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FABRICATION OF THE OPTICAL SYSTEM FOR CL AND PL MEASUREMENT USING CT PROBE WITH HIGH-SPACIAL RESOLUTION

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We aim at characterizing luminescence from nanometer area by cathode luminescence and photo luminescence measurement with high-spacial resolution using a conductive transparent (CT) probe. The CT probe can inject electrons or photons for excitation in the same nanometer area and detect weak luminescence with high efficiency. In this study, we fabricated an optical system for leading luminescence by the CT probe to a spectrometer and calibrated in advance the system of the spectrometry. We were able to measure Raman spectrum of diamond, indicating the correct operation of the spectrometer and of the optical system. Further, we can complete the new scanning probe microscope system by connecting the optical system to scanning tunneling microscope with the CT probe replaced from a metallic probe.

I. Introduction

Materials with atomic and nanometer size make it possible to show new properties of themselves derived from a quantum effect, which cannot be exhibited in the bulk structure. Therefore, many researchers have expected and investigated new functions appearing in the nanostructures so far. The structure difference in atomic level of individual nanoparticles causes a large energy shift in the electronic structure by an electron confinement effect in nanometer area. The large energy shift should necessarily be reflected to the energy shift and the intensity of the photoemission spectrum. As a result, the characterization of luminescence from the nanometer area is important to understand the property of nanomaterials.

Electron-hole pairs regarding the luminescence are generated by electron or light injection and excitation. Due to the difference of the basic properties between electron and photon such as electric charge, momentum, and propagation characteristics, the obtained information is quite different in the cathode luminescence (CL) and the photo luminescence (PL) phenomena. Therefore, complementary evaluations of nanomaterials by the CL and the PL methods are useful to completely investigate both electronic and optical properties of themselves. In ordinary scanning tunneling microscope (STM) system and near field scanning optical microscope (NSOM) system, however, we cannot measure electronic and optical properties as well as the atomic configuration simultaneously at the same position and the same period time.

In order to overcome these problems, a new scanning probe microscopy was designed and developed by Murashita *et al*. [1, 2]. In this new microscopy, electrons or photons can be injected into the sample through a conductive transparent (CT) probe in the same area, and weak luminescence is detected with high efficiency due to the large solid angle of detection. The typical structure of the CT probe is schematically depicted

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in Fig. 1.

In this study, we configured the optical components as shown in Fig. 2 and calibrated in advance the system of the spectrometry in terms of wavelength, sensitivity, and resolution. In the PL measurement, luminescence obtained by laser excitation through the CT probe is collected by the identical CT probe again; led to the spectrometer through the optical system. In the CL measurement, on the other hand, luminescence obtained by tunneling-electron injected from the CT probe is collected by the CT probe; led to the spectrometer. Both methods are alternatively measurable at the same position of the sample without optical re-adjustments.

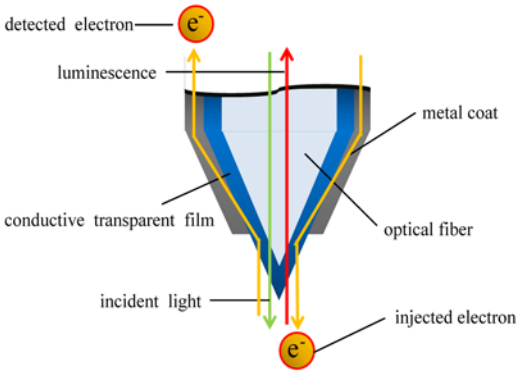

Fig. 1. Schematic image of CT probe. The inner optical fiber transmits light and the outer metal part transports electrons through conductive transparent film.

