Characterisation of the oxide effects on aluminium opacity targets

Contact *david.haddock@stfc.ac.uk*

D Haddock, C Spindloe, M Tolley

Science & Technologies Facilities Council, Rutherford Appleton Laboratory, Harwell Campus, Didcot OX11 0QX, UK

S White, D Riley

School of Mathematics and Physics, Queens's University Belfast, BT7 1NN, UK

Introduction

This paper presents analysis of an aluminium strip target made by the Target Fabrication Group at the Central Laser Facility (CLF). The production method is outlined alongside characterisation data of the foil flatness and relative abundance of contaminants present on the surface. An alternative production method is presented which was introduced to reduce contaminants and its suitability is discussed.

Techniques used to make the strip include coating plastic by Chemical Vapour Deposition, aluminium coating by Physical Vapour Deposition and oxygen plasma etching; analysis techniques include Light Microscopy with Dark-Field Illumination, Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy and White Light Interferometry. All resources used are part of the Target Fabrication capabilities at the CLF.

Target Specification

An experimental campaign undertaken in January 2017 on the Vulcan laser system required targets of aluminium 900nm thick, 500µm wide free-standing over a frame with 800µm gap. The form of the strip was specified to have no angles greater than 5 degrees with respect to the mount plane. A sketch of the design is shown in Figure 1.

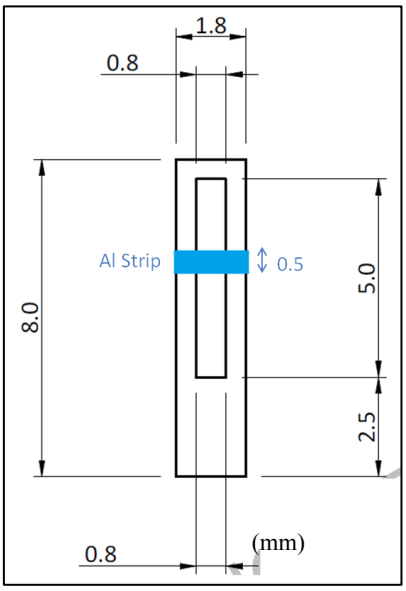

Figure 1. *Strip target specification sketch. All dimensions in mm. The aluminium strip is 900 nanometres thick. The supporting structure was made from chemically-etched steel*

Initial manufacture method

In order to easily place and support an aluminium strip whilst also achieving flatness across the area the chosen manufacture method included supporting the strip with a plastic underlayer that was removed by oxygen plasma etching.

A coating of Parylene-N ™ (trade name for a variety of chemical vapour deposited poly(p-xylene) polymers) of thickness 5µm was deposited onto a microscope slide by Chemical Vapour Deposition. The microscope slide is then coated with the desired thickness of aluminium by thermal evaporation in a thin-film deposition plant. The "strip" width of 500µm was defined by using a mask made of 100µm thick chemically-etched copper as shown in Figure 2. Placing the mask flat down onto the Parylene ™ coated microscope slide allowed for a coating with little shadowing as tested by a surface profilometer. The deviation in thickness coated across a microscope was considered well within reasonable error margins (±5% deviation from total thickness).

Figure 2. *Microscope slide with aluminum strips defined by a copper mask. The coating was performed with the mask overlaid on the slide to define the strips*

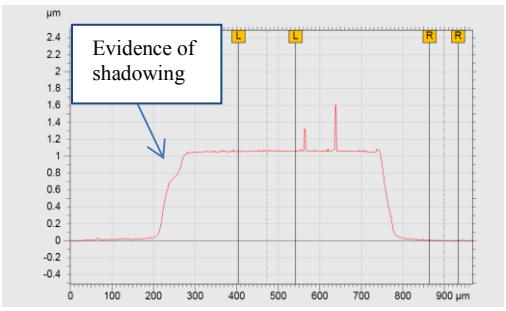

Figure 3. *Line out across width of strip measured by surface profileometer. Profile gives resultant coating thickness and shows shadowing from the copper mask*

The touch probe scanned across the width of each strip and resulted in the scan in Figure 3. This profile measurement also serves as the thickness measurement for the coating run.

