Photon Factory Activity Report 2022 #40 (2023)

BL-11D/2020G130

軟 X 線励起蛍光用顕微分光器の開発と評価

江島丈雄 [1,2*] 、佐々木英一 [2]
[1] 東北大学 国際放射光イノベーション・スマート研究センター
[2] 東北大学 多元物質科学研究所
〒980-8577 仙台市青葉区片平 2 丁目 1-1

Development and evaluation of micro-spectrometer for SX excited fluorescence

EJIMA, Takeo[1,2,*] and SASAKI, Eiichi[2]
[1] SRIS, Tohoku University
[2] IMRAM, Tohoku University
2-1-1, Katahira, Aoba-ku, Sendai, 980-8577, Japan

Scintillator, Eu:GGG, is being considered for implementing STED in the soft X-ray (SX) wavelength region, but the optimal wavelength for inducing fluorescence in SX excited fluorescence is still unknown. To address this issue, a new microscopic spectrometer has been developed with improvements such as precise pinhole placement and an external resonator for laser wavelength stabilization. Evaluation results show a decrease in emission intensity of the Eu:GGG scintillator due to laser irradiation, indicating suppression of induced emission in SX excited fluorescence.

1 Introduction

Certain scintillators can exhibit the stimulated emission suppression (STED) phenomenon when excited by laser light [1]. This phenomenon holds promise for further reducing pixel size in two-dimensional detectors [2]. Two scintillators, Eu:GGG and Tb:LSO, are particularly valuable for applying the STED phenomenon due to their high fluorescence intensity in the SX wavelength region [3]. The rare-earth ions in the scintillators demonstrate fluorescence levels corresponding to the number of f-electrons in their outer shell. However, the optimal wavelengths for inducing STED phenomenon in the scintillators remain unknown. To explore this, we developed a new microscopic spectrometer (micro-spectrometer) specifically designed for SX excited fluorescence and measured the fluorescence intensity [4].

During the STED experiments using the developed micro-spectrometer, the size of the SX beam, irradiated simultaneously with the laser, was approximately 0.2 × 0.2 mm^2. The beam size was significantly larger than the ϕ1 μm spot diameter of the laser beam. As a result, the SX irradiation area became excessively broad, resulting in an intense fluorescence that exceeded our measurement capabilities to assess the laser-induced decrease for STED. Achieving a matching irradiation area between the SX and the laser is ideal in the present scenario.

Furthermore, the STED laser employed a laser diode that matched the fluorescence wavelength of the scintillator. However, it was discovered that the oscillation wavelength exhibited slight variation of a few nm due to air conditioning of the experimental hall, necessitating improvements in this aspect. This study addresses these issues and demonstrates that induced emission at a wavelength diminishes the optical intensity of SX excited fluorescence at a specific wavelength.

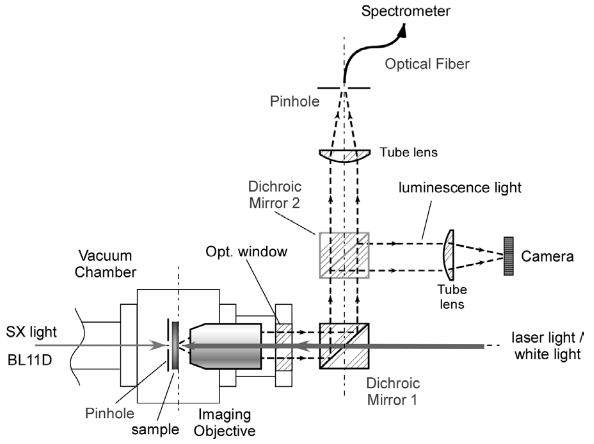

Fig. 1 : Layout of micro-spectrometer for SX excited fluorescence.

2 Improvements of a Fluorescence Micro-spectrometer

Figure 1 provides an overview of the developed a micro-spectrometer for SX excited fluorescence [4]. The figure illustrates the positioning of the sample at the center of the sample chamber in the micro-spectrometer, which is connected to the ICF70 nipple of the beamline. The fluorescence, excited by SX light, is collected and magnified by an objective lens within the square aperture. Subsequently, the fluorescence passes through an optical window, and a dichroic mirror separates it from the irradiation laser light. The separated fluorescence is then focused into a pinhole using an imaging lens. The fluorescence that passes through the pinhole is guided through an optical fiber to a commercially available compact spectrometer (BLACK-Comet, StellarNet Inc.). Additionally, the objective lens functions as the illumination optics for the STED laser beam, reducing the laser beam and other downstream light for sample illumination.

Two main improvements were implemented. Firstly, a high-precision pinhole was introduced in front of the sample to limit the range of SX irradiation and suppress stray light. The pinhole is designed to be easily replaced and aligned with an accuracy of tens of nanometers, utilizing a newly developed side-entry vacuum stage (Figure 2). This ensures sub-micrometer alignment of the optical axes between the SX beam and the optical axis of the micro-spectrometer.