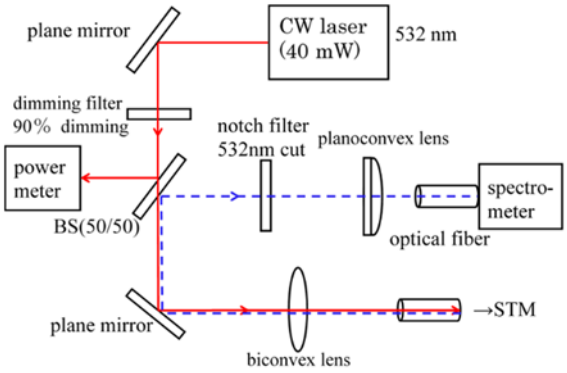

Fig. 2. Geometry of the optical system capable switching CL/PL mode. The solid lines show the path of excitation light and the dashed lines show the path of luminescence. PL mode uses the path indicated by the solid and dashed lines. On the other hand, CL mode uses the path shown by the dashed lines.

II. Experimental

In order to test the operation of the spectrometer, we first measured a fluorescent lamp spectrum and calibrated the wavelength indicated in the monochrometer. Secondly, we compared the spectrum of a miniature bulb with that of theoretical value assumed by the black-body radiation to calibrate the sensitivity of the optical system. Finally, we tried to improve the resolution by sufficiently narrow two emission lines observed from a sodium lamp. These light sources were installed in front of the incident port of the spectrometer and emitted photons were directly introduced into the spectrometer. We darkened the experimental room so that unrelated light should not be detected as a noise in the spectrometer. In order to evaluate the entire measurement system including the optical system and spectrometer, we measured Raman spectrum from diamond as a representative sample whose spectrum is widely known. In the measurement, an objective lens with a magnification of ×80 was used for making a solid angle large (see Fig. 3). Here, the laser light having a wavelength of 532 nm was adopted as the excitation source.

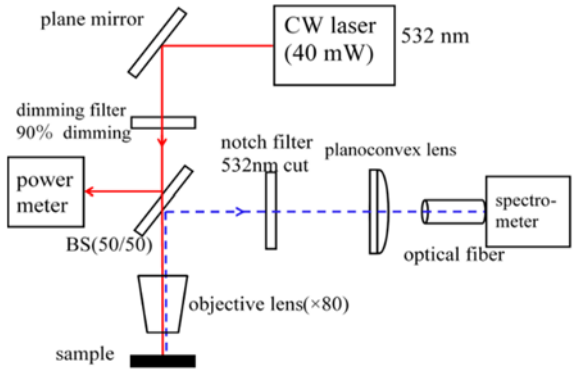

Fig. 3. Geometry of the optical system for Raman spectroscopy.

III. Results and Discussion

We first calibrated the wavelength indicated in the monochrometer by well-known line spectra observed in the light of a fluorescent lamp. Figure 4 shows the typical fluorescent lamp spectrum. 5-line spectra, which correspond to the emission from a mercury atom, observed on a continuous spectrum are clearly shown in the figure. The wavelengths of the measured line spectra are determined to be 404.3, 465.4, 545.8, 576.7 and 578.9 nm, which were quite consistent with those estimated from the energy levels of mercury: 404.7, 465.8, 546.1, 577.0, and 579.1 nm. It is indicated that the spectrometer in this system is well calibrated.

Next, we calibrated the sensitivity of the monochrometer depending on the wavelength by a miniature bulb spectrum. Figure 5 shows the spectrum and theoretical values estimated by the black-body radiation as follows,

$$F_\lambda = \Omega \frac{2hc^2}{\lambda^5} \cdot \frac{1}{\exp\left(\frac{hc}{\lambda k_B T}\right) - 1}.$$

Here, $F\lambda$: spectral radiation illuminance, h: Planck constant, c: velocity of light, λ: wavelength, k_B: Boltzmann constant, Ω: solid angle of detection and T: absolute temperature of black-body. The temperature was set at 3000 K. The observed values corresponded to the theoretical ones in the range of 400 – 700 nm. However, the intensity gradually decreased at 700 nm or more, deviating from the theoretical values. It is indicated that the sensitivity calibration is necessary for measurement in the infrared region. As long as one focuses on the visible region, the correction is found to be unnecessary.

