One hundred years ago, Wilhelm Conrad Röntgen discovered the x-ray. Over the subsequent three decades, researchers investigated the interaction of x-rays with matter, studying diffraction, refraction, and small-angle scattering effects as well as those of absorption and penetration. Development of practical and useful x-ray optical components based on these effects followed slowly, however; indeed, it is only within the last two decades that these phenomena have been exploited to produce a broad variety of x-ray optics and applications.

Bragg diffraction of x-rays from single crystals provided early demonstration of the wave properties of x-rays and remains the major tool for study of the microscopic structure of crystalline materials. Singly curved crystals for focusing x-rays were developed in the 1930s; in particular, they've been widely used in the past 30 years to focus and redirect x-ray beams from high-intensity synchrotron sources. In the 1950s, doubly curved crystal fabrication progressed to include crystals featuring different surface curvatures along orthogonal axes parallel to the surface. Researchers demonstrated the use of these doubly curved crystals (DCC) with standard laboratory x-ray sources to focus x-rays for analytical applications.

Curved crystals in theory
DCC optics are based on the Bragg law \( n\lambda = 2d \sin \theta_B \), where \( \lambda \) is the wavelength of the diffracted beam, \( n \) is the diffractive order from crystal planes of spacing \( d \), and \( \theta_B \) is the angle between the incident beam and the Bragg planes. For x-rays close to the Bragg angle \( \theta_B \), the efficiency for diffraction from the crystal can be nearly unity (with small effects from polarization, absorption, and crystal quality). The range of angles over which the diffraction yield remains high depends on the x-ray energy (or wavelength), the crystal quality, unit cell volume, and the particular planes involved in the diffraction. For highly perfect crystals, the angular...
range and associated wavelength range can be very small. The diffraction process thus not only redirects an x-ray beam but also acts as an efficient energy filter (monochromator).

DCCs can be formed of various crystal materials, including silicon, germanium, quartz, graphite, and mica. After production of a thin, flat crystal using standard growth, cutting, and polishing techniques, the crystals are elastically curved in two orthogonal directions and held in position. Curving the crystal allows it to meet the Bragg condition for a range of incident angles, for example, for x-rays emitted from a divergent (point) source. The most common geometry for curved crystal diffraction is based on the Rowland circle principle, which relies on the well-known property of a circle that an arc segment subtends a constant angle for any point on the circle. Applying this geometry for Bragg diffraction requires a crystal with a radius of curvature twice the radius of the Rowland circle. This means that crystal planes parallel to the surface of the curved crystal will be in contact with the circle over only a restricted but acceptable angular range (the familiar Johan geometry). For the case of crystal planes parallel to the crystal surface, the diffraction is symmetric such that the diffracted rays come to a focus on the opposite side of the Rowland circle from the divergent x-ray source (see figure 1).

Curving the crystal in a plane perpendicular to the Rowland circle provides an ability to capture a large angular range and focus the x-rays onto a small spot. In contrast, a singly curved crystal would focus onto a line rather than a spot. A toroidally bent crystal with the bending radius in the vertical direction $r_v=2R\sin^2\theta_B$, where $R$ is the radius of the Rowland circle, provides point-to-point focusing with the source spot $S$ imaged at the image point $I$ on the circle. Although the geometry shown in figure 1 is symmetrical, it is possible to place the crystal anywhere on the circle, which can effectively magnify or demagnify the source spot. In these cases, the crystal surface must be cut at an angle to the diffracting planes. Other curvature geometries such as elliptical, logarithmic parabolic, and log spiral can be used to accommodate different source or image geometries, though we’ll restrict this discussion to the symmetrical toroidal geometry.

DCC optics can produce an intense, highly monochromatic, focused x-ray beam with focal distances typically 75 to 200 mm in a small spot (50 to 150 $\mu$m) size. The capture angle $\theta$ in the dispersive plane (parallel to the Rowland circle) and the included rotational angle $\psi$ (perpendicular to the circle) determine the collection solid angle of a DCC optic. The capture angle in the dispersive plane typically varies from 1 to 5° but the included rotational angle can be large (10 to 30°) because of the rotational symmetry around the S-I axis in Figure 1. As a result of the large collection angle and the small spot size, the intensity of a monochromatic beam from a low-power (20 to 50 W) x-ray source can be comparable to that of a high-power (5 to 10 kW) rotating anode source with a conventional monochromator. Depending on the x-ray source used, DCC optics can produce focused beam spots as small as 20 $\mu$m, though spots of 50 to 150 $\mu$m are more typical.

