Selected Article

Development of X-ray Guide Tube

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X-rays entering a smooth surface such as glass at an extremely low angle reflect while maintaining the original strength (total reflection). An X-ray light-collecting device that produces a high-intensity X-ray microbeam using this phenomenon of X-ray total reflection is the X-ray guide tube. This article compares the X-ray guide tube with other X-ray light-collecting devices and outlines the process of X-ray guide tube development and the effects of the tube.

Introduction

Analysis of microscopic regions is very much sought-after amid the continuing promotion of advanced technologies such as development of materials with many advanced functions, miniaturization of electronic and electric parts, and growth of nanotechnology and biotechnology. Among various analytical techniques, X-ray analysis is an effective method of analysis for its many advantages including the capability of analyzing the internal structure of a sample by transmission X-ray, identifying the type and quantity of elements that constitute a sample, and analyzing the crystal structure of a sample in a non-destructive manner. But it is impossible for any given method as used to focus visible light in optical lens or mirror to focus X-ray because the refraction index of X-ray is extremely close to 1 and the reflectivity of normal incident light is extremely close to 0. With the method to focus X-rays by shielding them (collimator method), if the irradiation area of X-ray is reduced, the X-ray intensity will diminish in proportion to the area decreasing. Therefore, despite the impression of "microanalysis," the actual lower limit of analysis was a few hundreds of μm. Nowadays, we can focus strong X-rays to a small point because of the emergence of powerful X-ray sources such as large synchrotron radiation facilities and X-ray focusing devices such as X-ray mirrors using total reflection or multilayer films, X-ray guide tube, zone plate and refractive lenses. At present, we have a spatial resolution capability in a submicron level, and we see active research and development of X-ray microscope, X-ray telescope, X-ray lithography, or microscopic region analyzer using this technology[1]-[3].

Focusing of X-rays

The basic principle of X-ray focusing is to use X-ray diffraction, refraction and total reflection to bend and converge X-rays into a single point. Each element of the principle is described in the following sections.

Diffraction

When X-ray is radiated to crystals or multilayer films arranged in parallel and at even intervals, reflective X-rays from each plane of the lattice planes cause interference and mutually reinforce each other (Figure 1). This phenomenon is X-ray diffraction. The condition under which interference occurs is called Bragg condition and expressed by Equation (1).

$$n\lambda = 2d\sin\theta \quad \cdots\cdots (1)$$

λ : wavelength
d : interval of lattice planes
θ : glancing angle
n : positive integer

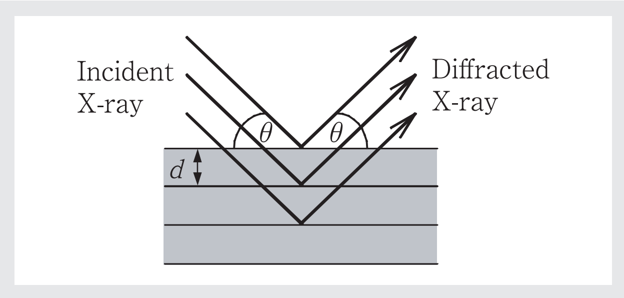

Figure 1 Diffraction

X-ray focusing devices based on X-ray diffraction include curved crystal mirrors, multilayer mirrors and zone plates[4], but their focusing efficiency is poor as they can only focus monochromatic X-rays because the diffraction conditions are determined by the wavelength of the X-rays. Therefore, those devices are primarily used in synchrotron radiation facilities where strong monochromatic X-rays can be produced.

Refraction

Since the refractive index of X-ray n is a value only slightly smaller than 1, refraction with a concave lens allows focusing of X-ray[5].

The refractive index is expressed by Equation(2). X-ray refraction is shown in Figure 2.

$$\text{Refractive index } n = 1 - \delta \quad \cdots\cdots\cdots \quad (2)$$

$$\delta = e^2 \lambda^2 \rho N_A Z / 2\pi mc^2 A$$

e : electron charge

λ : wavelength

ρ : density

N_A : Avogadro's number

Z : atomic number

m : mass of an electron

c : light speed

A : atomic mass

(δ is 10^{-5} to 10^{-6} in the X-ray region.)

