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Experiments in Metal Patterning of Epoxy Substrates Through the CO₂ Laser Reduction of Copper(II) Oxide

Henry [REDACTED]

Abstract—Laser induced selective metallization (LISM), a process which uses lasers to fabricate metal patterns or images onto substrates, has been the subject of recent research in its application in circuit board fabrication. My research investigates the feasibility of using CO₂ lasers in this process. Copper (II) oxide (CuO) serves as an activating agent which is reduced into copper metal after a process called activation. After integrating the CuO into an epoxy substrate, the material was activated and an image was patterned onto it by a CO₂ laser. The pattern was then reinforced by a copper plating process which creates a copper film on the activated regions. After analysis of the deposited copper films using microscopy, cross sectioning and resistivity, the quality of the copper films was determined. On all

but one sample, the activation failed. The sample that was successfully activated was processed under a 60 watt laser and had a resistivity of 6.57e-5 Ω/m. This resistivity is poor but still shows potential for a refined CO₂ LISM process.

I. Introduction

In the last several decades, there has been increasing interest in exploring the capabilities and applications of laser technologies in both academia and industry [1]. One such area of interest is in selective metallization of non conductive materials, the addition of a metal film onto specific areas of a substrate for functional or decorative applications. Several non-laser techniques for this process already exist, such as screen printing, photolithographic

exposure, and ink printing [2]. However, techniques such as these have significant drawbacks such as being cost prohibitive and time consuming along with their generally negative environmental impacts [2]. Another relatively new method, laser induced selective metallization (LISM), holds promise due to its sterility, low cost, repeatability and low environmental impact [1]. LISM is a three step process which uses lasers to fabricate copper traces, or wires, onto a substrate. This process is shown in Figure 1 below.

LISM has found an experimental use in non-standard circuit board fabrication [2]. Printed circuit boards (PCBs), a ubiquitous material in modern electronics, are commonly fabricated in a chemical etching method in which copper plated sheets are selectively etched to isolate copper traces. This use of hazardous chemicals can lead to environmental issues and high cost of production. The etching process is also mainly used on fiberglass substrates which limits the choice of materials for engineers. The field of laser metallization holds some promise for this issue, so an effort to develop a CO₂ laser induced metallization process that increases accessibility to small scale PCB fabrication may help to ease this problem. This makes LISM an attractive process as a relatively green and cheap process to produce copper traces on a variety of substrates.

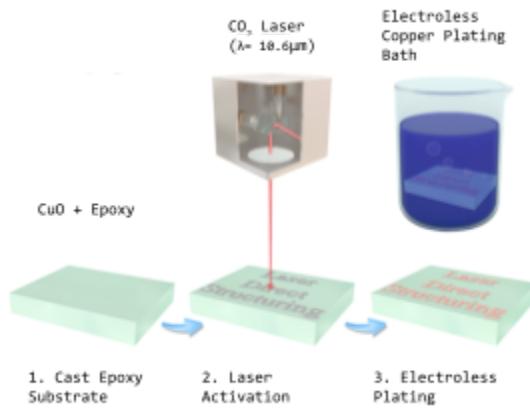


Figure 1. The general process for LISM. A substrate is cast or molded to include an activating agent (CuO), activated by a laser and then copper plated in a plating solution. [2]

II. Literature Review

A. CO₂ and Fiber Lasers

The two types of lasers used in LISM are fiber and CO₂ lasers. The most important distinction between them is the wavelength of light that they operate on and the influence this has on their applications. Wavelength is the length of the wave of light emitted by the laser and influences which materials they can process, or cut [1]. Fiber lasers often operate in the near infrared (NIR) region which corresponds to a wavelength between 700 and 2200 nanometers (nm). This makes fiber lasers important in processing materials such as metals which generally have a high absorption of NIR light. CO₂ lasers on the contrary operate on a much longer wavelength, around 10.6 μm , which makes them ideal

for processing many common polymers such as plastics [1].

There are also several properties which are relatively consistent between the platforms. Generally, for both lasers, the width of the beam is operator tuneable and can achieve diameters less than $200\mu\text{m}$ which allows for the manufacturing of traces with small feature sizes [1]. Additionally, both platforms will generally have variable wattage, pulse frequency and translational speed across the X and Y axis [1], [3]. These properties give lasers great versatility in how they process materials.

