Schwarzschild microscopes in vacuum ultraviolet and soft X-ray regions

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Abstract Microscopes in vacuum ultraviolet and soft X-ray regions using a normal incidence type of Schwarzschild objective are reviewed. The objective consists of a concave mirror and a convex mirror coated with a high reflectance multilayer, having a large numerical aperture comparing with other objectives. The microscopes have been used to diagnose inertia-confinement-fusion plasmas, and to investigate small samples or microstructures of inorganic and organic materials by imaging them using laboratory light sources. Synchrotron radiation has been also used to obtain a microbeam for a photoelectron scanning microscope with a spatial resolution of 0.1 μm. The structure and performance of two laboratory microscopes developed at Tohoku University are demonstrated. One of them is a soft X-ray emission imaging microscope. An image of an artificial pattern made of W and SiO_2 on Si wafer by focusing Si *L* emission was presented. The other is an ultraviolet photoelectron scanning microscope using a He (helium) gas discharge lamp. The valence band spectra of a microcrystal of $FeWO_4$ were presented. Furthermore other applications such as demagnifying optics for lithography and optics to gather fluorescence for emission spectroscopy are introduced.

Key words Microscope, Schwarzschild objective, Multilayer, Vacuum ultraviolet, Soft X-ray, Photoelectron
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1 Introduction

Recently, investigations of small materials, materials composed of small grains, artificial superlattices, living cells and other samples having microstructures have been required to clarify their macroscopic features from microscopic characteristics. There are many kinds of microscopes and among them the most popular ones are far field optical microscopes using visible light. They have advantages of imaging without scanning and having long working distances to be utilized for various treatments of samples. Their spatial resolution is restricted by so called diffraction limit ($\Delta x = 0.61\lambda/NA$, *NA* : numerical aperture)[1] and of the order of submicrometers. (The resolution of near field microscopes such as a near field luminescence microscope is a little smaller than the limit.) Therefore the efforts have been made to realize much smaller resolution using the light with short wavelength such as vacuum ultraviolet, soft X-ray and even hard X-ray.

In early days, the attention was not paid to the microscopes using short wavelength light because of the difficulty to fabricate precise optical elements and the lack of bright light sources as well. Nowadays, however, the precise machinery has been developed and the bright light sources such as plasma light sources and synchrotron radiation appear, so that the microscopes using short wavelength light have been developed considerably and still being developed more.[2,3] There are five typical objectives for short-wavelength-light microscopes. They are classi-

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fied into three categories.

The first ones are objectives using mirrors. They are of Schwarzschild type used in infrared, visible, ultraviolet, vacuum ultraviolet and soft X-ray regions shown in Fig.1,[4] of Wolter type shown in Fig.2 (a) and of KB (Kirkpatrick-Baez) type shown in Fig.2 (b). The Schwarzschild type is a normal incidence objective composed of a concave mirror and a convex mirror. They are coated with high reflectance multilayers when they are used in vacuum ultraviolet and soft X-ray regions. The Wolter and KB types are grazing incidence objectives usually used in hard X-ray region. The second one is zone plate used both in soft and hard X-ray regions shown in Fig.2 (c). The third one is the refraction lens used in hard X-ray region shown in Fig.2 (d),[5] the shape of which is concave, because the refraction index of every material in hard X-ray region except around core absorption edges is slightly smaller than 1. Among the above-mentioned five objectives, the Schwarzschild type has the largest numerical aperture and the microscope using the zone plate has achieved the smallest spatial resolution of 10 nm. In vacuum ultraviolet and soft X-ray regions, microscopic images have been obtained often in transmission mode with smaller resolutions comparing with those of visible microscopes. In addition, the information of highly excited states of atoms and valence band structures of materials is available. In magnifying configuration, the Schwarzschild objective has a small F-number because of a large numerical aperture, so that it has an advantage of imaging objects by focusing the light of which divergence angle is large. The light is, for instance, the emission from plasmas, and the soft X-ray emission from materials excited by light or electron beams. Furthermore, the measurements of photoelectrons using vacuum ultraviolet and soft X-ray also give the information of valence band structures. In demagnifying configuration, the Schwarzschild objective is useful to obtain a so called microbeam for photoelectron scanning microscopes because of having a large working distance to situate the photoelectron energy analyzer comparing with other objectives. However the bright light is required, because the F-number is large.

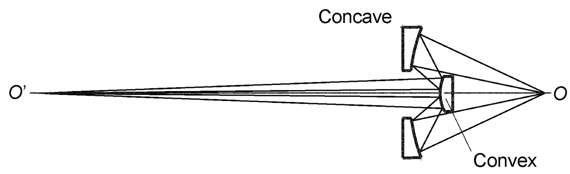

Fig.1 Schematic of Schwarzschild objective.