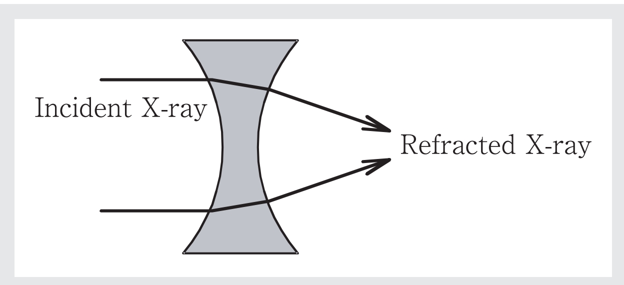

Figure 2 Refraction

X-rays are refracted, but the refraction angle is very small. Therefore, dozens or hundreds of concave lenses have to be combined. As an X-ray runs through the lenses, the X-ray is absorbed and its intensity decreases. Since the refractive index is determined by the wavelength of the X-ray, a monochromatic X-ray source has to be used. Thus, this kind of lens assembly is used at synchrotron radiation facilities that can radiate powerful monochromatic X-rays.

Total Reflection

The refractive index of the X-ray is only slightly smaller than 1, as explained above. Thus, if an X-ray enters to an extremely smooth plane at an extremely small angle, the X-ray can be reflected without losing its intensity. This phenomenon is called total reflection (Figure 3). Techniques using the principle of total reflection have high focusing efficiency as they are capable of totally reflecting X-rays whose incident angle is below a certain angle (critical angle θ_c), and therefore, it is possible to not only apply those methods to synchrotron radiation facilities but also use them in combination with conventional X-ray tubes[6]. The critical angle θ_c is calculated approximately using Equation(3).

$$\theta_c = 2.04 \times 10^{-2} \sqrt{\rho} / E \text{ (rad)} \quad \cdots\cdots\cdots \quad (3)$$

ρ : density (g/cm^3)

E : energy (keV, $E = 12.4/\lambda$)

λ : wavelength (Å)

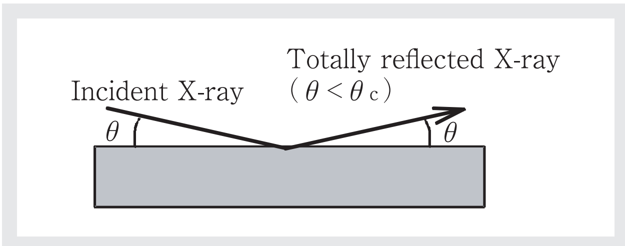

Figure 3 Total reflection

Development of X-ray Guide Tube

Background

The X-ray guide tube is a system that lets X-rays enter a tube that has an extremely smooth internal surface and causes total reflection inside to lead the X-rays to the output end[7][8]. Use of the phenomenon of total reflection enhances the effective solid angle of X-rays compared with the conventional collimator method, thereby it can reinforce the X-ray intensity. Note that the efficiency of total reflection is dominated by the surface roughness and that glass is generally used as it allows smoothing of the internal surface.

HORIBA developed a scanning X-ray microscope capable of analyzing samples at a spatial resolution of 10 µm as a research project for Research Development Corp. of Japan[9]-[11]. This microscope was chiefly applied to R&D,

but HORIBA felt the need to answer a strong market demand for shorter measurement time. Therefore, the company worked on development of an X-ray guide tube to realize high-intensity micro X-ray beam.

Polycapillary and Monocapillary

A polycapillary is straight type monocapillaries bundled and formed into a focusing shape (Figure 4)[12].
Although depending on the number of monocapillaries to bundle, aperture and shape, the polycapillary can improve luminance by two digits compared with the straight type monocapillary. But X-rays emitted from each capillary are spread, and it is difficult to achieve a spatial resolution of 10 µm. In addition, because of a wide spread of the beam, once the X-ray is off the focal point, the spatial resolution suddenly drops (Figure 5). Thus, we aimed to produce beams that have high luminance and are less spread by improving the efficiency of the monocapillary.

