Exploitation of Microchannel Plate Optics

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Thesis submitted to the University of Leicester for the degree of Doctor of Philosophy.

January 2000

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Abstract

This thesis contains work on microchannel plate (MCP) optics as used for X-ray focusing, and can be split into two sections; research and applications.

Research into improving the reflectivity of MCPs is presented which includes results obtained at the Daresbury Synchrotron, and electron microscope analysis. Different treatments performed on Nova Scientific channel plates were shown only to make a improvement to reflectivity in the case of annealing. Evidence for a 300Å layer of silica on the surfaces of the microchannels, a result of the acid etching process, was discovered and modelled. Measurements showing nickel coating onto the walls of the channels to a depth of around 25Å were also made.

The method of bending, or slumping MCPs to a spherical form by Photonis and Nova has been assessed, and X-ray images using slumped plates are presented. The accuracy and reproducibility of the process was not found to be excellent (within 10% of the target radius), but were acceptable for the plates slumped to date.

A comprehensive report is given of the application of channel plates as the imaging device in an Imaging X-ray Fluorescence Spectrometer, firstly at the Rutherford Appleton Laboratory and subsequently in the laboratory in Leicester. The spectrometer successfully imaged a multi-element target, resolving both elementally (down to Fluorine, Z=9) and spatially (to under 2mm) in a 34 hour integration. The concept of Bragg reflection imaging is examined as another use of the spectrometer.
I hereby declare that no part of this thesis has previously been submitted to this or any other University as part of the requirements for a higher degree. Work described here was conducted by the undersigned except for the contribution of colleagues indicated in the text.

Adrian Martin
December 1999
Acknowledgements

I would like to thank my supervisor Prof. George Fraser for his assistance and support throughout the duration of my PhD in Leicester, especially for his unerring enthusiasm for the subject. Also colleagues from our team; Nigel Bannister, Adam Brunton, John Lees, Jason Page, Sarah Pearce, Jim Pearson, Gareth Price and Rob Rideout for the enlightening discussions on any topic; scientific or otherwise. Esso Flyckt, J-P Boutot and Ray Fairbend looked after me very well on my trip to the Photonis factory in France, and provided many ideas for chapter 4 in spite of my ignorance of French.

Much other input was provided by George McTurk (SEM pictures), Magnus Johnson (lobsters), Dick Willingale (X-ray analysis software), Tony Abbey and Adam Keay (CCDs), John Spragg (space hardware), John Hennessy, Nick Boldra and Andy Underhill (workshop) and Barry Towell (draughting).

The staff of the Laser Plasma Source at the Rutherford Appleton Laboratories, including Edmond Turcu and Ric Allott, were invaluable during our time at the labs, as was Andrew Peele who joined us at the Daresbury Synchrotron for one of the three experimental runs there during my PhD.

The brave men and women of the Leicester University Mountaineering Club who have provided me with many an epic to describe of a Monday morning.

I acknowledge the financial support of Photonis (formerly Philips Photonics) and PPARC.
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Some of the experimental results reported in this thesis have been incorporated in the following papers. The chapters to which these papers refer are given in brackets.


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Abbreviations

AFM  Atomic Force Microscope
ASM  All Sky Monitor
AXAF Advanced X-ray Astrophysics Facility. (NASA's X-ray observatory, renamed Chandra (1999–))
CCD  Charge Coupled Device.
EN   Electroless Nickel
EPIC European Photon Imaging Camera. (One of XMM's instruments built in Leicester.)
EPSRC The Engineering and Physical Science Research Council
ESA  The European Space Agency.
ESCA Electron Spectroscopy for Chemical Analysis.
ESTEC European Space Research and Technology Centre.
EUV  Extreme Ultra-Violet.
FWHM Full Width at Half Maximum.
GRB  Gamma Ray Burst.
GSFC Goddard Space Flight Center.
HRC  High Resolution Camera (on Chandra)
HXT  High energy X-ray Telescope
IPA  Iso-Propyl Alcohol.
ISS  International Space Station
IXRF Imaging X-ray Fluorescence.
JPEX Joint Astrophysical Plasmadynamic Experiment
MACS The Miniature AXAF Calibration System (in Leicester)
MCP  Microchannel Plate.
NAG  Numerical Algorithm
NIST National Institute of Standards of Technology.
NRL  Naval Research Laboratory.
PET  Polyethylene terephthalate.
PPARC  The Particle Physics and Astronomy Research Council.
PRT  Platinum Resistance Thermometer
PSF  Point Spread Function.
QE  Quantum Efficiency.
RAL  The Rutherford Appleton Laboratory (near Didcot, Oxon)
RoSAT  Röntgen Satellite. (X-ray observatory (1990–1999)).
SEM  Scanning Electron Microscope.
SIMS  Secondary Ion Mass Spectrometry.
SPIE  Society of the Photo-optical Instrument Engineers.
SMART  Small Missions for Advanced Research in Technology.
          (An ESA concept similar to NASA’s SMEX)
SMEX  Small Mission Explorer.
SRS  Synchrotron Radiation Source.
TRP  Technology Research Programme (ESA’s concept of developing new ideas)
TTF  The Tunnel Test Facility in Leicester.
VTF  The Vacuum Test Facility in Leicester.
VUV  Vacuum Ultra Violet (Softer than EUV but harder than violet)
XEUS  X-ray mission for Evolving Universe Spectroscopy
          (One of ESA’s “Cornerstone” missions (1999–))
XRF  X-ray Fluorescence.
Z  Atomic number or charge.
Chapter 1

Microchannel Plate X-ray Optics

1.1 Introduction

MICROCHANNEL plates (MCPs) are a new type of X-ray optic which in the last 10 years have been developed for X-ray astronomy. In these optics, part of a larger family of capillary optics, X-rays are diverted by reflection from the inside surfaces of tiny tubes making up the plate. This differs fundamentally from the usual optical focusing method of refraction which has been ignored until only very recently (Snigirev et al., 1998; Lengeler et al., 1999) because in the X-ray band, the refractive indices of every known material are nearly unity.

This chapter starts with a summary of how channel plates focus light (section 1.1.2) and effects seen due to their unusual reflection, rather than refractive, focal method. A brief history of MCP optics (section 1.2) and other reflection optics (section 1.3) are followed by the manufacturing process of Photonis (section 1.4). Applications of MCPs in astronomy (section 1.6) and other areas (section 1.7) are presented, some of which have been realised whereas some are studies. Finally, specifically my own contributions to the work are detailed, and a review of the rest of the thesis, is in section 1.8.

1.1.1 A note on units

The widely used SI system of units is not observed in the astronomical community and to a certain extent in the X-ray community also. In this thesis, I have tried to adopt the use of SI units such as nm instead of Å, mrad instead of arcminutes and mm not cm. However many references
Figure 1.1: A Scanning Electron Microscope image of a face of a square-pore microchannel plate from Nova Scientific Inc. Scale is indicated by the bar at the bottom; each channel is about 30μm square. Note the multifibre boundary running down the centre of the frame; this dislocation will not have an adverse effect on the plate's focusing abilities.

Figure 1.2: A selection of channel plate optics. From right to left: a round pore Photonis plate spherically slumped to a radius of 70mm; a rimless Nova Scientific plate with square channels of length to diameter ratio L/D=40:1; a reduced Photonis plate and a 20mm square Nova Scientific plate with L/D≈100. Scale is indicated by a 20p coin.
list quantities in incompatible units (such as density in g/cm³ and absorption length in Å), so on occasion I have quoted both. To help assist translation from wavelength to energy the following formula is useful;

\[ E = \frac{hc \times 10^7}{e\lambda} = \frac{12.4}{\lambda} \]  

(1.1)

where \( E \) is in keV and \( \lambda \) is in Å.

### 1.1.2 MCPs as X-ray optics

The MCP optic works by grazing incidence reflection from the surfaces of its square channels. Figure 1.3 shows the conventional two dimensional focusing case of the flat MCP in point-to-point mode. Rays are also focused orthogonally onto a spot.

![Figure 1.3: Point to point focusing with a flat MCP](image)

In astronomy, the source will be at infinity, so this focusing element is not adequate. Instead, a slumped or curved MCP is used (figure 1.4), which obeys the lens equation (Brunton et al., 1995, p40):

\[ \frac{1}{l_s} - \frac{1}{l_i} = \frac{2}{R} \]  

(1.2)

where \( l_s \) and \( l_i \) are the source and image distances respectively, and \( R \) the radius of MCP curvature, which is positive when the source is on the concave side of the plate.

The slumping method and results from slumped plates are discussed in detail in chapter 4.
Figure 1.4: Focusing from infinity (right to left) or beam expansion (left to right) with a slumped MCP

X-rays are reflected at grazing incidence up to the critical angle $\theta_c$, whose value depends on the material making up the channel walls and the X-ray energy. For lead glass the empirical relation (Willingale et al., 1998, p283);

$$\theta_c = a E^{-1.04}$$  \hspace{1cm} (1.3)

applies where $a=2.4$ when $\theta_c$ has units of degrees and the unit of energy is keV. This value drops with energy (as in figure 2.4), and improvement by coating the channels with a metal is desirable. X-ray reflectivity of MCP glass is dealt with in detail in chapter 2.

For ease of manufacture, MCPs are built with the channels square packed. One undesirable feature of this geometry is that their point spread function (PSF) is not point-like, but cruciform. This is due to photons being reflected in one axis only (as the channels are of finite size) whereas an odd number of reflections are needed in each axis for true focusing – see figure 1.5 (Chapman et al., 1991). An example of an MCP X-ray PSF is shown in figure 1.6 (and in figure 4.5). Deconvolution of the PSF by image processing techniques is possible (Martin et al., 1999a, p569) but is complicated and slow. Packing channels radially rather than in a square pack grid (as in figure 1.20) will eliminate this cruciform structure, and work is well underway at Photoni s to achieving this. Note that the true focus is many times brighter than the crossarms which contain about 25% of the flux. Another component of the PSF arises from those photons which pass unreflected through the plate. These form an unresolved background, but it is sometimes possible to eliminate them by slightly tilting the plate out of the optical axis yielding no straight through path (as is the case in figure 1.6).
Figure 1.5: A single channel, where $\theta_t = D/L$, the channel diameter to length ratio for the plate. (a) At $\theta = 0^\circ$, all rays pass through the channel undiverted. (b) At $0^\circ < \theta > \theta_t$, some rays pass straight through, and some are reflected. (c) At $\theta_t > \theta > 2\theta_t$, no rays pass straight through, but some are singly and doubly reflected. (d) At $2\theta_t > \theta > 3\theta_t$, rays are doubly and triply reflected. Note that an odd number of reflections in one dimension give focused rays, while an even number (including zero) contribute to a diffuse, unfocused background.
Figure 1.6: Top: PSF of a spherically slumped (R=0.5m) square packed MCP (Brunton et al., 1999). The "true" focus is the bright spot. The broken line foci, a function of channel plate geometry, can be seen, as can the background "straight through" component. Below is a cut along a crossarm of the picture. Note the true focus is considerably brighter in terms of photons/unit area than the cross arms.
The channel’s length-to-diameter ratio, $L/D$, or equivalently, the transparency angle;

$$\theta_t = \tan^{-1} \left( \frac{D}{L} \right) \sim \frac{D}{L}$$

(1.4)
determines the angle at which rays are cut off from passing through the MCP without being reflected. It is desirable that the channel walls are still reflective at this angle, and preferably beyond. Figure 1.5 schematically shows a single channel reflecting a ray one, two and three times.

The pore size of channel plates (typically $10\mu m$) is only an order of magnitude greater than that of visible light ($0.5\mu m$) so diffraction effects will dominate when focusing any radiation whose wavelength is greater than about $100nm$. X-rays can be considered to have energies above $100eV$ and wavelengths shorter than $12.4nm$ so diffraction effects can be effectively ignored.

### 1.2 History of MCP optic development

The lineage of these plates deserves some explanation as it is somewhat complicated. Channel plates were originally described in the 1960’s (Wiza, 1979) by teams from Russia, the Bendix Research Laboratories in the USA and the Mullard Research Laboratories in the UK for use as electron multipliers in image intensifier tubes, and have since found favour as electron detectors and, either uncoated or with a high efficiency photocathode, UV and X-ray detectors. Indeed, the High Resolution Camera (HRC) on NASA’s Chandra observatory, formerly the Advanced X-ray Astrophysics Facility or AXAF (Winkler et al., 1993) launched in July 1999, could be regarded as the peak of microchannel plate detector development to date, with its $25\mu m$ (0.5 arcminute) resolution, large area ($100 \times 100mm^2$), CsI photocathode and even a new formula of channel plate glass free from the $^{40}K$ of commercial glasses which give rise to high levels of $\beta$-induced background noise (Fraser et al., 1987). The HRC was developed by the Harvard–Smithsonian Center for Astrophysics, the University of Leicester and the Osservatorio G. C. Vaiana, Palermo. These “detector channel plates” as they might be termed, are typically made from glass, have round pores of 6–12.5$\mu m$, are 1–1.5mm thick and have channels that are biased at up to 13° to the normal to prevent ion feedback in two stage MCP detectors. MCP multiplier plates are shown in figure 3.6; much more detail on their operation can be found in the literature (Wiza, 1979;
Square channel glass collimator arrays with 150\(\mu\)m pores were made for Leicester University in 1979 by Galileo Electro-Optics (now Burle) for EXOSAT's Medium Energy Detector Array (Turner and Smith, 1981). The collimators were a good approximation to an idea, originally by Angel (1979), for focusing X-rays using square arrays. Angel however, cannot be credited with first thinking up the concept, because lobsters (figure 1.9), as noted by Land (1978) already have eyes that work on the same principle (God, 4004BC). The paper by Wilkins et al. (1989) could probably be cited as initiating the concept of X-ray focusing using MCPs with square channels. Further, the authors suggested MCPs might focus, collimate and condense neutrons and \(\gamma\)-rays, as well as promoting their slumping to control the focusing action.

Research interest has continued in square pore channel arrays in Leicester, Melbourne and Colombia (since lapsed) Universities and recently NASA's Goddard Space Flight Center (GSFC) and the European Space Research and Technology Centre (ESTEC), while the manufacturers are Photonis (formerly Philips Photonics formerly Mullard) in Brive, France and Nova Scientific in Sturbridge MA, USA.

Square pore channel plates, or “focusing channel plates” are the subject of this thesis, and reference to MCPs can be taken to be referring to square pore focusing MCPs rather than round pore detector MCPs unless specifically stated. A selection of focusing channel plates are shown in figures 1.1, 1.2, 1.7, 1.8, 1.13 and 1.14, some with manufacturing defects. Note the uniformity of the channel stacking which is essential for good focusing.

### 1.3 Other X-ray optics

In 1947, Ehrenberg (section 4.2.1) first proposed the idea of focusing X-rays using reflection from polished surfaces, an idea which was refined by Kirkpatrick and Baez in their 1948 paper. The Kirkpatrick-Baez configuration uses two orthogonal, spherical reflecting surfaces (as in figure 1.10a) – a channel plate can be taken as an approximation to this geometry. Schmidt
Figure 1.7: An SEM view towards the edge of a channel plate manually cleaved using a razor. Note the small shards of glass originating in the broken channel walls visible next to the cleavage. Such impurities can make analyses of the channel's surface difficult. Scale is indicated by the 13.2 μm mark.

Figure 1.8: A low magnification SEM view of the cross section of a cleaved channel plate with a channel aspect ratio L/D of about 30:1. Scale is indicated by the 217 μm mark.
Figure 1.9: An SEM image of the eye of the squat lobster Munida rugosa. Photons are reflected from the walls of square packed "cones" onto the rhabdom (a type of retina). The lobster brain must see a point source of light as a cross in the same way as an MCP X-ray focus is a cross. The scale bar is 10μm. From Gaten (1994).

(Schmidt, 1975, 1981) proposed a telescope using plates focusing onto a cylinder in a type of 1-d Kirkpatrick-Baez form, but this design was not a true imaging device so was quickly abandoned. It was left to Angel (1979) to miniaturise the two orthogonal reflecting surfaces concept in his seminal lobster eye telescope paper.

In 1952, Wolter (Wolter, 1952a,b) proposed three independent systems of mirrors to focus X-rays by a process of two reflections. The Wolter type 1, 2 and 3 systems use two mirrors of conic sections, mounted end to end to achieve a focus, and one of them (Wolter type 1) forms the basis for the major X-ray telescopes today (see section 1.6.3 for more benefits of the Wolter type 1 system). To fabricate the conic sections, a process of replication is most often chosen, in which a highly accurately formed mandrel shaped to the Wolter type 1 form is electroplated with a good reflecting metal (usually gold and/or nickel). Once the mandrel has been removed, the "shell" has the exact form of the mandrel (Gondoin et al., 1995, 1996) and will focus X-rays. Shells are nested to give a larger collecting area – XMM has 58 shells per mirror module.

Capillary optics are bundles of glass fibres arranged as a light guide to funnel light from one
place to another (figure 1.12). The Kumakhov lens (Kumakhov and Komarov, 1990), a type of capillary optic like MCPs, is suitable for X-ray pseudofocusing by having X-rays bounce along the capillary fibres while keeping the incident grazing angles low enough so as not to attenuate the flux significantly. The fibre bundles can be made to a wide range of shapes; inverting, enlarging, focusing or physically bending or offsetting the flux in space. However, because each fibre has to be set by hand, they are very expensive and small. Single polycapillary fibres are made by X-ray Optical Systems.

Due to their geometry, MCPs will also focus any particles; such as neutrons. Work on cold neutron focusing has been undertaken, lately by the National Institute of Standards and Technology using high boron capillary glass with some success (Chen et al., 1994).