Oxygen plasma removal of plastic

Individual strips are cut from the slide manually using a scalpel and glued over the open area of the mount using a small amount of epoxy glue. This is placed inside a Plasma-Enhanced Chemical Vapour Deposition plant where oxygen plasma removes plastic by hydrogen abstraction [1]. A flow rate of 30sccm oxygen is maintained at a pressure of 100mT by a gate valve to a turbo vacuum pump. A power of 150W is maintained by a discharge between two electrodes, creating plasma. This oxygen plasma removes plastic at a rate of ~0.1µm/min. Choosing a time of ~50 minutes ablates ~5µm of plastic. This method has been verified by taking a control substrate (glass microscope slide) with plastic and etching under the same conditions. No remaining plastic is detectable by surface profileometry. The sample is then taken out and visually inspected to ensure all plastic is removed, the dimensions fit that of Figure 1 and there is no visible debris or deformity. The mount with the strip can then be placed onto a CLF standard aluminium post ready for laser irradiation in the target area as shown in Figure 4.

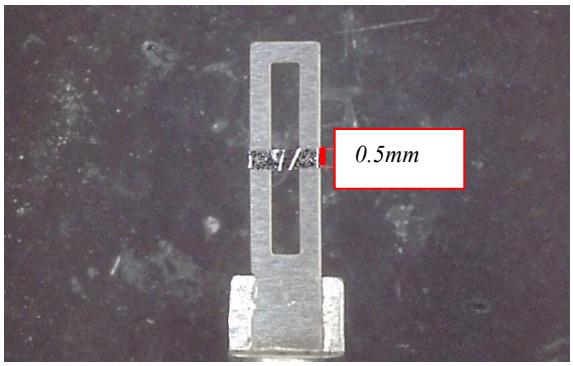

Figure 4. *The completed target. A 900nm thick, aluminium strip supported at both end over a 0.8mm wide aperture*

The result using this production method yields a flat and reproducible strip that can be batch produced. When measured by interferometry all targets were within specification and could be produced with high levels of consistency.

Alternative production method

The alternative production method was chosen to remove any source of plastic and include no oxygen plasma etching during processing. The initial manufacture method created a contaminant layer, detailed later in the paper.

Firstly a release layer of sodium chloride was deposited on a substrate using Physical Vapour Deposition in a thermal evaporation coating plant. Then, using the same strip mask as in the first production method, aluminium is coated through the mask to define the strips onto the sodium chloride. When the substrate is lowered into de-ionised water the salt dissolves and the aluminium is released from the surface and is suspended on the surface of the water. The strip can then be lifted out of the water directly on to the steel target mount. This process was done manually by an experienced fabricator. It was labour intensive because each strip had to be processed by hand and produced varying results in terms of the flatness of the strip across the aperture.

Each strip had its form measured over the open area to test against specification; White Light Interferometry gave a surface scan that allowed the form to be assessed. Due to the strict specified criteria for form around half of the targets produced were rejected. Figures 5 and 6 show an interferometry scan of the surface profile with a line-out across its length.

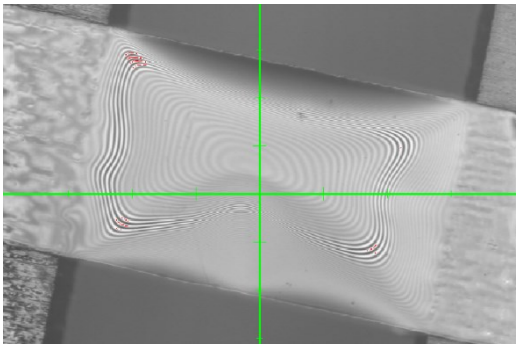

Figure 5. *Interferometry image of a target strip produced by float off. The interference fringes show the form of the strip*

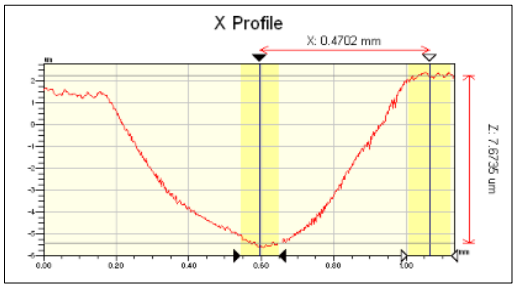

Figure 6. *Resultant profile along the length of the strip, measured using a White-Light Interferometer. Note the scale is exaggerated. The strip falls 7 microns in the middle relative to the supported ends*

Oxide observation by X-ray absorption

During the experimental campaign the user group noted an unexpectedly high X-ray absorption through the oxygen-etched sample strips indicating there may be a much greater oxide layer than there would be natively in aluminium; oxygen plasma has been shown to create a deep oxidation in aluminium [5]. As a result the user group took two identical shots: one on a standard production target and one produced via the alternative method. The results showed much greater X-ray absorption in the standard manufacture target indicative of a thicker-than-native oxide layer. The X-ray absorption on the alternative produced target was considerably less and was characteristic of a native aluminium oxide layer.

