Characterisation of Tape-Drive targets using X-Ray Fluorescence Spectroscopy

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Introduction

Tape-drive targets are commonly used in high-repetition rate experiments as they provide a simple but robust method of delivering 2.5D laser targets to the interaction point of the laser while remaining as close to the focal spot position as possible. This removes the time-costly need to refocus the laser on each shot [1]. The demand for these targets is increasing to support the future tape-drive target demands of the 'Extreme Photonics Applications Centre' (EPAC) facility, which will be operational in 2025. [1]

Tape-drive targets usually consist of a metal coating on Kapton, (a polyimide tape). Polyimide has advantageous mechanical and insulative properties regarding high-power laser experiments.

However, the characterisation of tape-drive targets is a difficult process due to some other properties of polyimide. Polyimide is compressible, translucent, blocks ultraviolet frequencies and has a relatively high surface roughness compared to the target thickness. For these reasons, most common optical and physical characterisation methods are ineffective. This led to the exploration of X-ray Fluorescence (XRF) spectroscopy, as this method would effectively 'ignore' the polyimide tape and measure the metallic coating. X-Ray Reflectivity (XRR) characterisation was also investigated, but the measurement was severely disrupted by the surface roughness of polyimide.
There are various machines that can measure coating thickness using XRF spectroscopy. However, this experiment established a method to train thickness coating models using any XRF machine.

Objectives

The main objective of these experiments was to identify the relationship between an XRF spectrum and metal coating thickness. A machine learning model was then be trained to quantify this relationship and provide a method of characterising metal coatings. The coating thickness measurement must be within a 10% tolerance for commonly used target materials. The measurement must also not be affected by the properties of the polyimide substrate. The characterisation method must be possible at high-repetition rates to support tape target fabrication.

Hypothesis

XRF spectroscopy can be simplified into the following process. Firstly, an incoming X-ray removes an electron from one of the orbitals around the nucleus. This leads to a 'hole' in the orbital, meaning the atom is in an unstable configuration. Finally, an electron falls from a higher orbital to fill this hole, releasing excess energy as fluorescent X-rays. The energies of the fluorescent X-rays are characteristic to each element [2]. The intensity at these specific X-ray energies is proportional to the number of atoms being irradiated, which can be linked to the thickness of a metal coating. The most common XRF spectroscopy machines use Energy-Dispersive XRF, which cannot detect elements with a lower atomic number than Aluminium. As the elements in polyimide are all below this threshold, so the substrate will be ignored during analysis.

Initial experimentation provided a feature to extract from the XRF spectrum; the maximum peak counts per second (MCPS). The MCPS value could be obtained by taking the maximum intensity value from the expected intensity peaks at the characteristic X-ray energies. This feature was successfully obtained with Copper and Gold, two commonly used target materials.

Method - Training

To provide training data, several Copper coating samples were prepared on a glass slide substrate. The coating thickness at the centre of the slide was measured using a Bruker Contour DektakXT profilometer. The thickness of each sample can be seen in Table 1.

Copper Sample	Copper Thickness (nm)	Copper Sample	Copper Thickness (nm)
1	121	**2**	207
3	316	**4**	493
5	636	**6**	742
7	833	**8**	992
9	1011	**10**	860
11	1459	**12**	1050
13	1458	**14**	1805
15	1807		

Table 1 - Thickness of training Copper samples.

The XRF spectrum for each sample was obtained using a Hitachi FT110A benchtop XRF analyser seen in Figure 1. The samples were analysed using the parameters seen in Table 2. Four measurements were taken for each sample and a rolling mean filter (window size of 8) was applied to the spectra to reduce noise.

Figure 1 - Hitachi FT110A XRF machine.

Measurement Time (s)	90	**Tube Voltage (kV)**	50
Collimator Size (mm)	0.2	**Tube Current (mA)**	1.0
Filter	1st	**Focus**	Normal

Table 2 - XRF analysis parameters.

The MCPS values were extracted from each XRF spectrum and used to train a machine learning model, using linear regression and Leave-One-Out K-Fold cross validation.

