Volume 4, Issue 6, 2014, 891-894 ISSN 2069-5837
Biointerface Research in Applied Chemistry
www.BiointerfaceResearch.com
Original Research Article Open Access Journal
Received: 15.10.2014 / Revised: 28.11.2014 / Accepted: 30.11.2014 / Published on-line: 15.12.2014

Influential factors in the adhesion of a metal layer deposited on an epoxy-resin plate in aqueous solution

Cunhua Chen [1,*], Xiaopeng Qu [1], Zhimou Xu [2]
[1]Department of Chemistry, Central China Normal University, Wuhan 430079, PR China
[2]Huazhong University of Science & Technology Wuhan 430074, PR China
*corresponding author e-mail address: ***cunhuachen@mail.com***

ABSTRACT

In this work, an Ag metal layer was deposited on an epoxy resin board using low power CO_2 laser-induced deposition in an aqueous solution. The Cu layer was rapidly deposited on the Ag metal layer due to Ag crystallite activity. This technique can also be used for sterilization in biology .The micro-structure of the metal layer was investigated using Electric Probe Micro-Analysis (EPMA) and an Atom Force Microscope (AFM). Adhesion between the metal layer and substrate was investigated using a Material Tensile-Ergometer. It was concluded that the adhesion of the metal layer was influenced by the surface morphology, the diffused depth of the Ag layer on the epoxy-resin board and surface roughness. It provides a new method for the preparation of biological engineering materials.

Key words: *CO_2 laser -induced, metal depositing, sterilization , adhesion, AFM analysis.*

1. INTRODUCTION

Plating silver or copper on biological resin material, it not only can help sterilization, but also can increase the circulation utilization. It is a major improvement for the traditional biological engineering materials.

In micro-electronics, efficient and low cost manufacturing techniques for high performance metal deposits on epoxy resin board can provide reduced cost PBC (Printed -Circuit Board). In recent years, there has been increased interest in local metallization on the surface of PBC or polyimide (PI) for laser-induced depositing metal lines (such as Cu or Ag line, etc.) [1-3]. Now plastic instruments replace metal instruments,they can do better to reduce the survival rate of bacteria and disinfection. Previous research has rarely utilized short-wave lasers, such as Argon lasers, helium, neon and Nd: YAP with selective energy absorption capacities for some metal ions, due to their high maintenance costs; nevertheless, some reported instances include Cu induced-deposition by an argon laser at 514.5nm and Ni induced–deposition by a Nd: YAP laser[4-7]. The development of a low power CO_2 laser (wavelength of 10.6 μm) for the induction of Ag and Cu deposits on PI (or epoxy resin) substrate from aqueous medium has significant potential as a new, low cost and versatile method. The factors influencing the adhesive properties of the metal layer on PI (or epoxy resin plates) obtained using low power CO_2 laser-induced deposition in an aqueous solution are also largely significant.

2. EXPERIMENTAL SECTION

The experimental device is shown in Figure.1. The mixed solution was composed of $AgNO_3$ (analysis) and other pure compounds; it was homogeneous and colloid. The CO_2 laser (produced by Huagong Science and Technology Industry Company) was 15W. The laser was a TEM_{10} model with a spot diameter of 0.1～1mm.