The presence of the oxide layer made the standard targets unsuitable for the rest of beam time. Target Fabrication undertook some retrospective characterisation research and chose the alternative production method for the remainder of the experimental campaign.

Contaminant characterisation

To image the contaminant both dark-field and bright-field light microscopy were used as well as Scanning Electron Microscopy (SEM). To characterise the contaminant Energy Dispersive X-ray Spectroscopy was used.

Figures 7 and 8 are optical images taken of the strips after etching. Figure 7 is a bright field image and figure 8 is a dark field image. Figures 7 and 8 clearly show plastic remaining around the strip despite prolonged etching.

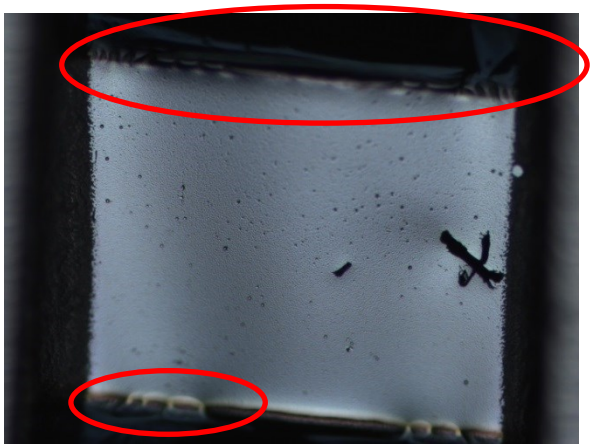

Figure 7. *Bright field image of aluminium strip produced using a plastic layer removed by oxygen plasma etching. There is some plastic visible at the top and bottom circled in red*

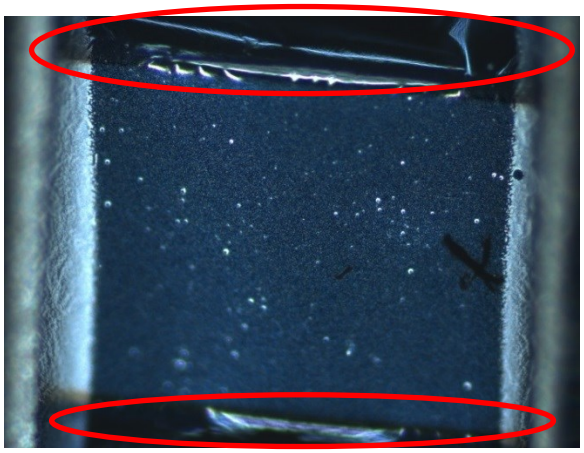

Figure 8. *Dark-field image of the strip. Dark-field microscopy excludes un-scattered light from the final image. Under dark field illumination plastic contaminant can be seen clearly in areas at the top and bottom circled in red*

Assessing the extent of the plastic contaminant using optical microscopy is difficult due to the plastic's transparency but SEM allows imaging irrespective of optical properties. Figure 9 shows the extent of the remaining plastic which is clearly visible despite prolonged exposure to the oxygen plasma.

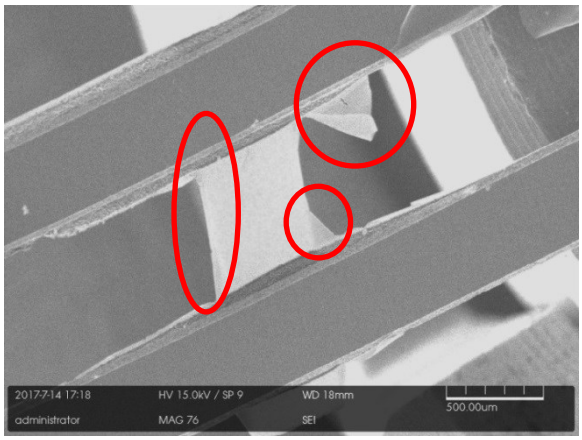

Figure 9. Scanning Electron Microscopy image of the same strip as figure 5. Plastic can be clearly seen around the strip

For comparison, Figure 10 shows a strip made by the alternative production method.

