

# X-Ray and Neutron Optical Systems

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**Abstract.** Although X-rays and neutrons can provide different information about samples, there are many similarities in the ways in which beams of them can be manipulated. The rationale behind bringing experts in the two fields together was the desire to find common solutions to common problems. The intention of this brief introduction is to give a flavour of the state-of-the-art in X-ray and neutron optics as well as an indication of future trends.

## 1.1 X-Ray Optics

There is a growing need for the determination and characterization of elements at trace concentrations that can be well below one part per million by weight. This is true in many fields of human activity, including the environmental sciences and cultural heritage as well as the more obvious physical and biological sciences. Although for quantitative as well as qualitative investigations, X-ray microanalysis is an established method for determining elemental composition, this is now often insufficient, a distribution map of each element being much more useful. However, this can be achieved only with *large flux*, *optimal excitation energy*, and *high lateral resolution*. For these to be satisfied appropriate optical elements must be developed to transport radiation from source to sample, providing powerful, highly concentrated and possibly monochromatic X-ray beams. As a result *X-ray optics* has grown rapidly in recent years as an important branch of physics and technology.

The phrase “X-ray optics” encompasses a wide range of optical elements exploiting reflection, diffraction, and refraction – or combinations of these – utilizing sub-micrometer and sub-nanometer artificial structures and natural crystals to focus, monochromate or otherwise manipulate X-ray beams. Historically, natural crystals can be regarded as prototypes of many of the artificial structures now in use or proposed. The development of multilayer interference mirrors for the nanometer wavelength range which provides efficient reflection at angles close to normal incidence was a great step forward.

These mirrors can be stable in ultrahigh power X-ray beams and can be also used for broadband high-flux monochromators providing energy resolutions  $E/\Delta E \sim 25$  in the range  $\sim 100 \text{ eV} - 15 \text{ keV}$ . Developments in the design and manufacture of multilayers have also allowed their capabilities as broadband polarizers and analyzers to be explored. In addition, recent technological progress now allows the manufacture of highly aspheric multilayer-coated optics. These preserve the efficiency, flux, and ease of alignment of spherical or partially aspheric mirrors, while eliminating spherical aberration, thus allowing high collection and convergence angles and small spot sizes.

A compromise between the reflected flux and the necessary energy resolution can be achieved by the choice of a suitable crystal or multilayer monochromator. Low-resolution monochromators can be also built from diffractive elements such as transmission and reflection zone plates. Zone plates as focusing elements and X-ray waveguides to relay sources of nanometer size are recognized as significant optical elements in the nano-world. The great majority of X-ray microscopes and microprobes currently use zone plates, and this has allowed such devices to become available as laboratory instruments and at synchrotron radiation facilities. However, during the last decade conventional zone plate technology has reached the theoretical limit of spatial resolution, with volume diffraction effects in the outer zones (with sizes comparable to X-ray wavelengths) providing the fundamental limitation of zone plate resolution.

Further development of micro and nanofabrication techniques, in particular for planar nanometer-scale structures with sizes of the order of X-ray wavelengths, as well as the deposition and growth of thin films of different materials, has enabled the manufacture of a new generation of diffractive optical elements. In a similar fashion, the fabrication and successful tests of a synthesized X-ray hologram on a crystal have been reported. With such improvements in nanotechnology, mostly for microelectronic applications, methods have been developed to create nanostructures and multilayer films for the effective control of X-rays to provide sub-micrometer spatial resolutions. These include two- and three-dimensional Fresnel and Bragg-Fresnel optical elements based on *zone plates*, with lateral resolutions as good as 15 nm, and diffraction gratings in combination with natural crystals or artificial multilayer structures. The recent development of graded crystals allows simultaneous focusing and enhancement of the spectral flux at the sample by several orders of magnitude. All these optical elements are related via the basic principles of Bragg, Bragg-Laue, or Bragg-Fresnel diffraction on artificially made volume structures and differ from other types of optic through combinations of optical properties. *Refractive/diffractive X-ray optics* were first realized in 1986 and have successfully been used with third-generation synchrotron radiation sources, as they are ideal for high-energy undulator radiation characterized by low divergence in both the vertical and horizontal directions.

