

## Development of micro-optics for high-resolution IL spectroscopy with a proton microbeam probe



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### ABSTRACT

Confocal optics for ion luminescence (IL) was developed for the precise analysis of the chemical composition of microscopic targets with an external proton microbeam probe. Anti-reflection-coated confocal micro-lens optics with an effective focus area of approximately  $800 \times 800 \mu\text{m}$  was installed on the microbeam line of a single-ended accelerator. Chromatic aberrations of the confocal optics were examined at wavelengths of 300–900 nm. An electrically-cooled back-thinned charge coupled device spectrometer with a wavelength resolution of 0.5 nm was used for the microscopic spectroscopy and IL imaging of microscopic mineral targets. Simultaneous microscopic IL and micro-PIXE analysis were performed using an external 3 MeV  $\text{H}^+$  microbeam with a current of less than 100 pA. A spectral resolution of 3 nm was achieved for a single IL peak which corresponded to  $\text{Cr}^{3+}$  impurities in a single-crystal of aluminum oxide. The use of IL spectroscopy and imaging for aerosol targets revealed microscopic distributions of the chemical and elemental composition in the atmosphere.

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### 1. Introduction

The atmosphere contains many different types of microscopic particles, which have a profound impact on global ecological systems and human health [1,2]. To improve our understanding of the particle effects and behavior in the atmosphere, it is necessary to investigate the surface chemical composition of individual particles [3,4]. Various heavy elements can be carried by particle surfaces that are dispersed in aerosols [5]. Also, these microscopic particles can act as a propagation medium for organic materials, including microbes and viruses [6,7]. Bulk analysis techniques are usually used for aerosol particles; however, analyzing individual particles is also important for determining the nature of the surface interactions between the carrier microscopic particles and surface contaminants. Therefore, the surface chemical characterization of these particles, particularly those sampled from ambient atmospheric conditions, is attracting considerable interest.

Microscopic imaging and analysis using ion microbeams is an effective analysis tool for the individual microscopic targets because of its high sensitivity. In-air micro-particle-induced X-ray emission (PIXE) analysis is a reliable technique for characterizing and visualizing the elemental composition of the target materials on a micrometer scale [8]. Aerosols are a typical target for PIXE analysis with a microbeam probe, and elemental distribution

imaging has been demonstrated with various systems [9–11]. However, because PIXE relies on the X-ray emission interactions between MeV ions and the inner-shell electrons of target atoms, the only chemical information it can gather about the targets is the elemental composition. A new complimentary technique for PIXE is required to determine the chemical composition and structural distribution in microscopic targets.

The excitation of the target atoms with MeV ions can also induce ion luminescence (IL) through the interaction of the ions with the outer shell electrons. The observed IL bands can be ascribed to each chemical compound and structure [12,13]. This technique provides information about the chemical composition or structures of the target materials [14–16]. IL analysis and imaging from mineral targets has also been successfully accomplished with ion microbeam probes [17–19]. However, the detection efficiency and signal-to-noise (S/N) ratio of IL analysis must be improved in order to analyze low-density micrometer-sized targets.

Currently, we are developing an IL analysis system for analyzing the chemical composition of micrometer-sized targets [20]. Pan-chromatic and monochromatic IL images of micrometer-sized particulate aerosol targets were obtained from IL measurements using a thermoelectrically cooled (TEC) photon detector [21]. The experimental results obtained from the prototype system suggested that the detection efficiency of IL for high-sensitivity spectroscopy and microscopic imaging could be improved by introducing high-efficiency optics [22].

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In this paper, we report the development of confocal micro-optics for external microbeam-driven IL analysis. The focal point of the ion microbeam and the optics of IL were aligned in a confocal system which we designed for IL imaging and spectroscopy. A spectrometer and a photon counting unit were used for the IL spectroscopy and the microscopic IL imaging at specific wavelengths. The IL signal processing procedure was evaluated for several particulate targets with known chemical compositions, and then potential uses of IL analysis were investigated by examining IL spectra from individual aerosol particles.

