IEEE Photonics Journal
An IEEE Photonics Society Publication

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Invited Paper

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Volume 6, Number 2, April 2014

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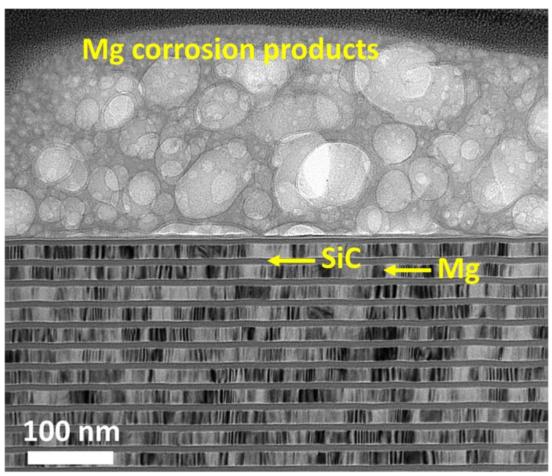

DOI: 10.1109/JPHOT.2014.2309640
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Breakthroughs in Photonics 2013: X-Ray Optics

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(Invited Paper)

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DOI: 10.1109/JPHOT.2014.2309640

Manuscript received February 11, 2014; accepted February 25, 2014. Date of current version April 30, 2014. This work was supported by the U.S. Department of Energy through the Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. Corresponding author: R. Soufli (e-mail: regina.soufli@llnl.gov).

Abstract: This review discusses the latest advances in extreme ultraviolet/X-ray optics development, which are motivated by the availability and demands of new X-ray sources and scientific and industrial applications. Among the breakthroughs highlighted are the following: i) fabrication, metrology, and mounting technologies for large-area optical substrates with improved figure, roughness, and focusing properties; ii) multilayer coatings with especially optimized layer properties, achieving improved reflectance, stability, and out-of-band suppression; and iii) nanodiffractive optics with improved efficiency and resolution.

Index Terms: X-ray optics, multilayer interference coatings, metrology, EUV, X-ray applications.

1. Introduction

Several exciting developments have fueled rapid advancements in the field of X-ray optics in the last few years. The emergence of novel X-ray sources [1]–[3] with unprecedented brightness and coherence (4th generation synchrotrons, free-electron lasers, tabletop lasers, high-harmonic generation, and attosecond sources) has ushered a new era in the fields of materials science, chemistry, plasma physics, biology, and life sciences and has imposed completely new demands for the performance of X-ray optics. There are also continuing needs for increasingly efficient and sophisticated X-ray optics for space-borne telescopes for solar physics and astrophysics, radiation detection and medical imaging applications, high-energy physics experiments, and technologies for semiconductor photolithography. In their interactions with matter, X-rays possess unique attributes that differentiate them from light in other portions of the spectrum [4], [5]. The refractive index of materials is less than 1 at X-ray wavelengths, leading to the phenomenon of "total external reflection," where the reflectivity of a single material surface approaches 1 at very shallow grazing incidence angles and rapidly falls towards zero at steeper grazing angles. Significant portions of the X-ray spectrum coincide with electronic absorption edges, causing materials to become highly absorbing upon illumination at certain X-ray wavelengths. In order to correctly design and model the performance of X-ray optical elements, accurate and comprehensive knowledge of the refractive index of materials in the soft X-ray spectrum is essential, although it remains elusive, due to challenges in its experimental determination. Because of the laws of diffraction and scattering scale according to the wavelength of light, X-ray optics are extremely sensitive to loss of efficiency, resolution, and wavefront coherence due to micro-roughness and figure errors on their surface. Depending on environmental conditions, X-ray optics can experience severe degradation in their performance due to contamination, corrosion, and damage effects. The above attributes often make the design and fabrication of efficient X-ray optical elements extremely challenging. For these reasons, when new experimental facilities, space instruments, and industrial technologies are first designed and proposed, one often comes to the realization that the X-ray optics needed to field these applications are beyond the state of the art. This ongoing quest for X-ray optics that "do not yet exist" has acted as a driving force constantly pushing the feasibility limits in X-ray optical technologies. This review presents a selection of highlights on recent progress towards innovative X-ray optics development from research groups around the world. The topics covered include optical substrates, multilayer mirrors and gratings, and nano-diffractive optics. The photon energy range of operation of these elements encompasses the region from a few eV (extreme ultraviolet, EUV) to several hundreds keV (soft gamma-ray). Precision metrologies are an essential part of the development and fabrication of X-ray optics and have been included in the material presented below and in the References. A report on the status, motivation, challenges, and future needs for X-ray optics for basic energy sciences facilities (including the topics of refractive lenses and crystal monochromators/ analyzers that are not covered in this review) can be found in [6].