### 1.4 MCP manufacture

The manufacture of square pore channel plates is essentially identical to that of round pore plates (e.g. Brunton, 1995, p11), but with different channel packing regimes. Figures 1.15 and 1.16 schematically show the process I observed at Photonis SAS (formerly Philips Photonics) in Brive as part of my CASE studentship.
A square shaped, 5mm thick, 1m long hollow tube of high lead content cladding glass (typically Corning 8161 or Philips 297) has the core glass (typically Philips 274) inserted in it, having first been made square. The core glass is softer and considerably more soluble than the cladding glass, and it is also slightly pink. The combined couple is hung at the top of an 8m high drawing tower and slowly heated in an oven until it "necks", dropping a blob of glass to the base of the tower, when drawing of the main fibre can commence. Draw speed is controlled by two gripper wheels towards the bottom of the tower, and varied such as to keep the force on the loadcell above the couple constant. The first draw fibres are about 1mm wide and cut to be 500mm long.

The fibres are then manually stacked into multifibre bundles and drawn again (the second draw). The multis are cut to suitable lengths about 100mm and are in turn assembled into a block with a section which has the dimensions of the final channel plate.

The block is ram-fused with high pressure and temperature, and slices sawn from it are ground and polished prior to etching with a 1M solution of constantly flowing hydrochloric acid for several hours.

Both Nova Scientific in the USA and Photonis in France also make boules rather than blocks where the assembled multis are encased inside a glass envelope prior to the final heating process.
Figure 1.12: A polycapillary fibre (left) – the fibre is 1.1 mm across flats. A 150 mm long Kumakhov lens (right) manufactured from polycapillary fibres for gathering flux from a source onto a detector 200 mm away. By permission of XOS.

Plates sawn from boules have solid rims which must be ground or cut off before use.

1.5 The Technology Research Programme

In 1996, the University, the European Space Research and Technology Centre (ESTEC), PhotoniX and ERA Technology began a “Technology Research Programme” (TRP) contract to develop channel plate technology for X-ray detectors and optics (Contract No. 12193/96/NL/SB). The TRP is intended by ESA (the European Space Agency) to help fledgling technologies mature into reliable and trustworthy methods for use in space programmes within ESA, and are typically three years long and worth about £0.75M. The guiding application for our contract is the optics for a high energy telescope (see section 1.6.3). Parts of the TRP concerned with MCP optics (rather than MCP detectors) are:

- Large format MCP production (54×54 mm² and channel length to diameter ratio of 500:1) for high energy focusing
- Radial packing of channels rather than square packing to better approximate the Wolter type I configuration
Figure 1.13: A low magnification view of an MCP manufactured by Nova Scientific with “fiducial” markers in every multifibre – special channels made from non-etchable glass used to check mechanical uniformity of a plate. Scale is indicated by the 222μm mark.

Figure 1.14: A large pore (100μm) Nova Scientific MCP with an exceptionally high open area. Here, the channels have suffered twisting, possibly caused by the obstacle such as that visible at the lower left – a stone in the glass. Such a plate would be useless for focusing. Scale is indicated by the 20.6μm mark.
Figure 1.15: A fibre drawing tower
Figure 1.16: Overview of the fabrication process of square pore channel plates. The drawing–stacking process is usually repeated once, but can be done more often (up to 4 times has been reported).
• Slumping of thick (500:1) MCPs to make Wolter type 1 lenses
• Improvement in surface roughness, channel alignments, twist and other manufacturing imperfections
• Silicon channel plates (see section 7.2)

Deliverables and results from the programme have arrived too late for inclusion in this work, but much of the investigation for chapters 2 to 4 is directly relevant to it, and much of the recent progress in MCP technology can be attributed to it.

1.6 Astronomical applications of MCP optics

In the Leicester X-ray astronomy group, the main direction of research has traditionally been towards astronomy, but funding limitations and the urge to diversify have necessitated a shift towards more commercial applications. However, there have been three very different instruments based on MCP optics proposed for astronomical applications in recent years, as well as several new ground based applications of the technology.

The first attraction of MCP optics for astronomy lies primarily in their mass. Fraser (1997) has defined a figure of merit, \( F \), for any X-ray telescope;

\[
F = \frac{\text{Telescope effective area (in cm}^2\text{)}}{\text{Telescope optic mass (in kg)}}
\]  

(1.5)

\( F \) (table 1.1) is about 1-10 for replicated (XMM) type optics and about 1000 for the MCP optics proposed for the All Sky Monitor (ASM) mission (Fraser et al., 1990). This advantage is due to the larger open area of MCPs and their small pore size which can constrain their length to only a few mm.

Additionally, MCPs have no preferred optical axis, so their field of view could, mechanics and shadowing aside, be \( 4\pi \) steradians. With such an advantage (XMM has a 30arcminute square
field of view) it was realised the ideal application for MCPs would be an All Sky Monitor satellite, which would be able to view a large part of the sky at once, and when spun appropriately, regularly map the heavens entirely. The LOBSTER mission is the latest of these designs.

1.6.1 LOBSTER

An all sky monitor mission (ASM) was unsuccessfully proposed (Fraser et al., 1990) to ESA in 1990 and following significant developments in channel plate manufacture, the idea surfaced again. As LOBSTER it was proposed for NASA’s SMEX (small mission explorer) call for proposals in 1997 (Priedhorsky, 1997). Favourably reviewed in terms of science, but eventually rejected on grounds of the technical readiness of the gas microstrip detectors and the MCP optics, it consisted of a series of “eyes” (figure 1.17), each of which could see $30^\circ \times 30^\circ$ of sky, and made of 144 slumped MCPs aligned to focus X-rays onto imaging proportional detectors. Previous all sky monitors have imaged a small (few square degrees) of sky at a time, building up a full sky map in hours or days by spinning the spacecraft. Though not of the high sensitivity or resolution of XMM or Chandra, LOBSTER could monitor multiple sources over the whole sky for long periods which would be impractical using the heavy and narrow angular view shell optics of RoSAT or XMM. Astronomers are interested in the light curves of many sources over long (years) periods of time, data which currently is not available for X-ray objects. Additionally, LOBSTER could spot the X-ray afterglows of Gamma Ray Bursters (GRBs) and report their location quickly to other observatory class instruments. LOBSTER has been successfully proposed in the Flexi mission round (ESA) in Jan 2000 as a International Space Station (ISS)

<table>
<thead>
<tr>
<th>Mission</th>
<th>$A_{\text{eff}}$ at 2keV (cm$^2$)</th>
<th>Mass of optics (kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chandra/AXAF, Pearce (1998)</td>
<td>780</td>
<td>950</td>
</tr>
<tr>
<td>XMM, Gondoin et al. (1994)</td>
<td>4000</td>
<td>420</td>
</tr>
<tr>
<td>ASM (proposal), Fraser et al. (1990)</td>
<td>2700</td>
<td>1 (optics)+3 (support)</td>
</tr>
</tbody>
</table>

Table 1.1: Effective areas ($A_{\text{eff}}$) and optic masses for X-ray telescopes.
attached payload, and will have a further six months of technical assessment before entering the final selection process.

1.6.2 Imaging X-ray spectrometer

SMART (or Small Missions for Advanced Research in Technology) is ESA’s programme to try new technology in space, and the first mission, aptly called SMART-1 (Martin et al., 1999a), is to prove the effectiveness of solar electric propulsion following the success of NASA’s Deep Space 1 launched in December 1998 (Lehman, 1999). As proposed (and selected) for SMART-1, the IXS’s function was twofold; to provide astronomers with the long term variation data on a few cosmic X-ray sources during the cruise to a near-earth asteroid, and to image X-ray fluorescence generated by an asteroid from excitation by the sun on arrival. The IXS instrument used four MCPs (see figure 1.19) to focus X-rays onto a CCD (similar to the Imaging X-ray Fluorescence spectrometer described in chapter 5) which had an excellent energy resolution capability giving elemental (Fe, Ca, Al, Si, S) mapping to a spatial resolution of 100m. Due to be launched at the end of 2002, the spacecraft’s mission objective has been changed to the Moon and unfortunately, ESA finally chose a non-imaging spectrometer for the mission, but there may be an opportunity for the resurrection of IXS on other missions, such as NEAP (the Near Earth Asteroid Prospector)*.

1.6.3 Hard X-ray telescope

The HXT is a hard X-ray (1–100keV) imaging telescope which was proposed for ESA’s M3 mission in 1993 (Fraser et al., 1993d). The HXT uses MCP optics for focusing, but their configuration is unlike those used for ASM or any other MCP focusing schemes to date. The harder X-rays encountered by HXT can only be reflected up to small angles (θc, the critical angle being a few arcminutes) so for optimum focusing, channels must have an aspect ratio of at least \(1 / \tan \theta_c\) or about 1000:1 (Holland et al., 1995; Willingale et al., 1998).

*Currently on http://www.spacedev.com/
Figure 1.17: The LOBSTER spacecraft. The MCPs are in three clusters at the lower end of the satellite. The spacecraft spins at 6 rpm about an axis aligned directly towards the sun indicated by the red arrow, so covering most of the sky in 5s and all but a circle around the sun every 10s.

Figure 1.18: The SMART-1 spacecraft observing an asteroid. The IXS instrument is looking down directly towards its surface gathering fluorescent X-rays excited by the sun. The large solar panels were required for the solar electrical propulsion whose testing was the primary mission objective.
The Wolter type 1 configuration (as described in section 1.3 and shown in figure 1.11) uses a two stage, two reflection optic in which the reflection angle is halved for each reflection, which is clearly desirable in this situation. To approximate the Wolter type 1 system, two slumped MCPs, whose radii of curvature are R and R/3 where the focus is at R/4, can be placed end to end (figure 1.20d). The two optics are each 500:1 thick. Additionally, the channels are packed radially, rather than in a square array (as in figure 1.20a and c) which gives a higher X-ray cross section and removes the cross from the focus (UK patent application 9311134). The ESA TRP (section 1.5) is principally guided towards such an application of MCP optics by making its end product a radially packed, 1000:1 (in 2 x 500:1 plates) Wolter type 1 optic.
Figure 1.20: (a) and (b) The conventional square pack, and (c) and (d) the novel radial pack focusing channel plate structure, where the MCPs have a radius of curvature of $R$ and $R/3$ for a focus at $R/4$. The radial design, though harder to make, has a higher effective area than the square pack, and does not produce the cruciform image shape (Willingale et al., 1998).
1.7 Terrestrial applications of MCP optics

1.7.1 Imaging X-ray fluorescence

Further to the astronomical applications of channel plates for imaging, a terrestrial fluorescence imager has been made in prototype, and recently (Summer 1999) a substantial grant won from EPSRC to develop it further. The device is described in detail in chapter 5 (see figure 5.1), relying on a channel plate for its capability to produce elemental maps to <0.5mm resolution, but replacing the sun with a conventional X-ray source and the asteroid with a laboratory sample. Such a device will be of use in geology, archaeology, metallurgy and medical science, or any situation where knowledge of elements' abundance and their cartography is needed. Additionally, slumped channel plates will give magnification of both less and greater than unity.

Another application of this technology is measuring changes in the angle of Bragg reflection from crystal planes (chapter 6). This enables stresses in crystals (eg. stress caused by an aluminium track being laid onto silicon) to be mapped for the semiconductor industry, a task presently achieved by microbeam scanning at a synchrotron – hardly a portable X-ray source.

1.7.2 X-ray concentration

Flux collection, or the gathering of photons over a wide area onto a small detector without the need for imaging, is particularly suited to channel plates, as samples of modest resolution can be manufactured cheaply (several hundred pounds). Capillary optics, usually Kumakhov lenses (Kumakhov and Komarov, 1990), which funnel light along, or between fibres, have been used for this, notably by Wollman et al. (1997), but their price (several thousand pounds) is prohibitive for all but the deepest of pockets. Bede and Shimadzu, manufacturers of X-ray spectroscopy equipment, and others have expressed an interest in using MCP concentrators, but to date, perhaps surprisingly, progress has been slow.
1.7.3 X-ray lithography

X-ray lithography – or the science of printing with X-rays instead of the more usual UV or visible light – needs a wide, parallel beam of X-rays to form small features. Printing on silicon wafers with such a procedure might give feature sizes of <0.12μm in mass production. Currently, electron beam lithography techniques can do this, but the process is not suitable for mass production. To generate the wide, parallel beam, a MCP can be mechanically profiled and used in beam expander mode; *i.e.* the left to right mode of figure 1.4 (Brunton *et al.*, 1996).

1.8 Overview

During my PhD I participated in two period of beamtime at the Daresbury Synchrotron which resulted in the work for chapter 2. I also took part in a visit to the Laser Plasma Source at the Rutherford Appleton Laboratory to produce chapter 5. For both of these allocations I shared responsibility with colleagues for engineering, operations and planning, but did most of the data analysis myself. The second, Leicester based fluorescence experiment (chapter 6) was more of a solo effort on my behalf. Additionally, I was responsible for designing and expediting nearly all of the smaller tests and the work on slumping.

Chapters 2 and 3 explore the theory of operation of MCP optics and some technical advances made in the manufacture and treatment of MCPs to help them to operate more efficiently. Chapter 4 is concerned with the mechanical bending or *slumping* of MCPs, which opens up a broad range of applications. Since the last thesis on the subject (Brunton, 1995) progress in the development of MCPs for focusing has been significant, and they are no longer mere curiosities. Some of the applications of MCP focusing have been presented in sections 1.6 and 1.7, and one important new application – imaging X-ray fluorescence – is fully explored in chapters 5 and 6. Finally, chapter 7 examines what might be possible in the next ten years of microchannel plates development.
Chapter 2

Channel Plate Reflectivity

2.1 Introduction

The reflectivity, or the probability that a material reflects light incident upon it, is the property of a channel plate that controls the ability to focus light, for without it, all rays would be absorbed or travel straight through the plate undeviated and unfocused. In the X-ray band, which is the subject of this work, specular reflection only occurs at grazing angles of, at the most, a few degrees for all materials, so any degradation in reflectivity as a result of surface roughness is undesirable and likely to have a large effect on focusing efficiency and intensity uniformity.

Reflectivity occurs as a result of interactions between photons and atomic electrons, so broadly speaking, any element that has more electrons is a better reflector, and any material that is dense — i.e. has more atoms per unit volume — is also better. Metals are examples of good reflectors owing to their high density and potentially high atomic number. Unfortunately, it is unlikely to ever be possible to make channel plates from metals due to their mechanical properties, leaving only glass (section 1.4) and silicon (section 7.2) as the only materials to date from which MCPs have been made.

The addition of lead ($Z=82$) to glass improves its reflectivity at higher incident angles, but it is only present at low number density. Alternatively, if the reflecting channels could be overlaid with a metal to an adequate depth, the X-rays would “see” the MCP as being made entirely from that metal, and show an improvement in reflectivity. Additionally, a coating onto the glass walls might smooth out any surface roughness and cover chemical inhomogeneities in the glass — both factors that adversely affect the reflectivity.
In this chapter X-ray reflectivity theory is first reviewed, then section 2.3 reports a synchrotron study of how different chemical treatments of bare glass affect reflectivity. Section 2.4 describes the first results of coating an MCP with silver. An examination of nickel surface coating at the Daresbury Synchrotron is described in section 2.5.

2.2 Theory

The reflectivity of a channel plate’s walls is the parameter which defines its ability to collect and focus light. It depends on incident energy, angle of incidence and the material from which the structure is made. The reflectivity can be calculated by the method of Henke (1981) from Fresnel’s equations as follows.

The atomic scattering factors, \( f_1 \) and \( f_2 \), are obtainable from the atomic cross-sections in the database of Cromer and Liberman (1970), and are given by;

\[
f_1(E) = Z + \frac{1}{\pi r_0 h c} \int_0^\infty \frac{e^2 \sigma_e(\epsilon)}{E^2 - \epsilon^2} d\epsilon + \Delta f_r
\]

\[
f_2(E) = \frac{E \sigma_e(E)}{2\pi r_0 h c}
\]

where: \( Z \) is the atomic number; \( r_0 \) the first Bohr radius; \( h \) Planck’s constant; \( c \) the speed of light; \( \sigma_e(E) \) the photoelectric cross-section at photon energy \( E \); \( \epsilon \) the energy for integration. \( \Delta f_r \) is a relativistic correction, only significant for heavier elements and given by Henke et al. (1988) as;

\[
\Delta f_r = 1.32 \times 10^{-6} Z^3 + 6.2 \times 10^{-5} Z^2
\]

The complex dielectric constant, \( k \), is given in term of decrements from unity;

\[
k = 1 - \alpha - i \gamma
\]

where \( \alpha \) and \( \gamma \) are small compared with unity.

\[
\alpha = \frac{r_0 \lambda^2}{\pi} \sum_q n_q f_{1q}
\]

\[
\gamma = \frac{r_0 \lambda^2}{\pi} \sum_q n_q f_{2q}
\]
where $\lambda$ is the X-ray wavelength. While chemical bonds affect the outer electrons of atoms, X-rays interact chiefly with the inner electrons, so the scattering factors $f_{1q}$ and $f_{2q}$ can be summed in proportion to their number density per unit volume $n_q$ for each element $q$ irrespective to which other atoms it may be bonded to. It is convenient to define a parameter $a$, where;

$$a^2 = \frac{1}{2} \left( \sin^2 \theta - \alpha + \sqrt{\sin^2 \theta - \alpha^2 + \gamma^2} \right)$$  \hspace{1cm} (2.6)$$

Here $\theta$ is the grazing incidence angle. $R_{\parallel}$ and $R_{\perp}$ are defined as the ratios of reflected intensity to incident intensity for the polarised components with the electric field vector parallel to, and perpendicular to the plane of reflection respectively (Henke, 1981);

$$R_{\parallel}(\theta) = \frac{4a^2(\sin \theta - a)^2 + \gamma^2}{4a^2(\sin \theta + a)^2 + \gamma^2}$$  \hspace{1cm} (2.7)$$

$$\frac{R_{\parallel}(\theta)}{R_{\perp}(\theta)} = \frac{4a^2(a - \cos \theta \cot \theta)^2 + \gamma^2}{4a^2(a + \cos \theta \cot \theta)^2 + \gamma^2}$$  \hspace{1cm} (2.8)$$

So finally this gives the reflectivity for unpolarised X-rays as;

$$R(\theta) = \frac{R_{\parallel}(\theta)}{2} \left( 1 + \frac{R_{\perp}(\theta)}{R_{\parallel}(\theta)} \right)$$  \hspace{1cm} (2.9)$$

Moreover, surface roughness changes reflectivity in a way dependent upon energy and angle, and can be incorporated by an exponential Debye-Waller factor;

$$R' = Re^{-\frac{1}{2}(4\pi\sigma \sin \theta / \lambda)^2}$$  \hspace{1cm} (2.10)$$

where $\sigma$ is the RMS surface roughness.