**Curved Crystals in Action**

X-ray fluorescence, in which an x-ray or electron-beam source triggers x-ray emission from a sample, has become an important tool for non-destructive analysis. Monochromatic x-ray microbeams provide several advantages over conventional micro-x-ray fluorescence (MXRF) techniques, which use electron beam or polychromatic x-ray excitation. Electron excitation, which requires a vacuum environment, has a detection limit of parts per thousand, which is limited by a bremsstrahlung radiation background arising from slowing of electrons in the sample. Polychromatic x-ray excitation suffers from a detection limit of parts per million, constrained by a background of scattered x-rays. In contrast, monochromatic excitation eliminates the x-ray scattering background under the fluorescence peaks and therefore gives a much improved measurement sensitivity. A monochromatic MXRF system with a low-power x-ray source and 600-s data acquisition times can detect bulk contaminants at...
levels of parts per billion, or surface concentrations of medium- to high-Z elements at femtogram levels.

A comparison of spectra measured by an MXRF system fitted with a pinhole collimator and a monochromatic MXRF system demonstrates the enhanced sensitivity of the monochromatic system (see figure 2 on page 15). The pinhole, which defined the spot size of the x-ray beam on the sample, was the same size as the DCC focal spot (50 µm) and was located the same distance from the source as the curved crystal (120 mm). A 25-mm² semiconductor diode detector positioned 15 mm from the sample captured the signals.

If one or a few impurity peaks are of primary interest, the use of one or more DCC optics between the sample and the detector can provide greater element specificity, reduced background, and higher sensitivity. The monochromatic MXRF configuration also allows use of less expensive non-energy-dispersive detectors. A sensor for on-line measurement of sulfur in diesel fuel and gasoline has recently been developed using such a configuration.4

Total-reflection x-ray fluorescence (TXRF) provides high-sensitivity trace analysis of particles, residues, and impurities on smooth surfaces. In TXRF, an x-ray beam intersects the surface under test at grazing incidence to achieve total external reflection. Surface contaminants, if present, interact with the x-ray beam to generate characteristic x-ray fluorescence lines. Semiconductor manufacturers use the technique to monitor
wafer surface contamination during chip fabrication, although
the large beam size of conventional TXRF systems does not
permit the acquisition of localized data.

By leveraging the focusing capabilities of DCC optics, engi-
neers can perform localized TXRF (see figure 3). To minimize
fluorescence from the sample and meet the total external re-
fection condition, the system features a slit that restricts beam
divergence in the scattering plane to less than the critical angle
for substrate reflectance. The flux density at the reflection sur-
face for a DCC-based system is several orders of magnitude
greater than that of conventional systems with higher power
sources. This increase yields a very high sensitivity for local con-
taminant detection as well as portability that enables in situ,
online process control.

X-ray reflectometry (XRR) offers a useful tool for characteriz-
ing thin film thickness, uniformity, surface and interface
roughness, and density. The technique has increased in impor-
tance as the thickness of applied films has fallen below the
capabilities of traditional optical ellipsometers. This application
involves measuring the reflectivity of monochromatic x-rays as a
function of the incident angle. Conventional XRR measurements
require highly collimated beams, with a reflectivity curve cap-
tured by sequentially scanning the incidence angle. A DCC optic
can provide a small focal spot and a range of incidence angles
that yield uniform illumination. Coupled with a position-sensi-
tive detector, the system can obtain an entire reflectivity curve
without the need for moving parts.

Combinations of compact x-ray sources and doubly curved
crystals can help generate high-intensity, monochromatic x-ray
beams with uniform and controlled convergence, making possi-le for the first time new portable, remote, in situ, highly
sensitive measurements of composition, contamination, and
thin-film structure. Only recently, however, have techniques
been developed that permit precise and reproducible figure
control for doubly curved crystals with a variety of diffracting
crystals, and short enough focal lengths to be practical for stan-
dard laboratory use. As development moves forward, new
applications and commercial systems will emerge.

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