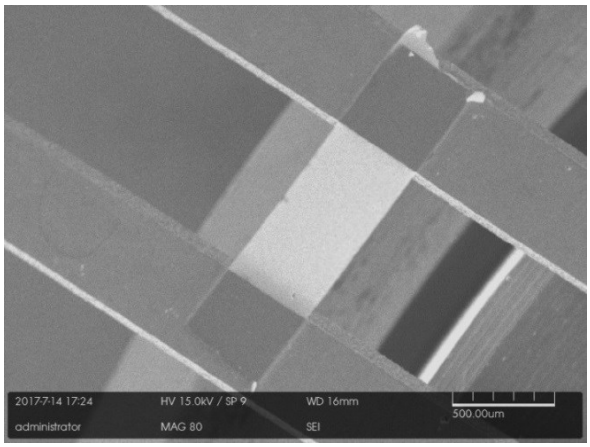

Figure 10. Scanning Electron Microscopy image of a strip produced by the alternative production method process performed without plastic coating or oxygen plasma processing. It can be clearly seen that the strip is free of contaminants

Identifying the contaminant

Energy-dispersive X-ray spectroscopy of strips with contaminant and without contaminant can be used firstly to rule out high-Z contaminant, and secondly to give a relative abundance of carbon and oxygen content and thereby estimate the extent of the contaminant. (Only relative abundance of low-Z elements can be obtained using EDX and cannot be used for accurate quantitative analysis.)

Using a beam voltage of 5KeV and choosing an area of ~50µm^2 in the centre of the strip a 100,000 counts spectrum was taken. The results for the samples in Figure 9 and Figure 10 are shown in Tables 1 and 2.

Table 1. ***EDX element data of relative abundance by weight and atomic number for a strip that had plastic removed by oxygen plasma (Figure 7, 8 and 9)***

Element	Weight %	Atomic %
Al	53.29	37.31
C	27.52	32.50
O	19.19	30.19

Table 2. ***EDX element data of relative abundance by weight and atomic number for a strip that had neither plastic or plasma processing (Figure 10)***

Element	Weight %	Atomic %
Al	76.24	60.99
C	8.27	11.16
O	15.49	27.84

Strips from both production methods contained no detectable high-Z contaminant. Relative carbon content is far higher in the strips with the plastic coating that has been subjected to oxygen plasma. This shows that there is plastic remaining on the surface of the strip as well as around the edges. It also shows a slightly higher abundance of oxygen, implying that the oxygen is reacting chemically with the carbon to form carboxylic groups, which has been show to occur in partially oxygen-etched Parylene TM [1]. Carboxylic groups are resistant to further reaction with oxygen plasma, which suggests why some plastic remained on the surface.

Modelling of electron interaction area and X-ray volume

To estimate a thickness of contaminant on the surface of the aluminium a CASINO simulation was performed as shown in Figure 11. CASINO software is a "Monte CArlo Simulation of electroN trajectory in sOlids" specially designed for low energy electron interaction in a bulk and thin foil [2] CASINO calculates the electron trajectory by a series of separate scattering events, after which the electron is absorbed and energy is released as electromagnetic radiation. The depth of the electron "tear-drop" shape is dependent on a number of factors including density of material and initial electron energy. This technique has been used to estimate the thickness of thin film layers [3],[4]. Using a simulated electron beam that is absorbed in a characteristic teardrop shape it is possible to analyse where electrons are absorbed and released as X-rays. A 5KeV electron beam was modelled entering an aluminium substrate with a carbon/oxygen contaminant on the surface. A 225nm contaminant layer of element ratios 4C:O:3H and density 1.06 g/cm^3 is modelled below. This approximate ratio was chosen to be empirically representative of Parylene TM with carboxylic acid functional groups. The basis for the reaction of Parylene TM with oxygen plasma in the creation of carboxylic groups is explored in depth in R Callahan and G Raupp [1]. Different thicknesses of contaminant were trialled to match the EDX data which could then be used to estimate the thickness. Using a contaminant layer of 225nm in the simulation the abundance of element matches the data collected from the contaminated strip, suggesting the contaminant thickness is of this order of magnitude.