2. Optical Substrates

Optical substrates, employed alone or combined with thin film coatings to be used as mirrors or gratings, are essential elements in many X-ray optical systems. Commonly used substrate materials range from single-crystal silicon for synchrotron and laser sources to ultra-low thermal expansion glasses for solar physics, astrophysics, and photolithography applications. Silicon carbide and other composite lightweight materials, as well as thermally formed glass segments, have emerged in recent years as viable substrates for mirrors in space borne solar physics and astrophysics telescopes [7]–[9].The applications discussed in Section 1 are imposing truly daunting specifications for the figure and roughness of X-ray optical substrates and often drive the selection process for the materials and fabrication processes involved. For example, photolithography at EUV/soft X-ray wavelengths requires sub-diffraction limited optical systems employing aspherical mirrors with diameters reaching hundreds of mm, to achieve efficient, high-resolution, and low-aberration imaging. The first micro-exposure systems for photolithography at 13.5 nm wavelengths with numerical aperture $(\mathrm{NA}) = 0.5$ (vs. the earlier $\mathrm{NA} = 0.25$) have been underway during the last year [10], involving fabrication of large-area mirror substrates made of ULE™ material with aspheric departures in the 50 μm range and figure/roughness specifications in the 0.1 nm rms range. Another example is the new synchrotron and free-electron laser facilities, whose photon energy range and operating distances from the X-ray source require steering and focusing mirrors in the meter-long range with figure/slope error specifications on the order of 1 nm rms / 0.1 μrad rms and micro-roughness around 0.25 nm rms, as required to preserve the X-ray source properties (coherence and brightness) and to achieve the desired focusing [11]. Such specifications, especially when applied on meter-long surfaces, are challenging the state-of-the–art in fabrication, mounting, and metrology technologies [12]–[15]. Novel polishing techniques (Electron Emission Machining) and metrologies have demonstrated figure and roughness errors on the order of 0.1–0.35 nm rms on 400 mm-long clear apertures [16]. Even more recently, a 450-mm long deformable X-ray mirror made of super-polished single-crystal silicon and equipped with actuators along the tangential axis has been demonstrated. With the use of high-precision visible-light metrology and control algorithms, the entire mirror surface was actuated and flattened to a 0.7 nm rms controllable figure error (compared to the initial figure error of 18 nm rms, after assembly). This result represents the first sub-nanometer active flattening on a substrate longer than 150 mm [17]. These developments are promising towards the realization of meter-class X-ray optics satisfying the aforementioned specifications. Experimental methods have been demonstrated for optimal tuning of mechanically bendable X-ray mirrors for diffraction-limited soft X-ray nano-focusing. Using wavefront-sensing tests, focal ray errors of $\sim$80 nm (peak-to-valley) were achieved on a horizontally deflecting synchrotron mirror, thus exceeding

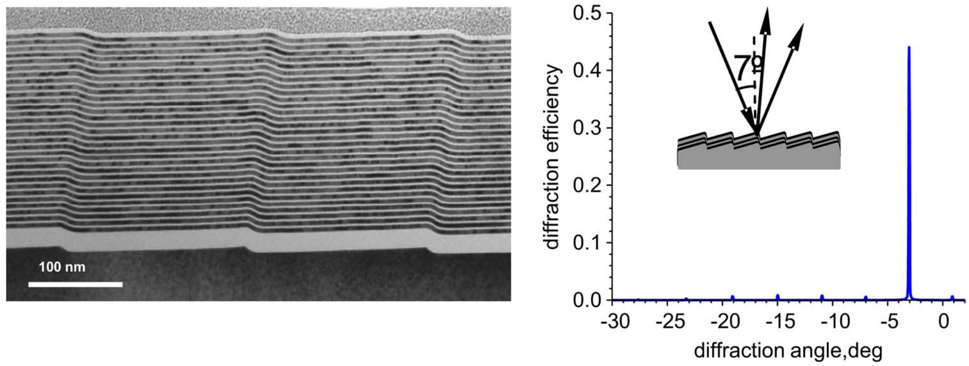

Fig. 1. From [20]. (Left) Cross-sectional Transmission Electron Microscopy (TEM) image of a Mo/Si coated blazed grating. (Right) Near-normal incidence diffraction from the grating at 13.1 nm wavelength. Courtesy of D. Voronov, Lawrence Berkeley National Laboratory.