### 2.3 Reflectivity of chemically treated channel plates

The reflectivity of four differently treated MCPs was measured at the Daresbury Synchrotron. The plates, specified as in table 2.1 and treated as described in table 2.2 were procured from Nova Scientific.

Previously, there has been some indication from atomic force microscopy measurements that hydrogen reduction reduces surface roughness (from 50Å to 22Å, Fraser et al. (1993c)), which
<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Schott Fiber Optics (Southbridge, MA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference</td>
<td>Boule 21555</td>
</tr>
<tr>
<td>Glass Type</td>
<td>Corning 8161</td>
</tr>
<tr>
<td>Size</td>
<td>$21 \times 21\text{mm}^2$</td>
</tr>
<tr>
<td>Pore size</td>
<td>$8\mu\text{m} \text{ square}$</td>
</tr>
<tr>
<td>Channel pitch</td>
<td>$12\mu\text{m}$</td>
</tr>
<tr>
<td>Open area fraction</td>
<td>0.44</td>
</tr>
<tr>
<td>Channel length</td>
<td>0.8mm</td>
</tr>
<tr>
<td>L:D</td>
<td>100:1</td>
</tr>
</tbody>
</table>

**Table 2.1:** Test MCP geometry.

<table>
<thead>
<tr>
<th>Plate</th>
<th>Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Hydrogen reduction at 430°C</td>
</tr>
<tr>
<td>B</td>
<td>Hydrogen reduction at 430°C</td>
</tr>
<tr>
<td>C</td>
<td>Air Bake at 430°C</td>
</tr>
<tr>
<td>D</td>
<td>Vacuum Bake at 250°C</td>
</tr>
</tbody>
</table>

**Table 2.2:** Treatment of the four plates. Plates B–D also had extra “post-cleaning” after etching compared with plate A.
would be detectable in the reflectivity curves, and it was also thought that annealing the glass would smooth it further (Corning 8161 MCP glass anneals at 430°C) by removing stresses and tiny surface bumps. These measurements set out to test these conjectures. Channel plate surface roughness has been estimated to be 50Å (Fraser et al., 1993a) by examination of the “wings” of an out-of-focus X-ray image using an unreduced Philips plate, and 57Å (Kaaret et al., 1992) from atomic force microscope (AFM) measurements of a hydrogen reduced Galileo MCP.

Beamline 9.3 at the Daresbury Synchrotron was used for the experiments. It can give 8-25keV X-rays, adjustable by a water cooled, double Si (220) crystal harmonic rejecting monochromator, and has a ∼3mrad horizontal acceptance angle. Fortunately, any work at this energy can be carried out in air, with suitable safety interlocks. The beamline is shown schematically in figure 2.1.

![Schematic diagram of beamlines 3.4 and 9.3 at the Daresbury Synchrotron (not to scale).](image)

Figure 2.1: Schematic diagram of beamlines 3.4 and 9.3 at the Daresbury Synchrotron (not to scale).

A 0.25 × 3mm² area (the multifibre size was 0.192 × 0.192mm²) of each MCP was illuminated by stopping down the primary beam at the entry slit. The entry and exit ion chambers were operated with 50mbar and 360mbar partial pressures of argon respectively, the balance to 1bar being helium. They were cross calibrated without an MCP being present, which allowed us to ignore ion chamber response changes with energy and X-ray absorption by the air and ion chamber windows. An MCP was inserted with the exit beamstop driven out of the optical path, and the signal from the exit ion chamber maximised by tilting it longitudinally and transversely to an accuracy of 0.001° (17μrad) to find the “straight through” position. The open area (previously calculated as 0.44 from SEM studies) was confirmed.

Reflectivity was measured as:
• a function of angle (0 to $\theta_t$, the transmission angle, $\tan^{-1} \frac{D}{L} = 10\text{mrad}$) at a constant energy of 8keV, and

• a function of energy (8 to 25keV) at a constant angle of 2.5mrad.

For each curve, a pair of measurements was necessary to give both the reflected and unreflected beam. First by removing the tungsten exit beamstop, we measured the summed intensity of the two beams. Then we drove the beamstop up (resolution $1\mu\text{m}$) until it intercepted the transmitted beam, and we measured the reflected beam alone. Using the following analysis, we calculated the reflectivity:

$$F_0 = \frac{(F_r/R + F_t)}{A_{\text{open}}}$$  \hspace{1cm} (2.11)

where $R$ is the reflectivity, $A_{\text{open}}$ the open area ratio, $F_0$ is the incident flux on the MCP, $F_r$ is reflected flux and $F_t$ is the unreflected flux. In terms of the ion chamber currents:

$$kI_F = \frac{(I_r^R/R + I_t^R)}{A_{\text{open}}}$$  \hspace{1cm} (2.12)

where $I_F$ is the front ion chamber current, $I_r^R$ is the contribution to the rear ion chamber current due to the reflected X-rays, and $I_t^R$ is the contribution to the rear ion chamber current due to the unreflected X-rays. $k$ is the ratio of the currents in the rear and front ion chambers with no MCP present. We can measure $I_r^R$ and $I_r^R + I_t^R = I_t^R + I_{r+4}$, so in terms of measurable quantities:

$$R = \frac{I_r^R}{(kI_F A_{\text{open}} + I_r^R - I_t^R)}$$  \hspace{1cm} (2.13)

Theoretical reflectivity curves, assuming zero surface roughness, were calculated, for comparison with the experimental data, by the refvang and XOPT programs written in Leicester using the Cromer and Liberman database for the materials in table 2.3.

Figures 2.2 & 2.3 plot reflectivity against angle and figures 2.4 & 2.5, reflectivity against energy. Apart from plate D it can be seen that the curves are very similar.

Plates A-C were all subjected to post etch thermal treatments which raised them to the glass annealing temperature of 430°C, while plate D was only heated to 250°C. We conclude that heating to 430°C caused some annealing which smoothed the reflecting surfaces. A temperature of
<table>
<thead>
<tr>
<th>Glass Type</th>
<th>Pseudomolecule</th>
<th>Density (kg/m(^3))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silica</td>
<td>Si(_2)O(_2)</td>
<td>2200</td>
</tr>
<tr>
<td>Corning 8161</td>
<td>Si(<em>6)O(</em>{17})Pb(_2)K</td>
<td>4000</td>
</tr>
<tr>
<td>Philips</td>
<td>Si(<em>5)O(</em>{12})KNaPb</td>
<td>3300</td>
</tr>
<tr>
<td>Philips Reduced</td>
<td>Si(<em>5)O(</em>{12})KNaC</td>
<td>3300</td>
</tr>
</tbody>
</table>

**Table 2.3:** Glass compositions. The MCPs under study here were made from Corning 8161 glass.

250°C was not sufficient for such annealing to occur leading to a poorer surface finish in plate D.

There are bumps visible in the steep part of the reflectivity versus angle curves which are “Kiessig fringes” (Kiessig, 1931; Peele *et al.*, 1998) resulting from the structure of the MCP reflecting surfaces. Kiessig fringes occur when X-rays are reflected from a layered material. At certain combinations of layer thickness, angle and energy, rays can penetrate the layer, reflect from the substrate surface, penetrate the layer once again and, at the detector, interfere with rays reflected directly from the layer surface to give apparently anomalous reflectivity values. This, of course, is the principle employed in multilayer X-ray mirrors. In the production of functioning MCP electron multipliers the basic MCP glass (in this case Corning 8161) undergoes a series of chemical processes, of which the most important are etching of the MCP to remove the core glass and hydrogen firing to reduce, hence activate, the surface. This treatment leads to a variation in glass composition with depth below the channel surface. Several authors have reported composition versus depth profiles for activated MCP glass using Auger electron spectroscopy (Hill, 1976; Praček and Kern, 1993), Electron Spectroscopy for Chemical Analysis (ESCA) (Siddiqui, 1977) or Secondary Ion Mass Spectrometry (SIMS) (Then and Pantano, 1990). These authors agree, qualitatively, on the surface and sub-surface structure (see figure 2.6). At the surface is a very thin (~ mono-layer) region (2), rich in the alkali metals, especially K. Below this is a lead free region (3), 100 to 500 Å thick. Below this is a thick semiconducting region where some of the lead oxide has been reduced to metallic lead (4); in this region the overall elemental composition appears to be that of the bulk glass. From the perspective of X-ray reflectivity there
Figure 2.2: Measured reflectivity against grazing angle at 8keV for the plates A-D. Note the Kiessig fringe "bumps".

Figure 2.3: Calculated reflectivity against grazing angle at 8keV. The measured curve for sample A is shown for comparison.
Figure 2.4: Measured reflectivity against energy at 2.5 mrad grazing angle. Unfortunately, an experimental error made sample D’s data unusable.

Figure 2.5: Calculated reflectivity against energy at 2.5 mrad grazing angle. The measured curve from sample A is shown for comparison.
are, therefore, two important layers: the silica like lead-depleted region (3) and the bulk glass composition region (4) beneath it.

![Diagram of MCP surface structure](image)

**Figure 2.6:** Layered structure of an MCP surface that forms Kiessig fringes. The layers are (1) Vacuum/air, (2) alkali metal monolayer, (3) lead-depleted silica layer and (4) bulk MCP glass.

The reflectivity data here has been modelled by Brunton *et al.* (1999) using the REX program (Crabb *et al.*, 1992) for the analysis of X-ray reflectivity data, and fitted to a two layer model to give values for the layer thicknesses and their surface roughnesses in table 2.4.

REX calculates the reflectivity of multilayers using the formulae of Parratt (1954), deriving parameters from the database of Cromer and Liberman according to the method of Henke (1981). Surface roughness is modelled using the scalar theory of Beckmann and Spizzichino (1963) according to Cowley and Ryan (1987) with the Fresnel coefficients multiplied by the Debye-Waller factor of equation 2.10. Beam divergence is accounted for by averaging reflectivity over a Gaussian beam profile. Least squares fitting within specified bounds using the Numerical Algorithm (NAG) function E04JAF to the data is carried out with layer thickness, RMS surface and interface roughnesses and also angular offset (not tabulated) as free parameters. The workings of the REX program are described more fully in Crabb *et al.* (1993).

The results in table 2.4 show a marked distinction in glass (layer 3) roughness between the annealed plates (A, B and C) and the unannealed, though still baked, plate D. The roughness, though not as good as mirrors such as the <4Å RMS of XMM’s gold coated shell optics, is comparable with more conventional X-ray mirrors.
Table 2.4: Values of parameters fitted by least squares to the reflectivity data.

<table>
<thead>
<tr>
<th>MCP</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
</tr>
</thead>
<tbody>
<tr>
<td>Layer 3 thickness (Å)</td>
<td>359</td>
<td>448</td>
<td>352</td>
<td>444</td>
</tr>
<tr>
<td>Layer 3 interface roughness (Å)</td>
<td>11.3</td>
<td>11.7</td>
<td>11.0</td>
<td>17.6</td>
</tr>
<tr>
<td>Layer 4 interface roughness (Å)</td>
<td>25.5</td>
<td>19.1</td>
<td>21.8</td>
<td>21.8</td>
</tr>
</tbody>
</table>

2.4 Silver coating of a 67μm MCP

In 1996, two square-pore channel plates (see table 2.5) were supplied to the University by Nova Scientific Inc. One had been silver coated by Nova using a proprietary process, which was assumed to be the silver mirror test or Tollen’s test (Lister, 1995), where silver is precipitated from silver ions mixed with ammonia when in the presence of an aldehyde;

\[ \text{CH}_3 - \text{CHO} + 2\text{Ag(NH}_3)_2^+ + 2\text{OH}^- \rightarrow \text{CH}_3 - \text{COO}^- + 4\text{NH}_3 + 2\text{H}_2\text{O} + 2\text{Ag (solid)} \]

The other was geometrically identical, but had no silver treatment. Our testing of the plates involved two tasks; electron microscope analysis and X-ray reflectivity measurement.

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Nova Scientific</th>
</tr>
</thead>
<tbody>
<tr>
<td>Size</td>
<td>10 × 10mm²</td>
</tr>
<tr>
<td>Pore size</td>
<td>67μm square</td>
</tr>
<tr>
<td>Channel length</td>
<td>2.4mm</td>
</tr>
<tr>
<td>L:D</td>
<td>36:1</td>
</tr>
</tbody>
</table>

Table 2.5: Specification of the plates used in the silver coating tests.

The scanning electron microscope and its X-ray fluorescence attachment in the Medical Sciences Building at Leicester University was used to look at the two MCPs for surface quality and elemental composition. Figures 2.7 & 2.8 are end views of the coated and uncoated specimens. The thin surface layer of silver can be seen flaking off in the coated figure, and it appears to be
Figure 2.7: The channel ends of the silver coated MCP. Note the peeling, granular nature of the silver deposit.

Figure 2.8: The channel ends of the uncoated MCP. Note the ‘radiused’ channel vertices.
granular or lumpy, and certainly much rougher than the glass under it. Such a surface finish is highly detrimental to X-ray reflectivity. On the microchannel structure itself, small pits along the centre of the channel walls, and considerable corner radiusing are evident, symptomatic of over-etching, also bad for X-ray performance. The corner (2 × 2mm²) of the silver MCP was cleaved off with a razor blade to expose channels interior to the plate (see figure 2.9) and the SEM’s 15kV microprobe positioned at the 3 points marked to search for evidence of silver penetration into the channels.

![Cross section of the silver coated plate cleaved with a razor blade.](image)

**Figure 2.9:** Cross section of the silver coated plate cleaved with a razor blade.

We can conclude that from the counts observed (table 2.6) there is some silver, even in the centre of the channels, but the layer is not very thick. The penetration depth of the 15kV electrons from the SEM can be determined by the empirical formula of Burke (1977);

\[ R = k(E + b)^n \]  

(2.14)

where;
Table 2.6: X-ray counts corresponding to silver and silicon at the 3 points shown in figure 2.9 from the SEM’s microprobe.

<table>
<thead>
<tr>
<th>Point Position</th>
<th>Ag–L (2903–3065eV)</th>
<th>Si–K (1664–1812eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A Left</td>
<td>10464</td>
<td>35134</td>
</tr>
<tr>
<td>B Centre</td>
<td>8191</td>
<td>39859</td>
</tr>
<tr>
<td>C Right</td>
<td>22766</td>
<td>43312</td>
</tr>
</tbody>
</table>

The parameter $k$ is determined by fitting $R$ to $E = 10\text{keV}$ with $R = 6.824 \times 10^{-6}Z + 2.566 \times 10^{-4}$. For silver, $Z = 47$, $\rho=10.50\text{g/cm}^3$, $E = 10\text{keV}$, so $k = 1.228 \times 10^{-5}$.

For $E = 15\text{keV}$, the energy of our spectra, $R/\rho = 1.04\mu\text{m}$. Knowing this, and that the underlying silicon can be seen all the way along the channels, we can be confident that the thickness of silver is not greater than 1\mu m. However, the silver may have been deposited in blobs leaving gaps through which the glass channel walls can be seen.

2.5 Synchrotron measurements of Ni-coated MCPs

In 1998 measurements using synchrotron X-rays were made on two further Nova Scientific MCPs (coated and uncoated) to investigate their reflectivity. Now, however, the covering material was
Table 2.7: Details of the uncoated and nickel coated MCPs tested at the Daresbury SRS.

<table>
<thead>
<tr>
<th>MCP</th>
<th>E</th>
<th>F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Manufacturer</td>
<td>Nova Scientific</td>
<td>Nova Scientific coated by Nanosystems Inc</td>
</tr>
<tr>
<td>Size</td>
<td>$10 \times 10\text{mm}^2$ with fiducial markers</td>
<td>$10 \times 10\text{mm}^2$</td>
</tr>
<tr>
<td>Pore size</td>
<td>$30\mu\text{m}$</td>
<td>$10\mu\text{m}$</td>
</tr>
<tr>
<td>Channel length</td>
<td>$1.2\text{mm}$</td>
<td>$0.3\text{mm}$</td>
</tr>
<tr>
<td>L:D</td>
<td>$40:1$</td>
<td>$30:1$</td>
</tr>
<tr>
<td>Coating</td>
<td>None</td>
<td>Nickel</td>
</tr>
</tbody>
</table>

nickel. Beamline 3.4 at the Daresbury SRS was used for the experimental run which was conducted in a similar way to the 1996 run on station 9.3 (section 2.3).

Station 3.4 comprises a water-cooled double crystal scanning monochromator which can cover the energy range 800-3500 eV using different crystal pairs. For our purposes, InSb(111) was used giving a useful energy range of 1680-3000eV. X-ray detection was using ion chambers, in which gas was free to circulate with that of the main chamber. The chamber was filled with 200mbar air for measurements of 2keV or greater, and 180mbar He on top of the 20mbar air remaining in the chamber after pumpdown for measurements under 2keV. The beam was stopped down to around 2mm high and 1mm wide using horizontal and vertical slits close to the sample. The beam divergence was estimated to be 0.04°. The MCP sample could be tilted in the vertical plane to an accuracy of 0.001°, and the entry and exit slits adjustable vertically to 1μm. Energy changes were possible by specifying a monochromator angle, after which the pitch of the top monochromator crystal had to be adjusted to give maximum output flux.