B. State of the Art

1) Substrates and Sensitizing agents: One of the main benefits of LISM is its application on a wide variety of substrates as shown in previous research [2]-[6]. For laser processing, ABS plastic is commonly used due to its low cost, versatility and high resolution patterning, but its decomposition into poisonous hydrogen cyanide poses a limitation. HDPE and PP plastics, while non-toxic in their decomposition, suffer from low cutting resolution and ablation rate, or the rate at which material is vaporized, which detracts from their viability [4]. Due to the shortcomings of these common plastics, I used an epoxy (Art N' Glow clear epoxy resin) for this project. Epoxy resin, a liquid which hardens into

a solid plastic, allows for the easy integration or mixing of a sensitizing agent into the substrate and cuts and engraves cleanly[10], [11]. These properties make it a good candidate for a versatile LISM process.

2) LISM Processes: Due to the relatively recent widespread availability of laser processing equipment there has been lots of research done on optimizing and exploring the LISM process. The first step in the three step process involves creating a substrate with a sensitizing or activating agent. The next step, called activation, uses a laser to etch or pattern the substrate. This reduces the activating agent into copper metal. The final step is to plate these activated areas with copper metal to improve their electrical characteristics and make them conductive [2]-[5], [7]-[9]. Figure 1 above shows this process.

a) Fiber LISM: Over multiple studies, Zhang et al. along with other groups, have outlined a general process for fiber LISM, shown in Figure 1 and described above, and the applications of such a process in the real world [2]-[5], [7]-[9]. Specifically for fiber LISM, during the activation step the activating agent absorbs the NIR light and decomposes into copper metal [7], [8]. Zhang et al. showed that the quality of the LISM activation is dependent on different laser parameters, the most

important of which were laser pulse frequency and power [7]. There has been a particular focus in this research to find an optimized compound that, when decomposed, allows for the best catalyzation of the plating reaction [2]-[5]. Additionally, these studies showed success with a wide variety of different activating agents and produced copper films with good electrical characteristics [8]. The main reason that research has thus far focused on fiber lasers, is due to their availability in commercial and lab settings [2].

b) CO₂ LISM: While fiber laser LISM is more common than CO₂ LISM, CO₂ lasers have also been used in the process. The main difference in the process is which material (activation agent or bulk substrate) absorbs the laser light. In CO₂ LISM the substrate absorbs the laser light which then reduces the activating agent [1]. In the 1970s, Suh et al. patented a process which showed that a carbon-containing polymer mixed with CuO worked in an LISM process. After creating a 75% copper oxide and polymer substrate and activating the sample, copper traces were created that could then be reinforced by electroless plating [10]. These traces exhibited characteristics identical to those of pure copper [10]. This process used a 50 watt laser with unspecified laser parameters. Another independent project showed that CuO powder mixed into an

epoxy resin could be selectively reduced by a CO₂ laser into elemental copper [11]. In this study, the resin was mixed 1:1 by mass with the CuO and produced clearly visible copper traces. Although copper traces were visibly created, they were not plated afterwards and as such had poor electrical characteristics [11].

Another study contradicted these findings and found that CO₂ LISM was not feasible as, after a failed activation of the sample, it was noted that the ablation rate of the CO₂ laser was substantially less than that of the fiber laser also used in the process [3]. It was proposed that this was the primary reason the substrate failed to activate, but no further research was performed using CO₂ lasers in the study [3]. It should also be noted though that the laser power used in this study was lower than in other studies and was maxed out at only 8 watts. This contradiction in the literature shows the need for more research to be done on a CO₂ LISM process.

C. Gap

This contradiction presents another question: Is a CO₂ LISM process using copper (II) oxide as a sensitizer feasible for the fabrication of copper plated substrates? Based on the agreement between Suh et al. and Krasnow I hypothesize that CO₂ LISM can be accomplished on a copper(II) oxide loaded epoxy

substrate and the deposited film should have electrical properties similar to those of elemental copper.

This question will help demonstrate the feasibility of a CO₂ LISM process which may open up the accessibility of LISM to more groups and potentially find use in small scale circuit board fabrication and will help inspire more research into the field [2].

