

Electron beam excitation assisted optical microscope with ultra-high resolution

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Abstract: We propose electron beam excitation assisted optical microscope, and demonstrated its resolution higher than 50 nm. In the microscope, a light source in a few nanometers size is excited by focused electron beam in a luminescent film. The microscope makes it possible to observe dynamic behavior of living biological specimens in various surroundings, such as air or liquids. Scan speed of the nanometric light source is faster than that in conventional near-field scanning optical microscopes. The microscope enables to observe optical constants such as absorption, refractive index, polarization, and their dynamic behavior on a nanometric scale. The microscope opens new microscopy applications in nano-technology and nano-science.

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1. Introduction

In conventional near-field optical scanning microscopes[1, 2, 3] (NSOMs) capable of sub-diffraction limit resolution, confinement of optical illumination area on the specimen is realized by small aperture of metal-coated fiber probe[4, 5, 6, 7], or apertureless tip probe[8, 9], optically-trapped gold particle[10], solid immersion lens[11], non-optically probing technique[12]. Images are acquired by scanning the small aperture near specimen surface while keeping a constant distance from the specimen surface. A common method of keeping probe-specimen distance is detection of shear forces between the end of the near-field probe and the specimen surface[13, 14]. In shear force mode, a tuning fork is mounted on the fiber probe and the fiber probe is oscillated at its resonance frequency[15, 16, 17, 18, 19]. The amplitude of the probe oscillation is damped by forces between the fiber probe and the specimen. Low throughput[5] of the small aperture limits the scanning speed, because accumulation of signal is required in order to obtain a higher signal-to-noise ratio. Throughput of the fiber probe is given by $\sim (d/\lambda)^4$ where d is the diameter of the aperture and λ is wavelength. Signal-to-noise ratio decreases in proportion to the fourth power of the diameter of the aperture, and worsens rapidly with increasing the resolution. Therefore, the scanning speed of the fiber probe is restricted by difficulty of keeping the probe-specimen distance and low signal-to-noise ratio. The low frame rate of the NSOMs limits the applications to observe dynamic process of living biological specimens or other fast phenomena.

Here, we propose electron beam excitation assisted optical (EXA) microscope in which electron beam focused on a luminescent thin film excites nanometric light source near the specimen. The light emission is well known as cathodoluminescence[20, 21]. It is possible to excite a light source of a nanometric size, because electron beam can be focused to a few nanometers. EXA microscope combines scanning electron microscopy that has nanometric resolution and optical

microscopy that is advantageous to dynamic observation of living biological specimens.

2. Principle of electron beam excitation assisted optical microscope

Figure 1A shows schematic diagram of the proposed EXA microscope. An electron beam is focused on a luminescent film. A specimen is put on the luminescent film directly. The inset in Fig.1A shows magnified image of the luminescent film and the specimen. Nanometric light source is excited in the luminescent film by the focused electron beam. The nanometric light source illuminates the specimen, and the scattered or transmitted radiation is detected with a photomultiplier tube (PMT). The light source is scanned by scanning of the focused electron beam in order to construct on image. Figure 1B shows the structure of sample holder used in EXA microscope. A square aperture of $100 \times 100 \mu\text{m}^2$ size was fabricated on silicon substrate and sealed by SiN film with a thickness of 50 nm.