The reflectivity measurement method was again based on equation 2.13.

The Ni coating was applied by Nanosystems Inc. to the MCP using the electroless nickel (EN) deposition technique. In contrast to the electroplating technique, the electroless nickel plating
process, also known as chemical or autocatalytic nickel plating, works without an external current source (Skanaluminium web site, Dec. 1999). The plating operation is based upon the catalytic reduction of aqueous nickel ions on the surface being plated, from a solution containing sodium hypophosphite (NaH$_2$PO$_2$.xH$_2$O), an acid at a pH of 4.5 to 5.0 and a temperature of 85-90°C. The deposits contain between 3-13% phosphorus by weight depending on the chemical composition of the solution and operating conditions. The phosphorus content affects its chemical and physical properties, but will not have a significant effect on the X-ray properties, phosphorus being a low Z element of smaller X-ray cross section than nickel. Once the catalytic surface, in this case glass, is covered by metal, plating will continue, but at a reduced rate. The EN process is especially suited to applications where the sample is irregularly or awkwardly shaped, such as threaded parts, holes or internal surfaces such as the channels of MCP.

It was expected that a thin layer of Ni would be coated onto the basic channel structure of figure 2.6, as in figure 2.11, which would be noticeable in the reflectivity curves. Figure 2.10 shows the reflectivity seen at 1.8keV for the uncoated (E) and coated (F) plate. The kink at 1.1° is a multilayer Kiessig fringe due to the silica layer as explained in figure 2.6. An enhancement in reflectivity at large angles and a reduction at small angles can be seen due to the Ni coating, but
by integration of the areas under the curves the enhancement to reduction ratio is $9.7:2.6$.

Measurements for distinct energies were modelled as three layers and fitted for layer thicknesses and roughness and angular offset (not tabulated) as free parameters using the REX program Crabb et al. (1992, 1993) as shown in table 2.8.

### 2.6 Conclusions

Considering the plates E and F in section 2.5 have come from different boules the fitted roughnesses and thicknesses agree very well and comparing with samples A-D in section 2.3 provides a good correlation for silica layer thickness and roughness.

The acid etching for plates A-D looks to have been more severe than both E and F as the silica layer (3) is thicker; these plates have been reduced so have had more opportunity to lose lead and widen their silica layer. The thinness of nickel coating is to be expected and its roughness is of similar size to the layer thickness, perhaps indicating a patchy coating of Nickel or else a very thin coating – the EN process coats thinly using the glass itself as the reaction catalyst, so when covered, deposition slows. Little weight should be placed on the high roughness of the bulk glass layer – the model’s sensitivity is poor to changes of roughness at such depths from the outer surface. Figure 2.12 shows the effect of changing the nickel layer thickness on reflectivity modelled using parameters similar to those fitted in table 2.8. The shape of the measured reflectivity of MCP F corresponds well when the nickel is taken to have a thickness of 25Å.

It would appear that channel plate glass always has a layer of lead-free silica on its surface. Even plate E, the most unprocessed plate having been etched and not reduced, coated or suffered any other treatment, has a fringe (see figure 2.10; dashed line) which has been modelled to be silica on lead glass. Further, Then and Pantano (1990) and later D’Souza and Pantano (1996) report a silica layer on etched, unreduced lead channel plate glass after SIMS depth profiling which was 20-50nm (200-500Å) thick. Kanunnikova et al. (1995) suggests it is sintering with soluble core glass and etching that is responsible for the reduction in lead in the surface layer as the lead
Figure 2.11: Layered structure of a coated MCP surface. The layers are (1) nickel, (2) alkali metal monolayer, (3) lead-depleted silica layer and (4) bulk MCP glass.

<table>
<thead>
<tr>
<th>Energy (keV)</th>
<th>1.8</th>
<th>2.0</th>
<th>2.3</th>
<th>2.8</th>
</tr>
</thead>
<tbody>
<tr>
<td>MCP</td>
<td>E</td>
<td>F</td>
<td>E</td>
<td>F</td>
</tr>
<tr>
<td>Thicknesses (Å)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Layer 1</td>
<td>-</td>
<td>31</td>
<td>-</td>
<td>23</td>
</tr>
<tr>
<td>Layer 3</td>
<td>231</td>
<td>195</td>
<td>258</td>
<td>195</td>
</tr>
<tr>
<td>Roughnesses (Å)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Layer 1</td>
<td>-</td>
<td>17</td>
<td>-</td>
<td>16</td>
</tr>
<tr>
<td>Layer 3</td>
<td>9</td>
<td>12</td>
<td>7</td>
<td>16</td>
</tr>
<tr>
<td>Layer 4</td>
<td>0</td>
<td>42</td>
<td>13</td>
<td>40</td>
</tr>
</tbody>
</table>

Table 2.8: Values of fitted parameters to the reflectivity data.
atoms diffuse out of the silica glass layer faster than they diffuse through the bulk lead-silicate glass, giving a more abrupt than a gradual boundary as modelled here, and the Kiessig fringes.

The exact chemical or electrochemical process responsible for the lead removal is far from clear. Kanunnikova et al. (1995) suggest diffusion, Then and Pantano (1990) suggest electron stimulated desorption (ESD) and electromigration whereby ions are moved electrically by externally applied field from primary or secondary electrons. Alternatively, the soluble core glass could leach the heavy ions from the cladding or the acid etch treatment might remove them.

In any case, the surface of MCP channels appear to be a lattice of silica which lead ions used to populate. The departure of lead is undesirable as it is just the material required for high reflectivity at high grazing angles, but if it is the acid etch or core glass removing it, is it difficult to see an alternative. The success of the nickel coating by the electroless technique is a major breakthrough. Nickel’s reflection characteristics are comparable with those of gold (see figure 2.13), and it does not readily deteriorate after contact with air. Manufacturers should examine
Figure 2.13: Comparision of Nickel and Gold (with 0Å surface roughness) as reflectors at an X-ray energy of 1.8keV.

this technique in detail if progress is to be made in MCP focusing, particularly at high energies.
Chapter 3

Miscellaneous X-ray Optic and Detector Tests

3.1 Introduction

This chapter describes a number of tests undertaken during the PhD on channel plates' form and function or alternative X-ray focusing technologies. Section 3.2 describes the use of the Leicester long beam facility to test Lobster eye optics fabricated by a competing technology to glass MCPs. Sections 3.3 and 3.4 describe investigations of MCP detector technology, while section 3.5 briefly describes some metrology on glass MCP optics performed under the auspices of the CASE studentship and the ESA TRP.

3.2 Hudec optics

The Lobster eye X-ray optic geometry can be realised not only using slumped, square pore MCPs, but also by stacking parallel gold coated flats. This section describes X-ray testing of such a “competitor” optic.

R. Hudec* and A. Inneman* from the Czech Academy of Sciences visited Leicester to test their replicated Lobster eye and Wolter type 1 X-ray optics (Inneman et al., 1999) in the Leicester

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X-ray beamline. The principles of these focusing techniques are described in chapter 1.

The Leicester tunnel test facility (TTF) is a 20m long vacuum beamline with a 1.5m diameter, 2m long detector/optic chamber. It is evacuated to a pressure of $10^{-8}$mbar by a helium cryopump at the detector chamber end and a turbomolecular pump at the source end. A $100 \times 100 \text{mm}^2$ MCP detector with a resistive anode readout (Lees and Pearson, 1997) is used to acquire X-ray images; the source was an oil-cooled, 0-5kV filament device with a Cu anode. Collimation was provided with a 1mm pinhole placed immediately in front of the source.

By dint of the large (~mm) spacing of the reflecting surfaces, the Czech Lobster eye optic could focus visible light as well as X-rays, giving it a considerable advantage in optical alignment, compared to MCPs.

### 3.2.1 Lobster eye optic

![Lobster eye optic](image)

**Figure 3.1:** The Lobster eye optic, shown with the two optical elements side by side instead of back to back, and with the case removed.

The “Lobster Eye”, or more correctly, Kirkpatrick-Baez optic, shown in figure 3.1, consisted of two perpendicular arrays of 36 and 42 double-sided flats, each $100 \times 80 \text{mm}$, and spaced by about 2mm (as in figure 1.10). The flats are float glass squares, 0.3mm thick and gold coated on
both sides. The focal length is 400mm from the midplane, and the field of view is about 6.5°. The device is 220mm long, 140mm wide, 150mm high, and weighs about 660g without its case.

The optic was positioned in the TTF and aligned in air using a filament light bulb as a source. Figure 3.2 and 3.3 show the best Cu $L_\alpha$ (0.93keV) X-ray focus found by remotely moving the optic in the Z (beam-axial) direction. The gain, defined as the ratio of the flux level within the FWHM to that of the straight through flux (as seen in the first small grey area immediately to the left of the true focus in figure 3.2) was 142. The width of the true focus was determined to be 2.22mm FWHM, which compares well with the 2mm inter-flat spacing, theoretical minimum.

The 2mm focus corresponds to an angular resolution of 5mrad (or 17arcminutes), which even considering the flat spacing could be reduced to perhaps 1mm, is poorer than the 2mrad (or 6arcminutes) FWHM demonstrated for MCP optics already (Brunton et al., 1995). Reducing the focal size by decreasing the plate spacing would be at the expense of the open area of the lobster eye optic, which falls quickly (75% for 2mm to 60% for 1mm spacing). The mass of the optics alone without support (660g) compares badly with a similar sized MCP (100g).

3.2.2 Wolter type 1 optic

The Wolter type 1 optic was also made by the Astronomical Institute of the Czech Academy of Sciences. The device was 130mm long and 50mm in diameter, and had a single mirror shaped to the Wolter type 1 configuration (see figure 1.11) made by the electrodeposition of nickel onto a highly polished glass former, which had been removed. The nickel shell was then gold plated. The focal length was about 450mm (a similar length to the Kirkpatrick-Baez arrangement of the previous section) and was aligned in a similar manner to that of the Lobster eye optic of the previous section using visible light. Figure 3.4 shows the best focus, again found by moving the optic in the Z direction. The gain is the ratio of the maximum flux of the focused X-rays, divided by the unfocused flux level – not visible in the figure – and was 1040.

The central focus was 0.54mm which corresponds to 1.2mrad (or 4.1arcminutes) FWHM – a big improvement compared to the 5mrad of the Lobster eye optic of the previous section, and an
Figure 3.2: The focus of the replicated Lobster eye optic made from two orthogonal sets of coated glass flats. The true focus is the point at the centre of the cross and is made from photons that have been reflected by both sets of plates; photons that have passed through the lens undeviated form the light grey chequered background, whereas those that have been reflected by only one set of plates contribute to the darker crossarms. Double and triple reflections can be seen starting at the periphery of the image.

Figure 3.3: An intensity cut through the above image, binning events in the box shown.
appreciable improvement on the 2mrad of MCPs to date. However, from an aperture of 50mm, only an annulus of width of a few mm was available for focusing, putting the open area fraction at around 0.2 (MCP open area fractions are around 0.65). Nesting of Wolter 1 shells is possible for increased collecting area, but alignment errors will increase the focus size.

These optics appear to function very satisfactorily, and the Wolter type 1 device looked rugged and strongly built. Indeed, the Academy of Sciences is marketing the optics commercially with Bede Scientific Instruments.

### 3.3 Acid etch MCP metrology

The University has built a channel plate detector for the Joint Astrophysical Plasmadynamic Experiment (JPEX) (Bannister et al., 1999), an NRL sounding rocket payload to study the EUV spectrum from the helium atmosphere of a particular white dwarf. The quantum efficiency (QE) defines the sensitivity of a channel plate detector, and as flux and observation time were limited, its increase was desirable.

Following the paper by Hemphill et al. (1997), which describes an improvement, by a factor of ~2, in quantum efficiency in the 256Å to 1024Å band, of detector plates when treated with 12M nitric acid, we undertook a reconstruction of the experimental treatment in full. A 12µm round pore, L/D=40:1 circular MCP, manufactured by Galileo, and supplied to us by NRL, was sacrificed for the experiment. A preliminary experiment to test the etching protocol was carried out on 1 July 1997 in the Chemistry and Physics departments of the University.

The MCP was cracked into two roughly equal parts – one for a control, and one to be treated. Then the experimental sequence described by Hemphill et al. (1997) was performed;

- The two plate halves were solvent cleaned and dried.
- One half was placed in 12 molar nitric acid, at 80°C for 3 hours.
- After removal, it was rinsed with distilled water several times.
**Figure 3.4:** The Wolter optic focus. The true, central focus is from rays reflected twice and the 'halo' around it, from single reflections. The arc in the image is caused by photons shining through the optic without being reflected, showing the optic is not mounted exactly co-axially to the X-rays. The two nicks in the arc are shadows of supports holding the reflecting surface.

**Figure 3.5:** The cut of the image above, binning events in the box shown. The FWHM of the central focus is 0.54mm or 4.1arcminutes.
Figure 3.6: Untreated plate at low magnification. Scale is indicated by the 2.94μm tick mark

Figure 3.7: Acid etched plate at low magnification. Scale is indicated by the 5.29μm tick mark
Figure 3.8: Untreated plate at high magnification. Scale is indicated by the 730nm tick mark

Figure 3.9: Acid etched plate at high magnification. Scale is indicated by the 1.61μm tick mark
• Then it was rinsed in an IPA/methanol 50:50 mixture.

• The two halves were vacuum baked at 120°C for 2 hours. Details of the vacuum bake were inferred from the paper by Siegmund (1989).

The test was successful, in that the MCP structure survived the rigorous treatment mechanically intact. Both fragments were imaged in the Scanning Electron Microscope (SEM) in the University Medical School. The pictures, first at low magnification, show little or no difference in either structure or surface quality – see figure 3.6 (untreated) and figure 3.7 (treated). The treated fragment had not suffered mechanical breakdown or cracking.

On further magnification, (see figures 3.8 and 3.9) it is possible to infer that the acid has had some effect in removing small surface blemishes; the untreated plate has more ~100nm particles on its surface. Additionally, the treated plate looks to be slightly smoother, but it is hard to say for sure. One hypothesis for the improvement in EUV quantum efficiency is that the acid roughens the MCP channel surfaces, reducing reflectivity and increasing the chances of secondary electron emission. The test proved that channel plate glass is capable of surviving the harsh chemical treatment asked for mechanically, and with physical change only visible on very small scales.

Absolute QE measurements were taken by Bannister et al. (1998) using a calibrated channel electron multiplier and a stacked channel plate detector. The lower plate was untreated, and the upper plate subjected to an identical treatment as above. They found that QEs at four wavelengths (161 Å, 256 Å, 304 Å and 584 Å) showed no significant change following the acid processing, but as they note, the plates are made of differing glass types – their plates, as ours, were of Corning 8161 glass, and Hemphill et al.’s were of Philips 297 composition. More investigation will be needed to understand the effect of acid etching on channel plate function.

### 3.4 Heat sunk detector

The operation of conventional MCP detectors at high count rates, encounters the problem of current limitation in the channel walls, which lowers plate gain with increasing count rate (Trem-
A detector using a low resistance (~1 MΩ compared with the more usual 50 MΩ) plate bonded to a large heatsink can support a larger current, but inevitably gets hot, and with channel plate glass having a negative temperature coefficient of resistance, runs the risk of “thermal runaway” – local melting of the channel matrix.

Two detectors, shown schematically in figure 3.10 and manufactured by Nova Scientific Inc. were supplied for testing at high rates under UV illumination in Leicester. The MCP in the first detector cracked and partially separated from the lower electrode due, we assume, to poor thermal contact between the MCP and the heatsink causing it to expand and break. The second detector with an improved bonding glue and a 0.5 mm thick, 40:1, 10 μm, round pore circular MCP, was mounted in the MACS vacuum system and illuminated with a mercury vapour UV lamp, focused through a sapphire window 300 mm from the detector. A 10 mm diameter spot on the detector produced events which were counted on the high voltage supply connections. Figure 3.11 shows the change in resistance with voltage increasing and decreasing. It should be noted that the data was taken as quickly as possible (35 minutes) to prevent a large rise in the detector’s temperature – the rise was limited to 24.0°C to 26.0°C as measured by a thermocouple on the heatsink – but the small difference in resistance between the rising and falling voltage is a consequence of that rise. Plate resistance at 0.5 V, as measured by a digital multimeter, was found to be 1.15 MΩ, and maximum power dissipation was 4.25 W at 1700 V.

Figure 3.12 shows the count rate varying strongly with operating voltage as expected (Trem-sin et al., 1996, p143). The noise level was 9 counts per sq. cm per second at 1600 V.

Because it was suspected that changes in temperature and resistance might cause count rate instabilities, these parameters are plotted against time in figure 3.13. From cold, the detector was
quickly powered up to 1600V, and kept there for 100 minutes while being illuminated with the UV lamp. No changes were made to the equipment during the 100 minutes, during which the power dissipated in the detector rose from 3.55W to 3.76W. As expected, the temperature shows a logarithmic rise. It might be expected that the count rate would show a similar increase, but at 20 minutes the rate quickly slowed, showing little previous or subsequent change.

When the voltage across the plate was raised to above 1800V in an attempt to increase the count rate still further, the front electrode became detached from the plate bringing the tests to an end.

The bonding method is of concern as both detectors were disabled by its failure, but otherwise they survived the increased temperature without mechanically distorting, melting or thermally running away.

### 3.5 Long etch test

In the first six months of 1998, a wedge of scrap 10μm round pore channel plate glass was left to etch in a corner of the weak acid bath usually used by Photonis to etch all the MCPs at Brive. The wedge was 62mm long and 22mm wide, 7mm thick at its thin end, rising to 21mm at the thick end, corresponding to a range of aspect angles of 700:1 to 2100:1. The MCPs required for
Figure 3.12: Counts rate per unit area vs bias voltage.