III. Methods and Experimentation

To explore the feasibility of a CO₂ LISM process, an experimental design with a mixed method was used in this study. In this experiment, a plate made of copper oxide and epoxy was patterned using a CO₂ laser. After patterning and activating the surface, the sample will be placed in a copper plating bath which will deposit copper onto the activated areas. By visually comparing the surfaces and cross-sections of the samples after activation and after plating, the quality and effectiveness of this process was determined. Additionally, the resistivity, or electrical resistance per unit length, of the samples was also measured to determine the effectiveness and feasibility of the process.

A. Materials

A two part epoxy resin purchased from Art n' Glow was used as the substrate of choice for the experiments due to the ease of integration of the sensitizing agent. This resin is mixed in a 1:1 ratio by weight of hardener to resin and cures over the course of 24 hours. CuO or Copper (II) Oxide was chosen as the activation in this study due to its low cost and ease of accessibility. The CuO was purchased from Alpha Chemicals with a purity of >95% and a mesh size of 325. For use in the electroless plating solution, glyoxylic acid was purchased from Sigma Aldrich in a 50% by weight solution, copper sulfate pentahydrate, sodium potassium tartrate, ethylenediaminetetraacetic acid (EDTA) solution and sodium hydroxide of reagent grade were purchased through Flinn Scientific. Distilled water was used for all experiments. All chemicals were ordered with the help of a chemistry teacher and the school office.

B. Preparation of Substrates

To prepare the substrates, equal parts epoxy hardener and resin were mixed together. The CuO powder was then added and stirred until fully combined. The resin CuO mixture was then poured into a mold, heated by hot air for several minutes to allow the resin to degas, and allowed to cure for 24 hours. At the end of the curing process I demolded

the epoxy. Several samples were prepared with varying ratios including 33% CuO by weight and 50% CuO by weight.

To ensure that the samples were clean, I cleaned the samples following the activation using distilled water and physical abrasion. A large amount of black charred film was removed during this step. Then after plating, I again cleaned the samples using 91% isopropyl alcohol and again more residue was removed.

C. Laser parameters and activation

For the activation of the substrates, an Epilog Zing 24 CO₂ laser was used. This laser is a 60 watt model and the power and speed of the laser can be adjusted before processing. The raster setting was the preferred technique for processing the substrates. In the raster mode, the laser carriage scans across the substrate and selectively activates it. The Epilog Zing 24 uses a DPI (dots-per-inch) setting to control the frequency of the laser pulses in its raster mode. For example, in the 100 DPI setting, the laser is pulsed 100 times across a one inch line. Based on the findings of prior research, the most successful activation occurred with a high laser frequency so the DPI was set to its maximum of 1000 DPI for all experiments [8]. The speed and power settings were adjusted throughout the experiment from 25-100%

and 20-80% of their maximum settings respectively. The power was varied from 12-48 watts and the speed from 200-1000 mm/s. Immediately prior to activation, the substrates were wiped down with water. During activation the image shown in Figure 2 below was patterned onto the substrate. This pattern helps show how different laser parameters (speed and power) affect the successfulness of the laser activation.

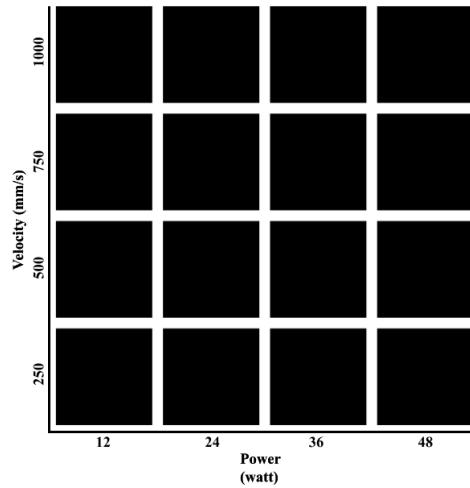


Figure 2. The image patterned onto the sample substrate was a 4x4 grid with axis corresponding to power of the laser and the velocity of the laser carriage. Each square was patterned according to the parameters at that square on the grid with a global setting (applied across all samples) of 1000 DPI.

During an initial test, it was found that the 60 watt power of the laser cutter combined with the lowest speed for the laser would cut through the sample (see Appendix 2) so, in subsequent

experiments, the power was limited to a maximum of 48 watts and the thickness of the substrate was increased.