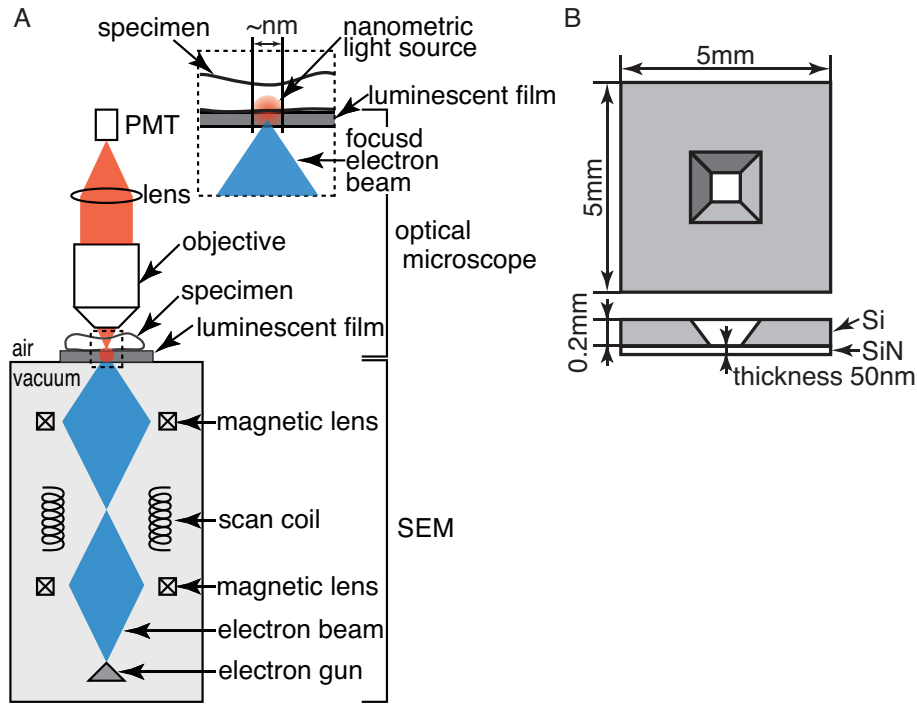


Fig. 1. (A) Schematic diagram of EXA microscope. The scanning electron microscope (SEM) is used for excitation and scanning of nanometric light source. The optical microscope is used to detect the light. (B) Layout of silicon nitride membrane. Silicon nitride membrane on a silicon substrate for separation of vacuum from atmospheric pressure.

In EXA microscope, focusing of the electron beam confines the size of optical spot instead of small apertures used in conventional NSOMs. A few tens nanometer size optical spot is produced easily, by electron beam focused in a few nanometers area. The microscope has a potential to observe dynamic activities of living biological specimens with video frame or faster frame rate, because electron beam can be scanned with modulation of magnetic or electric field without any mechanical moving parts. Higher signal-to-noise ratio is possible, by using highly efficient luminescent film for electron beam irradiation.

Another unique advantage of EXA microscope is the possibility to observe specimens in various environments, such as air, gases, liquids, as well as vacuum. The luminescent film

separates air or liquids surrounding specimens from vacuum where electron beam is focused. Since electron beam energy is converted to light at the luminescent film, and scattered and transmitted radiation from the specimen is detected as signal, vacuum is not required on the specimen side. If the luminescent film does not have enough strength for the separation, one may support the film with transparent thin film.

3. Experimental setup

To verify feasibility of the high resolution EXA microscope, we used silicon nitride (SiN) film of 50 nm thickness as a luminescent material[22] and all optical components were inserted in vacuum chamber of scanning electron microscope (SEM) (JEOL, JSM-6390). SiN film emits blue to ultra-violet (UV) light by irradiation of electron beam. We confirmed in a preliminary experiment that SiN film of 50 nm thickness had enough strength to separate 1 atm air pressure from vacuum.

In the experiment, electrons were accelerated with 10 kV voltage and focused to a 8nm diameter spot on the SiN film to excite nanometric light source. Scattered or transmitted light from the specimen was collected by a lens with numerical aperture 0.65 and detected with a photomultiplier tube (PMT) (Hamamatsu Photonics, R7400-U20). Images were reconstructed from the signal detected with raster scanning of electron beam using a computer. We used polystyrene latex spheres dispersed on the SiN film directly as specimens for resolution verification. The latex spheres were dispersed in monolayer, which was confirmed with an atomic force microscope (Seiko, SPI-3800).

4. Experimental results

Figure 2A and B show observation images acquired with the SEM and EXA microscopes, respectively. The two images were acquired successively. The scale bars in the images represent 100 nm length. The acquisition time of EXA image was 16 minutes at 512 by 512 pixels. The resolution and signal-to-noise ratio of the SEM image in Fig 2A is poor because the SEM image was acquired through the SiN membrane. Each latex sphere of 100 nm diameter was observed clearly and its position in the EXA image was identified with that in the SEM image. This allows us to conclude that EXA microscope has resolution higher than the diffraction limit[23].