Method - Testing

Once the model was obtained, further unseen copper samples were used to test the performance of the model. The thickness of each sample can be seen in Table 3. Samples 1-4 and samples 5-9 were coated onto glass and polyimide substrates respectively.

Copper Sample	Copper Thickness (nm)	Copper Sample	Copper Thickness (nm)
1	210	**2**	411
3	619	**4**	819
5	254	**6**	555
7	765	**8**	1041
9	50		

Table 3 - Thickness of unseen Copper samples.

The MCPS values were obtained for each sample using the same methodology as before, with the results put into the machine learning model to estimate the coating thickness. The performance of the model was then evaluated using these results.

Results - Training

The following spectra show the range of MCPS values obtained by the XRF analysis. The first measurement for each sample can be seen in Figure 2 to indicate the trend of the results.

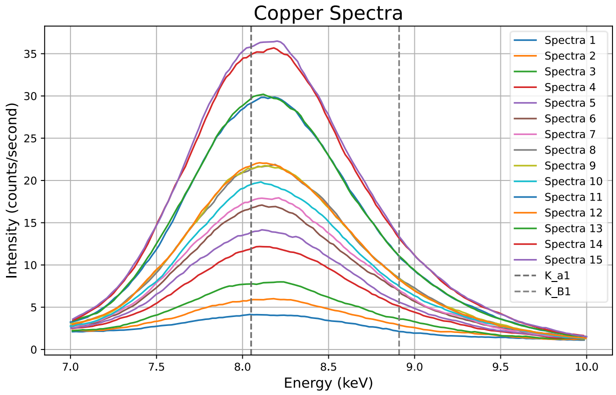

Figure 2 - XRF spectra for Copper samples.

The final MCPS was obtained by taking an average of each MCPS for the four sample measurements, seen in Figure 3.

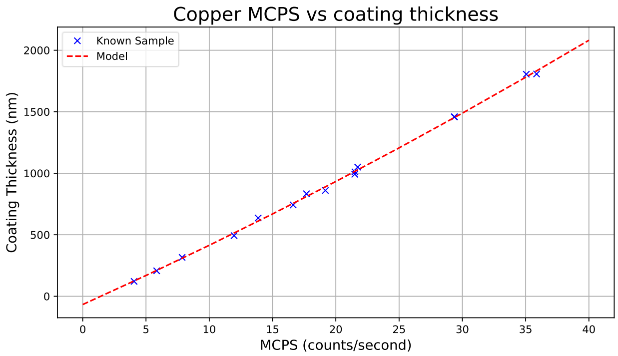

Figure 3 - Relationship between MCPS and coating thickness.

The machine learning model was then trained with the results shown in Equation below and Table 4. The model had a R^2 value of 0.99 and a Mean Average Error (MAE) of 19.27nm.

$$\textit{Predicted Thickness} = \alpha_2 * \mathbf{MCPS}^2 + \alpha_1 * \mathbf{MCPS} + \beta$$

Model Constant	Value
α_2	0.1991735100963794
α_1	45.72680844297905
β	-64.49529540257527

Table 4 - Coating thickness model constants

Results - Testing

The measurement of the unseen Copper samples provided the results seen in Table 5.

Sample	Predicted Thickness (nm)	Actual Thickness (nm)	Error (%)
1	213.69	210	1.76
2	444.85	411	8.23
3	658.37	619	6.36
4	878.96	819	7.32
5	245.19	254	-3.47
6	544.50	555	-1.89
7	824.03	765	7.71
8	1055.21	1041	1.36
9	39.32	50	-21.36

Table 5 - Model performance results

Analysis

The model performs poorly at thicknesses below 100nm, which is expected as the model was not trained below this point. However, the predicted thickness is within a 10% tolerance for higher thicknesses, meeting the initial objective. Furthermore, an optical attenuation method may be more suitable at this these thicknesses, as the Copper coating would become translucent.

The testing results also showed that the predicted thickness remained accurate on glass and polyimide. This means the characterisation method meets another initial objective and 'ignores' these substrates.