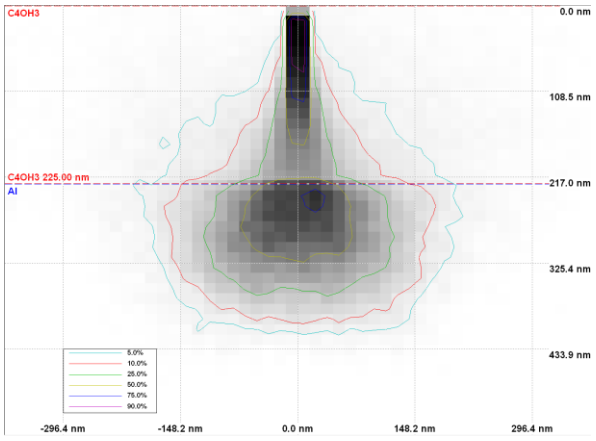

Figure 11. *A simulation of 5KeV electrons in a 225nm plastic contaminant containing a ratio of 4C:O:3H and a density of 1.06g/cm^3 on top of solid aluminium*

Conclusions

Self-supported aluminium strips of 900nm in thickness and 0.5mm width were batch produced by using a Parylene TM support film and were acceptably flat across the open area as measured by interferometry. The oxygen plasma etch to remove the plastic support film introduced a contaminant layer of carbon and oxygen atoms which rendered the targets inadequate for experimental use. The contaminant layer was confirmed by EDX to be a significant proportion of carbon with some inclusion of oxygen.

Although chemical bonding could not be assessed with the equipment it is proposed that the oxygen plasma reacted with the plastic support layer and produced carbon-oxygen functional groups which were resistant to further etching. A 225nm 4:1 carbon-oxygen contaminant on the surface of aluminium was sumilated under a 5KeV beam; the energy emitted as X-rays from each element was comparable to data gathered from EDX of a contaminated strip. Increased contaminant occurred more readily than expected because of the geometery of the coatrer; poor conduction with the baseplate inside the process chamber caused the sample to charge.

Because of contaminant layers an alternative production method was used. Coating the strips onto a release layer of sodium choride succeeded in producing a product with no significant contaminant, however, this process was labour intensive and produced inconsistent flatness across the open area. The criteria for flatness required each target to be quality checked and approximately half of those produced were suitable.

In future to further analyze the chemical nature of the contaminant, X-ray photoelectron spectroscopy could be employed. This technique can be used to analyze the surface chemistry of a sample and would give insight into the oxygen containing functional groups present on the surface as a result of oxygen plasma etching.

Acknowledgements

The work presented in this article was performed in collaboration with Queens University Belfast whose target design and specifications were the basis for the research. The experiment took place in January 2017 at Target Area West.

All production resources used are owned by Target Fabrication at the Central Laser Facility.

References

1. Callahan R R A, Raupp G B, and Beaudoin S P "Effects of gas pressure and substrate temperature on the etching of Parylene-N TM using a remote microwave oxygen plasma", *J. Vac. Sci. and Technol. B: Microelectronics and Nanometer Structures*, **2001**, 19(3), pp 725-31
2. Gołda C M, Brzychczy-Włoch M, Faryna M, Engvall K and Kotarba A, "Oxygen plasma functionalization of Parylene-C TM coating for implants surface: Nanotopography and active sites for drug anchoring", *Materials Sci. and Eng. C*, **2013**, vol. 33, pp 4221-7
3. Canli S, "Thickness determination of thin films by energy dispersive x-ray spectroscopy", **2010.** A thesis submitted to the Graduate School of Natural and Applied Sciences of Middle East Technical University.
4. Myint K, Barfels T and Kuhr J, "EDX depth profiling by means of effective layers", *Fresenius J Anal. Chem.*, **1998**, vol. 361(7), pp 637-639
   Baier-Saip J, Babrera A, and Zarate R, "Deep oxidation of aluminum by a DC oxygen plasma", *Surface and Coating Tech.*, **2005**, vol.195, pp 168-175