In this article, the Schwarzschild type is explained briefly with an introduction of a photoelectron scanning microscope using synchrotron radiation. Furthermore, the structure and performance of two laboratory microscopes developed at Tohoku University are demonstrated with experimental results. They are a soft X-ray emission microscope and an ultraviolet photoelectron scanning microscope. Furthermore, the applications of the Schwarzschild objective to optics other than microscopes are introduced. They are demagnifying optics for lithography and optics to gather fluorescence for emission spectroscopy, which have been developed in other laboratories.

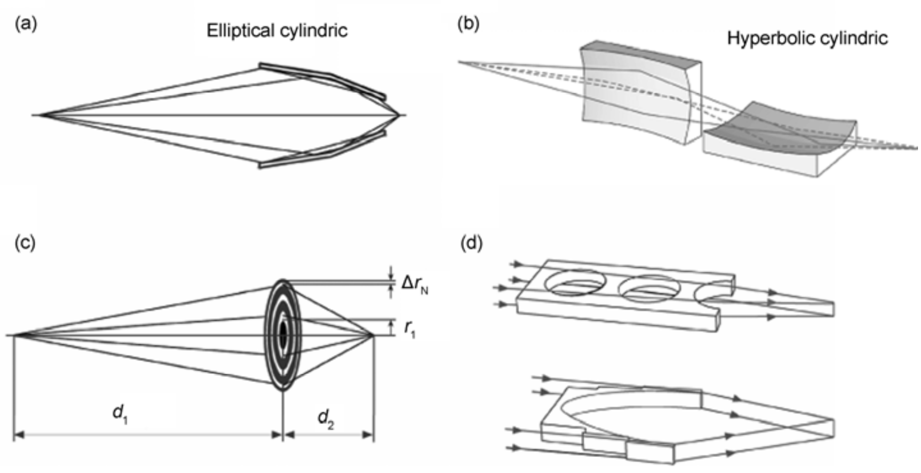

Fig.2 Schematics of objectives. (a) Wolter type, (b) Kirkpatrick-Baez type, (c) zone plate, (d) refraction lens.

2 Review of Schwarzschild microscopes

2.1 Schwarzschild objectives

In the Schwarzschild objectives, the centers of radii of curvature of two spherical mirrors, one of which is concave and the other is convex, are positioned at the same point to reduce the third order spherical and coma aberrations[6] or separated a little according to the further consideration on aberration, as will be discussed in **3.1**. The Schwarzschild objective works as a magnifying optics, when an object is at point O and an image at point O', while the objective works as a demagnifying optics, when an object is at point O' and an image at point O (see Fig.1). In the former case, imaging microscopes can be constructed. In the latter case, when a pinhole is situated at point O', one can obtain a small spot, that is a microbeam, at point O, and scanning microscopes can be constructed. Recently, a type using non-spherical mirrors is discussed, which has an advantage of a small resolution with a relatively large tolerance for setting the mirrors.[7]

2.2 Multilayers

The Schwarzschild objective is used in the normal incidence. In the wavelength region below 30 nm, the normal incidence reflectances of usual materials are less than 5%, so that high reflectance coating materials are required. Recently multilayers have been developed extensively as the high reflectance coating materials.[8] The multilayer is composed of two materials as shown in Fig.3, which are piled periodically by magnetron sputtering or ion beam sputtering instruments as usual. The reflectance is enhanced at the wavelength λ, which satisfies the Bragg equation $n\lambda = 2d\sin\hat{\theta}$, where $\hat{\theta}$ stands for grazing angle and d, period-distance of the multilayer.

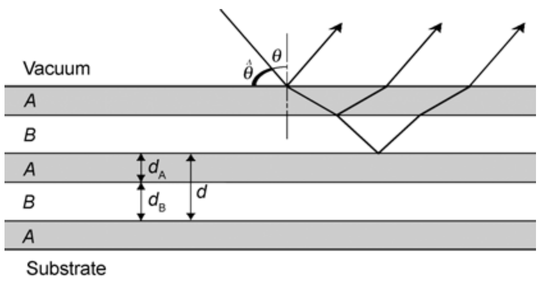

Fig.3 Structure of multilayer.

The two materials having large difference in refraction indices with small extinction coefficients have to be selected to realize high reflectance. The optical constant tables are filed in the home page of Center for X-ray Optics, Lawrence Berkeley National Laboratory.[9] Further experiments to supplement the above-mentioned optical constant table have been made.[10] The highest reflectance of about 70 % has been achieved by Mo/Si and Mo/Be around 13 nm as shown in Fig.4.[11] In this case, the layers of B_4C are inserted to prevent diffusion in between Mo and Si, and in between Mo and Be.