## 2. Materials and methods

### 2.1. Confocal IL optics

Fig. 1 shows a schematic diagram of the IL spectroscopy setup combined with an external micro-PIXE system on a 3MV single-ended accelerator at the Takasaki Advanced Radiation Research Institute, Japan Atomic Energy Agency [23]. A biconvex lens assembly with effective wavelengths ranging from 300 to 900 nm was placed inside the vacuum chamber at an angle of  $40^\circ$  to the microbeam beam axis at a distance of 150 mm from the irradiation target point. An 800- $\mu\text{m}$ -diameter optical fiber was installed at the opposite focal point of the same biconvex lens. The other end of the fiber was connected to a UV–vis spectrometer (Spectra Co-op, Solid Lambda) for IL spectroscopy. A high-efficiency grating dispersed the IL photons with an effective wavelength range of 200–980 nm and a resolution of 0.8 nm. IL photons were then detected by a TEC back-thinned charge coupled device (CCD) array sensor (Hamamatsu Photonics, S7031-1006). IL photon signals for each wavelength were acquired continuously with an analog-to-digital converter and transmitted to a PC-based system. A high S/

N ratio of 10,000:1 and a good thermal stability of less than 10% were achieved with this experimental setup for continuous IL measurements over several hours.

Fig. 2 shows comparisons of our confocal micro-optics and conventional optics for the IL spectra. Our confocal optics increased the IL collection efficiency by approximately 1000-fold compared with the conventional optics. For the conventional optics, the IL emissions from the micrometer-sized samples were not sufficient to be detected by CCD sensors. In contrast, IL spectra were obtained from same sample with the same detector using our confocal optics, which increased the detection efficiency by approximately 100-fold.

The chromatic aberrations of the IL photons were also examined for an effective IL range of 300–800 nm (Fig. 3). The position of the

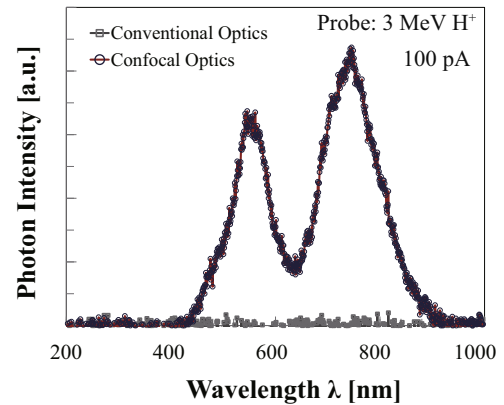


Fig. 2. Comparison of IL spectra obtained using general micro-optics and using confocal micro-optics designed for IL spectroscopy.

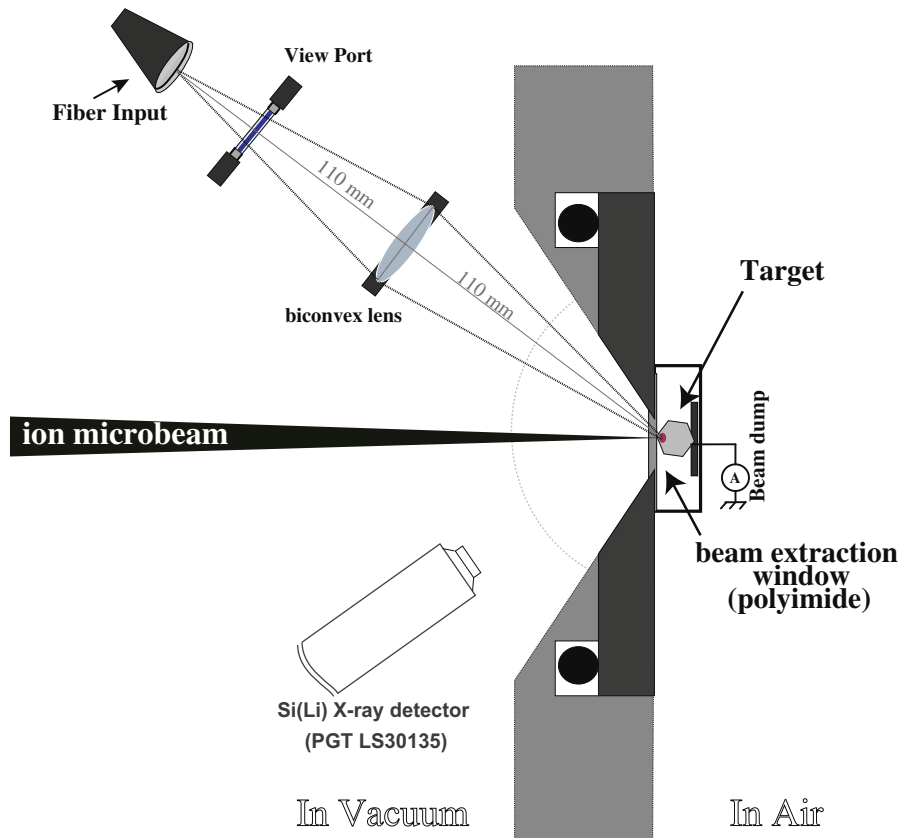


Fig. 1. Schematic of the confocal micro-optics for IL spectroscopy and imaging with an external proton microbeam.

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