Figure 3.13: Temperature, Resistance and Counts per second vs Time.
The construction of a high aspect (up to 500:1) ratio MCP has been necessary for the high energy (up to 100keV) focusing requirements of the ESA TRP driven by X-ray astronomy needs. Etching is clearly only one of the hurdles to be overcome in this goal, but its viability has to be proven.

SEM photographs of the thinner end showed channels etched normally, with no sign of blocking or over-etching. The thicker end exhibited blockage after about 300:1 (figure 3.14) and some intriguing crystals after that (figure 3.15), but no over-etching.

On the evidence of this work, etching of a 500:1 plate would be expected to be possible, though maybe taking longer than usual, as it is 250:1 from each end, and etching to 300:1 is satisfactory. The removal of dissolved core glass appears to be the principal problem that halts channel etching, as it can be seen core glass has been dissolved but not carried away in figure 3.15 – indeed recrystallisation has occurred here. Once channels have “broken through”, liquid can pass freely along them, clearing the used, saturated acid for fresh – Photonis use the rule of thumb that the etching time should be three times the “break through” time. This cannot occur in blocked channels. Old acid should be regularly changed for fresh in the bath, and flowing it over the MCP
would help circulation. In extreme cases, removal and drying of a plate would force fresh acid along the channels. Smaller pore sizes will inevitably make the situation worse.

### 3.6 Conclusions

The Lobster eye optic of Hudec demonstrated considerably worse performance than that already achieved by MCP focusing technology (focus FWHM size of 5mrad compared to the MCP's 2mrad), as well as being much larger and heavier so cannot seriously be considered to be an alternative. The Wolter type 1 optic performed better, but still at a considerable mass disadvantage to MCPs. Wolter type 1 optics continue to be the lens of choice of astronomical observatories, but at the cost of very heavy shells and the associated support structure. Channel plates can offer very considerable advantages here.

The acid etch experiment’s findings, which were contrary to those of Hemphill et. al., appear to be backed up by recent work by Bannister et al.. The area of QE improvement by treatment of glass is poorly understood and more work is underway at the University in regard to this.
Chapter 4

Slumping of Microchannel Plates

4.1 Introduction

The slumping of channel plates is essential in X-ray astronomy as flat plates have the severe limitation of only being able to focus to a point equidistant from the plate as the source (as in figure 1.3). Clearly, a source at infinity, such as a star, cannot be focused by such a plate (figure 1.4). As part of the ESA Technology Research Programme (TRP), the University and its partners have been developing hard X-ray MCP optics, and spherically curved or slumped MCPs form a significant part of the programme (see chapter 1).

The ability to slump channel plates opens up the possibilities of realising both the Kirkpatrick and Baez (1948) and the Wolter type 1 (Wolter, 1952a,b) X-ray lens configurations, the latter with two optical elements of different slump radii placed next to each other. With lightweight channel plates, rather than the heavy shell optics used on the Chandra, XMM and other orbiting observatories, X-ray telescope missions could become considerably more versatile in terms of bandpass and field of view. However, the slumping of channel plates has a very limited history, and the TRP requirements are unlike those tried before.

4.2 Summary of previous slumping efforts

Attempts to construct slumped X-ray optics in the past fall into two categories; curved glass strip reflectors and curved channel plates.
4.2.1 Bending glass strips

Ehrenberg (1947, 1949) first suggested that glass could be used, especially when coated with a metal (e.g., gold), to reflect X-rays in one dimension to a line focus. He successfully tried bending thin glass strips (40 x 15 x 4.5mm) by applying a constant screw pressure as in figure 4.1. The glass was to take the form of a cylinder ($R > 20m$ between the two supports) and its focal length was adjusted by altering the screw’s position.

![Figure 4.1: Ehrenberg's glass bending apparatus](image)

Following Kirkpatrick and Baez’s invention of their optical system using orthogonal mirrors to produce a point focus with minimum distortion, Lucht and Harker (1950) made a microscope from elastically bent glass in a Kirkpatrick-Baez configuration with each mirror having a radius of curvature of about 20m. Again, a constant force was applied from a screw to a rod central to the plate to deform it. In the end, these authors decided to use tungsten carbide instead of glass for its superior X-ray reflecting properties, but still elastically bent it in the same manner.

Gorenstein et al. (1971) investigated the Cygnus loop supernova remnant from a sounding rocket using eight glass strips, bent to parabolae by mounting them directly onto a machined metal former (see figure 4.2, after Miyatani et al. (1981)). Such systems are unsuitable for microchannel plates with at least low energy X-rays, as the mounting obscures the X-rays from passing through the plate, but higher energy rays might only be weakly attenuated.

Underwood (Underwood and Turner, 1977; Underwood, 1978) described how a strip could be bent to any shape by suitable width profiling and application of end couples. None of these researchers discussed bending MCPs or even bending a single glass sheet to a spherical profile rather than a strip to a cylindrical one, and all left the glass under stress without annealing it.
4.2.2 MCP slumping

The concept of bending channel plates to provide them with a finite focal length relies on the fact that the plate is curved spherically, but the channels stay straight, parallel and radially directed. In 1992, David Emberson of Philips Components, Mitcham (now Photonis) succeeded in slumping 0.5–4mm thick, 46mm diameter round pore etched channel plates to a 1.4m radius by elastically deforming them with pressure delivered via an annular metal spring with screw pressure, over a curved steel former. Then, to remove stresses internal to the glass, the jig, with channel plate under load, was heated to anneal the cladding glass for 6 to 9 hours; after cooling the radius of curvature remained in the channel plate (Emerson, 1994). Emberson’s apparatus is depicted in Figure 4.3.

These round pore plates were tested in Leicester (Fraser et al., 1993a,c) in beam expander mode giving X-ray results broadly in line with prediction from Monte-Carlo models. It is not known to what extent distortions were introduced to the channels by dint of slumping etched plates, as opposed to a solid pre-etch microchannel plate slice, but relaxation of the glass, an effect studied
by Underwood (1978, p50) would not be significant following annealing because the glass is no longer under stress.

In 1995, the first square pore focusing results (Brunton et al., 1995) on a Nova Scientific 21 × 21mm², 11μm, 75:1 plate slumped after etching to a 1m radius were poor, because the plate had significant corner radiusing – a manufacturing defect whereby the channel corners are not square but rounded. An MCP will obey the lens equation;

$$\frac{1}{l_s} - \frac{1}{l_i} = \frac{2}{R}$$  \hspace{1cm} (4.1)

where \(l_s\) and \(l_i\) are the source and image distances respectively, and \(R\) the radius of MCP curvature. Figure 4.4(a) shows the image from an illumination of the plate from \(l_s=464\)mm and with \(R=1\)m, almost satisfying the point source to parallel beam condition to make \(l_i=\infty\) and (b) has the Monte Carlo ray trace simulation of an ideal plate, showing reasonable agreement with experiment. Profilometer investigations revealed that the plate was slumped accurately to within \(\sim 1\mu\)m RMS.

![Figure 4.4](image_url)

*Figure 4.4:* (a) An image from Nova Scientific MCP FE-7 slumped to a radius of 1m in beam-expander configuration with C-K illumination and (b) a simulation using an ideal MCP in the same configuration.
Figure 4.5 shows two images using the same Nova Scientific plate (reference FE7) firstly (a) unslumped in point-to-point configuration and secondly (b) after slumping, in focus in the long beam line facility in Leicester. The similarity between the images is striking, showing the slumping has had little or no effect on the image quality.

**Figure 4.5:** Full face illumination of the same Nova Scientific plate before (left) and after (right) slumping to a radius of 1m. Straight though rays are cut off in the right image as the plate is not exactly on axis.

For thicker plates, or plates of small radii of curvature, bending stresses internal to the glass will exceed its breaking stress (see section 4.4). In such situations, a multistage (load and anneal ...) process can be envisaged to reach the final form.

The rest of this chapter is concerned with profile measurements on slumped channel plates (section 4.3), then a feasibility assessment of the slumping of thicker plates to tighter radii (section 4.4) and finally some other ideas concerning slumping.

### 4.3 Measurement of slumped MCP profiles

Profile measurements of MCPs have been made using the University’s Engineering Department’s Surfcom stylus profilometer. The profilometer drags a tip of known radius (typically 0.1mm)
lightly across the surface to be analysed, and vertical movement is detected and plotted on a continuous paper output. The horizontal scan rate and vertical gain are adjustable, as are screws to translate the sample in the scan (x) and vertical (z) directions, and tilt in the xz plane. Data is read from the paper trace and entered into more conventional software for analysis and plotting. Unfortunately, this instrument has been designed to measure surface roughness and the large changes in height that can occur with tightly slumped plates can go offscale.

Square plates are scanned by orthogonal profilometer scans, each parallel to a side of the plate, and passing through the plate’s centre to within an accuracy of 0.5mm. Circles are fitted to the data using the following method;

A circle is;

\[
x^2 + y^2 = R^2 \tag{4.2}
\]

\[
y = \sqrt{R^2 - x^2} \tag{4.3}
\]

By binomial expansion;

\[
y = R \left(1 - \frac{x^2}{2R^2} - \frac{x^4}{8R^4} - \frac{x^6}{16R^6} - \cdots \right) \tag{4.4}
\]

Assuming \(x\) is small compared with \(R\), then only terms up to \(x^2\) are important which gives;

\[
y = R - \frac{x^2}{2R} \quad \text{For a circle centred on the origin}; \tag{4.5}
\]

\[
(y - q) = R - \frac{(x - p)^2}{2R} \quad \text{For a circle centred at } (p,q) \tag{4.6}
\]

Expanding;

\[
y = R + \frac{-x^2 + 2xp - p^2}{2R} + q \tag{4.7}
\]

The \(x^2\) coefficient is equal to \(\left(-\frac{1}{2R}\right)\), so \(R\) can be calculated, once the profilometer scan data is fitted with a polynomial fitting routine. The slump radius \(R\), measurement radius \(a\) and saggital depth \(w\) are related by;

\[
R = \frac{1}{2} \left( w + \frac{a^2}{w} \right) \tag{4.8}
\]

\[
R \approx \frac{a^2}{2w} \tag{4.9}
\]
Knowing the reading error in $w$ (the profilometer’s height), we may plot minimum and maximum radius curves (see figure 4.6) to yield the total error.

### 4.3.1 Spherical reconstruction of Nova Scientific MCPs

The detailed figure of a slumped channel plate was examined on a plate slumped by Nova Scientific using a proprietary process. 18 × 10 horizontal scans were meshed to a surface, compared with a sphere of the target slumped radius (0.5m) and to two single, orthogonal scans – figure 4.7. The overall shape is spherical, but the radius of curvature is not the same in the two orthogonal scans shown in the figure.

Three other, ostensibly identical plates were measured by the selfsame method with the results in table 4.1. For each plate, the radii of the two orthogonal arcs are close in value; well within experimental error. However, there appear to be two distinct pairs in the set of four MCPs, the first with radii at, or very near, 500mm and the second with radii of about 540mm. The reason for this grouping is not clear and more work is required to perfect the manufacturing process.
Figure 4.7: Top: Contour plot of a slumped Nova Scientific MCP (left) and a sphere of radius 500mm (right). The vertical separation between successive contours in both figures is 8µm. Bottom: Orthogonal scans through the centre of the same plate. The respective fitted radii are indicated. One scan has been offset vertically from the other for clarity.
<table>
<thead>
<tr>
<th>Plate Number</th>
<th>Orientation</th>
<th>Radius (mm)</th>
<th>error ± (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>11-13D</td>
<td>1</td>
<td>537</td>
<td>16</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>549</td>
<td>16</td>
</tr>
<tr>
<td>11-6A</td>
<td>1</td>
<td>505</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>497</td>
<td>15</td>
</tr>
<tr>
<td>11-7B</td>
<td>1</td>
<td>491</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>495</td>
<td>15</td>
</tr>
<tr>
<td>11-12C</td>
<td>1</td>
<td>546</td>
<td>16</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>540</td>
<td>16</td>
</tr>
</tbody>
</table>

Table 4.1: Profilometer slump radii of four nominally identical channel plates. The plate in figure 4.7 is 11-13D.

4.3.2 Photonis 70mm slumped plates

We received four channel plates which had been slumped by Ray Fairbend of Photonis in Brive for Oswald Siegmund of Space Science Lab, University of California, Berkeley, as part of the FUSE MCP detector development programme (one of these plates can be seen in figure 1.2). The method of slumping was as described in section 4.2.2 above (Figure 4.3), but with a concave–convex system of steel formers rather than the steel spring and convex former. Additionally, a sacrificial plate was placed between the target plate and the convex former to spread the bending load and prevent cracking of the target plate. Often damage was seen on this scrap plate at the point of first contact.

The convex curvature profile was measured using the Leicester University Surfcom stylus profilometer, described in section 4.3, to calculate the curvature and corresponding error.

Due to the very small radius of curvature (70mm) that the plates have been slumped to, the newer, more accurate profile equipment in the Engineering department could not be used – the range of heights to be measured is larger than the maximum deflection of the scanning head which has been designed to measure surface roughnesses. The older machine can tolerate a large deflection...
Table 4.2: Description of Photonis 70mm slumped plates measured for curvature

<table>
<thead>
<tr>
<th>Plate</th>
<th>Appearance</th>
<th>Channel length (mm)</th>
<th>Plate diameter (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Darkest (<em>reduced?</em>)</td>
<td>1</td>
<td>46</td>
</tr>
<tr>
<td>B</td>
<td>Slightly pink (<em>unetched?</em>)</td>
<td>1</td>
<td>46</td>
</tr>
<tr>
<td>C</td>
<td>Clear – with scribed arrow</td>
<td>0.5</td>
<td>45.5</td>
</tr>
<tr>
<td>D</td>
<td>Clear – with scribed square</td>
<td>0.5</td>
<td>45.5</td>
</tr>
</tbody>
</table>

(up to about 0.35mm), but can still only profile the central 13mm of the 45mm diameter plates. A more complex 4 or 5 scan “montage” can be envisaged, but this was ruled out as being too time consuming and complicated. The plates are described by appearance in table 4.2.

As noted above the slumping process involves using a second, sacrificial plate to protect the primary plate from excessive stresses. Plates C and D may have had this function. A small arrow and square were scribed on the perimeter of the plates to distinguish between them. Plate B looked to be unetched and had the pinkish colour of the core glass. Plate A was significantly blacker than the rest, probably denoting its having been etched and reduced.

Figure 4.8 shows the results of the scans compared with the desired radii of curvature (70mm). A circle of 70mm radius is also shown displaced for profile comparison (dotted). The two scans on each plate (showed as solid and dashed lines) are orthogonal, but cannot be correlated between the plates as there is no circumferential reference point. Table 4.3 summarises the derived radii of curvature for all plates.

The radius error was calculated to be ±1.7% of 70mm, or about ±1.2mm by the method described in Brunton *et al.* (1999).

Plates A and D have achieved the required radius in both axes within the measurement error. Plate B has been curved slightly too much, but plate C is over-slumped by a significant amount.

These results are encouraging as they indicate that the degree of accuracy obtainable in slumping plates of such small radii is good. More work will be needed to measure curvature of the entire
Figure 4.8: Talysurf scans of the four 70mm channel plates. The dotted line is a circle of radius 70mm, offset vertically by -0.1mm for clarity. The solid and dashed lines are orthogonal scans across the plate.
<table>
<thead>
<tr>
<th>Plate Number</th>
<th>Orientation</th>
<th>Radius (mm)</th>
<th>error ± (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A 1</td>
<td>70.0</td>
<td>1.2</td>
<td></td>
</tr>
<tr>
<td>A 2</td>
<td>68.5</td>
<td>1.2</td>
<td></td>
</tr>
<tr>
<td>B 1</td>
<td>66.0</td>
<td>1.2</td>
<td></td>
</tr>
<tr>
<td>B 2</td>
<td>68.6</td>
<td>1.2</td>
<td></td>
</tr>
<tr>
<td>C 1</td>
<td>59.3</td>
<td>1.2</td>
<td></td>
</tr>
<tr>
<td>C 2</td>
<td>57.2</td>
<td>1.2</td>
<td></td>
</tr>
<tr>
<td>D 1</td>
<td>70.6</td>
<td>1.2</td>
<td></td>
</tr>
<tr>
<td>D 2</td>
<td>70.2</td>
<td>1.2</td>
<td></td>
</tr>
</tbody>
</table>

Table 4.3: Measured radii of curvature of plates described in table 4.2, slumped to a nominal radius of 70mm.

plate, or to measure the concave side, if required.

### 4.4 Slumping of thick plates to large radii

The ESA TRP programme requires the construction of a Wolter type 1 configuration optic for the imaging of high energy (10–100keV) X-rays. The Wolter type 1 configuration (Aschenbach, 1985) has 2 optic elements, mounted back to back (as in figure 1.20) whose radii are $R$ and $R/3$, where the focal length of the combination is $R/4$. Unfortunately, the maximum grazing angles that high energy X-rays allow are very small (< 0.5°), so only plates slumped to a large radii will be able to focus radiation by grazing incidence reflection. This makes for long telescopes, and more importantly from the slumping point of view, thick MCPs to give a good field of view. To fabricate this arrangement, it has been decided to attempt to slump, in the first instance, plates of the sizes given in table 4.4.