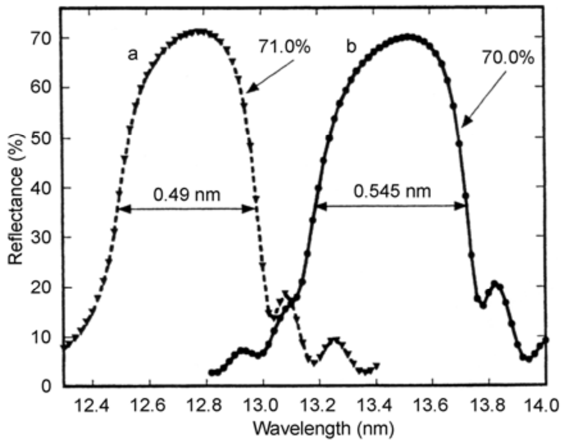

Fig.4 Reflectance spectra of (a) Mo/Si and (b) Mo/Be multilayers.[11] In between Mo and Si, and in between Mo and Be, layers of B_4C are inserted to prevent diffusion.

Recently Mo/Si multilayers bearing against high heat load are fabricated with insertion of thin SiC or SiO_2 layers in between Mo and Si.[12] Around 60 eV, Al/YB_6 is one of good pairs;[13] and around 400 eV in so called water-window region, Sc/Cr is promising.[14] By a multilayer, the reflectance of light at a certain wavelength is enhanced, but there is a requirement for the multilayer to reflect the light at more than two wavelengths. For instance, for the photoelectron spectroscopy in laboratory, it is preferable to use He- I (58.4 nm, 21.2 eV) and He- II (30.4 nm, 40.8 eV) resonance lines from a He (helium) gas discharge lamp. However, there were not any coating materials to have high reflectance for both resonance lines. Therefore, the multilayer having high reflectance for both lines called two-color multilayer was developed.[15] It is one kind of so called supermirror. The structure and the reflectance spectrum of $SiC\text{-}Mg/Y_2O_3$ two-color multilayer are shown in

Figs.5 and 6, respectively.

The multilayer has more than 10% reflectance in the wide photon energy range between 10 and 40 eV. Therefore it is useful for the use of not only other resonance lines of Ar-Ⅰ(11.6 eV) and Ne-Ⅰ(16.7 eV), but also the continuum light of synchrotron radiation with a monochromator. When the multilayer is coated on the mirrors, its period-distance must be increased in the radial direction from the center of the mirror to its outside with the increase in the angle of incidence. The method to control the period-distance in such a way has been developed.[16] In China, big efforts have been also made to develop high reflectance multilayers for Schwarzs- child objectives.[17,18]

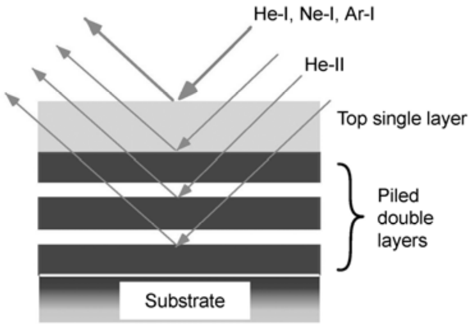

Fig.5 Structure of two-color multilayer for He-Ⅰand He-Ⅱ resonance lines.[15]

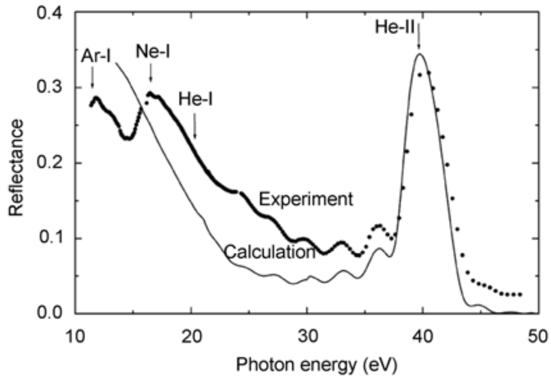

Fig.6 Reflectance spectrum of SiC-Mg/Y_2O_3 two-color multilayer.[15] Arrows indicate energy positions of not only He-Ⅰ and He-Ⅱ, but also Ar-Ⅰand Ne-Ⅰresonance lines. θ=10°.