D. Metallization

To metallize the samples, a homemade electroless plating solution was used. The solution is composed of copper sulfate pentahydrate (8 g/L), sodium potassium tartrate (30 g/l), citric acid (3 g/L), EDTA (3 g/L) and glyoxylic acid (12g/L) [12], [13], [14]. The pH of the solution was adjusted between 12 – 12.5 using sodium hydroxide. 500 ml of the solution was prepared by combining the materials in a 1 liter beaker. After adjusting the pH of the solution and heating it to 40°C, the solution is ready [12]. The samples were submerged for 30 minutes after which they were removed and dried. The procedure followed is shown in Appendix 1 and notes from the experiments are shown in Appendix 2.

E. Methods of Characterization

The end product of the LISM was characterized by the analysis of the electrical properties and cross sectioning. In addition, optical microscopy was used to observe the surface of the substrate before and after activation along with examining the deposited layer.

I) Resistivity Measurements: The electrical property measured was the resistivity of the sample. Resistivity can be measured using a 4-wire or Kelvin resistance probe and the dimensions of the respective area [8], [14]. An instrument was constructed to allow for the consistent and repeatable measurement of the resistance of the surface of the samples. A voltage is applied across the two outer pins and the current passing through the completed circuit is measured using an ammeter. The voltage across the film is then measured across two interior pins. The resistivity (ρ) of the sample is given using the equation

$$\frac{VA}{I\ell} = \rho_f$$

where V is voltage across the sample, A is the cross sectional area of the film, I is the current through the film and ℓ is the length of the sample measured, or the distance between the probes. This method of determining the resistivity is used as it largely eliminates the resistance of the probe wires.

2) Cross Sectioning: To cross section the films for inspection under a microscope, a perpendicular cross sectioning method was chosen [15]. The samples were encased in a layer of epoxy to protect the surface, and then cut perpendicular to the surface to expose a cross section of the layers. Because the newly exposed area is generally

damaged due to the cutting, it is not suitable to observe this cut until the area is sanded to a smooth surface. To fix this, the cut area is then sanded with increasingly fine sandpaper until there is a smooth exposed area [15]. In this experiment, the grit varied from 300 to 2000 grit. The sample is then examined from the cut and sanded side under a microscope.

3) Microscopy: Optical microscopy is useful in observing the surface of the substrate both after activation and after plating. This is helpful in understanding how different laser activation parameters affect the substrate and then how that affects the plating results. The optical microscopy was done with a stereo or dissecting microscope.

F. Research Ethics and IRB approval

Since this project did not involve any subjects or other participants from which data was collected, IRB approval was not needed. In addition, due to the non-toxic components of the electroless plating solution, filtering units on the laser, proper safety measures such as PPE and supervision by school faculty, there was negligible potential for harm while performing the experiments. The main ethical concern of the experiment was with the safe and ethical disposal of the electroless copper plating solution. Therefore, to dispose of the solution, the

copper was precipitated out, the solution was neutralized with HCl acid under a fume hood and the top water layer was decanted. This left copper metal in the bottom of the beaker and a water layer. The water layer was discharged into the sewer and the copper precipitate was washed and disposed of as waste.

IV. Results and Analysis

A. Activation Analysis

After the laser activation of the samples, magnified photos were taken and I made several observations. The most noticeable effects of activation, as shown in Figure 3 (a) below, are the reddening of the samples and also the formation of deep grooves which are particularly evident in the lower speed (250mm/s) samples. These are both signs that the activation was successful as the grooves or trenches formed are a product of the epoxy being vaporized by the laser and the discoloration a result of copper oxide being reduced. When vaporized, the epoxy decomposes to form hydrocarbons that act as a reducing environment. This indicates that the red discoloration present on the samples is most likely a result of the copper oxide being successfully reduced into copper metal. I also noted that, as the power of the laser increases during activation along the x axis,

a deeper discoloration becomes apparent in the samples and the grooves become more prominent.