Improvement of Monocapillary

The monocapillary used in the conventional devices is a rotational paraboloid in shape. It was designed to focus X-rays of parallel components but, since the X-ray tube serving as the source of X-ray is a divergent light source, its focusing efficiency is restrictive and the majority of them used multireflection X-rays as with the straight type. The reality of total reflection is that the intensity decreases as the light is reflected, and that means the less reflections, the more efficiency. And making a divergent light source to focus light improves focusing efficiency. So we shaped the monocapillary into a spheroid that can focus light from the divergent light source just with a single reflection, and the microfocus was chosen for an X-ray source (Figure 6). We also determined the best shape (long axis, short axis, distance between focuses) that matches the equipment installed onboard based on the simulation using the light tracking method.

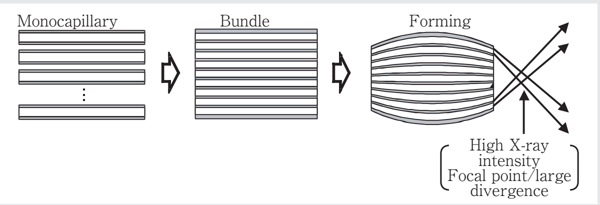

Figure 4 Polycapillary

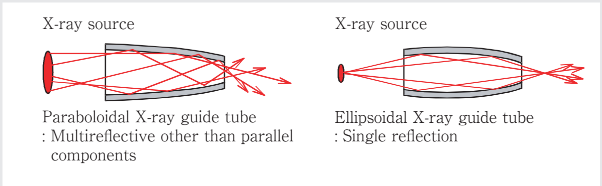

Figure 6 Improvement of monocapillary

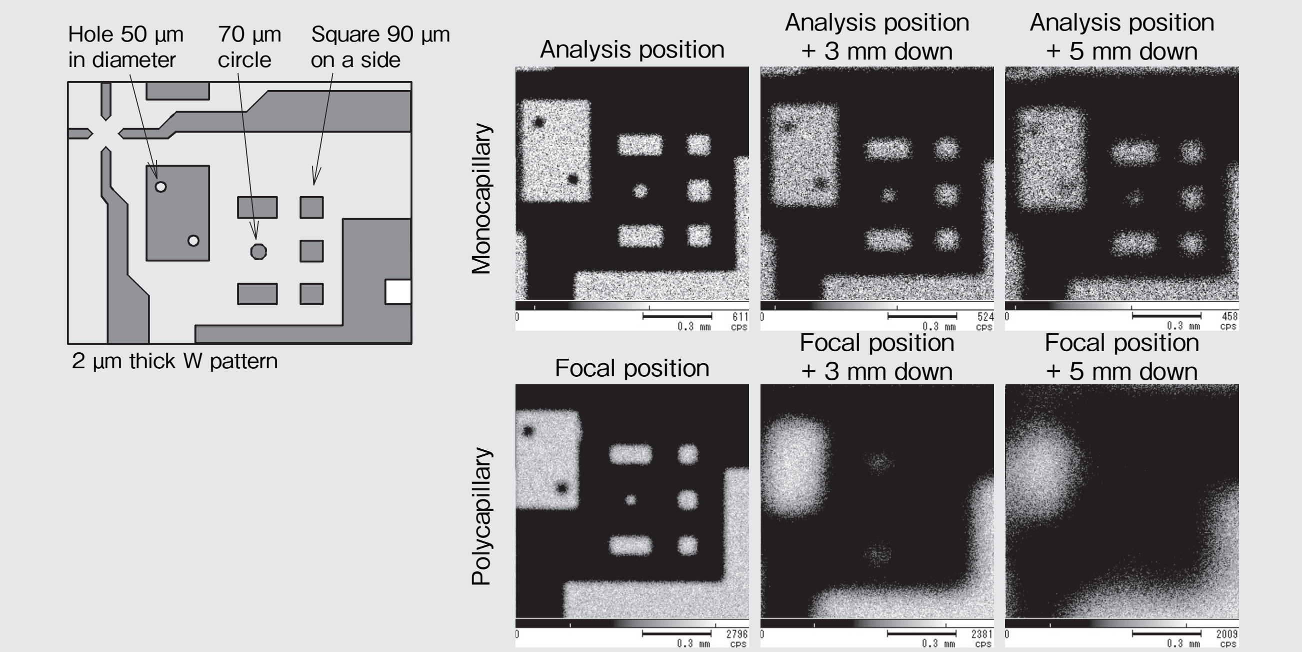

Figure 5 Comparison of beam divergence between monocapillary and polycapillary
Comparison made using the fluorescent X-ray mapping image of 2 µm thick tungsten (W) pattern on the sapphire substrate (measured by changing the distance from the standard measuring position)