2.3 Use of Schwarzschild microscopes

In vacuum ultraviolet and soft X-ray regions, the Schwarzschild microscopes have been used for diagnosis of inertia-confinement-fusion plasmas confined within a space of 1 mm diameter.[19] The objectives have been also used for observation of small materials and microstructures in transmission mode using laboratory light sources such as laser produced plasma with smaller resolutions comparing with those of visible microscopes.[20] The microscopes aiming at the observation of living cells under natural environment in the water-window region between 4.4 nm (C *K* absorption edge) and 2.3 nm (O *K* absorption edge) have been under way. The photoelectron scanning microscope using synchrotron radiation was initiated by Margaritondo et al.[21] and followed by the researchers in Elettra Facility.[22] They constructed the microscope having three pairs of Schwarzschild objectives coated with Mo/Si (74 eV), Mo/Si (95 eV) and Ru/B_4C (110 eV), which can be interchanged in situ. The Ga 3d core photoelectron images of cross section of p-n GaAs superlattice are shown in Fig.7. The two top images are obtained by tuning the energy analyzer at the kinetic energy of the Ga 3d peak of (a) n-type GaAs and (b) p-type GaAs. The layers of n-type GaAs appear bright in (a) and dark in (b). Image (c) is the difference between the two top images. In Fig.7 (c), the bright and dark parts correspond to the p- and n-types of GaAs, respectively. The spatial resolution is estimated as 0.1 μm.

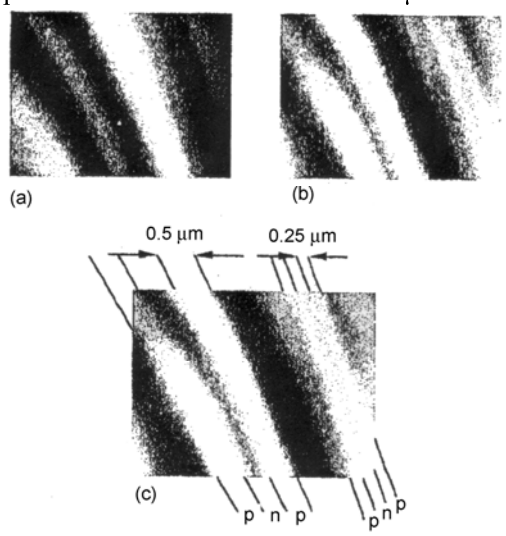

Fig.7 Cross-sectional image of an MBE-grown p-n GaAs superlattice with different periods obtained by sample scanning.[22] The two top images are obtained by tuning the energy analyzer at the kinetic energy of the Ga 3d peak of (a) n-type GaAs and (b) p-type GaAs. The layers of n-type GaAs appear bright in (a) and dark in (b). Image (c) is the difference between the two top images.

3 Schwarzschild microscopes at Tohoku University

At Tohoku University, two kinds of Schwarzschild microscopes have been developed. One of them is a soft X-ray emission imaging microscope and the other, an ultraviolet photoelectron scanning microscope.

3.1 Soft X-ray emission imaging microscope

When materials are irradiated by photons or electrons with higher energies than the binding energy of a certain core level, X-ray emission can be observed due to the transition of electrons from occupied states to the core hole level. The emission is element-specific because its energy is characteristic of the element, so that the image of a material gives the information on the distribution of the element in the material. The schematic of the soft X-ray emission imaging microscope focusing Si *L* emission is shown in Fig.8.[23] It consists of a sample holder, a Schwarzschild objective and a detector. In the sample chamber, an electron gun is equipped for the excitation of samples. The samples are irradiated by an electron beam of 1 μA accelerated to 2.5 kV. At the focusing point, a resistive-anode-type position- sensitive detector is situated. An ion sputter pump and a turbo molecular pump are attached to evacuate the sample and the objective chambers.

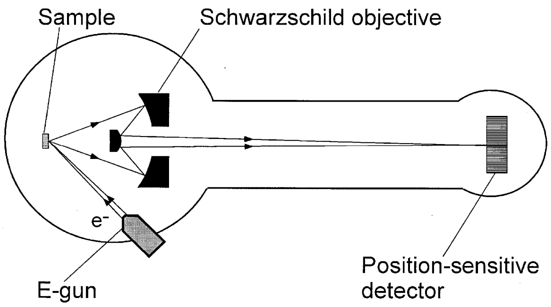

Fig.8 Schematic of soft X-ray emission imaging microscope.[23]

When two mirrors are aligned in a concentric geometry, the third order spherical and coma aberrations are simultaneously reduced,[6] while the eccentric geometry has an advantage of diminishing higher order aberrations though the third order coma aberration is not eliminated. As a result of ray tracing, the latter was employed because it gave a smaller focusing spot, which was 30nm on the optical axis in ideal case. The distance *s* between the centers of curvature of two mirrors was decided as 0.69mm. The size and configuration of two mirrors are given in Fig.9. The distance from the object to the focusing point was 980.969mm. Magnifying factor was 50 and numerical aperture was 0.25. The mirrors were fabricated from fused silica. Shape errors were estimated using an optical interferometer (Zygo, MARK II) as $\lambda/10$ (λ=633 nm) and $\lambda/6$ for the concave and convex mirrors, respectively. The surface roughness was measured using a surface profiler (WYKO, TOPO-2D) to be about 0.3nm for both mirrors. The error of *s* was within 40μm, while the allowed error was 50μm as estimated from ray tracing. The two optical axes of both mirrors were adjusted using an autocollimator as the distance between the two axes was smaller than 13μm.

