Factors Affecting the Quality of Electroplated High Power Laser Target Foils

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Introduction

The Central Laser Facility has continuously sought to develop new materials for high power laser experiments as a way of enabling the user community to access new areas of science.

Metal foils and structures machined from metal blocks are common place targets used at many laser facilities. Another method of fabrication is electroplating. This is a well-established process and the focus of this work has been directed to assess the factors that affect the quality of copper plated foil on to aluminium substrates, which are common laser target components. This work has been carried out to develop capabilities to reduce the need for machining of expensive metals, potentially provide glue-free targets which are a focus of intense investigation across the community, and to obtain intricate target designs with reduced components.

In some situations artefacts present in plated materials include pits, peaks or nodules, and uneven plating across a flat surface. Reducing these artefacts can allow us to plate substrates with intricate and complex structures. Some of the artefacts encountered could be due to the use of aluminium as the substrate. Plating on to aluminium, where an oxide layer is inevitable, may give rise to these irregularities. In this preliminary run aluminium is the metal of choice as it is a commonly used to machine very standard target component.

Further research could focus on using seed layers to plate onto non-metals and non-conductive materials to produce multilayer foils as adhesive-free layered targets but this area has not been investigated for this report.

Experimental Setup

There were many areas that could be investigated, five were chosen; seed layer thickness effects, sulphuric acid effects, solution mixing, current applied, and solution temperature.

1. Aluminium Preparation

250um thick and 99.0% purity aluminium sheets were purchased from commercial suppliers. The sheets were then cut into 5x1cm strips. All aluminium strips were cleaned with sodium hydroxide solution, 10%. Following cleaning a seed layer was applied to all strips. The strips were coated with 240nm copper on one side (side A) and 180nm copper on the other (side B) using Kurt J. Lesker plasma sputter coating. The seed layer thicknesses were arbitrary and the thickness applied was used.

When coated strips were not in use following this process they were kept under nitrogen. This was done to prevent copper oxide forming on the treated surface.

2. Experiment Standards

For the duration of this investigation solution concentration was kept constant, ie, a 1mol dm⁻³ concentration of copper sulphate was made fresh every time.

The copper source was a commercially purchased foil. A 2mm thick, 5x5cm copper slab at 99.0% purity was used each time.

3. Time Allocation

Plating time was 2 hours for each strip tested.

Experimental Factors Investigated

1. Standard plating

A test strip was plated with no seed layer in $1 \text{mol dm}^{-3} \text{ CuSO}_4$ solution at 10 mA with continuous stirring using a magnetic stirrer

This test was carried out to establish plating parameters to investigate. This test sample did show plating had occurred. The result was highly irregular, uneven, highly nodular copper plating. Copper had not plated across the whole surface. The edges were preferred.

Where plating had occurred, copper nodules ran along furrows present in the aluminium strip. In these areas plating was uneven, and these areas were random across the surface. No usable foil or flat area was achieved

Nodules formed varied greatly in size in X, Y, and Z. The heights ranged from 3um to 154um.

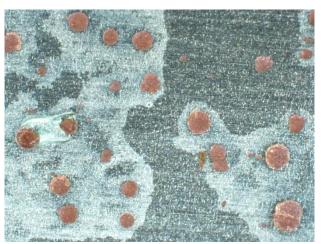


Figure 1: Seedless copper plating on aluminium substrate, 10mA applied, 2hours, 1mol dm⁻³ CuSO₄, 2.5x MAG.

2. Seed Layer

Seed layers of copper were applied using the plasma sputter coater were of arbitrary thickness. Side A was plated with 240nm copper, and side B 180nm. It was important to have layers with different thicknesses because it demonstrated that seed layer thickness appeared to not have consistent effects on the resulting plated foil. Currents applied were 5, 10 and

15mA. There are differences in the thickness of plated copper on sides A and B for most strips tested at each particular current. There are too many inconsistencies with the thickness of plated foil and the thickness of seed layer across the currents applied.

The presence of the seed layer did however decrease the number of irregularities on the plated surface compared to tests without the seed layer. Figure 2 shows the surface has been plated and it less nodular that without the seed layer in figure 1. Further work may be carried out on seed layers with greater difference



Figure 2: Copper plating with seed layer, 10mA applied.

3. Solution temperature

The aim of investigating solution temperature was to assess if the thickness of the copper foil plated differed across a temperature range. Three temperatures were investigated; 10, 18 and 40°C. Room temperature of the lab used is 18°C.

Temperature effects could not be fully investigated; all samples tested at 40°C delaminated to some degree. Side A (240nm seed layer) delaminated more often and in larger pieces than side B. These samples were characterised where possible but the inconsistency of foil success and unknown plating time leaves these samples void in this investigation.

