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Casting Technique for the Fabrication of Pinholes for X-ray Radiation

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Pinholes with diameters down to 4 µm have been made in gold plates of a few hundred µm thickness. The fabrication method used is based on a casting technique. The pinholes are well suited for the collimation of (hard) X-rays from a synchrotron radiation source.

Keywords: pinholes; coherent X-rays; speckle patterns; Fraunhofer diffraction.

1. Introduction

In recent years, micrometre-sized pinholes have been used as diaphragms for the selection of transverse coherent X-ray beams from high-brilliance synchrotron radiation sources (Sutton *et al.*, 1991; Grübel *et al.*, 1994). Such a coherent beam, when scattered from an inhomogeneous sample, may generate static or fluctuating speckle patterns. Observations of time-dependent correlations in X-ray speckle patterns allow for the probing of dynamical phenomena in samples on length scales inaccessible to visible light (Brauer *et al.*, 1995; Dierker, Pindak, Fleming, Robinson & Berman, 1995). Given the growing importance of X-ray photon correlation spectroscopy (XPCS) at third-generation synchrotron radiation sources, there is increasing demand for micrometre-sized beam diaphragms.

For the collimation of visible light and for electron microscopy, micrometre-sized pinholes are commercially available. These holes are either drilled mechanically in the form of a tapered aperture (product from Pelco, Redding, CA, USA) or are made by high-precision laser drilling of a thin metal foil (see *e.g.* the catalogue of Melles Griot, Irvine, CA, USA). For the collimation of X-rays of energy larger than ~ 8 keV, such diaphragms are often unsuitable; the thin material immediately surrounding the pinhole is semi-transparent and changes the phase and amplitude of the wavefront in an uncontrolled way. This results in transmission of a partially incoherent beam.

A diaphragm for X-rays should preferably have the form of a long straight channel in a strongly absorbing material. If we consider attenuation of a ~ 10 keV beam in the material by a factor of 10^{10} to be acceptable, then a plate of Au should be of at least 100 µm thickness. For a diaphragm of 4 µm diameter, this requirement leads to straight channels having a length-to-diameter (L/D) ratio of at least 25, which is difficult to achieve with conventional drilling techniques. In several studies this problem has been solved by stacking a number of laser-drilled pinholes, but aligning them is inconvenient. Recently, X-ray diaphragms with large L/D ratios and smooth interior walls have been fabricated using the LIGA lithography process (Lehr & Ehrfeld, 1994), but this technique is not readily available. Commercially available motorized slits are also of use, provided the slit blades are mechanically polished and carefully aligned (De Vries, Vlieg, Alvarez & Ferrer, 1996). Finally, one may intercept a coherent portion of the beam by pairs of reflecting mirrors under extreme grazing incidence (Ferrer *et al.*, 1995). The latter method is complex since it requires very careful alignment. In this communication we present a casting method for the fabrication of Au pinholes suitable for the collimation of hard X-rays up to at least 40 keV.

Irrespective of their method of production, diaphragms having a size smaller than the transverse coherence length of the beam should give a clear Fraunhofer diffraction pattern in the far field (Sutton *et al.*, 1991; Grübel *et al.*, 1994). These patterns serve as direct proof that the transmitted beam is coherent. In addition, possible irregularities in the pinhole shape show up as deviations of the measured Fraunhofer pattern from the theoretical pattern. We present diffraction patterns from our cast pinholes.

2. Casting pinholes

The fabrication method is simple. In brief, a stretched thin tungsten wire is immersed in a crucible containing molten gold. After solidification, a slice is cut from the Au ingot. The piece of W wire, which runs through the centre of the slice, is then selectively etched. This results in a long straight hole having the diameter of the W wire. Drawn W wires have been used with diameters ranging from 4 to 20 µm (Goodfellow, Cambridge, UK).

