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Development of Novel Near-field Optical Fiber Probe Using Photonic Crystal Fiber for Highly Sensitive Fluorescence Lifetime Measurement

Daichi Seto, Yoshihiro Taguchi, Toshiharu Saiki and Yuji Nagasaka

Keio Univ.
3-14-1, Hiyoshi, Kohoku-ku Yokohama-shi,
Kanagawa 223-8522, Japan
Phone: +81-45-563-1151(ext. 43125) E-mail: seto@naga.sd.keio.ac.jp

1. Introduction

As highly integrated nano- and micro-scale devices have been developed over the few decades, thermal design become quite important. In the field of nano-bioengineering, the thermometry with nanoscale spatial resolution becomes a powerful tool to investigate the interaction of biomaterials. But nanoscale temperature measurement has some problems. Some contact-mode methods can achieve nanoscale spatial resolution; however, the contact causes breaking nanoscale structure at sample. In contrast, optical methods enable to prevent sample broken by non-contact-mode measurement, however it is difficult to achieve nanoscale spatial resolution because of diffraction limit.

From above aspects, we have developed a non-contact and nanoscale temperature measurement technique, namely Fluorescence Near-field Optics Thermal Nanoscopy (Fluor-NOTN) [1] using near-field light and fluorescence.

In this paper, in order to enhance sensitivity of the temperature measurement, we purpose a novel near-field optical fiber probe using Photonic Crystal Fiber (PCF) and fusion splicing.

2. Measurement principle of Fluor-NOTN

A Schematic image of Fluor-NOTN is shown in Fig. 1. In Fluor-NOTN, the micro-fabricated near-field optical fiber probe with nanoscale aperture is utilized. The excitation laser diode (wavelength: 473 nm) is coupled with near-field optical fiber probe. The near-field light is excited in nanoscale aperture at the near-field optical probe tip. The size of near-field light is same as the diameter of the nanoscale aperture. The near-field light illuminates the fluorophores (Qdot655) modified on the sample surface, and the fluorescence (wavelength: 655 nm) is excited. The fluorescence is collected by the nanoscale aperture at the tip of the near-field optical fiber. In our research, the temperature dependence of the fluorescence lifetime is measured by the frequency domain method. In this method, a sinusoidally modulated laser is utilized as the excitation light. In this case, the excited fluorophores are forced to emit fluorescence at the same frequency. Due to the fluorescence lifetime, the fluorescence emission is delayed relative to the excitation, and the delay can be measured as the phase lag of excitation light and fluorescence. The fluorescence lifetime is expressed by the following equation.

$$\tau = \frac{\tan\phi}{2\pi f} \qquad (1)$$

τ: Fluorescence lifetime[s], ϕ: Phase lag[deg.], f: Modulating frequency[Hz]

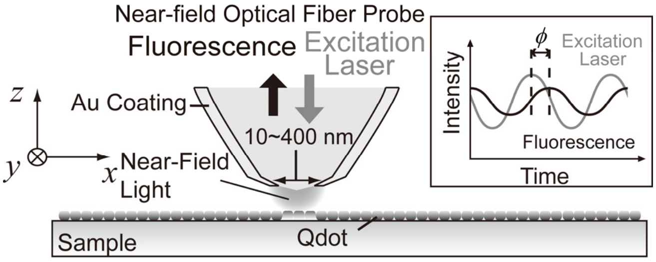

Fig. 1 Schematic image of measurement principle of Fluor-NOTN.

2. Measurement principle of Fluor-NOTN

A schematic diagram of experimental apparatus of Fluor-NOTN is shown in Fig. 2. In the Fluor-NOTN, the experimental apparatus is divided into two parts as a signal detection system and a distance control system. In the signal detection system, the excitation laser is coupled into near-field optical fiber probe and generates the near-field light in the proximity region of the probe tip. The fluorophores on the sample surface are illuminated by the near-field light, and the excited fluorescence is collected by the same fiber probe. The fluorescence is detected by the cooled photomultiplier tube (PMT) via a pinhole and band-pass filter. The phase lag of the detected fluorescent signal is measured by a lock-in amplifier. In the distance control system, since a near-field light is a nonpropagation light that is excited in the proximity region of the probe tip, the distance between the near-field optical fiber probe tip and the fluorophores on the sample should be controlled at the nanoscale (less than the size of aperture). For the purpose of distance control, the quartz crystal tuning fork is attached on the near-field optical fiber probe, and the distance is controlled by shear-force detection using PID control.

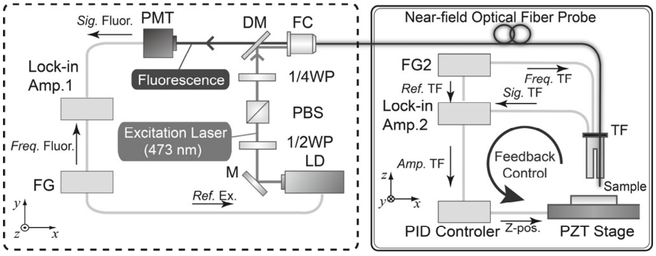

Fig.2 Experimental apparatus of Fluor-NOTN.