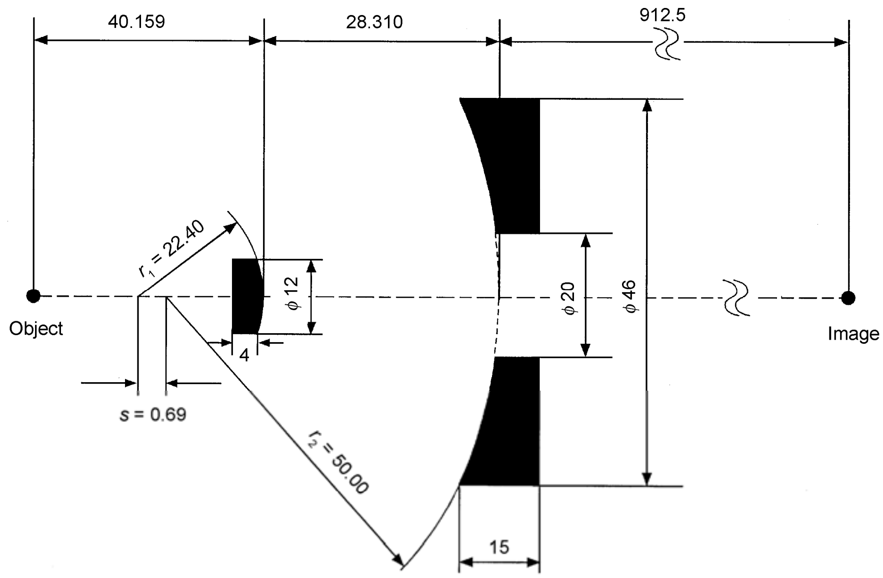

Fig.9 Schwarzschild objective in eccentric arrangement.[23]

The multilayers were Mo/Si made by the use of the magnetron sputtering instrument. At each position on the mirror in the radial direction, the most appropriate period-distance of the multilayer has to be chosen corresponding to the angle of incidence. However in this study, the period-distance was constant, because the width of the reflectance peak was not so sharp for the change of the angle of incidence, that the decrease of the reflectance from the peak value was small. The overall throughput of the objective was 14% at a peak wavelength of 13.3 nm.

At the first time, the spatial resolution was checked by the use of visible light from a W lamp and it was about 1 μm close to the diffraction limit. The test sample for the soft X-ray emission microscopy was lithographically patterned stripes of SiO_2 and W, both 5 μm wide and 50 nm thick, on a Si wafer. Fig.10 shows the SEM (scanning electron microscope) image of this stripe pattern and Fig.11 is its Si *L* emission microscopic image. The intensity at the detector was 10^5 photons • s^{-1}, which corresponded to 400 photons • s^{-1} • mm^{-2}. The image was taken in the recording time of 6 min. The yellow area corresponds to SiO_2, while the red area to W. The stripe pattern is distinctly observed. From the figure, the Si *L* emission emitted from the Si wafer under W layer seems to be partially observed. The escape depth of the soft X-ray emission is of the order of 100 nm, so that this microscope can be used for the observation of Si pattern even when it is covered by other thin materials (buried layer).[24] The spatial resolution, which was limited by the pixel size of the resistive-anode-type position-sensitive detector of 50 μm, was estimated as 2—3 μm. To get better resolution, a detector with smaller pixel size, an X-ray film or an imaging plate having small grain size have to be used. This microscope can be applied to observe materials including other elements such as Be, B, C, Mg, Al, P, S by choosing appropriate multilayers to detect the soft X-ray emission from these elements effectively and selectively. The structure of an absorption spectrum near a core edge of a certain element is specific for its chemical states, that is, for the kinds of compounds, so that one can excite a certain compound selectively choosing the photon energy. Synchrotron radiation is a continuum light source. Therefore by its use, one can obtain images specifying the kind of compound.

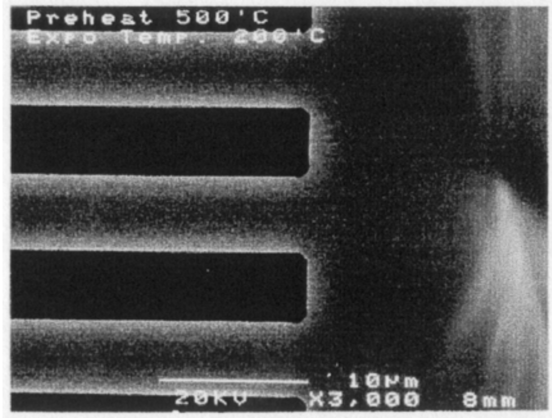

Fig.10 SEM image of the test sample.[23] The dark area is SiO_2 and the bright area is W, both 5 μm in width.