the diffraction-limited focal size at wavelengths < 0.5 nm [18]. Techniques based on direct-write electron beam and optical lithography have also been developed for the production of anisotropically etched Si blazed grating substrates. These methods offer superior accuracy and flexibility, compared to the more traditional mechanical ruling and interference lithography techniques for grating fabrication [19].

3. Multilayer-Coated Mirrors and Gratings

Multilayer coatings, in periodic or aperiodic configurations, achieve high reflective performance due to constructive wave interference phenomena [4], [5]. They are often designed to operate at photon energies just below an electronic absorption edge of one of their constituent materials, where absorption is low, thus resulting in enhanced reflectivity. Multilayer mirrors enable operation at normal incidence angles at EUV/soft X-ray wavelengths. They also offer design flexibility and allow for efficient operation at grazing incidence angles larger than the critical angle, thus greatly easing the fabrication, mounting, and alignment of grazing-incidence X-ray optical systems. During the last couple years, several exciting advances have taken place within the field of reflective multilayer coatings at wavelengths ranging from the EUV to the soft gamma-ray regions of the spectrum. Multilayer-coated reflective gratings are essential elements in EUV/X-ray spectrometers; nevertheless, depositing a reflective multilayer film on a saw-tooth (blazed) grating substrate presents a dilemma: the deposition conditions that produce the smoothest layers (and thus the highest multilayer reflectivity) also cause smoothening of the saw-tooth pattern, resulting in reduced diffraction efficiency. By especially optimizing the aforementioned two competing mechanisms, Mo/Si-coated gratings with record efficiencies in the EUV and X-ray regions were recently demonstrated [20] (see Fig. 1). Motivated by semiconductor photolithography applications seeking to maximize reflective performance at 13.5 nm wavelength while suppressing unwanted reflections at 100–400 nm (which are simultaneously emitted by the light source), normal-incidence Mo/Si multilayers equipped with special anti-reflective coatings on top have been developed, and the performance of these coatings has been recently optimized using design rules guided by the thickness and optical constants of the constituent materials [21]. Moreover, due to next-generation lithographic technologies being proposed for operation at wavelengths > 6.6 nm (the Boron K absorption edge), the performance of La/B and La/B_4C-based multilayers has been significantly improved, with measured normal-incidence peak reflectivities now approaching 60% (compared to 40–50% reported just a couple years ago) [22]–[24]. In the field of EUV solar physics instrumentation, bi-periodic structures consisting of tri-material (Al/Mo/B_4C or Al/Mo/SiC) multilayer coatings with enhanced reflectivity have been developed and implemented to achieve high reflectivities in two bands (17.4 and 30.4 nm) simultaneously. The peak reflectivity of over 56% is the highest reported to date at 17.4 nm wavelength [25]. The performance of Al/Zr multilayers operating at wavelengths > 17.1 nm (the Al $L_{2,3}$ edge) has been enhanced by the introduction of Si layers, aiming to interrupt the