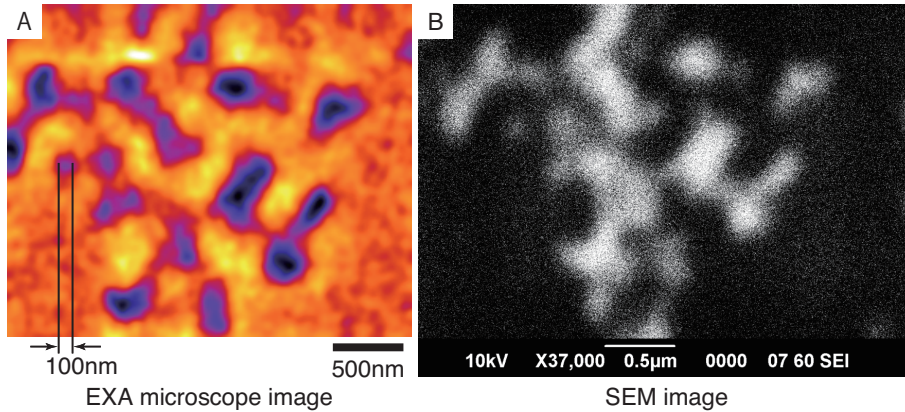


Fig. 2. (A) EXA microscope image of 100 nm latex spheres. (B) SEM image of the same area as in (A). Each latex sphere of 100 nm diameter was observed clearly and its position in the EXA image was identified with that in the SEM image. The latex spheres were dispersed in a monolayer on SiN surface.

Figure 3A shows observation results of latex spheres of 50 nm diameter with EXA microscope. Figure 3B shows intensity distribution on the solid line indicated in Fig.3A. It is recognized that EXA microscope can resolve aligned spheres of 50 nm diameter clearly. We believe that the developed EXA microscope has a potential to achieve a few tens nanometer resolution because electron beam can be focused in a few nanometers.

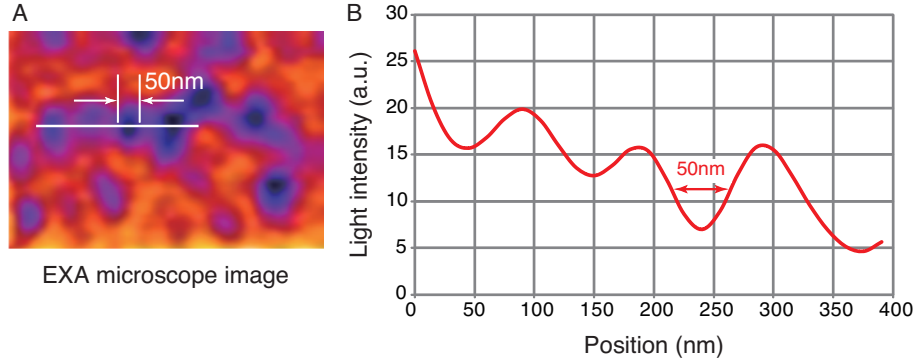


Fig. 3. (A) Observation image of 50nm diameter latex spheres with EXA microscope. (B) Intensity distribution on the solid line indicated in (A). EXA microscope can resolve aligned spheres of 50 nm diameter clearly. The latex spheres were dispersed in a monolayer on SiN surface.

5. Simulation results of electron scattering in the silicon nitride medium

We have simulated electron scattering in the luminescent film in order to evaluate optical resolution in EXA microscope. Scattering is one of main factors that degrade the resolution. Figure 4 shows Monte-Carlo simulation[24] results of electron trajectories in SiN film. Thicknesses of SiN membrane were 30, 50 and 75 nm and acceleration voltages of the incident electron were 1 kV, 5 kV and 10 kV. Electron beam was focused to a spot with diameter of 2 nm on the surface of SiN. It was found that electron scattering decreases with increasing acceleration voltage and decreasing thickness of SiN film. In the case of acceleration voltage of 10 kV, and 50 nm thickness of SiN film, electrons are scattered in the area of 12 nm width. The spot size of electron beam is small enough for achieving resolution above the diffraction limit, even if we take into account electron scattering.