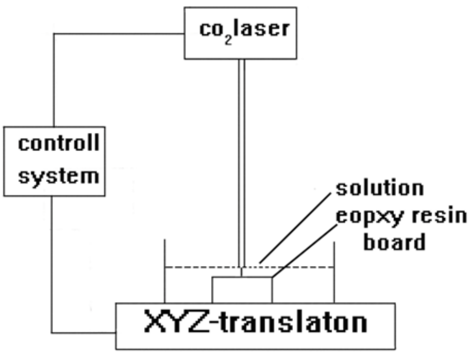


Figure 1. Setup for co_2 laser-induced deposition from aqueous solution

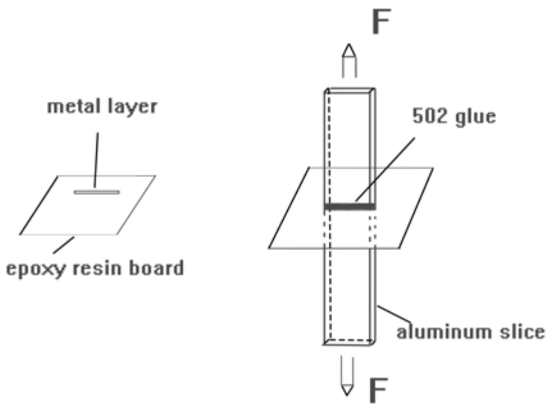


Figure 2. Measurement of adhesion of Cu layer with epoxy resin board

All of the process parameters were controlled through a computer. The epoxy resin plate was 40×20×1mm. First, cleaned surface specimens were immersed in a composite solution under room temperature conditions. The laser-induced deposition of Ag on the surface of epoxy-resin plate was then performed under different conditions before the electroless plating of copper. Metal surface topography, thickness of deposition and adhesive force were examined using an EPMA (electron-probe micro-analyzer, JXA-8800R) and AFM (Atom Force Microscope, SPA-400). The adhesive force tests is shown in Figure 2. The thickness and length of the two aluminum slices were processed according to the size of metal line/layer. Cross-section areas equal in size to wire/layer surface areas were connected with glue.They were left static and maintained at room temperature for 24 hours. The adhesive force was tested by a Material Tension Machine (Japan SGM-50kN). The tensile speed was controlled at 10cm/min.

3. RESULTS SECTION

3.1. The Effect of Silver Metal Layer Thickness on the Adhesive Force of Cu/Ag Line.

It is essential that the surface of a non-metal matrix such as epoxy resin first be activated in $SnCl_2$ - $PdCl_2$ colloid solution before the electroless plating of copper. As a result, dispersive particles such as Sn^{2+}/ Pd^{2+} absorbed and then formed a catalytic centre on the surface of the matrix, which provided easy Cu deposition via the electroless plating of copper. Ag^+ is cost economical as it not only activates the surface of the nonmetal matrix, but also replaces Sn/Pd. In this research, an Ag layer was deposited on the epoxy-resin surface via CO_2 laser-induced deposition, which was then covered by Cu deposition. In this process, the decomposition reaction was:

$$Ag^+ + 2NH_3 \cdot H_2O \rightarrow Ag(NH_3)_2^+ + 2H_2O \quad (1)$$

$$Ag(NH_3)_2^+ + NO_3^- \rightarrow Ag + 2\,NH_3 + NO_2 + O_2 \quad (2)$$

In chemical reaction (1), NH_3 was the complexing agent and catalyst, which enhanced the stability of Ag^+ in the solution with decreasing electrode potential $E_{Ag^+/Ag}$. In chemical reaction (2), NH_3 volatilized gradually and electrode potential $E_{Ag^+/Ag}$ increased with laser scanning. The Ag^+ stability was weakened and Ag^+ was reduced to metal Ag. At the same time, the local area melted due to energy absorption, and Ag crystallite gradually infiltrated into the epoxy-resin plate. Next, a “transitional area” of Ag crystallite and organic compound formed on the matrix surface. It was found that increased infiltration of metal particles led to a thicker “transitional area”.

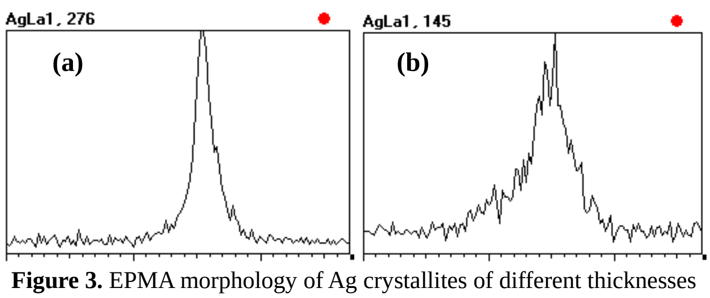


Figure 3. EPMA morphology of Ag crystallites of different thicknesses (line analysis); (a) laser power P=3W thickness: 5.5 um; (b) laser power P=3.5W thickness：9 um;