We now describe the casting process step by step with the aid of Fig. 1. Panel (1): the W wire is stretched over an alumina support tube (inner diameter 4.8 mm) using a pulley and a small weight. Notches in the tube fix the position of the wire. Panels (2) and (3): the tube with the stretched wire is immersed in an alumina crucible containing 0.3 cm^3 of molten gold at a temperature of 1373 K. Immersion of the tube into the crucible is carried out very slowly (*ca* 1 mm min^{-1}) so as to prevent breaking of the W wire on first contact with the Au melt. Panel (4): after solidification, the gold ingot, including the alumina support tube, is disengaged from the crucible. The support tube is cracked open and removed, while taking care that the W wire remains intact and clearly visible. Panel (5): the Au ingot is positioned on a goniometer head and the W ends at either side of the ingot are oriented with the aid of an optical microscope. Panel (6): the ingot is spark-cut in slices along planes perpendicular to the direction of the W wire to within a few tenths of a degree. The slices are then mechanically polished on both sides using 0.03 μm alumina grit. Panel (7): four indentations are made in the slice in order to mark the position of the hole. Panel (8): the W wire is removed by etching in an HNO_3/HF (1:1) mixture. To ensure complete dissolution of the W in the channel, the etching is performed in an ultrasonic bath for *ca* 48 h.

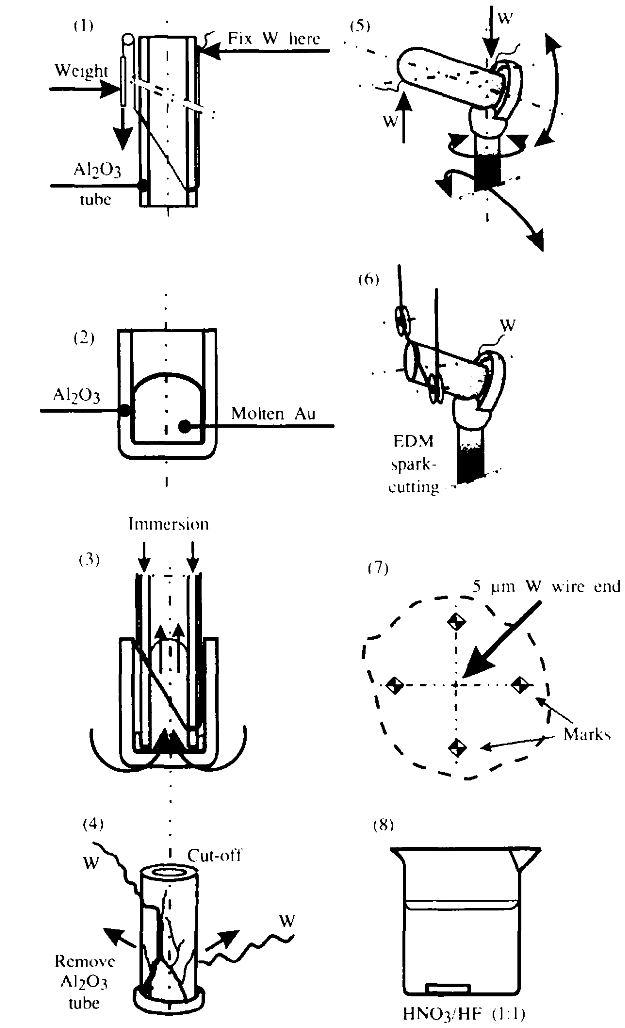

Figure 1
Pinhole manufacturing technique shown in steps (see text).

From a single ingot several slices can be obtained. Thick slices, of course, yield long channels, the limit being set by our ability to stretch the W wires perfectly straight over an extended length. We cut slices of up to 800 μm thickness, containing a W wire of 5 μm (L/D = 160). After etching, it was verified optically that the channel was straight. We note that a diaphragm of 800 μm thickness is suitable for photon energies up to 38 keV (using attenuation by a factor 10^{10} as a criterion). There should be no problem with the fabrication of pinholes of even larger thickness (a few mm). However, with increasing thickness their alignment with the beam direction becomes more difficult.

Using electron microscopy we found that the interior wall of the cast channel is a replica of the surface of the W wire, including any deviations from the ideal cylindrical shape. As is typical for wire drawn from a die, the surface is found to be somewhat corrugated, with the crests and valleys running parallel to the cylinder axis. The local deviations from the cylindrical surface are at maximum $\sim$0.2 μm.