The problem that is likely to be the limiting factor for Emberson’s (or any other slumping method) is if stresses internal to the glass rise above the breaking stress of the glass and fracture the plate.
### Table 4.4: Thick MCP dimensions

<table>
<thead>
<tr>
<th>Slump radius (m)</th>
<th>Radius of circular MCP (mm)</th>
<th>Channel length (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>30</td>
<td>5</td>
</tr>
<tr>
<td>10</td>
<td>30</td>
<td>5</td>
</tr>
<tr>
<td>6.67</td>
<td>20</td>
<td>5</td>
</tr>
</tbody>
</table>

### Table 4.5: Glass parameters according to different sources

<table>
<thead>
<tr>
<th>Young's modulus</th>
<th>( \nu )</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>( 5.4 \times 10^{10} \text{Pa} )</td>
<td>0.20</td>
<td>Shand (1958, p37) For lead glass</td>
</tr>
<tr>
<td>( 6.0 \times 10^{10} \text{Pa} )</td>
<td>0.20</td>
<td>Philips ‘G12’ type glass data sheet</td>
</tr>
<tr>
<td>( 5 \times 10^{10} \text{Pa} )</td>
<td>0.30</td>
<td>Emberson (1994, in his calculations)</td>
</tr>
</tbody>
</table>

The breaking stress for the glass is cited as; 41–103MPa (or \( 6–15 \times 10^3 \text{psi} \) by Shand (1958, p17)), or 50–90MPa (or 500–900bar by Emberson (1994, p7)). To calculate the internal stress, two bending cases were considered:

- A freely supported thin plate, bent under an evenly applied pressure
- A freely supported thin plate, bent under the pressure of a single force applied at the centre

The slumping method tried already will start using the second, and finish using the first of these cases as the plate elastically bends. For the following calculations, \( E \), the Young’s modulus of glass is taken to be \( 5 \times 10^{10} \text{Pa} \), and \( \nu \), the Poisson ratio, as 0.3.
4.4.1 Even loading of a disc.

From Benham et al. (1987, p447), the deflection obtained by evenly loading a freely supported thin disc, \( w \), is given by;

\[
\begin{align*}
\frac{w}{a^2} &= \frac{5 + \nu}{1 + \nu} \frac{pa^4}{64D} \quad (4.10) \\
\frac{w}{2R} &= \frac{a^2}{2R} \quad \text{if } a < R \quad (4.11) \\
D &= \frac{EL^3}{12(1 - \nu^2)} \quad (4.12) \\
\sigma_{\text{max}} &= \frac{3}{8L^2} (3 + \nu)pa^2 \quad (4.13)
\end{align*}
\]

where;

\[
\begin{align*}
\nu &= \text{Poisson's ratio} \\
p &= \text{pressure on the plate (Pa)} \\
a &= \text{radius of plate (m)} \\
D &= \text{flexural rigidity (Nm)} \\
L &= \text{thickness of plate or channel length (m)} \\
E &= \text{Young’s modulus of glass (Pa)} \\
R &= \text{radius of curvature of the plate (m)} \\
\sigma_{\text{max}} &= \text{maximum stress as a result of } p \text{ (Pa)}
\end{align*}
\]

Simplifying these equations gives;

\[
\sigma_{\text{max}} = \frac{(3 + \nu)E}{(5 + \nu)(1 - \nu) \cdot R} \cdot L \quad (4.14)
\]

which is independent of \( a \), the plate radius.

For values of interest, this gives the results in table 4.6 c.f. breaking stress of glass \( \sim 70 \text{MPa} \). The stress of 635MPa should have been easily sufficient to fracture the MCPs of section 4.3.2, but the slumping method used used by Fairbend, used two channel plates, as explained above, to lessen the stress. Emberson’s thicker plate didn’t fracture either, but it was etched which, perhaps, might give it a higher Young’s modulus.
<table>
<thead>
<tr>
<th>$L$ (mm)</th>
<th>$R$ (m)</th>
<th>$\sigma_{\text{max}}$ (MPa)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>20</td>
<td>9.62</td>
<td>MCPs for Wolter optics</td>
</tr>
<tr>
<td>5</td>
<td>10</td>
<td>19.2</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>6.67</td>
<td>28.9</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>1.4</td>
<td>31.9</td>
<td>Emberson’s trial plates</td>
</tr>
<tr>
<td>4</td>
<td>1.4</td>
<td>128.0</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>0.07</td>
<td>635</td>
<td>Plates described in section 4.3.2</td>
</tr>
</tbody>
</table>

Table 4.6: Stresses ($\sigma_{\text{max}}$) calculated to exist inside plates of thickness $L$ slumped to a radius of $R$.

### 4.4.2 Local loading of a disc

For the case of a single deforming force, first the situation of the force being applied in a small area central to the plate is discussed, the profile of the plate’s curvature derived, and then the effect of widening the contact area is investigated.

From Benham et al. (1987, p450);

$$w = \frac{F}{8\pi D} \left[ \frac{3 + \nu}{2(1 + \nu)} \left( a^2 - r^2 \right) + r^2 \ln \frac{r}{a} \right]$$

(4.15)

where $r$ is the radial distance from the plate centre (m), $F$ is the force applied to the centre of the plate (N) and $D$ is the flexural rigidity (Nm).

Figure 4.9 shows the profile produced from loading the plate enough to give a central deflection corresponding to $R=1.4$m. Also shown (dashed) is a circle of radius 1.4m. The profile, as expected, does not follow that of a circle, but more of a triangle. A channel plate with this profile would not be suitable for focusing. Benham et al. also quote stress values for force applied to a freely supported thin disc at a radius $b$.

$$F = \frac{EL^3w}{a^2c'} \quad w = \frac{a^2}{2R} \quad \sigma_{\text{max}} = \frac{c''F}{L^2}$$

(4.16)
Figure 4.9: *Profile of a centrally loaded circular plate*

where $c'$ and $c''$ are tabulated parameter values Benham *et al.* (1987) for $\nu=0.3$ and varying values of $a/b$. The results for $a/b=5$ are shown in table 4.7, cf. breaking stress of glass $\sim 70$MPa.

These figures show that while it is possible to slump a thicker plate with even pressure over all its surface without exceeding the breaking stress (see section 4.4.1), there might be trouble applying a force just in the centre. The force could build stress to the levels of plate fracture in the central area, but once the plate has started to deform, pressure and stress is spread out and lessened.

Slumping before etching is more likely to be successful because the presence of the core glass will stop channels deforming and becoming non-parallel or not normal to the plate’s surface. The process of annealing is fortunate in annealing only the cladding glass (Philips 297 glass anneals at 445°C) and not the core glass (Philips 274 glass anneals at 545°C). Hopefully this will mean that after annealing the cladding glass has the majority of the bending distortion, leaving the channels square and parallel which is essential for good focusing. It would be wise to apply an extra (*eg* 20%) load after the plate has been bent, but before annealing, to ensure it has taken the shape of the mandrel fully. Any extra load not used for bending at this stage will be harmlessly transmitted through the plate if it is fully in contact with the mandrel. The whole assembly would then be annealed whilst under pressure.
<table>
<thead>
<tr>
<th>L (mm)</th>
<th>a (mm)</th>
<th>R (m)</th>
<th>F (N)</th>
<th>$\sigma_{max}$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>30</td>
<td>20</td>
<td>221.9</td>
<td>20.8</td>
</tr>
<tr>
<td>5</td>
<td>30</td>
<td>10</td>
<td>443.9</td>
<td>41.5</td>
</tr>
<tr>
<td>5</td>
<td>30</td>
<td>6.67</td>
<td>887.8</td>
<td>83.1</td>
</tr>
<tr>
<td>1</td>
<td>23</td>
<td>1.4</td>
<td>25.37</td>
<td>59.37</td>
</tr>
<tr>
<td>4</td>
<td>23</td>
<td>1.4</td>
<td>1624</td>
<td>237.5</td>
</tr>
<tr>
<td>1</td>
<td>18</td>
<td>0.07</td>
<td>507.3</td>
<td>1187</td>
</tr>
</tbody>
</table>

Table 4.7: Calculated values of stress produced in a centrally loaded circular plate by a disc of radius $a/5$

### 4.5 Conclusions

Major progress has been made in the successful construction of slumped channel plates for the focusing of X-rays from 1995 to date, and the technique could be said to be accepted if not mature.

It has been shown glass can be made to a spherical form and slump radii are reproducible to a certain extent. Also, images have shown that the focusing of rays from infinity is possible, opening up the slumped MCP for use in applications usually reserved for shell optics, such as astronomy for which of course, the slumping was developed in the first place. Much work still remains to be done however in the area of slumping, in particular concerning uniformity of radii between and within plates, and the slumping of plates to smaller radii.

The uniformity of slumping (in table 4.1) has an error of up to 10% of the nominal slump radius, but for a given radius of curvature ($R$) and plate radius ($a$) the blur might be expected to be a circle of radius:

$$\frac{2a}{R} \cdot \Delta R$$

where $\Delta R$ is the error from the nominal slump radius. Given that $R \gg a$, the extent of blurring is small; i.e. MCPs have a large depth of field. For the plates measured in table 4.1, $a=10$ mm,
$R=0.5\text{m}$ and $\Delta R=0.05\text{m}$, giving the blur as 2mm or 8mrad. This error is unacceptably large as good MCP foci are of the order of 1mrad. The situation may improve for larger slump radii, but errors should be within a few percent if slumping is not to be the dominant factor in MCP focus size. Radial differences within plates were much less (within the measurement error) which might point to slumping errors being more of a channel plate problem than a slumping problem given that they are all slumped in the same manner.

A "variable lens" can be imagined where the focal length can be changed by tightening or slackening a rim band similar to a Jubilee clip around the circumference of the MCP to alter the radius of curvature. The thick MCPs slumped to large radii could be even constructed as a slumped block by applying an uneven tilting pressure to flow cladding glass along the channels during ram fusion to create radially aligned channels and a block that increases in size along the direction of the channels. The core glass does not flow until a higher temperature, keeping the channel walls parallel and straight.

The thesis thus far has been concerned with MCP construction and performance. Subsequent chapters are concerned with applications of channel plates.
Chapter 5

Imaging X-ray Fluorescence Proof of Concept Experiment

5.1 Introduction to X-ray fluorescence

Fluorescence is the process whereby longer wavelength light is emitted from a material as a result of absorption of shorter wavelength radiation; the emitted wavelengths are characteristic of the elemental composition of the material.

X-ray fluorescence (XRF), when the illumination and emission are in the X-ray band, is a widely used non-destructive technique for chemical analysis. In the simplest geometry, a sample is uniformly irradiated and an averaged fluorescence spectrum obtained from its entire area. One determines which elements are present and in what proportions, but obtains no information on their spatial distribution.

Imaging XRF (IXRF), the construction of 2-d elemental maps, has generally been performed by producing a narrow ($\leq 10\mu m$) tightly collimated beam of primary X-rays, and then scanning the sample on a motorised $x - y$ stage (Morton and Witherspoon, 1992). Such schemes, functionally similar to the elemental mapping performed in a scanning electron microscope, usually rely on a synchrotron source (Iida and Noma, 1995; Vincze et al., 1994) and produce the X-ray microprobe using single capillaries (Yiming et al., 1994; Bilderback et al., 1994; Yiming and Xunliang, 1993; Dozier et al., 1994), ellipsoidal mirrors (Hayakawa et al., 1990; van Langevelde et al., 1990) or grazing incidence Kirkpatrick-Baez X-ray optics (Underwood et al., 1988; Suzuki et al., 1988; Iida and Noma, 1993; Jones et al., 1988).
Rather than having to scan the sample relative to the primary beam, it is obviously attractive to uniformly illuminate the sample and re-image the fluorescence onto an energy dispersive, position sensitive detector, thus producing an imaging XRF spectrometer with no moving parts. Previous workers have used line (Gurker, 1979) and area scanning (Gurker, 1985) and coded mask arrays (Gurker, 1986) to produce images. These methods, although providing good spatial resolution, require fairly complex equipment: the reconstruction of coded aperture images is an involved, computer intensive, mathematical process.

In this chapter, we describe an experiment using a planar, square pore microchannel plate (MCP) to relay fluorescent radiation onto a 2-d charged coupled device (CCD), capable of recording both an image and providing energy information even for low Z elements such as C, O and N.

The picosecond pulsed laser plasma X-ray source at the Rutherford Appleton Laboratory was used as the primary source for the proof of concept experiment described below. The experiment is also described in Martin et al. (1999b).

5.2 IXRF experiment configuration

The experimental arrangement is shown in figure 5.1 and in the photograph, figure 5.3.

UV laser light ($\lambda = 248$nm) enters the target chamber as a stream of $8 \times 7$ps pulses, 2ns apart, and a power density of $\sim 10^{19}$W.m$^{-2}$ (Turcu et al., 1994, 1993). Each picosecond pulse contains an energy of 25mJ and is brought to a 10$\mu$m focus on a slowly moving target tape. At this focus, an X-ray emitting plasma is created with an electron temperature of $10^6$K. The line spectrum of the plasma depends on the elemental composition of the tape. The X-ray spectrum for steel (Fe, Z=26) tape shown in figure 5.2, is composed of lines from mainly Ne-like (Fe XVII) and F-like (Fe XVIII) ions, and a high energy Bremsstrahlung tail extending above 2keV.

The Fe X-rays illuminated the sample, which is at a distance of only 18mm from the plasma, exciting fluorescence isotropically. The microchannel plate optic, manufactured by Nova Scientific, was placed half-way between the sample position and the CCD. The MCP-sample dis-
Figure 5.1: Experimental geometry of the IXRF proof of concept experiment

Figure 5.2: Spectrum of Fe plasma produced by the UV laser. From Batani et al. (1991).
Figure 5.3: Photograph of the IXRF experiment chamber
tance $l_s$ was minimised within the existing experimental chamber to a value of 275mm, making the maximum incident angle to the channel walls 2°, approximately equal to the critical angle for 1keV X-ray reflection (see figure 5.4).

![Reflectivity graph](image)

**Figure 5.4:** Calculated reflectivity of ideal (zero surface roughness) Corning channel plate glass at 1keV. From the method in section 2.2.

In this position, by virtue of the equiangular, grazing incidence X-ray reflection off the interior channel walls, the MCP acts as a relay lens of magnification unity (Chapman *et al.*, 1991), focusing the fluorescent light onto the CCD’s surface. The MCP channel aspect ratio (30:1) gives maximum focusing efficiency at an X-ray energy of ~0.5keV (Chapman *et al.*, 1991). The experimental MCP geometry is summarised in table 5.1.

<table>
<thead>
<tr>
<th>MCP size</th>
<th>20mm×12mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Channel aspect ratio</td>
<td>30:1</td>
</tr>
<tr>
<td>Channel size</td>
<td>8.5μm square</td>
</tr>
<tr>
<td>Open Area fraction</td>
<td>50%</td>
</tr>
<tr>
<td>Manufacturer</td>
<td>Nova Scientific from Corning 8161 glass</td>
</tr>
</tbody>
</table>

**Table 5.1:** Microchannel plate geometry

The continuum emission from the laser plasma extends into the visible and infra-red, so to ensure
that only fluorescent X-rays were incident on the CCD, funnel shaped tubes surrounded the light paths, and all windows in the chamber were covered with thick black PVC.

It was found that two optical filters, each of 1\(\mu\)m polypropylene with 0.1\(\mu\)m evaporated aluminium, fitted to the front of the CCD eliminated visible and ultra-violet light reflected and fluoresced by the sample. A single filter contained pinholes large enough to transmit a quantity of light sufficient to be detected by the CCD. To confine the plasma debris, the main chamber was filled with helium to a pressure of 13 ± 2mbar. The calculated (Cromer and Liberman, 1970) X-ray transmission of the combination of the filters and the helium column is shown in figure 5.5. The composite transmission is dominated by the polypropylene, whose only function is to support the aluminium.

![Figure 5.5: Transmission of helium column and aluminised polypropylene filter (solid curve), and polypropylene alone (dashed curve). Note the narrow window below the C K edge (283eV).](image)

The CCD, produced by EEV under contract to the University of Leicester for the European Space Agency XMM EPIC instrument (Holland et al., 1996), was contained in a liquid nitrogen cooled chamber with its own rotary vacuum pump, separated from the main chamber by a gate valve. It was of a type optimised for low energy X-ray detection, having a front electrode that had been partially etched away to allow X-rays to penetrate through it into the semiconductor region below. Specifications of the device are given in table 5.2 and the quantum efficiency curve in figure 5.6. Note that the low value of the read noise gives this device, when operating in cooled, slow-
scan mode, a photon counting capability as low as Boron in atomic number ($K_\alpha$ emission energy 183eV, corresponding to a mean number of signal electrons of approximately 48). Full energy resolution in the CCD is only achievable if the photon arrival rate is less than one per pixel per frame – if two photons land in the same pixel in the same frame, their individual energies are lost, a condition known as pile up.