*Capillary X-ray optics*, including microchannel plates, have been successfully used with conventional X-ray sources. Straight glass monocapillaries are efficient in transporting X-rays from the source leading to increased radiation intensity on the sample. Tapered monocapillaries are used in synchrotron beamlines for focusing radiation into micrometer and submicrometer spots. Polycapillary arrays with curved channels can be used for transforming divergent radiation from a point source into a quasiparallel beam or for focusing a divergent beam onto a small spot. Straight polycapillary arrays have been used for X-ray imaging and for beam splitting and filtering. Recent developments have been in making arrays with different geometries to enhance the performances of such optics. Also, of late, elliptically bent *Kirkpatrick-Baez mirrors* have been used to produce submicrometer size X-ray beams. These optics are achromatic and have relatively long focal distances compared to capillaries. This property can be very important for microfluorescence applications in special environments, for example when the sample needs to be contained in a gas-filled temperature-controlled chamber. *Refractive X-ray optics* represent a rapidly emerging option for focusing high energy synchrotron radiation from micrometer to nanometer dimensions. These devices are simple to align, offer a good working distance between the optics and the sample, and are expected to become standard elements in synchrotron beamline instrumentation in general and in high energy X-ray microscopy in particular.

## 1.2 Metrology

Most synchrotron radiation facilities and large industrial companies have developed their own metrology laboratories to meet the needs of optical characterization in terms of microroughness, radius of curvature, slope errors, and shape errors. The instrumentation used consists mainly of commercial instruments: phase shift interferometers for microroughness characterization, Fizeau interferometers for bidimensional topography, and optical profilometers – for measurements of long optical components – such as the long trace profiler (LTP) or the nanometer optical component measuring machine (NOM). In this book an attempt is made to systemize recent knowledge in ultraprecise surface metrology. This is directly linked to instrument calibration, but up to now there is no standardization of calibration. In round-robin endeavor, typical X-ray mirrors – plane, spherical or toroidal – were examined by the various laboratories using their own instrumentation in order to better understand the accuracy achievable. The ultimate goal of this Round Robin was to create a database of the measurement results in order to provide these references as calibration tools available to the metrology community.

### 1.3 Neutron Optics

Because of their unique properties, neutrons are used to investigate a growing number of research areas, in both traditional and new fields and from fundamental science to technology; no end to this growth can be foreseen. Currently 4,000–5,000 European researchers are using neutron scattering for their scientific work. This demand drives the improvement of neutron instrumentation which, to a large extent, is related to neutron optics. During the last decade the main advancement has been the introduction of supermirrors (with lateral or transverse grading of the layer thicknesses, either quasiperiodically or aperiodically) for neutron transport in guides, while the next decade will see the increased application of focusing and polarizing devices which will also be based mainly on supermirror coatings. It is in this field of multilayers where there is much similarity between neutron and X-ray optics. Focusing systems for neutrons also have much in common with their X-ray equivalents; in particular, focusing tests using capillary optics and Fresnel zone plates have been performed with neutrons.

An important property of a supermirror is its critical angle,  $\theta_c$ , the glancing angle up to which it reflects efficiently. By convention  $\theta_c$  is measured in multiples,  $m$ , of the critical angle of nickel, which has the largest critical angle of all naturally occurring elements.

For multilayer production an important advance has been the reduction of stress development during the growth of the film coatings; this is important for the production of X-ray multilayers as well as those designed as neutron reflectors. By varying several parameters during the sputtering process, their influences on the stress development have been determined, leading to an order of magnitude decrease in the stress. Another important step has been progress in the production of polarizing and nonpolarizing supermirrors. For polarizing supermirrors the critical angle up to which they reflect neutrons has been increased and the magnetic field necessary to retain good polarization has been lowered. For nonpolarizing supermirrors the critical angle has also been increased, while the temperature and radiation stability as well as the corresponding crystal structures were characterized and the homogeneity increased for the coating of large areas. Phase space mapping of a neutron beam following neutron optical devices containing supermirrors has also been demonstrated.

Research on bent perfect crystals has been aimed at the development of the technique and demonstrating the properties of systems based on one or two components. One component enables ultrahigh resolution for monochromatization or analysis, while two components allow for an adjustable spectral resolution and collimation of  $\sim 10^{-3}$ – $10^{-4}$ . Such systems have allowed the realization and test of a multianalyzer module for a three-axis spectrometer consisting of an array of 31 individual channels, covering a scattering

angle range of  $75^\circ$ . This new device offers improved momentum resolution and enhanced data collection efficiency in experiments aimed at mapping of inelastic response over extended areas in momentum/frequency space and, at the same time, keeps the high incident flux and most of the flexibility of up-to-date triple axis spectrometry using doubly focusing crystal optics.