6. Conclusion

We proposed a new type near-field optical microscope and demonstrated its resolution higher than 50 nm. The concept of EXA microscope is an integration of a high-resolution SEM and an optical microscopy suitable for observation of living biological specimens and spectroscopic analysis of materials. Due to the conversion of the electron beam to light source, specimens can be placed in air or liquids, as well as vacuum. Since electron beam can be scanned at a fast speed, it is possible to observe dynamic behavior of specimens with fast frame rate. We expect that the EXA microscope can resolve the scanning speed limitation of conventional NSOMs due to low signal-to-noise ratio at higher resolution, and difficulty of maintaining the probe-specimen distance.

It is possible to apply EXA microscope in a variety of research fields by using functionalized thin film as the light emission layer. Fluorescent imaging can be obtained with UV emission materials, and spectroscopic properties of specimens can be analyzed by using a luminescent

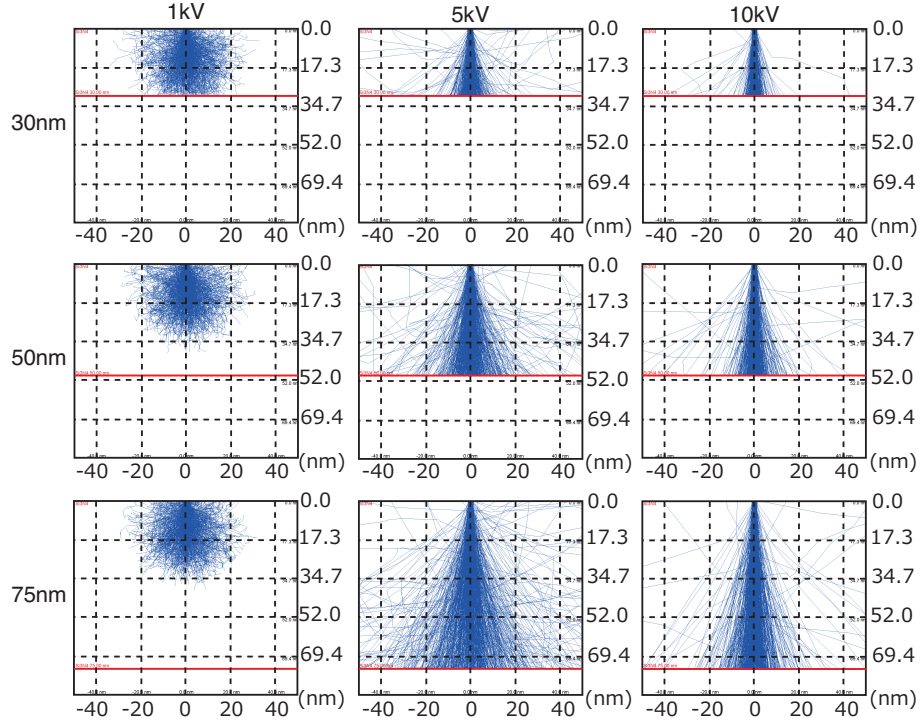


Fig. 4. Dependence of electron scattering on the thickness of SiN and the incident electron acceleration voltage. Thicknesses of SiN membrane were 30, 50 and 75 nm, and acceleration voltages were 1, 5 and 10 kV. Electron beam was focused to a spot diameter of 2 nm on surface of SiN. In the experimental case of acceleration voltage of 10 kV and 50 nm thickness of SiN film, electrons are scattered in the area of 12 nm width.

layer emitting in a broad wavelength region. Dot-array structure[25, 26], nanohole array[27] and nano-cylinder array structure[28] in luminescent films may be effective to reduce electron scattering in luminescent films.

As shown in Fig. 4, some electrons penetrate to specimens and might cause damage. We need optimization of acceleration voltage of electron beam, thickness and structure of luminescent film, in order to minimize the damage in specimens. From the simulation results, we have estimated the resolution of EXA microscope in various conditions. It is important to take into account near-field interaction between the specimen and SiN film in order to evaluate the resolution precisely. We believe that EXA microscope opens new microscopy applications because it realizes a new tool in nano-technology and nano-science.

7. Acknowledgment

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