSOM, the sample is loaded directly onto the prong surface of the bare tuning fork and then dithered with the vibrating tuning fork. In this scheme, as the fiber probe tip closely approaches a point on the sample...
FIG. 1. Conceptual description of the conventional NSOM (a) and proposed NSOM (b), where the sample loaded on the prong surface of a tuning fork is dithered but an optical fiber probe is not dithered.

surface, the shear force interaction between the sample and tip will become significant, which can be electrically detected via the tuning fork. Because the fiber probe is not glued to the tuning fork, the resonant characteristics of the bare tuning fork enable an extremely high Q factor which can be used for the shear force detection. Consequently, the new NSOM system has higher sensitivity than that of the conventional NSOM. In addition, the NSOM construction can be simplified because it is not necessary to use ungainly techniques for gluing the fiber probe to the tuning fork.

Figure 2 shows the schematic diagram of the experimental setup for NSOM measurement. The height of the probe tip was controlled by the shear force feedback control method using a tuning fork. The vertical position of the fiber probe is changed using a piezoelectric translator (PZT, P-611.3 from Physik Instrumente) that has a 0.1 nm resolution; its lateral position (along the x- or y-axis) is also scanned using a PZT with a 10 nm position repeatability against a 100 μm total moving range. The light coming out of the laser diode (at a wavelength of 405 nm, RDE4070 from SUSEMICON) was focused onto the sample via an objective lens. The near-field optical signal close to the sample surface was then measured using the optical fiber probe and converted into an electric signal by a photomultiplier tube (PMT, R2027 from Hamamatsu) that was placed at the opposite end of the fiber.

The fiber probe was a commercially available Al-coated tapered fiber (MF001/10 from NT-MDT) having an aperture diameter of 100 nm. Finally, the height and optical information were acquired using an express data acquisition board (PCI-6229 from National Instruments).

As mentioned above, the high Q factor of the bare tuning fork severely deteriorates when the fiber probe is glued to one of its prongs. This deterioration is mainly due to dissipation of the vibration energy that is imparted to the tuning fork. A common explanation of the dissipation is both the asymmetric vibration of the two prongs and the difference in natural frequencies of the tuning fork and the fiber probe. In addition, the resonant frequency \( f_0 = \frac{(k/m_e)^{1/2}}{2\pi} \) of the tuning fork glued with the fiber probe changes owing to both the additional effective mass \( m_e \) and the additional stiffness (spring constant, \( k \)) imparted to the prong by the fiber probe. It is also well known that the Q factor decreases with an increase in the resonant frequency shift (\( \Delta f \)). Accordingly, the NSOM probe usually used in a conventional NSOM has a Q factor in the range of 100–1700.

For comparison, three frequency response curves were measured. Figure 3(a) shows the frequency response curves for a bare tuning fork and a tuning fork with a sample loaded on it, and Figure 3(b) shows the frequency response curve of a conventional NSOM probe. In Figure 3(a), the resonant frequency shift due to the sample load was negligible, and the change of Q value was also insignificant, from 7783 to 7480. On the other hand, the resonant frequency shift and Q value...
of the conventional NSOM probe were measured to be $f/\Delta f = 58$ and $Q = 495$, respectively.

In order to verify the ability to sensitively detect the shear force interaction, the output voltage of the tuning fork in the proposed scheme was measured while approaching the fiber probe to a bare tuning fork vibrating at its resonant frequency. Because the two prongs connected to the base of the tuning fork vibrate in an anti-phase mode, as schematically drawn in Figure 4(a), the vibration displacement of the prong will vary with the distance from the approaching point to the base. Accordingly, the sensitivity of detecting the shear force will likewise depend on the position of the prong surface at which the fiber probe approaches. Thus, the approach curves were measured at the end, the center, and near the base of the prong. Figure 4(b) shows the measured approach curves. As expected, the steepest curve was obtained when the fiber probe approached toward the prong end. In other words, the most sensitive result would be attained, in the proposed NSOM scheme, when a sample was loaded on the prong end.