The initial experiments showed that the modelled relationship can also be seen for gold coatings, though a reliable model could not be obtained due to a lack of training samples. This observation proves that a thickness model can be obtained for other metal coatings.

Method - High Repetition

With a coating thickness model obtained and tested experimentally, the ability to characterise tape targets at a high-repetition rate must be developed. This would require a smaller, simplified Tape Drive that would fit inside an XRF machine, allowing for tape targets to be characterised automatically on a tape spool.

The FT110A has the following stage size: 500mm(W) × 400mm(D) × 150mm(H). A system called a 'Beam-Dump Tape Drive' (BDTD) fit this requirement and was used to test tape target characterisation.

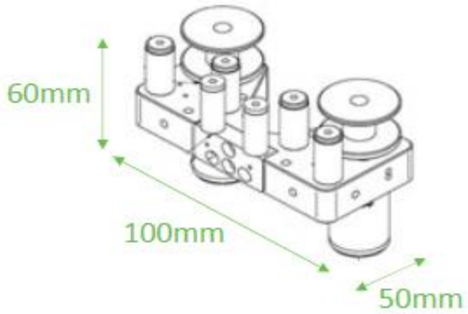

Figure 4 - The 'Beam-Dump Tape Drive' system.

To test this characterisation method, 24 Copper tape targets were analysed using a BDTD system. The average thicknesses of each target can be seen in Table 6.

Copper Tape Targets	Average Target Thickness (nm)
1-8	121
9-16	275
17-24	566

Table 6- Thickness of Copper tape targets.

The targets were analysed using the parameters seen in Table 7 and the 'Auto Focus' function was used to compensate for tape Z-axis variations. A rolling mean filter (window size of 8) was applied to the spectra to reduce noise.

Measurement Time (s)	45	**Tube Voltage (kV)**	50
Collimator Size (mm)	0.2	**Tube Current (mA)**	1.0
Filter	1st	**Focus**	Normal

Table 7 - XRF analysis parameters.

Results - High Repetition

The measurement of the unseen Copper samples provided the results seen in Table 8.

Copper Tape Targets	Average Target Thickness (nm)	Average Error (%)
1-8	121	-15.59
9-16	275	-6.14
17-24	566	-6.45

Table 8 - Model performance results

Analysis - High Repetition

The model consistently underestimated the estimated thickness of the tape drive targets. This can be attributed to error in the target thickness estimations and a difference in measurement conditions from the calibration conditions. As the base plate is made of Steel, the Iron content may contribute slightly to the Copper MCPS. Initial investigation also revealed that the presence of a glass slide may affect thickness measurement.

The measurement accuracy can be improved by ensuring that the measurement and calibration environments are more similar. This can be achieved by ensuring a glass slide and steel plate is placed behind the tape targets, as well as performing the calibration routine in the same position as the tape target measurement point.

Future Work

To improve the range of the coating thickness model, further Copper samples must be analysed, especially below 100nm. Also, the use of a wider collimator should be tested as it would provide a higher resolution. Furthermore, models for different XRF machines and various coating materials should be created.

To reduce the characterisation sample requirement, the effect of the atomic number should be modelled. This would mean that a single model could be used for all samples, with the atomic number as an input.

A 'Characterisation Tape Drive' system will need to be developed using standard tape drive spools and a BDTD controller. A program to control the XRF machine software and tape drive would need to be created.

Conclusions

The results showed that with a wide range of samples, the relationship between an XRF spectrum and metal coating thickness can be obtained for certain XRF spectroscopy machines. A coating thickness model for copper was trained and evaluated using unseen samples. The characterisation method shows promise for scaling to a high-repetition rate without extensive hardware modifications.

Acknowledgements

The author would like to thank the CLF Target Fabrication Group, the CLF Electrical/Mechanical Engineering Groups for their assistance and support during this experiment.

The author would also like to acknowledge the advice and support from RAL Space and ISIS Neutron and Muon Source departments.

References

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