<table>
<thead>
<tr>
<th>Pixel Number</th>
<th>820×1024 in full frame mode</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pixel size</td>
<td>27μm square</td>
</tr>
<tr>
<td>Pixel type</td>
<td>Open electrode</td>
</tr>
<tr>
<td>Operating Temperature</td>
<td>-100°C</td>
</tr>
<tr>
<td>Read noise at -100°C</td>
<td>4.5 electrons rms</td>
</tr>
<tr>
<td>Energy resolution</td>
<td>10% at 1keV, 2% at 6.5keV</td>
</tr>
<tr>
<td>Energy range</td>
<td>180 – 10000eV</td>
</tr>
<tr>
<td>Readout time</td>
<td>16s</td>
</tr>
<tr>
<td>Manufacturer</td>
<td>EEV</td>
</tr>
</tbody>
</table>

**Table 5.2: CCD specification**

### 5.3 IXRF experiment modelling

Using the low energy optimised CCD, it was our aim to detect low energy K shell X-ray emission of low Z elements such as C, N and O, and L shell emission of transition metals like Ni and Cu. The number of detected photons, $I_i$, per laser pulse train was estimated for C, O, Ni and Al samples using the “fundamental parameters” method (Mainardi and Barrea, 1996).

$$I_i = T_i A \frac{\Omega}{4\pi} \frac{\Omega_{mc}}{4\pi} G_i \ldots$$

$$\ldots \int_{E_{abs,i}}^{E_{max}} \frac{\mu_i(E)}{\mu_i(E) + A\mu_i(E_i)} \left[ 1 - \exp \left\{ -\rho h \left( \frac{\mu_i(E)}{\sin \psi_1} + \frac{\mu_i(E_i)}{\sin \psi_2} \right) \right\} \right] I_0(E) dE (5.1)$$
Figure 5.6: Quantum efficiency curve of the EPIC type CCD used in the experiment.

where:

\[ d \] sample size, or pixel size \[ = 27 \mu m \]

\[ h \] sample thickness \[ > 1 \text{mm} \]

\[ T_i \] composite transmission of filters and helium column (Figure 5.5)

\[ \Omega \] solid angle subtended by sample

(at 18mm from source) \[ = 3090d^2 \text{sr} \]

\[ \psi_1 \] primary X-ray incidence angle \[ = 45^\circ \]

\[ \psi_2 \] angle at which fluorescent X-rays are viewed \[ = 45^\circ \]

\[ A \sin \psi_1 / \sin \psi_2 \] \[ = 1 \]

\[ Q_i \] CCD quantum efficiency (Figure 5.6)

\[ \nu_i \] MCP focusing efficiency (Chapman \textit{et al.}, 1991) \[ = 0.05 \]

\[ \Omega_{mcp} \] solid angle subtended by MCP from sample

(12mm \( \times \) 20mm at 275mm) \[ = 3.17 \times 10^{-3} \text{sr} \]

\[ G_i \] excitation factors; \[ G_i = \frac{r-1}{r} \omega_i g \] (Mainardi and Barrea, 1996)

\[ g \] weight fraction of \( K_\alpha \) or \( L_\alpha \) line within the
\[ K \text{ or } L \text{ series} \quad (\text{Kaelble, 1967}) \]
\[ \omega_i \] fluorescence yield of element \( i \) \quad (\text{Krause, 1979})
\[ r \] jump ratio of K or L absorption edge \quad (\text{Cromer and Liberman, 1970})
\[ E \] energy of primary X-rays (keV)
\[ E_i \] emission line energy of element \( i \) (keV)
\[ E_{\text{max}} \] maximum energy of primary spectrum (keV)
\[ E_{\text{abs},i} \] absorption edge energy of element \( i \) (keV)
\[ \mu_i(E) \] mass absorption coefficient of element \( i \) at energy \( E \) (cm\(^2\)/g) \quad (\text{Cromer and Liberman, 1970})
\[ \mu_i(E_i) \] mass absorption coefficient of element \( i \) at energy \( E_i \) (cm\(^2\)/g) \quad (\text{Cromer and Liberman, 1970})
\[ \rho \] sample density (g/cm\(^3\))
\[ I_0(E) \] X-ray source intensity (Figure 5.2) = 8 \times 2.5mJ

For the microchannel plate operating, as here, with unity magnification, the appropriate value of \( d \) is the CCD pixel size, 27\( \mu \)m, leading to the count rates per pixel given in table 5.3. It was thus expected that one or two laser shots would be sufficient to obtain an energy resolved CCD image. The laser was therefore used at its slowest rate of 6Hz, and its shutter opened manually so that only one or two pulse trains were allowed to go into the chamber.

5.4 Results

Initial tests with a special target (figure 6.2) from Micro Analysis Consultants, containing B, C, N, O and F samples on an Al substrate proved to be unsuccessful, because of the excitation of a high background level of Al K fluorescence by the Bremsstrahlung tail of the Fe plasma. Therefore, a simpler binary target was constructed from aluminium (\( K_\alpha \) X-ray energy 1.49keV) and nickel (\( L_\alpha \) X-ray energy 0.85keV) as shown in figure 5.7.

It was found through trial and error that two laser shots indeed produced an acceptable count
rate for this target, with minimal “pile up” (the process whereby more than one photon lands in a pixel resulting in confusion over their energies).

The spectrum obtained from eight such images added together is shown in figure 5.8. Unfortunately, the lower energy detection efficiency is poor due to the lower CCD quantum efficiency, fluorescent yield and polypropylene filter transmission. However, carbon and oxygen emission (Kα X-ray energies 0.28keV and 0.53keV) from the polyethylene terephthalate (PET) tape holding the sample together is visible.

A small amount of pile-up can be made out at 1.0keV (2×O Kα) and 1.7keV (2×Ni Lα), showing that the incident flux is as high as it can be.

The ratio of nickel to aluminium implied in the spectrum is, before correction for the details of the CCD quantum efficiency versus energy curve, approximately 2.2:1; larger than the 1:1 expected from table 5.3. This disparity is most likely due to; (a) the deviation of the high energy tail of the laser plasma spectrum from the reference curve shown in figure 5.8, and (b) the actual energy dependence of the MCP focusing efficiency (Al K X-rays incident at the MCP perimeter

<table>
<thead>
<tr>
<th></th>
<th>C-K</th>
<th>O-K</th>
<th>Ni-L</th>
<th>Al-K</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E_i$ (eV)</td>
<td>277</td>
<td>525</td>
<td>851</td>
<td>1487</td>
</tr>
<tr>
<td>$T_i$</td>
<td>0.08</td>
<td>0.2</td>
<td>0.5</td>
<td>0.85</td>
</tr>
<tr>
<td>$Q_i$</td>
<td>0.25</td>
<td>0.55</td>
<td>0.7</td>
<td>0.85</td>
</tr>
<tr>
<td>$G_i$</td>
<td>2.65×10⁻³</td>
<td>7.84×10⁻³</td>
<td>10.4×10⁻³</td>
<td>32.7×10⁻³</td>
</tr>
<tr>
<td>$\omega_i$</td>
<td>0.0028</td>
<td>0.0083</td>
<td>0.0193</td>
<td>0.039</td>
</tr>
<tr>
<td>$r$</td>
<td>18.7</td>
<td>18.1</td>
<td>5.53</td>
<td>12.96</td>
</tr>
<tr>
<td>$g$</td>
<td>1.0</td>
<td>1.0</td>
<td>0.66</td>
<td>0.909</td>
</tr>
<tr>
<td>$\rho$ (g/cm³)</td>
<td>1.375</td>
<td>1.375</td>
<td>8.9</td>
<td>2.70</td>
</tr>
<tr>
<td>$\mu_i(E_i)$ (cm²/g)</td>
<td>2316</td>
<td>1168</td>
<td>1881</td>
<td>378.5</td>
</tr>
</tbody>
</table>

**Table 5.3:** Estimated CCD count rates for one eight-pulse laser train
would fail to be reflected, because the incident angle exceeds the critical angle of reflection at 1.49keV).

Energy resolved images in either aluminium or nickel X-rays, selected using a CCD pulse height window 100eV wide centered on the maximum of the relevant spectral peaks are shown in figures 5.9 & 5.10. These images have been smoothed using a top hat filter of $12 \times 12$ pixels to highlight the central foci. The line foci of the microchannel plate response can be seen, as can the “diffuse” unfocused background. The apparent size of the central object (about $5\text{mm} \times 3\text{mm}$) is slightly greater than that shown in figure 5.7 (about $3\text{mm} \times 2\text{mm}$), but the nickel and aluminium emission sites are clearly separated. The FWHM width of the central focus of the MCP used in these experiments was determined in the laboratory at Leicester to be $\sim 0.6\text{mm}$.

**Figure 5.7:** Fluorescent test target for spatially and spectrally resolved imaging.
Figure 5.8: Composite spectrum of eight images added together, with C $K_{\alpha}$, O $K_{\alpha}$, Ni $L_{\alpha}$, Al $K_{\alpha}$ emission lines marked. Windows for nickel and aluminium X-ray imaging are indicated by vertical broken lines.
**Figure 5.9:** Fluorescent image in Al X-rays. Cross (x=19mm, y=24mm) shows the position of the nickel centroid.

**Figure 5.10:** Fluorescent image in Ni X-rays. Cross (x=14mm, y=25mm) shows the position of the aluminium centroid.
5.5 Conclusions

The experiment has successfully demonstrated the principle of a novel imaging X-ray fluorescence spectrometer based on square pore MCP X-ray optics and photon counting CCDs.

The ultimate spatial resolution ($\Delta x$) of this instrument is determined first by the channel pore size, and second by the CCD pixel size. Prototype MCPs with channels as small as 6$\mu$m square have been fabricated. The current generation of such plates exhibits an angular resolution ($\Delta \theta$), limited by mechanical imperfections in manufacture, to around 4–5 arcminutes FWHM, corresponding to $\Delta x$=0.4mm for the present MCP–sample separation $l_s$ of 275mm. Present developments for X-ray astronomy (Fraser, 1997) should reduce $\Delta \theta$ to the 1-2 arcminute range within the next few years, making feasible a spectrometer with spatial resolution less than 100 microns FWHM. The cruciform nature of the point spread function of the presently available square-pore microchannel plates is obviously non-ideal. Approximately one third of the power in the image lies in the line foci (Fraser et al., 1993b; Chapman et al., 1991). A more radical design, also under development for X-ray astronomy, employs square pores which are radially packed* (Willingale et al., 1998), rather than packed on a square grid (figure 1.20). This design eliminates the line foci completely.

The ultimate spectrometer sensitivity to K-shell emission from ultra-light, low atomic number elements such as B, C, N and O, and to L-shell emission from transition metals in the same sub-keV energy band, is determined by $\Omega_{mcp}$, the solid angle subtended by the MCP at the sample, the MCP focusing efficiency and the quantum efficiency of the CCD. The value of $\Omega_{mcp}$ in the present experiment, ~3msr, is lower than the ~25msr typical of a non-imaging energy-dispersive spectrometer based on a Si(Li) detector (Frank, 1997), but could be increased by the use of a larger MCP (at least for the lowest X-ray energies) and/or reduced sample–MCP separation $l_s$. Calculations based on equation 5.1 using an attainable solid angle of 40msr (an MCP 50 x 50mm$^2$), and the other parameters as shown in section 5.3, indicate that a 10$\mu$m diameter particle of carbon (1180pg) would give $\sim$ 0.04 counts per laser shot, or 4 counts per second with the laser operated at its maximum repetition rate of 100Hz. According to Iida and Noma (1995),

*UK patent application No. 931134.2, (1993)
the carbon sensitivity of current X-ray microprobe techniques using synchrotron sources, is of a similar level (if one extrapolates figure 5.11 to energies below 1keV). Sensitivities to metals such as nickel are higher than this in proportion to their higher fluorescence yield and elemental density, as already indicated in table 5.3.

The Laser Plasma Facility, though providing a high flux source, and a well equipped laboratory for the experiment, was far from ideal. The high quantity of continuum emission from the plasma was not expected, neither was the large thermal load placed on the CCD by the helium column in the chamber. This load limited the exposure time, and hence the sensitivity of the instrument without waiting for the CCD to cool down again.

It was anticipated that in using steel tape, elements whose lines fall above iron ($L_\alpha = 0.71$keV) would not fluoresce, giving a high signal to noise ratio for low $Z$ elements that are hard to detect using other, more established techniques. A filament X-ray source with a suitable target material would be able to do this, and would give the experiment the additional advantage that it could be made in the laboratory in Leicester. This experiment is described in the next chapter.
Chapter 6

Imaging X-ray Fluorescence Development

6.1 Introduction

Following the success of the imaging X-ray fluorescence proof of concept experiment at RAL (chapter 5), and to prove the imaging scheme could be realised without the need for a large laser, we undertook some lab based work in Leicester.

The overall aim was to recreate the RAL set-up, but with a better quality MCP and hopefully lower energy X-ray source. The high energy X-rays present in the laser plasma spectrum were problematic in causing fluorescence of higher Z elements such as aluminium and silicon. These elements are typically those which form the substrates or backgrounds upon which impurities of lower Z – such as F, O, N and C which are the target for this study – are found. K-shell fluorescence from Al and Si will eclipse the “real” fluorescence signal from the lower Z material. Additionally, the density and fluorescence yield of the higher Z elements are greater, exacerbating the signal to background problem.

Limiting the maximum energy of the primary X-rays would inhibit the production of fluorescent lines to those below that energy, improving the ratio of “signal” X-rays (those from low Z fluorescence) to “noise” X-rays (mainly those fluoresced from background materials). This turned out to be difficult with the laser plasma source, but the spectrum of the filament X-ray source in our home laboratory is more easily controlled by setting the anode voltage to a level below the background material’s relevant fluorescent line (e.g. < 1487V for Al).

This chapter starts by describing the experiments undertaken in Leicester, then explores the potential for exploiting Bragg reflection imaging, serendipitously discovered during the fluorescent
observations.

Configuration for fluorescence spectroscopy
Indirect illumination

Liquid Nitrogen Tank

Configuration for source spectroscopy
Direct illumination

Figure 6.1: Laboratory fluorescence experimental configurations.
6.2 Fluorescence equipment

The laboratory test chamber was configured as in figure 6.1 firstly in the direct illumination configuration for source spectroscopy, then in indirect, fluorescence mode for sample analysis. Chamber pressure was kept to $< 10^{-6}$ mbar by turbomolecular pumps. The channel plate was a 30µm pore 100:1 aspect 10×10mm$^2$ active area (sample number 020498-01) from Nova Scientific Inc., and black fiducial solid glass markers in the channel matrix (the plate is shown in figure 1.13). This "tartan" plate had been previously tested in point to point focusing mode, and its focus found to be 0.8mrad or 3arcminutes FWHM and had a gain of 46 (Peele et al., 1998).

The CCD was an EPIC demonstration model type (#5401-12-5) with 600×600, 40µm × 40µm pixels, and an image area of 24mm × 24mm. The device was mounted in a ceramic package. The device was similar to that used in chapter 5, with an open electrode quantum efficiency curve as in figure 5.6. An on-chip frame store was used, into which charge is rapidly shifted from the image area at the end of each frame. To prevent X-ray interactions inside the frame store, it was covered with 2mm lead sheet. Liquid nitrogen and active heating kept the CCD at -100°C, as measured by a PRT mounted inside the copper cold finger immediately behind the ceramic package the CCD is mounted in, for all the frames that were recorded.

The sample was a Micro Analysis Consultants microstandard XRF target (figure 6.2) with six samples placed in 1.5mm diameter holes on centres 2.5mm apart. The bulk material was a 1” aluminium disc, which was mounted so its normal was at 45° in the $xy$ plane, and was adjustable in the $x$ direction. The X-ray source was an in house, oil cooled 0–5kV filament source with a copper anode to which different targets could be attached or painted. Collimation was provided with a 0.5mm pinhole to prevent flux fluorescing the chamber’s stainless steel walls. A filter was installed between the source and sample to reduce the considerable quantity of UV and visible light flux generated by the filament source, to which the CCD is very sensitive. The filter was 1µm Lexan with aluminium evaporated onto it to a thickness of 0.12µm, as measured by a piezoelectric crystal, and was glued to a copper annulus which doubled as the vacuum gasket for the joint. Unfortunately, this also formed an airtight join which necessitated separate and careful pumping for the source and sample volumes.
Figure 6.2: An SEM picture of the fluorescence target - scale is shown by the bar. The samples are, clockwise from top left: Carbon, Carbon, Boron Nitride (BN), Silicon, Aluminium Fluoride ($\text{AlF}_3$) and Aluminium Oxide ($\text{Al}_2\text{O}_3$) mounted in a one inch diameter aluminium disc. Note that the irregular individual samples do not fill their slots and are different sizes.
6.3 Fluorescence imaging

6.3.1 Source and MCP characterisation

To begin with, it was necessary to spectrally characterise the source and obtain a point spread function for the MCP for later analysis. The chamber was configured in direct illumination mode (figure 6.1). Figure 6.3 shows the CCD image and output spectrum for an uncoated Cu anode biased at 1250V and an emission current of 0.5mA; the image accumulation time was 15s. The FWHM was a disc of diameter 0.8mm, made up of the source pinhole size of 0.5mm and the intrinsic MCP focus size discussed in section 6.2. The darker area on the right of the image is a strip of lower gain on the CCD which afterwards was corrected by adjusting timing voltages. The high single pixel peaks are background events from cosmic rays. The oxygen line at 525eV in the spectrum is strong, but most of the flux is not contained in the Cu-L line at 930eV as might be expected, but instead appears as Bremsstrahlung, the anode voltage being not high enough to excite anything more than a small amount of line emission. A small high energy tail extends to 1250eV, the cutoff energy for the source.

6.3.2 Imaging of the fluorescent target

Once the system was reconfigured in fluorescence mode, it was quickly realised that lack of X-ray flux from our filament source was giving unacceptably long integration times, so the accelerating voltage was raised from 1250V to 3000V. This increase improved the flux below 1250V, but also excited the Al-K line. As most of the target was aluminium, the K-shell fluorescent flux at 1487eV dominates the total count rate, which is undesirable, but an acceptable concession for the lower energy intensification. Raising the voltage improved the total count rate by a factor of 9.2 – the bulk of that rise being within the Al-K line. The maximum flux that could be detected by the CCD with full energy resolution was governed by “pile-up” (see page 83), which is considered to be a problem when photons fall in more than 1% of pixels in any frame. For our case this was 3600 events per 45s frame.
Figure 6.3: Left: The Point Spread Function of the MCP (the detector area is 24 × 24 mm²), and Right: its spectrum. The narrow line at 370 eV is an anomaly generated within the CCD electronics which was corrected shortly after the image was taken. The peak of the Bremsstrahlung is about 750 eV which is close to the 830 eV predicted by equation 6.4 (anode voltage was 1250 V).

Secondly, the copper target was covered with a tungsten disc – tungsten has a higher atomic number and a higher X-ray production cross-section. Indeed, Dyson (1959) cites the empirical efficiency of continuous X-ray production by electron bombardment as:

\[ \eta = 1.1 \times 10^{-9} ZV \]  

(6.1)

where \( Z \) is the atomic number of the target and \( V \) the accelerating voltage. Increasing the atomic number of the target by 2.55 times, from Cu (29) to W (74) increased Al-K counts from 568 to 1597 per frame, or 2.81 times, a good correlation.