Then we optimized the resolution of the wavelength by adjusting the slit width. Figure 6 shows spectra observed for a sodium lamp with the different slit width of 75, 50 and 25 μm. Each spectrum has narrow two peaks at 589.0 and 589.6 nm. These peaks are observed at expected wavelength and line width corresponding to the transitions from two very close exited states of $3P_{3/2}$ and $3P_{1/2}$ to the ground state 3S. The resolution is strongly dependent on the slit width of the spectrometer; and therefore we adjusted the slit width at the optimum value so as to obtain the sufficient peak resolution and the practical intensity. The full width (FWHM) at half maximum of the emission lines for the slit width of 25 μm, which was the best condition indicating the separation of the two emission lines, are estimated to be 0.44 nm (12.7 cm^{-1}) and 0.38 nm (10.9 cm^{-1}).

Figure 7 shows a Raman spectrum observed for the diamond sample. A peak was

clearly observed at 573.7 nm by optimizing the whole optical alignment. The wave number for the peak was estimated to be 1366 cm^{-1}, corresponding to the typical Raman peak (1333 cm^{-1}) of diamond. The FWHM of the observed peak measured with a slit width of 25 μm was 0.51 nm (15.6 cm^{-1}).

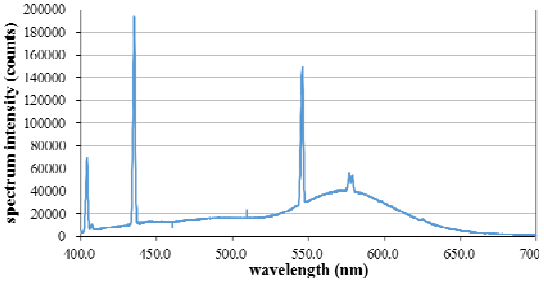

Fig. 4. Fluorescent lamp spectrum.

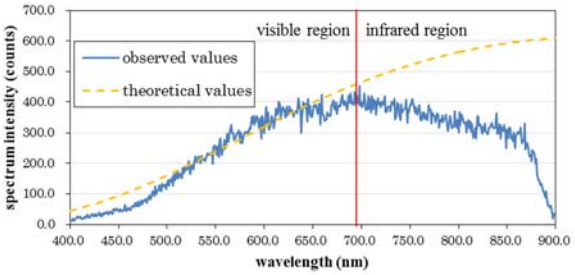

Fig. 5. Miniature bulb spectrum and theoretical values estimated by the black-body radiation.

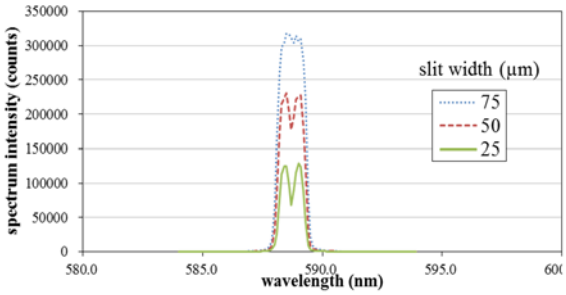

Fig. 6. Sodium lamp spectra with different slit widths.

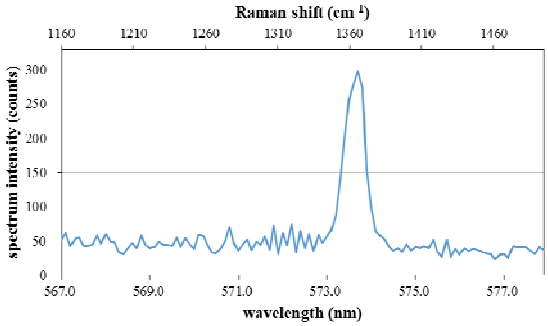

Fig. 7. Raman spectrum of diamond.

IV. Conclusions

We were able to perform the wavelength calibration, the sensitivity correction and improving the resolution of the spectrometer, and succeeded in measuring weak luminescence from the sample by using the established optical system. Therefore, the optical system was completed and the operation of spectrometer was confirmed. Further, as shown in Fig. 2, we can complete the new scanning probe microscope system by connecting the optical system and STM with the CT probe through an optical fiber.

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