The thickness of the transitional area and the Ag crystallite layer directly influenced the adhesive force between the subsequent Cu/Ag line and plate. A cross-section EPMA analysis was used to obtain the average thickness values and microstructures of Ag crystallite layers as shown in Figure 3, where abscissa indicates width value and ordinate indicates length value. The thickness of the metal layer and transitional area was shown to increase with increasing laser power. Consequently, the adhesive force of the metal line formed by the Ag layer and subsequent Cu coating was determined based on this width. As shown in Figure 4, the adhesive force of Ag/Cu line increased with increasing Ag crystallite thickness.

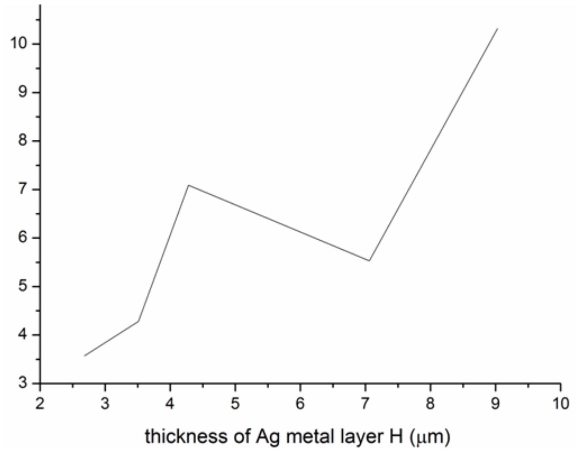
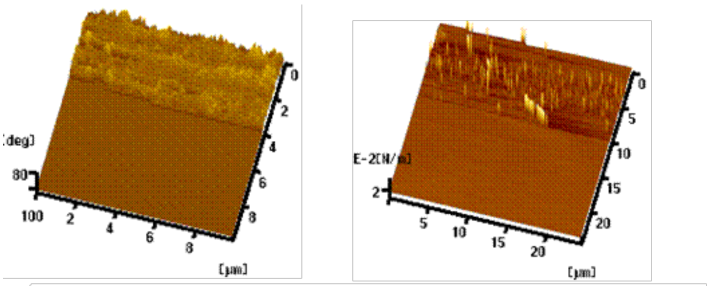


Figure 4. Curve of Cu layer adhesion and Ag crystallites thickness

3.2. The Influence of Ag Layer Surface Morphology on the Adhesive Force of Ag/Cu Line.

After the low power CO_2 laser, the catalytic activity of Ag led to deposition on the epoxy-resin plate. In the electroless plating of copper, the surface morphology of the Ag layer was highly influential on the adhesion of copper coating. Generally, the symmetrical and thin Ag crystallite layer was not only propitious for the inner pervasion on the matrix surface, but also to the tightness and uniformity of subsequent copper deposition. It was thus determined that a more symmetrical and thinner Ag crystallite layer provided good adhesion force. Conversely, an incompact and sparse layer could not be covered by tight and symmetrical Cu deposition. AFM was used to illustrate the surface topography of the Ag crystallite layer under different laser powers and scanning speeds, as shown in Figure.5. Two three-dimensional photos of compact and sparse metal layers are displayed. The performance of the Ag crystallite layers varied according to laser power, scanning speed and compound solution, among other properties. Excessive laser power resulted in evaporation and carbonization in the solution and plate surface. The right photo (b) in Figure.5 depicts a sparse Ag layer on a plate surface obtained using a small amount of low laser power, while the left photo (a) illustrates a good Ag layer obtained with optimum laser power. The identification of appropriate technical parameters was a vital issue in the processing of CO_2 laser-induced deposition.



a: (P=4W,V=0.85m/min) b: (P=2.5W,V=1.4m/min)

Figure 5. AFM surface morphology of Ag crystallites deposited by different condition