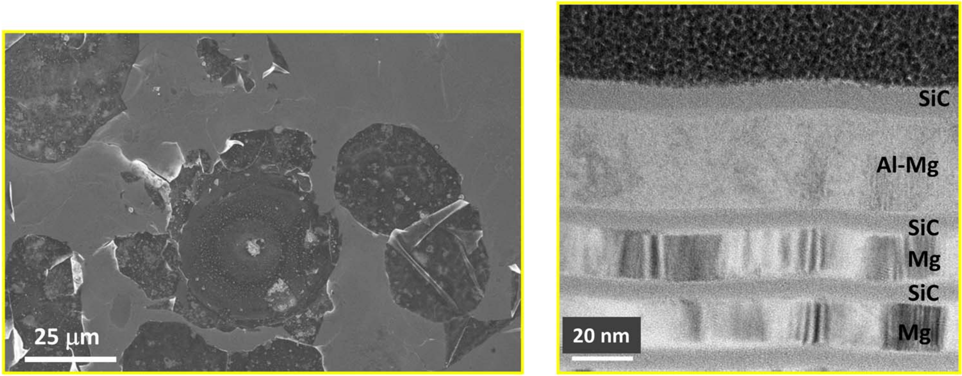

Fig. 2. From [27]. (Left) Scanning Electron Microscopy image obtained on the top surface of a standard Mg/SiC multilayer coating with advanced atmospheric corrosion. Portions from the top few layers of the film are shown to be delaminating, or are missing entirely. (Right) Cross-sectional TEM image of the top layers of a corrosion-resistant Mg/SiC multilayer. The corrosion barrier consists of a spontaneously intermixed, partially amorphous Al-Mg layer deposited underneath the top SiC layer. Crystalline Mg and amorphous SiC layers are also shown. The multilayer period thickness is designed for operation at wavelengths around 46 nm at near-normal incidence angles.

Al layer crystallization and to reduce the roughness at the interfaces between the Al and Zr layers [26]. In the 25-80 nm wavelength range, efficient Mg/SiC multilayers with corrosion barriers consisting of spontaneously intermixed Al-Mg layers have been demonstrated [27], [28] (see Fig. 2). This development addresses the long-standing issue of Mg-related corrosion that had been preventing the use of Mg/SiC (the material pair with the highest reflective performance in this wavelength region) in applications requiring mirrors with good lifetime stability. In the field of high-harmonic generation/attosecond sources, where aperiodic multilayer elements have been developed for temporal pulse shaping, it is crucial to have experimental knowledge of both the amplitude and phase reflected by the multilayer, as a function of wavelength. Direct determination of the phase remains a difficult problem due to limitations imposed by the wavelength and relevant length scales within the multilayer, involved in this type of measurement [29]–[33]. Multilayer reflectivity has been measured at the highest reported photon energies (384, 511, and 642 keV), and the effects of incoherent Compton scattering were quantified for the first time. WC/SiC multilayers with ultra-short-periods (1.5 nm) deposited on flat super-polished glass substrates were used for this purpose and achieved 52.6% peak reflectivity at 384 keV at a grazing incidence angle of 0.063 deg. These results foreshadow the use of multilayer reflective optics in soft-gamma-ray applications such as nuclear and medical physics and astrophysics [34], [35].

4. Diffractive Elements

Fresnel zone plates act as nano-diffractive elements in X-ray microscopes with current spatial resolutions in the 15–20 nm range (soft X-ray) and 50–70 nm (hard X-ray). The availability of new X-ray sources with unprecedented brightness, coherence, and temporal resolution opens up new opportunities for fundamental studies of materials and structures at the nm-scale level [6]. Pushing the limits of resolution (which is mainly determined by the width of the thinnest, outermost zone) and efficiency and doing so using methods that are practical, accessible, and relatively low-cost are the main challenges in the development of next-generation zone plates. Using electron-beam lithography for zone plate fabrication, soft X-ray imaging with 10 nm spatial resolution has been demonstrated [36]. Alternative zone plate nanofabrication methods such as ion-beam lithography [37] have also been demonstrated. Another promising method for fabricating high-resolution high-efficiency zone plates for EUV to hard X-ray energies is vertical directionality-controlled metal-assisted chemical etching, which is capable of producing large-area optics with ultra-high aspect ratio features in Si and a variety of other materials [38]. A fundamentally different method for zone plate fabrication (also favorable for high-aspect-ratio structures for hard X-ray nano-focusing) consists of deposition of "thick" aperiodic multilayer structures, with the thickness of each layer coinciding with the required Fresnel zone-plate width. A slice of the multilayer is removed using techniques such as focused ion beam or mechanical thinning and polishing and used in transmission mode as a zone plate "Laue lens." Using these structures, parallel measurements of fluorescence, absorption, and phase with spatial resolution better than 50 nm were achieved at 12 and 19.5 keV photon energies [39]. Even more recently, an 11-nm focal spot size has been measured with 15% efficiency at 12 keV from a multilayer Laue lens with 43.4 μm aperture [40].

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