3. Fusion-spliced near-field optical fiber probe

In order to gain the accuracy of the temperature measurement, the weak fluorescence generated from the surface-modified Qdot should be detected with a high S/N ratio. In our previous study, a custom-made Ge-doped silica core SMF was utilized to create a near-field optical fiber probe; however, the most critical noise was the auto-fluorescence generated from the dopant of SMF. Since the auto-fluorescence spectrum is broad, and the auto-fluorescence was simultaneously modulated at the same frequency of fluorescence emitted from Qdot, the auto-fluorescence cannot be eliminated by a lock-in amplifier. In order to decrease this noise, we utilized a Photonic Crystal Fiber (PCF) for the near-field optical fiber probe. PCFs have a triangular pattern of micro air holes to form the cladding; therefore, PCFs usually consist of a single material. Fig. 3 shows a SEM image of PCF. This aspect of PCFs is an important advantage for near-field fluorescence detection because the auto-fluorescence generated from the dopant is expected to be negligibly small. We proposed a fusion-spliced near-field optical fiber probe fabricated by splicing a PCF and a conventional SMF, aiming at sensitive fluorescence detection from Qdot. Fig. 4 shows in brief the fabrication process of the fusion-spliced fiber. **(a)** The SMF and the PCF are fusion-spliced. **(b)** The spliced point is protected by UV curing resin (3042C, Three Bond Co.) and then cleaved at the SMF side with the remaining length of the SMF up to 5 mm, in order to decrease the auto-fluorescence generated from the dopant of the SMF. **(c)** The probe tip is then fabricated at the end face of the SMF side.

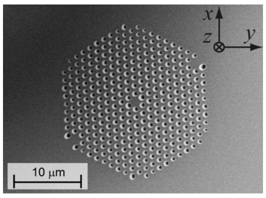

Fig. 3 SEM image of cross-section of PCF.

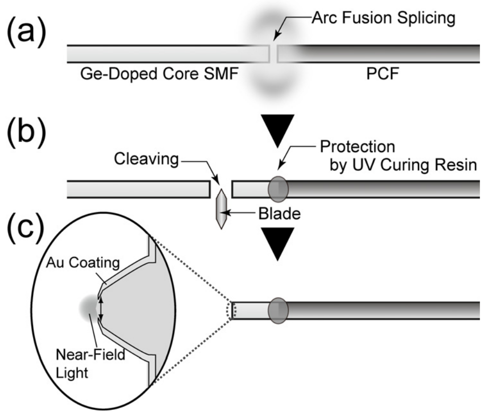

Fig. 4 Fabrication of fusion-spliced near-field optical fiber.

4. Results

As the materials for the fusion-spliced near-field optical fiber probe, we utilized NL-PM-750 (Nkt Photonics Co.) as the PCF, and the Ge-doped SMF for the near-field optical fiber probe because these two fibers have similar optical parameter. In the experiment of the fusion-splicing, an average splice loss of 3.2 dB and a minimum splice loss of 0.56 dB were achieved by optimizing the splicing parameters. Finally, the double tapered near-field optical probe was successfully fabricated in the tip of fusion-spliced optical fiber.

In order to evaluate the validity of the fusion-spliced fiber for the near-field fluorescence detection, the auto-fluorescence spectra of optical fibers were measured by the spectroscope. Figure 5 shows the fluorescence spectra of the Ge-doped core SMF, PCF and the fusion-spliced fiber. All fibers were the same length (1.0 m), and the auto-fluorescence intensities were normalized by the intensities of the excitation laser coupled to the fibers. The gray area is measurement waveband of Qdot655. The auto-fluorescence intensity of fusion-spliced optical fiber in measurement waveband dramatically small by 14 % compared with Ge doped core SMF.

We then measured near-field fluorescence lifetime of Qdot655 by using the fusion-spliced near-field optical fiber probe. Fig. 6 shows the phase lag between the excitation laser and the near-field fluorescence of Qdot655 detected at room temperature. The plots were fitted by the single-exponential decay model. The fluorescence lifetime of Qdot655 was estimated to be 8.91 ns. The near-field fluorescence lifetime was successfully measured using the fusion-spliced near-field optical fiber probe.

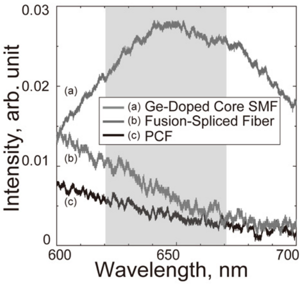

Fig. 5 Auto-Fluorescence spectra of (a) SMF, (b) Fusion-spliced Fiber, (c) PCF.

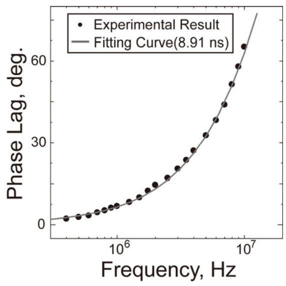

Fig. 6 Phase lag and fitting curve of Qdot655 measured by the fabricated probe.

4. Conclusion

We successfully fabricated the highly sensitive fusion-spliced near-field optical fiber probe. In addition,we confirmed this probe can be used our temperature measurement method.

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References

[1] T. Fujii, Y. Taguchi, T. Saiki, Y. Nagasaka, *SENSORS*, **11**, (2011), 8358-8369.