The Ag layer could be covered with Cu coating using electroless plating. The surface morphology of the Cu coating is shown in Figure 6, as illustrated with SNOM. A symmetrical and compact Ag crystallite layer was shown to result in symmetrical and compact Cu coating. The uniform and dense Cu layer provided a strong foundation for a good adhesive force. The adhesive force of the Ag/Cu coating (line) was tested using a Material Tension Machine. The adhesive force values are shown in Figure 6. Pictures (a) and (b) depict better adhesive force in more dense and compact copper coating than sparser and looser copper coating. Technical parameters were set to the following: P=4W, V=0.85m/min or P=2.5W, V=1.4m/min (laser power (P) and scan speed (V)). The adhesive force reached up to 10.18 kg/cm^2 or 2.52kg/cm^2.

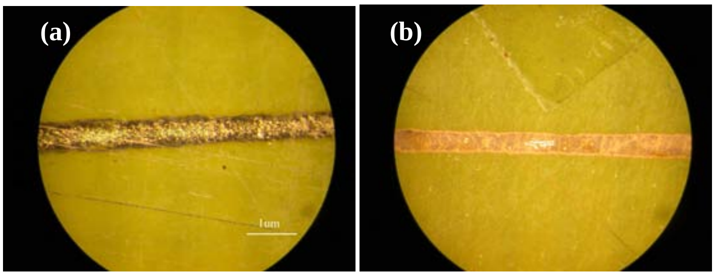


Figure 6. SNOM surface morphology of Cu deposited on the Ag by electro-less Plating. The adhesion of metal layer is: (a) f=10.18 Kg/cm^2; (b) f=2.52 Kg/cm^2;

3.3. The Influence of Epoxy-Resin Plate Surface Roughness on Cu Coating Adhesion.

It was determined that the adhesion of Cu coating varied according to factors such as physical structure, interface layer (i.e. transitional area) and substrate properties in addition to epoxy-resin plate surface roughness. It is known that the surface roughness of epoxy-resin plate can be improved using physical methods, such as cutting and polishing, and chemical methods, such as cleaning and etching. More recently, laser scanning activation has been proposed as a new method for processing nonmetal materials, such as epoxy-resin, PI and ceramics, among others. It is known that the surface roughness of epoxy-resin plate can be improved using physical methods, such as cutting and polishing, and chemical methods, such as cleaning and etching. More recently, laser scanning activation has been proposed as a new method for processing nonmetal materials, such as epoxy-resin, PI and ceramics, among others. However, chemical etching and laser scanning can change the physical structure and chemical composition of the surface.

The surface roughness and morphology of the epoxy-resin plates were tested by AFM, as shown in Figure 7. The three pieces of epoxy-resin plate were processed using different methods. The original surface (shown in Figure 7a) was smooth with less surface roughness. Chemical etching was used as an epoxy resin polymer surface treatment to provide a rough surface and strong "solution corrosion" (as shown in Figure 7b). Laser surface treatment provided strong local melting and enhanced surface roughness (as shown in Figure 7c). Further laser interaction with the surface of epoxy resin provoked a coarser surface (Figure7c).