The second improvement involved the addition of an external resonator to stabilize the wavelength of the STED laser [5]. Furthermore, temperature control was implemented to ensure a stable light intensity of the laser. As a result, the wavelength stability was within ±0.3 nm/h, which corresponds to the minimum measurable step width for the utilized spectrometer. The normalized variability of light intensity, expressed as the ratio (%) of the standard deviation to the mean value of light intensity per hour, improved from 12.7% to 0.3%.

3 Evaluation Results

We evaluated the improved fluorescence micro-spectrometer by irradiating the Eu:GGG with 140 eV SX light using BL11D beamline and obtaining fluorescence spectra. Additionally, we attempted to suppress the induced emission of the SX-excited fluorescence by irradiating a 708-nm laser beam under the same conditions. We compared this modified fluorescence spectrum with the original SX-excited fluorescence spectrum. The results demonstrated a reduction in emission intensity due to laser irradiation (Figure 4).

Acknowledgments

We are particularly grateful to Prof. T. Higashiguchi and his students in Utsunomiya Univ. and to Prof. K. Mase in KEK for their assistance in the measurements. The high-precision pinhole manipulator was manufactured at machine shop in IMRAM, Tohoku Univ. This work was supported by JSPS KAKENHI (grant numbers 19H04391).

References

[1] M. S. Alekhin, J. Renger, M. Kasperczyk, P.-A. Douissard, T. Martin, Y. Zorenko, D. A. Vasil'ev, M. Stiefel, L. Novotny, and M. Stampanoni, Opt. Express **25** (2017), pp.1251-1261. doi: 10.1364/OE.25.001251

[2] T. Ejima, T. Wakayama, N. Shinozaki, M. Shoji, G. Hatayama, T. Higashiguchi, Sci. Rep. **10**, 5391 (2020). doi: 10.1038/s41598-020-62100-0

[3] T. Ejima, S. Kurosawa, A. Yamaji, T. Hatano, T. Wakayama, T. Higashiguchi, M. Kitaura, J. Luminescence, **219** (2020) 116850. doi:10.1016/j.jlumin.2019.116850

[4] T. Ejima, Photon Factory Activity Report 2020 #39 (2021) 109.

[5] Y. Tanaka, Kogaku **41**(6) (2012) pp.340-341 (Japanese).

* takeo.ejima.e7@tohoku.ac.jp

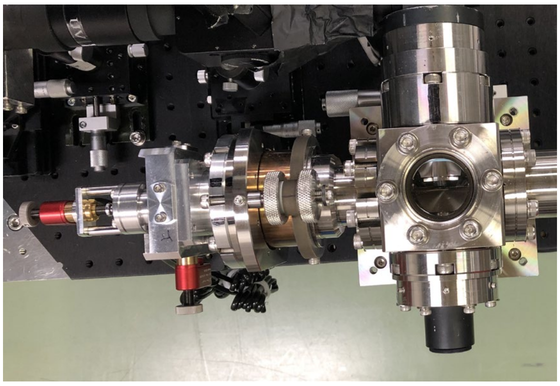

Fig. 2：The high-precision pinhole manipulator shares a similar structure to the side-entry manipulators employed in TEMs. Specifically, inside the vacuum chamber, there is a pinhole attached to an arm extending from the left side in the atmosphere. The pinhole is manipulated by utilizing Picomotors^{TM} to push and pull the tip of the arm.

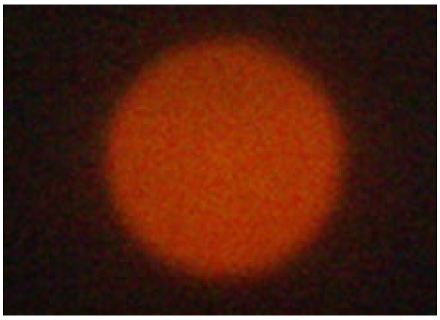

Fig. 3：Fluorescence of the scintillator when it is irradiated with 140eV SX light passing through a ϕ20 μm pinhole positioned in front of it.

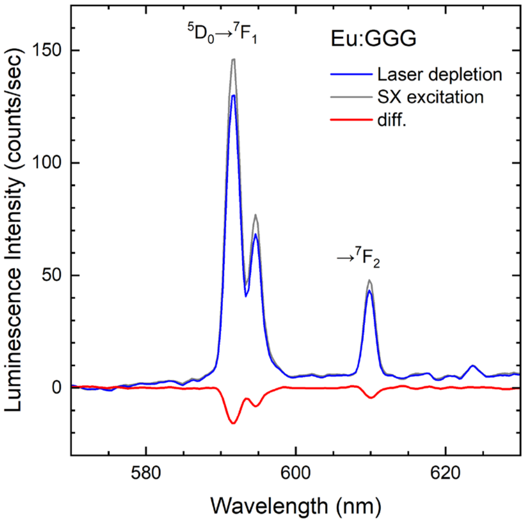

Fig. 4：The fluorescence spectra are represented by the blue and grey curves, corresponding to the excitation with 140 eV SX light with and without 708 nm wavelength laser irradiation, respectively. The red curve demonstrates a decrease in spectral intensity due to the laser irradiation.