At 10°C there was little to no change to the foil thickness. The temperature difference may not have been enough to give a result. Without access to a freezer, temperatures below 10°C could not be maintained.

4. Current Applied

The current applied to the aluminium strips was 5, 10, or 15mA.

Where 5mA were applied in a 1mol dm⁻³ CuSO₄ solution for 2 hours, without continuous mixing, at 18°C poor results were obtained. In these instances the copper deposited only as nodules. No foils were plated. Nodules ranged from 25um-110um in height. The nodules post-plating appeared bright, the seed layer however, was blue in colour. Figure 3 shows one of the results of this plating attempt.

No overall thickness measurements were taken for these samples as there were no areas where foil had been plated.



Figure 3: *Image of 5mA no mixing, nodules can be seen across the surface. No foil success.*

Where 10mA were applied to aluminium strips in 1mol dm⁻³ CuSO₄ solution, for 2 hours, with no continuous mixing, at 18°C poor quality foils were achieved. These foils differed in thickness on sides A and B. Side A samples showed copper thicknesses of 18-20um, where side B were 6-8um. Further investigation will need to be carried out to address these results.

The roughness was measured at 3.8um on side A, 1.8um side B. White light interferometry was used to measure the roughness of the foils. Foils achieved here were difficult to remove from the aluminium substrate. There was some discolouration on the surface. Discolouration was striped along the width of the sample. This is the orientation of the grooves found in the aluminium pre-processing. Aluminium foil could be polished prior to plating to help remove this effect. This area has not been investigated at this time.

EDX analysis was carried out on the sample. The discoloured areas appear darker in colour, EDX analysis shows a greater oxygen content suggesting the copper had oxidised. Figure 6 shows the surface structure of the 10mA sample. Although the resulting foil is more consistent than the 5mA partner (figure 5), it does have greater surface area, allowing for oxidation to readily occur.

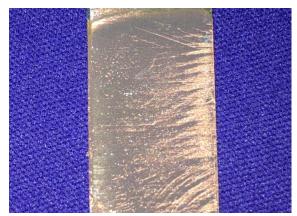


Figure 4: 10mA applied, solution was not continuously mixed.

Where 15mA were applied to aluminium strips in 1mol dm⁻³ CuSO₄ solution, for 2 hours, with no continuous mixing, at 18°C foils were achieved. These samples gave thicknesses of 0-28.8um on side A. Side B was uneven giving coating of 0um-17um. Both sides showed areas where plating had not occurred. The thickness across the surface was inconsistent. Although a foil was plated, there were large areas of thick, nodular, dull

copper and areas of thinner, flatter, brighter copper. The thicker areas appeared darker in colour, much like the foils achieved at 10mA. The range of thicknesses across the surface does not give a usable foil. Foils achieved on side B under these conditions were also dull and nodular on the thicker areas and brighter on the thinner areas. The foils achieved at 15mA, under these conditions, did not come away from the substrate easily. Small pieces of copper were released from the aluminium substrate, averaging 1mm². The plated foil would flake away when pressure was applied to release the foil for analysis.

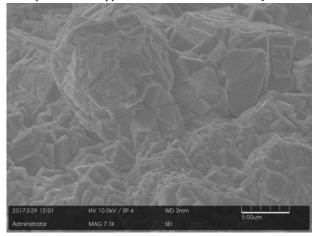


Figure 5: SEM Image of copper nodule formed on aluminium strip. Plating conditions: 18° C, 1mol dm^{-3} CuSO₄, not stirred throughout plating time, 5mA applied.

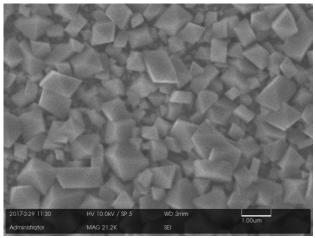


Figure 6: SEM Image of copper surface formed on aluminium strip. Plating conditions: 18° C, 1mol dm^{-3} CuSO₄, not stirred throughout plating time, 10mA applied.

5. Continuous Mixing

Tests were carried out to examine the effect of continuous solution mixing. For these tests all other factors remained constant. Current applied was 5, 10, or 15mA in a 1mol dm⁻³ CuSO₄ solution, for 2 hours, at 18°C.

All test strips showed a marked improvement in uniformity across the surface when the solutions where continuously mixed. The range of thicknesses across the surface had been dramatically reduced. Dull areas remained, this is where the copper was rougher compared to the appearance of brighter foil sections. Thicknesses achieved can be seen in table 1. These foils were again flaky when removed from the aluminium surface. Copper was adhered well to the surface, making foil removal difficult.

