

Parameters optimization of electroless deposition of Cu on Cr-coated diamond

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Abstract: Electroless copper plating on diamond particles precoated with 1% Cr was carried out to evaluate the effects of various experimental parameters on coating quality and deposition rate to obtain the optimized reaction parameters. The formulated samples under optimized parameters were characterized by X-ray diffraction, scanning electron microscopy, energy dispersive spectroscopy, X-ray photoelectron spectra and optical microscopy. The best parameters, where uniform and maximum coating thickness was achieved, are etching with 20% NaOH for 30 min, sensitization and activation with SnCl₂ and PdCl₂ for 5 and 20 min, respectively. The composition of the copper solution bath was 16 g/L CuSO₄·5H₂O, 35 mL/L formaldehyde (HCHO), 23 g/L KNaC₄H₄O₆ at 60 °C, pH=13 and stirring at (350±15) r/min under ultrasonication.

Key words: electroless copper plating; Cr-coated diamond; parameter optimization

1 Introduction

The fundamental of electroless deposition is the reduction of metallic ions from the solution onto the substrate to be metallized without application of electric current. It is widely used in fabrication of printed circuit boards because of its low cost, easy deposition and very simple experimental setup [1,2]. Electroless metallic deposition has drawn a special attention in fabrication of fine metal patterns for microelectronics, wear and corrosion resistant materials, medical applications and battery technologies [3–5]. Generally, it requires a catalyst to initiate the autocatalytic metallization process. Selective and delicate metal deposition can only be done using this process, which is a challenge for electrolytic process [6]. Through this process, a uniform copper coating is obtained on a variety of substrates, like diamond particles [7]. The deposition rate and deposit properties of electroless copper plating depend on the copper complexing agent, reducing agent and bath temperature and pH value of the process [8]. The latest interest in copper electroless deposition has grown for it is used to fabricate ultra-large scale integrated circuits (ULSI) because of the higher conductivity of copper than

aluminum [9–12].

Electroless copper plating on diamond has rarely been mentioned in previous literatures because of the cost and poor or zero wettability of diamond with other elements. No methods were available to produce diamond synthetically on commercial basis. However, recently, some economical methods have been developed to produce diamond synthetically like CVD diamond [13,14] and to catalyze the surface chemically for electroless copper coating. Recently, it has drawn the attention of researchers around the globe when the need of a material with high thermal conductivity and compatible coefficient of thermal expansion with semiconductors has become a vital requirement. Hence, it needs a careful study on the process so that the best coating could be achieved. For this, diamond's surface treatments are necessary prior to the ordinary electroless plating process to make diamond particles more susceptible to adhesion of the palladium/tin catalyst and enhance the adhesion of the successive copper plating on the diamond particles. A layer of catalyst particles gets attached in the surface micro-cavities formed during surface treatments, providing redox reaction catalytic sites for later initiation of the electroless copper plating process [15].

In the present work, diamond particles already coated with 1% Cr (mass fraction) layer were selected because it was more easy and economical to catalyze the surface than pure diamond and to get uniform coating thickness. Hence, a careful study on each step was carried out to investigate the influences of experimental parameters and then these parameters were optimized, by varying experimental conditions, through SEM analyses and using other very simple techniques to list down these optimized parameters, in order to establish a stable and higher quality deposit on Cr-diamond particles.

2 Experimental

The diamond type selected for electroless plating was MBD-8 (110 μm) with 1% Cr coating. Diamond particles were first pretreated to catalyze the surface, etching by 20% NaOH, sensitizing by SnCl_2 and activating by PdCl_2 . These pretreated diamond particles were introduced into copper solution bath, composed of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, $\text{KNaC}_4\text{H}_4\text{O}_6$, 20% NaOH and formaldehyde (HCHO) for electroless plating. The mass of coated diamond particles was calculated and then these particles were introduced into HNO_3 . After complete dissolution of deposited copper, diamond particles were dried and weighed again and hence the mass gain was calculated. Distilled water was used throughout to prepare these solutions and also to wash the coated diamond particles gravitationally.

3 Results and discussion

3.1 Pretreatment of diamond particles

During pretreatment, effect of each step on the surface morphology of the diamond particles was investigated and the best set of parameters was listed.

3.1.1 Etching effect on diamond particles

The main purpose of etching with 20% NaOH was to remove impurities, create micro cavities, oxidize the surface in order to increase the rate of sensitization and activation, which resulted in good ion-ion adhesion on diamond particles surface [16]. The electroless coatings with or without etching are shown in Fig. 1. The coating film thickness on diamond particles without etching was very thin as shown in Fig. 1(a) where the coating thickness was very high on the etched particles, as shown in Fig. 1(b). The best etching time after series of experiments was 30 min.