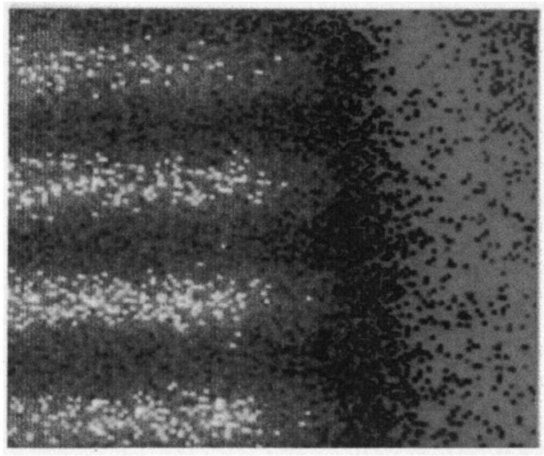

Fig.11 Si *L* emission microscopic image obtained for the test sample.[23] Yellow area corresponds to SiO_2, while red area to W. The observed area is the same as that shown in Fig.10.

3.2 Ultraviolet photoelectron scanning microscope

By the use of a demagnifying optics with a pinhole, one can obtain a microbeam. As a monochromatic microbeam irradiates a sample and photoelectrons from the sample are detected and their energies are analyzed, one can make photoelectron spectroscopy on a small area of the sample. One can also make the spectroscopy on small samples. This system can be used as the scanning microscope, as the sample is scanned with the photoelectron signal taken spot by spot with a fixed kinetic energy and registered in a computer, and after the scanning the signals are arranged to form the image. The photoelectron microscope using synchrotron radiation developed by Margaritondo et al. was such a microscope.[21,22]

In laboratory, there are two kinds of photoelectron spectorometers. They are XPS (X-ray photoelectron spectrometer) and UPS (ultraviolet photoelectron spectrometer). The difference is in the kind of light sources. In the former, an X-ray tube [Mg *K*α (1253.6 eV) and Al *K*α (1456.6 eV)] is used, while in the latter, a He gas discharge lamp [He-Ⅰ (21.1 eV) and He-Ⅱ (40.8 eV)] is equipped. In UPS, the energy resolution of photoelectron spectra is small and the excitation with photons of different energies may cause remarkable changes in spectral features in comparison with in XPS. Accordingly, the UPS microscope using the He gas discharge lamp was developed. (The Schwarzschild objective is not appropriate for the XPS microscope because of the lack of high reflectance coating materials in the normal incidence.)

The schematic of the ultraviolet photoelectron scanning microscope is shown in Fig.12.[25] The light emitted from the He gas discharge lamp was transmitted through a pinhole and focused using the Schwarzschild objective on samples. The Schwarzschild objective was used as a demagnifying optics. The demagnifying factor was 1/68. The objective and its performance resembled those of the soft X-ray emission imaging microscope described in **3.1**. The multilayer coated on the mirrors was the two-color multilayer similar to that described in **2.2**. Samples were mounted on a sample scanning stage in the main chamber and could be moved by 10 mm in *s*, *y* and *z* directions with the smallest step of 2 μm. The electron energy analyzer was of cylindrical retarding field type. This analyzer has an advantage of having a long working distance between its entrance and the position from which photoelectrons are emitted. The Schwarzschild objective has also a long working distance, so that it is easy to construct the photoelectron microscope. An Ar ion sputtering gun for cleaning the sample surface was attached to the main chamber. The chamber was evacuated by two ion sputter pumps, a Ti sublimation pump and a turbo molecular pump. Three-step differential pumping was employed in order to reduce the leakage of He gas into the chamber.

At the first time, the spatial resolution was checked by the knife edge test using visible light and a mesh-screen. The pinhole diameter was 300 μm. The beam size was estimated as about 5 μm, which was consistent with the calculated value. Secondly, the

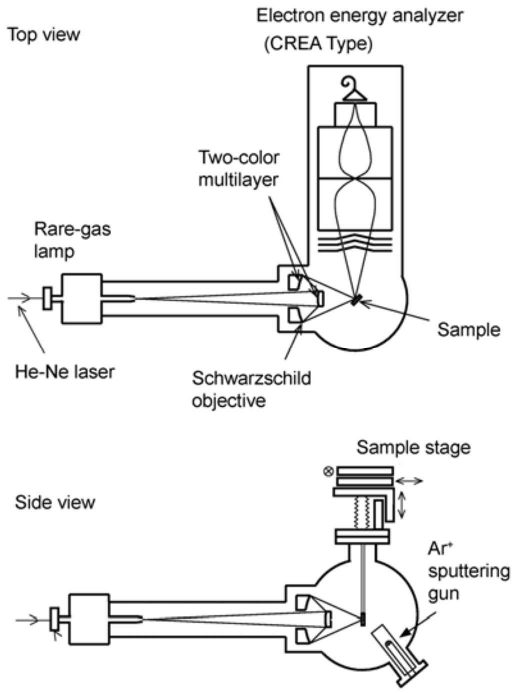

Fig.12 Schematic of ultraviolet photoelectron scanning microscope.[25]