Stephenson and Mason (1949) quote several authors who agree that a Bremsstrahlung spectrum is described by:

\[ \lambda_{\text{max}} = \frac{3\lambda_0}{2(1 + BZ\lambda_0/C)} \]  

(6.2)

which relates \( \lambda_{\text{max}} \), the wavelength of maximum intensity, to the atomic number \( Z \), two constants, \( B \) and \( C \), and \( \lambda_0 (= eV/hc) \) the minimum wavelength present. Usually \( B \) is small giving

\[ \lambda_{\text{max}} = \frac{3\lambda_0}{2} \]  

(6.3)
for the energy equivalent. With $E_0$ corresponding to 3000V, this puts the continuum peak at 2000eV, which is just above the silicon line.

Figures 6.4 and 6.5 have the spectrum and images from an exposure with 2500 frames each of 45s integration time, 3keV X-rays and 2.25mA of emission current. Fluorescent lines have been marked on the spectrum in their theoretical positions, as have the major pile-up lines which correspond to two or more photons' energy added together.

The spectrum shows peaks for Al, Si and F present in the sample, and the images in the corresponding elements’ fluorescent X-rays. In the case of Al, other elements prevent fluorescence excitation, so “holes” appear. The image spots are well defined for Si and F and their position corresponds well to the holes in the Al image as indicated by arrows. Also shown are O and C which though present in the sample, have not been imaged. Both elements would be expected as contaminants evenly spread over the surface of the Al; O as oxide and C deposited from organic contamination. Mg is present in the Al alloy. The small Cu peak must originate from Cu X-rays from the anode – which is only partially covered with W – elastically scattering from the fluorescence sample and focused by the MCP. Peaks above 2keV are all pile-up lines.

The quantity of flux in the focused points can be estimated by the following:

$$I = I_s \eta \frac{\Omega_s}{4\pi} \omega_K \omega_i \frac{\Omega_m}{4\pi} m Q_i \text{ photons.s}^{-1}$$  \hspace{1cm} (6.5) $$

where;

$I$  Fluorescent photons of a particular element detected s$^{-1}$

$I_s$  Source flux of 2.25mA (electrons per second)

$\eta$  Source efficiency = $1.1 \times 10^{-9} ZV$ (electrons:X-rays)

$f$  Filter transmission (figure 5.5)

$\Omega_s$  Angle that sample subtends from the source - from figure 6.2 at 310mm (sr)

$\omega_K$ (or $\omega_L$) $K$- (or L-shell) fluorescence yield (Krause, 1979)
<table>
<thead>
<tr>
<th>Element, $K_{\alpha}$-line (eV)</th>
<th>Si, 1740</th>
<th>F, 677</th>
<th>N, 392</th>
</tr>
</thead>
<tbody>
<tr>
<td>$I_s$ (e$^-$.s$^{-1}$)</td>
<td>$1.41 \times 10^{16}$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\eta$</td>
<td>$244 \times 10^{-6}$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$f$</td>
<td>0.8</td>
<td>0.3</td>
<td>0.01</td>
</tr>
<tr>
<td>$\Omega_s$ (sr)</td>
<td>$5.20 \times 10^{-6}$</td>
<td>$10.4 \times 10^{-6}$</td>
<td></td>
</tr>
<tr>
<td>$\omega_K$</td>
<td>0.050</td>
<td>0.013</td>
<td>0.0052</td>
</tr>
<tr>
<td>$w$</td>
<td>0.1</td>
<td>0.8</td>
<td>0.9</td>
</tr>
<tr>
<td>$\Omega_m$ (sr)</td>
<td>$1.21 \times 10^{-3}$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$m$</td>
<td>0.60</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$Q_i$</td>
<td>0.8</td>
<td>0.4</td>
<td>0.3</td>
</tr>
<tr>
<td>$I$ (photons.s$^{-1}$)</td>
<td>0.264</td>
<td>0.205</td>
<td>0.00231</td>
</tr>
<tr>
<td>Photons in windows of figure 6.4 s$^{-1}$</td>
<td>0.15</td>
<td>0.14</td>
<td>–</td>
</tr>
</tbody>
</table>

**Table 6.1**: *Estimated and measured CCD count rates per second for three elements.*

$w$: Proportion of incident X-rays that will excite an element’s fluorescence line.
Estimated from theory.

$\Omega_m$: Angle that MCP subtends from the sample – 1mm $\times$ 1mm at 287mm (sr)

$m$: Focusing efficiency of MCP

$Q_i$: Quantum efficiency of CCD (figure 5.6)
Figure 6.4: Spectrum of the fluorescence target at 3kV with notable peaks, and fluorine and silicon windows marked. The pile-up peaks above 2keV are multiple events; e.g., Al+Si is at $\text{Al-K}_\alpha + \text{Si-K}_\alpha = 1487 + 1740\text{eV}$. 
Figure 6.5: Images of the IXRF sample at the X-ray energies of the marked elements. Silicon and fluorine are easily distinguished as are the "holes" in the aluminium. The centroids of the Si and F targets are indicated on the complete image which reveals the target to be as in figure 6.2 but inverted.
There is good (within a factor of 2) agreement between the estimation and the measured fluorescence counts (table 6.1).

### 6.3.3 Bragg peak imaging

In an endeavour to increase the fluorescent flux, the anode voltage was increased still further from 3kV to 5kV. The most obvious effect of this was to produce an extra peak at 3720eV corresponding almost exactly to Ca-Kα (figure 6.8). However, it was quickly realised that this line was a Bragg reflection from the microcrystalline bulk aluminium that made up the target, and that this reflection could be used as a measure of lattice spacing and hence strain. Bragg’s law states that;

\[ n\lambda = 2d \sin \theta_t \]  

(6.6)

where \( \lambda \) is the incident wavelength; \( d \) the lattice spacing; \( \theta_i = \theta_f \) the incident and reflected angles and \( n \) the order of reflection (figure 6.6).

For our configuration, \( 2d=4.676 \times 10^{-10} \text{m} \) for Al(111) and the energy of the peak was 3.72keV, making \( \theta_i=45.4^\circ \); a close correspondence to the actual geometry. The uses of this feature were appreciated when two images were made from events in the windows A and B centred \( \pm 100\text{eV} \) from the peak (figures 6.7 and 6.9). They show that an energy separation can be mapped as an angular separation in accordance with the predictions of Bragg’s law.
6.4 Discussion and further work

Although the Leicester experiment succeeded in resolving two small samples, the low level of low energy flux required an integration time of over a day to do it. This flux paucity will be one of the main problems in further experiments of this kind, and its successful increase will be the key to the realisation of a productive IXRF instrument. Additionally, the design of a filter capable of only passing soft X-rays (those of ~100eV to ~1500eV) will be of fundamental importance. Absorption filters will always cut out low energy radiation in preference to high energy, and the requirement for very thin filter layers is bound to make pinholes – which with the CCD being so sensitive, are unacceptable also, unless used in pairs.

The two approaches so far are, on the one hand, high flux but poor spectral control (the laser plasma source) and low flux and good spectral control (the filament source). Investigations into different X-ray sources will be needed before an optimum solution is found, which may well be the plasma source, but the high energy X-rays must be eliminated by cooling the plasma, filtering or another method.

A second experiment at RAL in July 1999 has shown that it is difficult to alter the plasma’s spectrum, especially in a controlled way by either using different targets, or spatially or temporally spreading the laser. Ideally, an X-ray “notch” filter would be used, cutting out visible and UV
Figure 6.8: Spectrum of the fluorescent target at 5kV showing the Bragg reflection peak at 3.7keV.

Figure 6.9: Two images made from events in the windows A (left) and B (right) from figure 6.8. Note with the different energy the Bragg peak shift in angle across the detector. The images have been blurred slightly using a 2×2 pixel top-hat filter. The y direction is as indicated in figure 6.1.
light, as well as hard (>1500eV) X-rays. Such a filter does not exist and filters that cut transmis­sion >1500eV are generally metals which have to be supported on carbon based plastics which have poor transmission in the low Z elements’ K line region.

A different approach, and perhaps the most promising, would be to use a tilted MCP, or another reflector, as a low pass filter. The reflectivity of channel plate glass is fully explored in chapter 2, and an MCP used in a configuration such as that of figure 6.10 would completely cut out the X-ray flux above about 1500eV. The quality of such MCPs would not need to be high, neither would their area. A large pore (50μm) MCP would be adequate in which case it could focus visible light making alignment much easier. A visible light filter would still be needed, but could be made from Be or Si₃N₄ which are stronger and so do not require supports.

A substantial grant has been secured from EPSRC to develop a pre-commercial prototype IXRF system, but without concentrating on any one application. The X-ray source type and filter design will inevitably be the two areas requiring the most attention.

The high energy resolution of the CCD can, given sufficient flux, be used to calculate the centroid of a Bragg peak to an accuracy sufficient to monitor changes in the lattice spacing of a solid. Such a capability would make it feasible to realise stress maps over an area of lattice with the spatial resolution limited by the quality of the MCP. The sensitivity of the instrument to stresses
is estimated as follows:

\[ 2d = \frac{1.24}{E \sin \theta} \]  

(6.7)

where \(2d\) is the lattice spacing in nm and \(E, \theta\) the Bragg reflection energy (keV) and angle (°).

Differentiating:

\[ \frac{d(2d)}{dE} = -\frac{1.24}{E^2 \sin \theta} \]  

(6.8)

For a given \(\Delta E\) (keV), strain is:

\[ \Delta(\text{Strain}) = -\frac{1.24}{E^2 \sin \theta} \frac{\Delta E}{d} \]  

(6.9)

Stress is related to strain by Young’s modulus, \(Y\) (Pa):

\[ \Delta(\text{Stress}) = -\frac{1.24}{E^2 \sin \theta} \frac{Y}{d} \Delta E \]  

(6.10)

For Al(111), \(Y=76.1\text{GPa}\) and \(d=0.4676\text{nm}\), \(\theta = 45°\) and \(E=3.72\text{keV}\) as before, so;

\[ \Delta(\text{Stress}) = -2.06 \times 10^{10} \Delta E \]  

(6.11)

To estimate \(\Delta E\) the minimum detectable energy change, we can use the relation;

\[ \sigma_m = \frac{\sigma}{\sqrt{N}} \]  

(6.12)

where \(\sigma\) is the standard deviation of a distribution, \(N\) the number of counts in it and \(\sigma_m\) the standard deviation of the mean of the distribution. For a Gaussian peak,

\[ \sigma = \frac{\text{FWHM}}{\sqrt{8 \ln 2}} \]  

(6.13)

The FWHM of the Al-K\(_\alpha\) peak at 1487eV in figure 6.4 is 85.34eV and has more than \(4 \times 10^6\) counts, so \(\sigma_m < 36.3\text{eV}\). Figure 6.11 shows the deviation of the mean expected for a spectral peak to a \(3\sigma\) (99.7%) certainty against number of counts in the peak. Even for a modest (1000) number of counts, a \(\Delta E\) of 3eV or a minimum stress of 71MPa will be detectable.

These changes could occur when a thin layer of a metal (eg Al(111) with a \(2d\) of 0.468nm) is placed onto another material (eg Si(111) with a \(2d\) of 0.627nm) such as might occur on the surface of a silicon wafer or a micromachined component. It could be imagined that an image would show stresses being highest – or the lattice distorting the most – around the edges of the thin layer, and with some knowledge of the physics involved, a calculation of the exact stresses could be made.
Figure 6.11: Graph showing benefit of increased counts to peak resolution at Al-K$_\alpha$ energy.
Chapter 7

Conclusions and Future Work

Progress reported in this thesis can be split into two areas. Firstly, changes in the channel plate manufacturing process have been assessed. It has been found that annealing of the glass after etching reduces the MCP channel surface roughness by a significant amount, and that the acid etching process leaves the channel surfaces with a layer depleted in lead which is detrimental to X-ray focusing. Further, the slumping of channel plates has been shown to be reproducible and images of X-ray foci using slumped plates have been taken.

Secondly, imaging MCPs have had their first application – imaging X-ray fluorescence. It has been shown that a channel plate can collect and focus X-ray fluorescence from an extended sample onto a camera, a task normally either expensive or performed in a non-imaging manner. The interesting prospect of “stress imaging” is also presented.

Over the next few years, work on focusing MCPs will be concerned with:

- Hard X-ray focusing. Results from the TRP are promising, and high aspect ratio MCPs have been made that would be suitable for focusing X-rays up to 100keV. The Tunnel Test Facility (TTF) in Leicester has also been commissioned for testing these optics. It is a 20m long, high vacuum beamline equipped with an 100kV, 3kW source as well as provision to mount and adjust optics along its length. The first high energy (50–67keV) MCP focused image is shown in figure 7.1. One possible application of hard X-ray focusing is XEUS, see section 7.1 below.

- Imaging X-ray fluorescence. The provision of a grant from EPSRC has made it possible to build on the work in chapters 5 and 6 with a commercial prototype which is being assembled in Leicester. An MCP is the optical element.
• LOBSTER, the all sky X-ray monitor (section 1.6.1) has been proposed for the ESA F2/F3 opportunity as an attachment to the ESA Columbus module of the International Space Station (ISS) and accepted for further study. Using many slumped channel plates, it will see the whole sky every 90 minutes as the ISS rotates. Coaligning the MCPs using visible laser light reflected from the front surfaces of the MCPs to a common focus has been shown to work for a single channel plate, but positioning of many (~50) will be a significant challenge.

• Metallic coating. Work on improving the coating of glass microchannels' X-ray reflectivity is needed, especially if high energy focusing is to be efficient. Work presented here in section 2.5 has shown a significant improvement is possible, but the results are not as good as they could be.

• Silicon channel plates. In what may be the most significant development, MCPs have been made from a silicon crystal, which gives the potential of making channel plates of any channel arrangement. See section 7.2.

7.1 XEUS

XEUS or the X-ray mission for Evolving Universe Spectroscopy, has been proposed as an X-ray observatory (Turner et al., 1997) of the generation after XMM and Chandra. The telescope as it is currently envisaged, will be two spacecraft 50m apart (figure 7.2); one containing the X-ray mirrors and the other the detectors. The mirrors will be made from segments or petals which can be added in orbit (see table 7.1), and will be of a Wolter type 1 form. The International Space Station is being considered as the location to do this (Dasa et al., 1999). In this way, a very large (10m diameter) mirror can be made, and the space station can be put to scientific use.

MCPs are being considered as optics for the central part of the mirror (the circular area inside the petals) so high energy (up to 100keV) X-ray can be focused. The open area of the glass petals preclude them from use in this energy band. However, at the time of writing (December 1999) no firm decision has been made on this.
Figure 7.1: The focus of a 500:1 MCP in the long beam test facility in Leicester taken by Gareth Price and Adam Brunt. X-rays were generated using a tungsten anode at 80kV and filtered with 0.5mm Tantalum giving emission between 50 and 67keV, and are incident with the MCP half way along the 20m beamline in point to point focusing mode. The optic, reference FB001-A3 from the ESA TRP, is $54 \times 54\text{mm}^2$. The crossarm structure can be seen, but the focus is confused and not a point, possibly indicating misalignments in the multifibres of the channel plate. The focus is $7 \times 10$ arcminutes.
Figure 7.2: The two XEUS spacecraft in orbit close to the International Space Station where they will be partially constructed. Picture courtesy D. Watson, Leicester University.
<table>
<thead>
<tr>
<th>Mirror diameter</th>
<th>4.5m</th>
<th>10m</th>
</tr>
</thead>
<tbody>
<tr>
<td>$A_{\text{eff}}$ at 1keV (m$^2$)</td>
<td>6</td>
<td>30</td>
</tr>
<tr>
<td>$A_{\text{eff}}$ at 8keV (m$^2$)</td>
<td>3</td>
<td>3</td>
</tr>
</tbody>
</table>

Table 7.1: Effective area of XEUS before and after the extra mirrors have been added.

7.2 Silicon channel plates

The anisotropic etching of silicon along different lattice planes has been known for some time (e.g. Bean, 1978), and in the last few years there have been attempts to etch square channels in silicon to make focusing channel plates. Nanosystems Inc. in the USA have successfully made a $\sim0.3$mm thick, 6.5$\mu$m square pore channel plate for Goddard Space Flight Center (Peele, 1999) which was tested at the Daresbury Synchrotron along with the MCPs in section 2.5, and gave the reflectivity curve in figure 7.3. While the results are not excellent at this stage, it opens up the possibility of etching any pattern of channels onto a wafer of any shape and avoiding the processing problems inherent in the glass drawing method – a radially packed optic would be as easy to make as a square packed one. The X-ray reflectivity of silicon is not as good as that of glass so coating of the channels with a metal such as nickel will be needed for most X-ray applications. ERA Ltd. have also been making square pore silicon MCPs as part of the ESA TRP (section 1.5). Hopefully there will be significant developments in this technology over the next few years.

7.3 Summary

The most significant progress contained within this thesis with respect to microchannel plates can be split into two areas. Firstly an understanding of the layer structure of MCP channel walls has been achieved. A lead depleted layer has been consistently found on the surface of channels, with a detrimental effect on X-ray reflectivity. The reason for this depletion is unclear, but there
Figure 7.3: Reflectivity measured from a silicon MCP (solid) and simulated reflectivity from 50Å (best fit) and 0Å rough silicon (dashed) with 1.8keV X-rays

is some evidence that it is the acid etching process that removes lead. Also, a metal (nickel) has been successfully coated onto the channel walls, and its structure assessed.

Secondly, focusing MCPs have had their first application as the imaging element in an X-ray fluorescence spectrometer. Results from the prototype apparatus presented here are promising and we can look forward to significant progress with this instrument, and many other applications of focusing MCPs, over the next few years.

Also reported here is the bending or slumping of MCPs to a spherical form. While the reproducibility of the process is not excellent, the concept has been shown to work to give an acceptable shape without mechanical damage, while preserving the focusing abilities of the MCP.
Addresses

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Shimadzu Research Laboratory Ltd, Wharfside, Trafford Wharf Road, Manchester, M17 1GP, UK
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