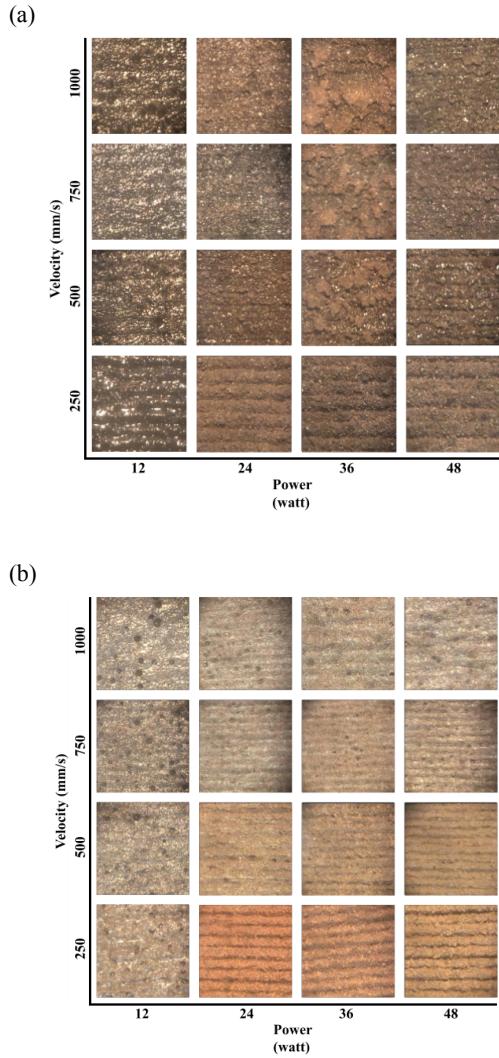


Figure 3. (a) The activated surfaces positioned at their respective coordinates under 15x top down magnification before electroless copper plating. **(b)** The activated surfaces after electroless copper plating under magnification.

By looking at the cross sectioned samples, several issues became apparent with the activation. As shown in Figure 4, where the highlighted gray

area indicates the outer area of the epoxy substrate, I noted the presence of copper beads embedded beneath the outer epoxy layer. Here the copper oxide was successfully reduced, but the epoxy seems to have melted and covered the metal. By covering the copper particles, the next step of the LISM process, the electroless plating process, is hindered and cannot be catalyzed. It appears from this analysis that the activation is only partially successful as some copper oxide was reduced, but that this copper oxide was embedded under the surface of the substrate.



Figure 4. In the 36 watt 250 mm/s cross sectioned sample shown above there are small copper beads deposited under the surface of the epoxy (colored gray). These beads were exposed after cross sectioning and were not evident from the initial microscopy.

B. Copper Plating Analysis

After plating and cross sectioning, measurements of affected depth from laser activation, height of copper deposition and other observations

were made. The most immediate change noticed was the brightening of the samples as shown in Figure 3 (b). Because of the heat and basicity of the electroless plating solution, it's likely that the substrate was partially cleaned which contributed to the brightening of the samples. Additionally, deposition of copper would also be a contributing factor to the increased brightness. This is especially noticeable in the 24 watt 250mm/s sample shown in Figure 3 (b). While the brightening seems indicative of successful plating, we would expect to see a reflective and uniform film of copper deposited as opposed to the matte surface I observed. To look at the different layers of the sample, I scratched the outer film of the 48 watt 250mm/s and a layer of copper was exposed, as shown in Figure 5 below. Unlike the surface of the samples which was matte, the newly exposed copper was reflective and brightly colored.

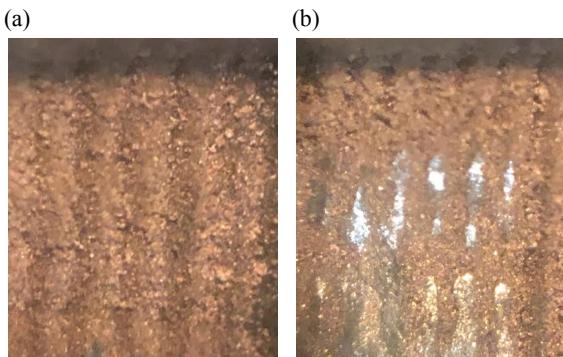


Figure 5. (a) The corner of the 250 mm/s 48 watt square. (b) The same corner as (a) after scoring with a tweezers.

By looking at the cross section of a plated sample in Figure 6 I noticed the non-uniformity and discontinuous nature of the deposited copper layer. This layer initially is very thick where the laser initially begins patterning, but the height of the layer decreases quickly to only several microns tall. This again becomes a discontinuous film with several noticeable breaks in the layer forming isolated regions of copper. This non-uniformity is a sign of poor activation and subsequent plating and may be a result of inconsistent laser power throughout the patterning. The effect of power on the height of copper deposited was also noticeable as higher power samples had around $>50\mu\text{m}$ of copper deposited. In the lower power and higher speed samples there was significantly less, or even no, copper deposited (Figure 7 below which shows the 500 and 750mm/s samples at 48 watts).