spatial resolution in the photoelectron measurement was checked. In this case, the pinhole diameter was 2 mm and the diameter of microbeam was 25 μm. The size of the microbeam was limited by the size of the pinhole, which was determined by the requirement of sufficient intensity of light or counts of photoelectrons. (Recently, a diameter of 15 μm has been obtained.) By the use of intense light from a He microwave-discharge lamp or synchrotron radiation, the spatial resolution of less than 1 μm can be achieved. By scanning, the microscopic image of the mesh-screen had been obtained. Furthermore, the valence band photoelectron spectra of a microcrystal of $FeWO_4$ having wolframite structure were measured for an example. The size of the sample was 0.7 mm × 0.5 mm as shown in Fig.13. Fig.14 shows its photoelectron spectra taken by the use of not only He-Ⅰ and He-Ⅱ, but also Ar-Ⅰ and Ne-Ⅰ. The photoelectron intensity is dependent on the absorption cross section due to the transition from constituent orbitals. The overall spectra for He-Ⅰ and He-Ⅱ are similar, while the peak *B* is enhanced for Ne-Ⅰ. On the other hand, the cross sections of O 2p and W 5d orbitals for He-Ⅰ and He-Ⅱ are almost the same and much larger than those of Fe 3d orbital for He-Ⅰ and He-Ⅱ. Furthermore the cross section of Fe 3d orbital for Ne-Ⅰ is much larger than those for He-Ⅰ and He-Ⅱ. Therefore, it was suggested that O 2p and W 5d orbitals contribute to the wide range of the valence band spectra from its top to bottom (peaks *A*, *C*, *D*, *E*) and Fe 3d orbital contributes to the structure around the binding energy of –2.5 eV (peaks *B*).

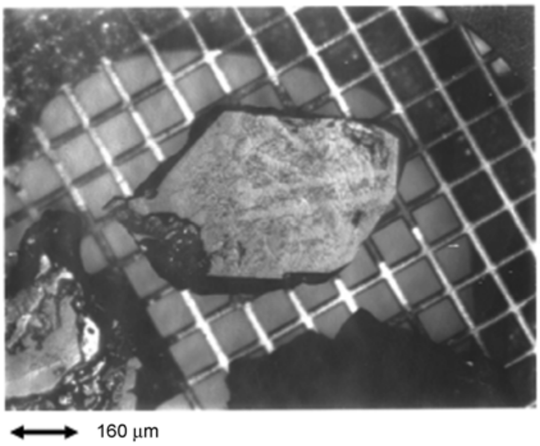

Fig.13 Microcrystal of $FeWO_4$.[25]

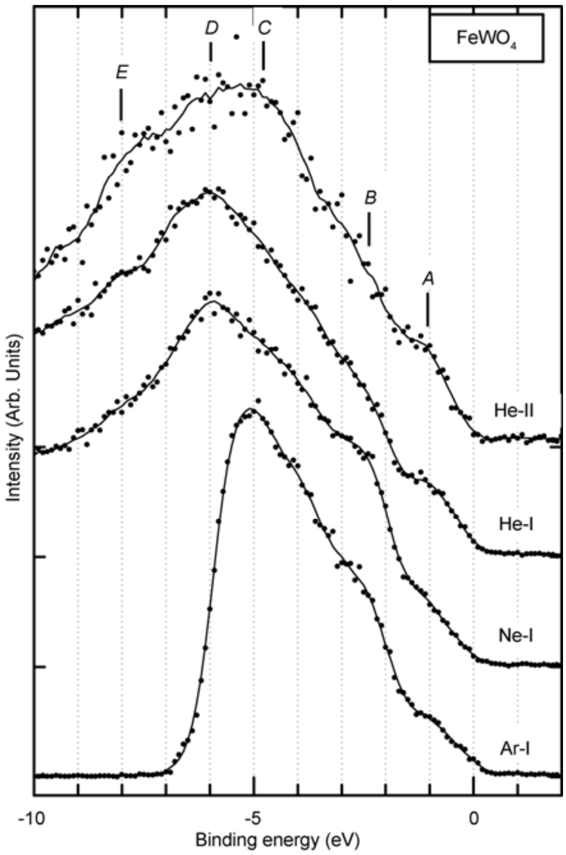

Fig.14 Valence photoelectron spectra of $FeWO_4$.[25]

At Tohoku University, the third Schwarzschild microscope using a laser plasma light source is under construction.[26] It is a transmission type, challenging to realize the spatial resolution of 0.05 μm using a new technique of correcting the error of the multilayer-coated surfaces of the mirror with observing the interference pattern and other new techniques.