Current	Solution	Solution	Not	Not	
applied	Mixed -	Mixed -	Mixed	Mixed	
(mA)	Thickness	Thickness	Thickness	Thickness	
	side A	side B	side A	side B	
	(um)	(um)	(um)	(um)	
5	5	14-15	N/A too	N/A too	
			many	many	
			nodules	nodules	
10	20-21	7-8	18-20	6-8	
15	9-10	10	0-28	0-17	

Table 1: Copper foil thickness using white light interferometry.

6. Effects of Sulphuric Acid

Sulphuric acid was added to the copper sulphate solution. All other conditions remained the constant. Current applied was 5, 10, or 15mA in a 1mol dm⁻³ CuSO₄ solution, for 2 hours, at 18°C. Due to the foil success achieved by continuous mixing, this was also kept constant.

 $200ml\ CuSO_4\ 1mol\ dm^{\text{-}3}$ was the standard solution used. 10ml H_2SO_4 at 50% was added to the standard solution.

The resulting foils across all currents applied were flatter, appeared bright copper in colour, plated more evenly than previously and were easily removed from the aluminium substrate by peeling. Figure 8 shows the comparison of foils with and without sulphuric acid. Complete foils were removed from the aluminium substrate leaving no copper, or copper seed layer behind. Figure 7 shows the flatness achieved at 5mA with sulphuric acid. Results achieved at 5mA seem to have given the clearest effects.

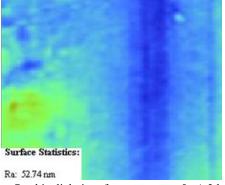


Figure 7: white light interferometry scan, 5mA 2 hours sulphuric acid. Roughness of 53nm achieved



Figure 8: comparison of plating results with and without sulphuric acid

Current	Copper	Copper	Copper	Copper	
applied	thickness	thickness	thickness	thickness	
(mA)	(um)	(um)	(um)	(um)	
	CuSO ₄ without H ₂ SO ₄	CuSO ₄ without H ₂ SO ₄	CuSO ₄ with H ₂ SO ₄	CuSO ₄ with H ₂ SO ₄	
	Side A	Side B	Side A	Side B	
5	N/A too	N/A too	2.5	2.5	
	many	many			
	nodules	nodules			
10	18-20	6-8	20	6	
15	0-28	0-17	5	15	

Table 2: Copper foil thickness characterisation for solutions with and without sulphuric acid

7. Roughness

Roughness of the foils achieved was also checked alongside thickness. The roughness improved with continuous mixing and with the addition of sulphuric acid. The foils achieved at 5mA current gave bright, almost mirrored foils.

Roughness scans taken here are carried out with the white light interferometer. If more detail is needed in the future we will investigate with an atomic force microscope for higher resolution.

Curr	Rough	Rough	Rough	Rough	Rough	Rough
ent	ness	ness	ness	ness	ness	ness
Appl	(nm)	(nm)	(nm)	(nm)	(nm)	(nm)
ied	CuSO ₄	CuSO ₄				
	No	No	No	No	With	With
mA	H ₂ SO ₄	H_2SO_4				
	No	No	Mixed	Mixed		
	mixing	mixing				
	Side A	Side B	Side A	Side B	Side A	Side B
5	N/A	N/A	210	180	N/A	53
	too	too				
	many	many				
	nodule	nodule				
10	3800	1800	1000	1240	240	240
15	N/A	N/A	790	760	400	470
	sample	sample				
	too	too				
	dark to	dark to				
	charact	charact				
	erise.	erise.				

Table 3: roughness scans using white light interferometry

Conclusion

There are many factors that can affect the quality, thickness and usability of plated metals. The five factors investigated here in this preliminary research shows each factor does affect the quality and thickness of copper plated metal on to aluminium substrates.

From the investigation so far, if layered copper aluminium targets are required, the best result would be achieved with foils that are electroplated with continuous mixing of the solution and the addition of sulphuric acid. This will give results with increased thickness uniformity with reduced risk of delamination, allowing for adhesive-free layered metal targets.

If thin copper foils, below 2um, were required the parameters could be set to achieve this. The benefit of achieving thin copper foils on aluminium substrates is we can produce foils over intricate target designs with the probability of removing them in one piece. This will again give glue-free targets but also reduces the number of components.

Further work will focus on the most effective ways of increasing the electroplated deposit quality and also begin to focus on plating onto micromachined target components to enable the group to offer this service to the community.