Figure 6. The cross section of the 48 watt 250mm/s sample shows how the activated and plated layers (discolored tan layers) decrease further into the sample away from the edge.

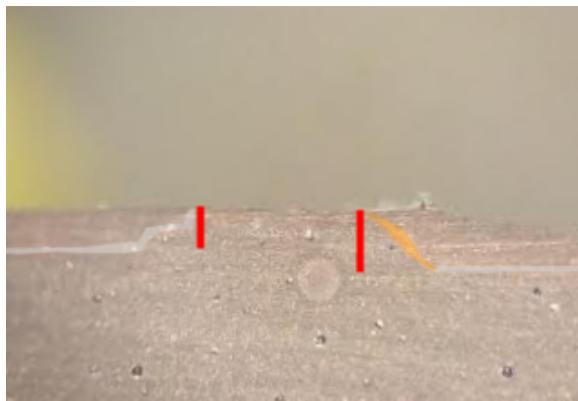


Figure 7. The 750 mm/s 48 watt (left) and 500 mm/s 48 watt (right) samples. The affected depth (red line) is significantly deeper in the 500mm/s sample compared to the 750mm/s sample.

C. Resistance

The resistance of the samples was determined using both a kelvin 4 wire probe and an AstroAI DT132A multimeter. Although this equipment should allow for the measurement of resistances up to several megaohms, the resistance of the activated and plated samples was too high to be measured. The plating process failed to create a film of copper across any of the activated samples and so, with limited interconnectivity between the substrate, the sheet resistance of the copper film on the samples was too high to be found. Because of the deep grooves produced during activation, there was most likely not enough time for the copper to deposit and fill in the grooves and form a continuous film.

D. Shear Cut Sample

While none of the 16 grid samples were successfully activated, one unintentionally activated sample was noticed and observations were made. This sample was found on the side of the substrate which had been cut using a 60 laser. As seen in Figure 8 (a) a layer of copper was deposited on the side of the sample. By cross sectioning the sample, the thickness of the film was measured as varying between 38 and 158 μm . While this sample was unintentionally created, it shows the results that would have been expected had the activation in the other samples been successful.

Although it was not possible to determine the sheet resistance of the 16 activated samples, the copper deposited on the side sample was conductive and the resistance of this sample was measured at 4.2 ohms. I determined the resistivity of the deposited layer was $6.57\text{e-}5 \Omega/\text{m}$ for the sample as compared to $1.68\text{e-}8 \Omega/\text{m}$ for pure copper [2]. The resistivity of the sample is much higher, likely due to low purity and non-uniform thickness of the sample. As shown in Figure 8, the copper is not of a uniform thickness although a consistent cross sectional area of $43800\mu\text{m}^2$ was assumed while doing calculations. It is likely that the area of the copper decreases further into the sample which leads to this discrepancy.

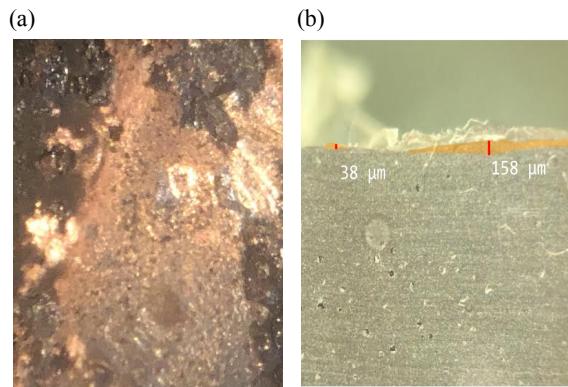


Figure 8. (a) Pictured is the laser cut and electroless copper plated edge of the sample. Under 15x magnification, the copper plated layer is clearly visible. (b) Cross section of the plated copper edge. There is a wide variation in the height of the deposited copper ranging from 38 μm to 158 μm . **Note:** Copper is highlighted in image (b).

E. Sample Defects

There were some defects noticed in the 36 watt samples that appeared as plateaus of epoxy and copper. These plateaus that appear after the activation step are likely a result of inadequate mixing in parts of the substrate that leaves clumps of epoxy and copper oxide. Then, after being laser etched or activated, they were unevenly reduced, leading to these flat surfaces.