4 Other applications of Schwarzschild objective

There are some applications of the Schwarzschild objective in vacuum ultraviolet and soft X-ray regions other than the microscope. One of them is demagnifying imaging optics which aims at the lithography to make ultra large scale integrated (ULSI) circuit as shown in Fig.15.[27,28] Strictly speaking, the optics in this case is not of Schwarzschild type, but similar. The surface of the mirrors are coated with Mo/Si multilayers and the surface of the mask as well. The next generation ULSI circuit will be fabricated by the use of excimer lasers, but further ULSI may be made by the use of soft X-ray around 13 nm with demagnifying optics.

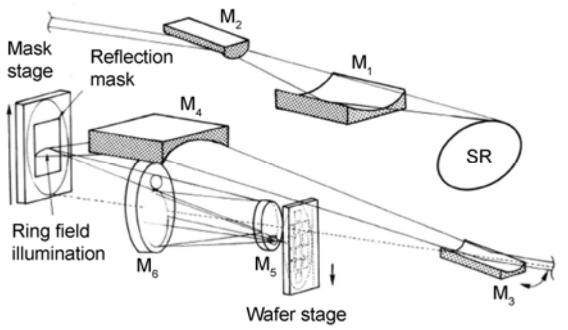

Fig.15 Schematic of demagnifying optics for fabrication of ultra large scale integrated (ULSI) circuits.[28] SR means synchrotron radiation source.

The other application is microscopic fluorescence spectroscopy on solids. At HASYLAB, it was performed by the use of a monochromatic microbeam as an excitation light (an elliptical cylindric mirror was used to make a microbeam) and the Schwarzschild objective,[29] which gathers the fluorescence and focuses it on an entrance slit of a fluorescence spectrometer. In this case, the surfaces of the mirrors are not coated with multilayers, but coated with a usual coating material, so that the measurements can be made continuously in wavelength in visible, ultraviolet and longer wavelength region of vacuum ultraviolet. By scanning the samples, one can get emission microscopic images.

5 Summary

The Schwarzschild objective consists of a concave mirror and a convex mirror having a large numerical aperture comparing with other objectives. The positions of the centers of radii of the two mirrors are situated at the same point or separated a little from the requirement of the spatial resolution. The mirrors are coated with the high reflectance multilayer, when the objective is used in vacuum ultraviolet and soft X-ray regions. Examples of the high reflectance multilayers are Mo/Si around 13 nm, Sc/Cr around 3 nm and Al/YB_6 around 20 nm. The two-color multilayer of $SiC\text{-}Mg/Y_2O_3$ is useful for He-Ⅰ (58.4 nm) and He-Ⅱ (30.4 nm). In magnifying configuration, the Schwarzschild microscopes are very useful to obtain microscopic images of objects emitting the divergent light. By the use of such divergent light of laboratory light sources in vacuum ultraviolet and soft X-ray regions, small materials and microstructures have been investigated in transmission mode with smaller resolutions comparing with those of visible microscopes. The microscopic studies have been also made by focusing such divergent light of the emission from highly excited states of atoms in plasma and the soft X-ray emission from materials due to the transition from the valence band to the core hole level. At Tohoku University, the Schwarzschild soft X-ray emission imaging microscope was constructed using a multilayer specifying the element of Si. The soft X-ray emission was generated by the use of the electron beam. The Si *L* emission image of the stripe pattern made of SiO_2 and W was clearly observed. The microscope enables one to observe the pattern even when covered with other thin materials (buried layer). In demagnifying configuration, the Schwarzschild objective is also useful to obtain a microbeam for the photoelectron spectroscopy which needs a large working distance. In this case, a bright light source is required. The world record of the resolution of 0.1 μm is achieved in the photoelectron microscopy by the use of synchrotron radiation at Elettra Facility. At Tohoku University, the photoelectron microscopy is realized even in laboratory by the use of the He discharge lamp. An image of a mesh-screen was obtained and the valence photoelectron spectra of a small sample of $FeWO_4$ were measured. The challenge realizing 0.05 μm resolution in transmission mode is underway at Tohoku University with developing new techniques such as correction of the error of the multilayer-coated mirror surfaces. Synchrotron radiation is intense continuum light, so that it can be useful for generation of the soft X-ray emission and the photoelectron as the excitation light with choosing its energy, which has an advantage to clarify the electronic states in detail with high resolution. By the above-mentioned achievements, the microscopes will be used to clarify macroscopic features from microscopic images in material and life sciences. The further ULSI will be made using demagnifying optics similar to the Schwarzschild optics. By gathering fluorescence from solids excited by a microbeam by the use of the Schwarzschild objective, the microscopic emission experiments have been available.

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