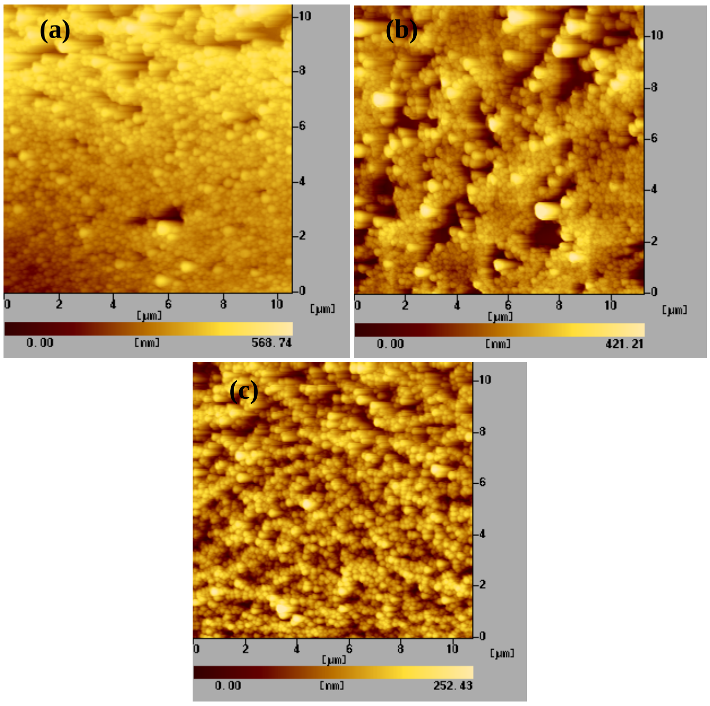


Figure 7. AFM surface morphology of the same substrate activated by different methods

The Ag/Cu coating was first deposited on the three afore mentioned plates using laser-induced deposition in aqueous solution. The plates were subsequently cleaned and dried. Adhesive force was tested in accordance with the abovementioned method. The roughness and adhesive force data is shown in Table 1. The adhesive force of the metal coating was seen to increase with increased roughness.

Table 1. The surface adhesion of metal layers with different roughness value

No. content	roughness data RMS(nm)	adhesion F(kg/cm^2)
a	8.00×10^0	0.047
b	1.48×10^2	1.58
c	4.02×10^2	2.31

4. CONCLUSIONS

In this work, the adhesive force of a Ag/Cu metal coating after CO_2 laser-induced metal deposition in an aqueous solution was shown to be influenced by series of factors, including thickness of the activated metal layer (Ag), surface topography of the Ag layer and surface roughness of the epoxy-resin plate. It was found that increased thickness in the activated metal layer provided enhanced adhesive force in the Cu coating obtained from electroless plating. Moreover, the surface morphology of the activated metal layer on the epoxy-resin plate was also seen to influence the adhesive force of Cu coating. This technology will be widely applied in biological science in the future. Optimized technical laser parameters provided a more symmetrical and compact Ag layer. Surface roughness was also found significant for enhanced adhesive force in the Ag/Cu layer. Generally, good surface roughness can be obtained using various activation methods for different materials.

5. REFERENCES

[1] Malgorzata N., Wojciech J.S, Maresk P., Dariusz Z., Zbigniew B., Jerzy B.A., Comparative study on the hydrogen absorption of thin films at room temperature deposited on non-porous glass substrate and nano-porous anodic aluminumoxide template, *International Journal of Hydrogen Energy*, 36, 18, 11777-11784, **2011**.

[2] Huang F., Lou Q., Dong J., Excimer laser treatment of fluorocarbon resin for improved adhesion, *Applied Surface Science*, 174, 1-6, **2001**.

[3] Shafeev G.A., Hoffmann P., Light-enhanced electro-less Cu deposition on laser-treated polyimide surface, *Applied Surface Science*, 138-139, 455-460, **1999**.

[4] Kordás K., Leppävuori S., Uusimäki A., George T.F., Nánai L., Vajtai R., Bali K., Békési J., Palladium thin film deposition on polyimide by CW Ar+ laser radiation for electroless copper plating, *Thin Solid Films*, 384, 2, 185-188, **2001**.

[5] Kordás K., Bali K., Leppävuori S., Uusimäki A., Nánai L., Laser direct writing of palladium on polyimide surfaces from solution, *Applied Surface Science*, 152, 149-155, **1999**.

[6] Kordás K., Nánai L., Bali K., Stépán K., Vajtai R., George T.F., Leppävuori S., Palladium thin film deposition from liquid precursors on polymers by projected excimer beams, *Applied surface Science*, 168, 66-70, **2000**.

[7] Békési J., Kordás K., Beleznai Cs., Bali K., Vajtai R., Nánai L., Uv-laser-induced etching and metal seeding on polymers, *Applied Surface Science*, 138-139, 613-616, **1999**.