I also noted the appearance of pitting or holes in the samples after the samples were removed from the plating solution. As seen in Figure 3 above, this is most notable in the 1000 mm/s samples. The pitting is most likely due to air bubbles being trapped in the epoxy while it was still a liquid. As the epoxy hardens, these bubbles form cavities that are exposed

only after being patterned. While they have little effect on my analysis and conclusions, they could impact the uniformity of a deposited copper film in an industrial or commercial setting.

V. Discussion and Conclusions

It appears that the activation process was, overall, very ineffective. While some copper oxide was successfully reduced to copper, there was both not enough reduced and not enough copper exposed on the surface to constitute a successful activation process. As a result of the epoxy melting instead of vaporizing, there was little reducing environment formed which prevented the reduction of more copper. Additionally, by melting, the reduced copper was covered by flowing melted epoxy which prevented the catalyzation of the copper plating process. Although the activation was poor, it appears that higher power samples were more successfully activated. This is especially true in the shear cut sample which underwent significant copper plating.

From this, it is evident that the CO₂ activation had a limited effect on the substrate. The parameters I intended to test, the 4x4 grid, were ineffective in activating the substrate primarily I suspect due to low laser power which prevented the successful reduction and exposure of copper oxide. While my parameters were ineffective, CO₂ LISM

still has potential as shown by the shear cut. The resistivity of the shear cut and the presence of deposited copper shows that, with more research, a process which uses CO₂ lasers to pattern copper oxide and epoxy is certainly feasible.

A. Limitations

There were several limitations in this project including time and equipment constraints. The largest limitation was the amount of time available in the lab. The instability of the electroless plating solution required the entire electroless copper plating process to be done in one continuous run which meant that the solution must be created, the sample cleaned and prepared, plated and then subsequently cleaned again in one time block. Due to the scheduling of school, the only time that this would work is after class so I was limited to 3 hour runs in the lab which allowed for 30 minutes of plating time. This lack of total plating time meant that it is harder to determine the effectiveness of the copper plating process and additionally, since this is not enough time for a continuous sheet to be deposited, makes it difficult to determine the resistance of the deposited copper. Another limitation was in the use of non ideal tools. The microscope used in my project had only a 15-30x stereo zoom which prevented very close examination of the samples. With a higher power microscope, the

morphology, or surface texture, of the samples could be examined much more closely. Additionally, in an academic lab setting researchers would have access to advanced microscopy techniques and x-ray analysis equipment. This equipment and these techniques would provide quantitative data that would make it easier to make objective conclusions about the feasibility of the process.

Additional experience and better equipment would better prepare me to face these limitations during the process and equip me to carry out my data collection and analysis.

B. Problems and Future recommendations

It's important to note the issues encountered with the electroless plating solution. The first solution I used suffered several issues including very short bathlife and the poor adhesion of the copper. The short bathlife was a problem as, if left overnight, the solution would decompose and the copper would precipitate out. It was noted that this process is accelerated if the solution is heated and would generally occur in less than half an hour. When the copper fell out of solution it would weakly plate the topside of any exposed surface. Due to these issues, the solution described in Karthikeyan's paper was replaced by a solution described in another paper [2]. The main difference between these solutions are the

chelating or complexing agents used but I was unable to determine whether the complexing agents were the sole cause of my problems in Karthikeyan's solution.

Based on these experiments there were several key takeaways and recommendations I have. Foremost is the need for a higher power laser. From these experiments it was clear that a major problem was the lack of vaporized epoxy which prevented the exposure and reduction of the copper oxide. A thorough cleaning of the substrate via both physical and solvent-based means after activation and before plating is also required to allow the best adhesion of the plated copper. A more homogenous integration of the activation agent into the substrate, along with vacuum degassing of the epoxy should provide more

consistent results and help prevent some of the pitting found in my samples. A final recommendation is to use more accurate, or higher resolution, and varied equipment to aid in data collection. Without adequate equipment it is hard to very accurately assess the success of activation.