Production of Spheroidal Monocapillary

With conventional kinds of multireflective monocapillary or polycapillary, incident X-rays are finally emitted out of the output end by repeated reflections despite some bend. But for a single-reflective spheroidal capillary, X-rays reflected on the inner surface of the capillary ought to be emitted directly out of the output end. Therefore, a remarkably higher level of precision than for a conventional forming machine is required for production of a spheroidal capillary. For a calculation example, a vertical section is set against the longitudinal direction of the capillary, the deviations from the target shape in two direction normal to each other on that sectional plane are measured, and the square root of the sum of squares of the measured values is added up every 1 mm along the total length of the capillary. The relationship between this value ("differential sum") and the output X-ray intensity is shown in Figure 7.

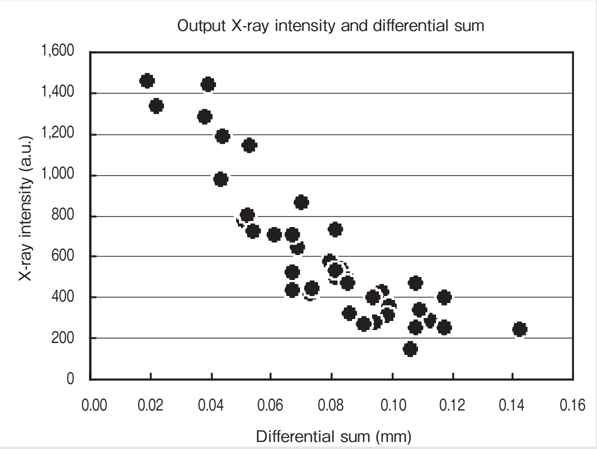

Figure 7 Output X-ray intensity and differential sum

Effect of Monocapillary Improvement

In addition to monocapillary improvement, we developed XGT-5000, an X-ray analytical microscope with its X-ray optical system optimized to match the improved monocapillary. It successfully improved the X-ray intensity by 20 to 50 times that of any conventional device at a spatial resolution of 10 µm (Figure 8). With those improvements, the product can greatly reduce measuring time and have attractiveness to new users such as quality control divisions of corporations in addition to universities and research institutes.

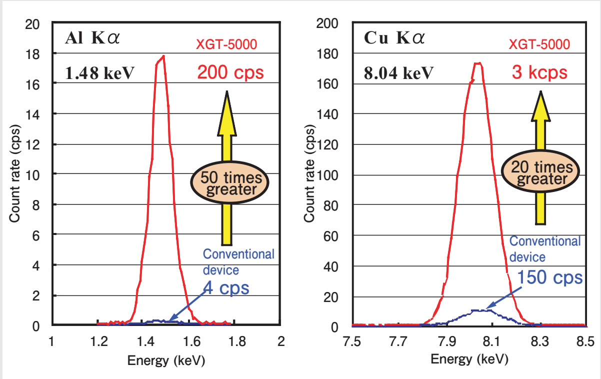

Figure 8 Comparison of output X-ray intensity

Conclusion

Development of an X-ray guide tube realized considerable improvement of the X-ray intensity of the X-ray analytical microscope. This success gave us the capability to analyze elements in microscopic parts of a sample without difficulty and greatly increase the number of subjects to analyze. The demand for high resolution performance and rapid analytical performance is more and more growing as a result of ongoing technical improvements including enhancement of material functions, miniaturization of electronic and electric parts, and advancement in nanotechnology and biotechnology. We intend to realize comprehensive development of X-ray focusing devices, X-ray sources, stages and detectors in order to promote enhancement of luminance of X-ray guide tubes and the miniaturization of their focal spot sizes.

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