3. Fraunhofer diffraction pattern

The pinholes were tested at the 'Troika' undulator beamline (ID10) of the European Synchrotron Radiation Facility, Grenoble. The photon beam was monochromatized with a single-bounce Si(220) crystal located at 45 m from the source. The monochromator was set to an energy of 9.1 keV (λ = 1.36 Å). An SiC grazing-incidence mirror eliminated higher harmonics from the beam. The pinholes, located at 46 m from the source, were mounted on a Eulerian cradle which allows for alignment of the pinhole channel with the incident beam direction. The integrated coherent flux through a 5 μm pinhole was $\sim 2 \times 10^7$ photons s^{-1}. Fraunhofer diffraction patterns from the pinholes were taken by measuring the scattered beam intensity through a second pinhole as a function of scattering angle in both horizontal and vertical planes. The analyzing pinhole, of 8.5 μm diameter, was positioned at a distance of 1.5 m from the scattering pinhole.

The measured diffraction pattern from a pinhole of 4 μm diameter and 300 μm thickness is shown in Fig. 2. One sees the maxima and minima characteristic for Fraunhofer diffraction of a plane wave from a circular hole. Superimposed on these maxima and minima are rapidly oscillating non-statistical variations in the intensity and a low structureless background. These variations and the background do *not* relate to diffraction from the pinhole but result from distortions in the primary wavefront and from partial incoherence caused by the optical elements along the beam path. The optical elements concerned are two Be windows and the SiC mirror. Recently, Snigirev, Snigireva, Kohn & Kuznetsov (1996) have identified the surface roughness of the Be window as a possible cause; the uneven surface of a non-polished Be window introduces interference structures in the beam, which may result in intensity changes of up to a factor of two and in reduced contrast in the diffraction pattern. Similar effects are expected to arise from an uneven mirror surface.

We have compared the measured Fraunhofer diffraction pattern with an Airy pattern (Born & Wolf, 1975) for a hole of 4 μm diameter, convolved with the resolution function of the analyzing pinhole (Fig. 2). We find good agreement between the measured envelope of the pattern and the theoretical one and note that incoherent background is the main cause for the difference between measured and calculated depths of the minima in the wings of the pattern.

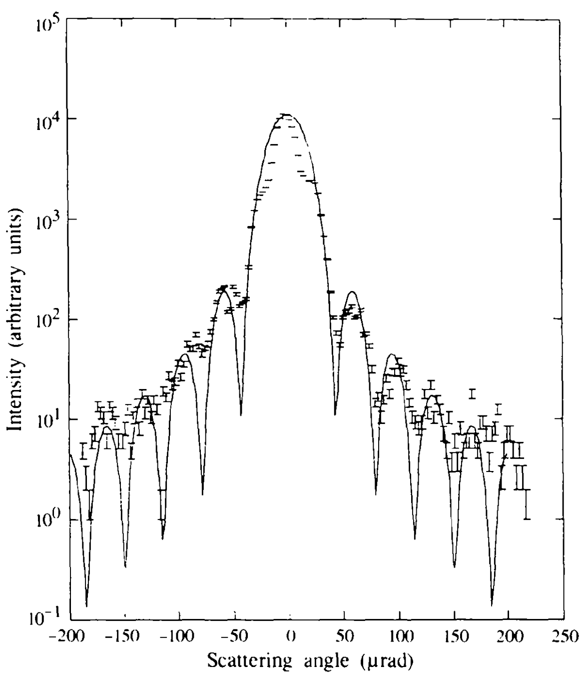

Figure 2
Fraunhofer diffraction pattern from a pinhole of 4 μm diameter and 300 μm thickness, measured at a wavelength λ = 1.36 Å. The solid curve represents the Airy function, convolved with the resolution function of the detecting pinhole.

4. Conclusions

For the fabrication of X-ray pinholes, the casting technique offers some advantages over other techniques. Firstly, thick diaphragms with very long straight channels can be fabricated, making them suitable for the collimation of hard X-rays. One may even consider going beyond a thickness of 1 mm for some special applications. Secondly, the hole shape is smooth and free of the irregularities that are usually found in laser-drilled holes of the smallest size. Thirdly, the casting technique is relatively simple and inexpensive.

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