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Appendix 1

Procedures for Electroless Copper Metallization Solutions

Preparing Solution (Method #1)

Karthikeyan, Vasudevan, Srinivasan & John

Materials (100ml)

Chemicals

Copper sulfate Pentahydrate	1.6 g
Sodium potassium tartrate	3 g
Triethanolamine (TEA)	3 mL
Glyoxylic acid	2 mL
Sodium hydroxide	1.5 g (pH adjust)
Deionized or Distilled water	

Equipment

Beaker	1 L
Container to store solution	>100 ml
Graduated cylinder	
Syringe	10 ml x 2
Weighing Boat	
Scale	
pH paper or electronic meter	
Stir plate	
Ice Bath	
Fume Hood	
Gloves/Goggles	

Procedure

Add 50 ml of DI water to a 1 liter beaker.

Weigh the copper sulfate and sodium potassium tartrate, measure the 2 ml glyoxylic acid and 3 ml TEA with separate syringes, adding each to the beaker after measuring.

Raise the volume of the solution to 100 ml using DI water.

Prepare an ice bath and place the beaker into it.

Slowly add sodium hydroxide to the solution until the pH reaches between 12-12.5 stirring until everything is dissolved.

Test use

Heat the solution to 50 C.

Weigh a sample of copper and then add to the solution.

Preparing Solution (Method #2)*Honma & Kobayashi***Materials (100ml)****Chemicals**

Copper sulfate pentahydrate	0.75 g (.03M)
Ethylenediaminetetraacetic acid (EDTA)	7.01 g (.24M)
	1.75 g (.06M)
Glyoxylic acid	1.96 g (.20M)
(Glyoxylic acid 50% w.)	1.46 ml
2,2' Bipyridine	10ppm
Potassium hydroxide	(pH adjust)
Deionized or Distilled water	

Equipment

Beaker	
Container to store solution	> 100 ml
Graduated cylinder	
Syringe	10 ml
Weighing Boat	
Scale	
pH paper	
Stir plate	
Ice Bath	
Fume Hood	
Gloves/Goggles	

Procedure

Add 50 ml of DI water to a beaker.

Weigh the copper sulfate and EDTA, measure the 1.5 ml glyoxylic acid with a syringe, adding each to the beaker after measuring.

Raise the volume of the solution to 100 ml using DI water.

Prepare an ice bath and place the beaker into it.

Slowly add potassium hydroxide to the solution until the pH reaches between 12-13 stirring until well dissolved.

Test use

Heat the solution to 50 C.

Weigh a sample of copper and then add to the solution.

Allow to plate for 30 minutes then remove, pat dry, rinse then dry again.

Reweigh the sample.

Preparing Solution (Method #3)*Zhang, Feng, Jia, Zhang, Zhang, Sun & Zhou***Materials (100ml)****Chemicals**

Copper sulfate pentahydrate	0.8 g
Sodium potassium tartrate	3 g
Sodium citrate	0.3 g
Ethylenediaminetetraacetic acid (EDTA)	0.3 g
Glyoxylic acid 50% w.	1.46 ml
Sodium hydroxide	(pH adjust)
Deionized or Distilled water	

Procedure

Add 25 ml of 0.04M EDTA solution to a beaker.

Then weigh/measure the remaining chemicals (not including the hydroxide) and add them to the solution.

Raise the volume of the solution to 100 ml using DI water.

Equipment

Beaker	
Graduated cylinder	
Syringe	10 ml
Weighing Boat	
Scale	
pH paper	
Stir plate	
Ice Bath	
Fume Hood	
Gloves/Goggles	

Prepare an ice bath and place the beaker into it.

Slowly add potassium hydroxide to the solution until the pH reaches between 12-13 stirring until well dissolved.

Test use

Heat the solution to 50 C.

Weigh a sample of copper and then add to the solution.

Allow to plate for 30 minutes then remove, pat dry, rinse then dry again.

Reweigh the sample to calculate the copper deposited.

Using the solution**Materials**

Thermometer
Plating solution
Samples to be plated
Hotplate
1 Liter beaker
Stir rod
Water
Paper towel

Plating

Pour solution into a 1 liter beaker.
Raise the temperature to 30°C and stir to ensure the solution is fully dissolved.
When the temperature reaches 30°C, add the samples to the bath and allow to sit undisturbed.
Remove the samples from the solution, rinse with water and pat dry.
Allow solution to cool and return to storage.

Appendix 2

Electroless Metallization Notes

Below is the link to a PDF of my journal used to record the progress of my experiments. It does not contain images from my analyzed results and includes mostly images from the plating process.