III. RESULTS AND DISCUSSION

Because the proposed method has an extremely high Q factor ($\sim 8000$), it was also expected to have a higher sensitivity than that of a conventional NSOM. Topographic images of the graphene sheets were observed with the conventional and proposed method to identify the sensitivity enhancement. The graphene sheets were derived from a graphene powder solution diluted with isopropyl alcohol. One drop of the graphene powder solution was poured on the prong surface of a tuning fork and dried in an incubator at 70°C for 1 min. In the first measurement, the conventional NSOM method was used (as shown in Figure 5(a)), in which the tuning fork with the graphene sheets loaded on its surface was not vibrated and another tuning fork with the glued fiber probe was dithered, which scanned over the sample. The tuning fork/fiber probe used in the conventional NSOM had a Q factor of 495. Next, the proposed method (shown schematically in Figure 5(d)) was used to obtain the topographic image of the same part of sample. By dithering the tuning fork with the graphene sheets on its surface horizontally, the non-vibrating NSOM probe then scanned over the sample. Figures 5(b) and 5(e) are the topography results of graphene sheets measured with the conventional and proposed method, respectively. The line profiles of topographic images are drawn in Figures 5(c) and 5(f) in order to compare the sensitivity of the two methods. In case of the conventional method, the graphene sheets on the tuning fork could not be clearly observed due to the low sensitivity in tip-sample distance control. Indeed, the line profile obtained by the conventional NSOM probe follows the actual geometry of the sample, but tended to become unstable by the overshoot signal when the tip encountered a ridge or a furrow in the sample. On the other hand, in case of the proposed method, the shape of graphene sheets was clearly observed and the line profile faithfully showed the actual geometry of the sample without noise. Until now, nano-materials/devices having sub-nanometer height were hardly observable with NSOM due to the limit of vertical resolution. However, from these results, it is confirmed that the proposed NSOM can simply measure the nano-scale sample with a higher sensitivity. Thus, the proposed system can be applied to investigate the surface, optical, and electrical properties of graphene devices such as graphene nano-ribbons and graphene transistors without damaging the samples.

In the second demonstration, the topographic and the near-field images were observed with the proposed NSOM to evaluate on the soft biomaterials such as cells. We used neural cells which were derived from a human neuroblastoma cell line. The cells were cultured for 2 days after plating on the culture dishes in vitro. For the sampling, the number of 12,000 neural cells was diluted in the media of 10 μl and one drop of the solution was poured onto the prong surface of a tuning fork. To attach the cells to the surface, the tuning fork
FIG. 6. Topographic and near-field images of the neural cells, (b) and (c) were observed with the proposed method (a), and the images, (e) and (f), observed with the proposed method (d) having different direction of line scan.

was incubated in the 5% CO₂ incubator at 37 °C for 4 h and preserved in 4% paraformaldehyde (PFA) solution at room temperature for 20 min and then washed out by a phosphate buffered saline (PBS) solution and distilled water. Finally, the cells loaded on the prong surface were exposed to the air in order to measure the cell surface. The surface profile and near-field signal of the neural cells were measured by illuminating with a 405 nm wavelength laser and scanned with an Al-coated 100 nm aperture NSOM probe. During scanning, the gap control between the cells with the fiber tip was performed by keeping the amplitude difference between the driving input and the tuning fork output signal constant. The intensity of the collected photons was then converted to an electrical voltage by a PMT. In the first measurement (shown schematically in Figure 6(a)), with the cells placed horizontally on the dithering tuning fork, the NSOM probe scanned a 10 μm × 10 μm area in the direction parallel to the dithering direction of the tuning fork. The clearly observed surface profile and the near-field image are shown in Figures 6(b) and 6(c). In the second method, the direction of the line scan was perpendicular to the dithering direction which was shown in Figure 6(d). The clear results of the second scan are shown in Figures 6(e) and 6(f). From the results obtained by the proposed method with two different directions of line scanning, it was concluded that the direction of the line scan had no correlation with the direction of dithering. Moreover, it suggested that the nano-range changes of the cell surface could be detected with high sensitivity. It could be an important imaging technique to detect cells pathologically because the various proteins and macromolecules were located on the cell surface and these molecules indicated cellular characterization and were altered at diseased conditions.

It should be mentioned that the proposed NSOM has an extremely high Q factor and thus, the scanning speed is relatively slow in comparison with a low-Q NSOM due to the long decay time-constant of the tuning fork. If necessary, this problem can be easily resolved by implementing an electronic Q-control method such as a phase-locked loop. Note that a critical parameter that determines the maximum scanning speed is the decay time-constant given by τ = 3/12Q/πf₀ (Q: Q factor, f₀: resonant frequency). In NSOMs, generally, the sample sizes are very small and the scan range is less than 100 μm². Thus the tuning fork can perform the role of a sample stage. However, the size of the sample can sometimes be large. For example, a small piece of silicon with something on it. In this case, an AT-cut quartz crystal resonator (QCR) can be used instead of a tuning fork because it has a sufficiently large vibrating area (10 mm of diameter). In addition, the AT-cut QCR offers a fast scanning rate because of the short decay time-constant due to its high fundamental resonant frequency f₀ in the radio-frequency range (1–100 MHz).

IV. CONCLUSIONS

In conclusion, we have proposed and demonstrated a new NSOM scheme that maintains a Q factor as high as that of a bare tuning fork. In the scheme, we used sample stage dithering feedback mechanism, where the fiber probe was not glued to the tuning fork, but rather a sample was loaded directly onto a prong surface of the bare tuning fork and dithered using the vibrating tuning fork. The gap control between the fiber probe and the dithering sample could be accurately performed by detecting the output voltage of the tuning fork, as in a conventional NSOM. From the measured images of graphene sheets and neural cells, it was subsequently confirmed that the proposed method could be more conveniently used with high vertical resolution than the conventional NSOM method. Therefore, it is expected that this new NSOM technique can be used to investigate various nano-scale samples which cannot be observed by a conventional NSOM.

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