3.1.2 Effect of sensitization and activation processes

Diamond particle without being catalyzed can not be metallized with any other elements due to its inertness. Sensitization and activation were carried out by

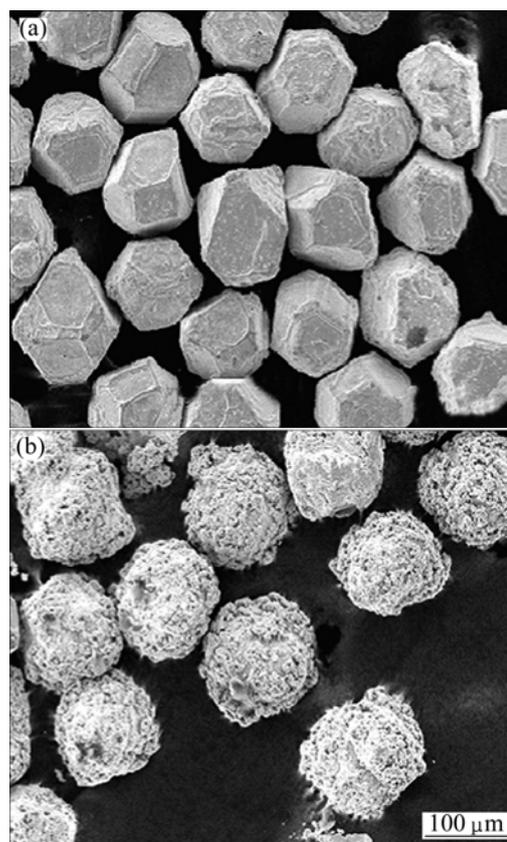
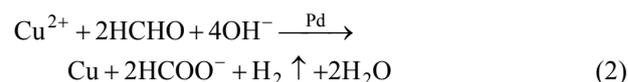


Fig. 1 SEM images of diamond particles: (a) Direct plating without 20% NaOH etching; (b) Plating after 30 min etching with 20% NaOH

immersing diamond in $\text{SnCl}_2 + \text{HCl}$ and $\text{PdCl}_2 + \text{HCl}$ solutions, respectively. During sensitization Sn^{2+} ions are attached to diamond particles at micro cavities, and during activation, Pd^{2+} ions are reduced by Sn^{2+} ions to Pd layer, which acts as catalyst for subsequent electroless deposition of copper [17]. The overall reaction of Pd reduction is as follows:



Then diamond particles were immersed in copper solution bath with formaldehyde as reducing agents. The redox reaction during electroless plating is as follows:



Pd and Cu^{2+} ions as catalyst were reduced by formaldehyde (HCHO). Pd deposited on the diamond surface was taken out by creating new nucleation sites for deposition of Cu^{2+} ions on the surface and hence deposition of Cu took place [18,19].

The effect of sensitization and activation time on the mass gain of electroless copper plating is shown in Fig. 2. Anchoring of Sn^{2+} ions did not take a long time to deposit on diamond surface; while the reduction of Pd^{2+}

ions by Sn^{2+} took a long time. Hence, after a series of experiments the best time for sensitization was 5 min and 20 min for activation.

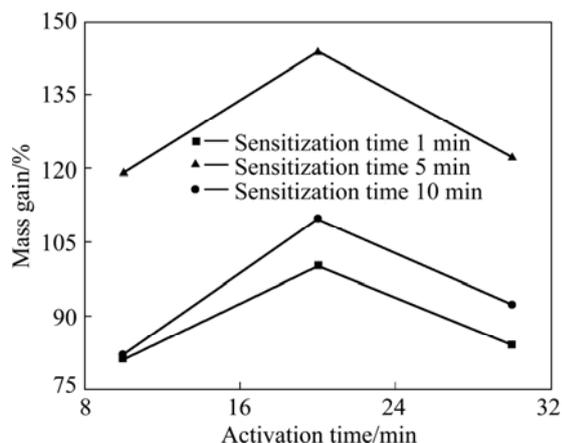


Fig. 2 Effect of sensitization and activation time on mass gain of electroless copper plating on Cr–diamond particles (110 μm)

The concentration of activation solution ($\text{PdCl}_2 + \text{HCl}$) greatly affected the plating phenomena as shown in Fig. 3. It shows that thickness and uniform coating on diamond particles first increase and then decrease, when the concentration of PdCl_2 was increased from

0.25 g/L to 1.0 g/L and HCl from 10 mL/L to 40 mL/L, while keeping all other conditions the same. The most uniform coatings and highest deposition rate were achieved when the concentration of activation solution was 0.75 g/L $\text{PdCl}_2 + 30$ mL/L HCl. It is due to the fact that the lower the concentration is, the lower the reduction activity of Pd^{2+} is, resultantly the lower the nucleation sites created are for Cu^{2+} ions to deposit on diamond surface. However, when the concentration of Pd^{2+} ions is too high, more Pd^{2+} ions are attached to Cu^{2+} ions, decreasing the reduction activity of HCHO on the diamond surface, suppressing the bath stability and plating phenomena [20].

3.2 Electroless copper plating

3.2.1 Effect of different complexing agents

It is compulsory to use complexing agents in order to avoid the precipitation of $\text{Cu}(\text{OH})_2$ under alkaline solution conditions. In contrast, both bath stability and choice of reducing agent are dependent on complexing agent, which not only affects the rate of crystal formation but also reduces its possibility if the formation constant is higher [19].

EDTA, TEA, citric acid and $\text{KNaC}_4\text{H}_4\text{O}_6$ are commonly used as complexing agents. The effect of

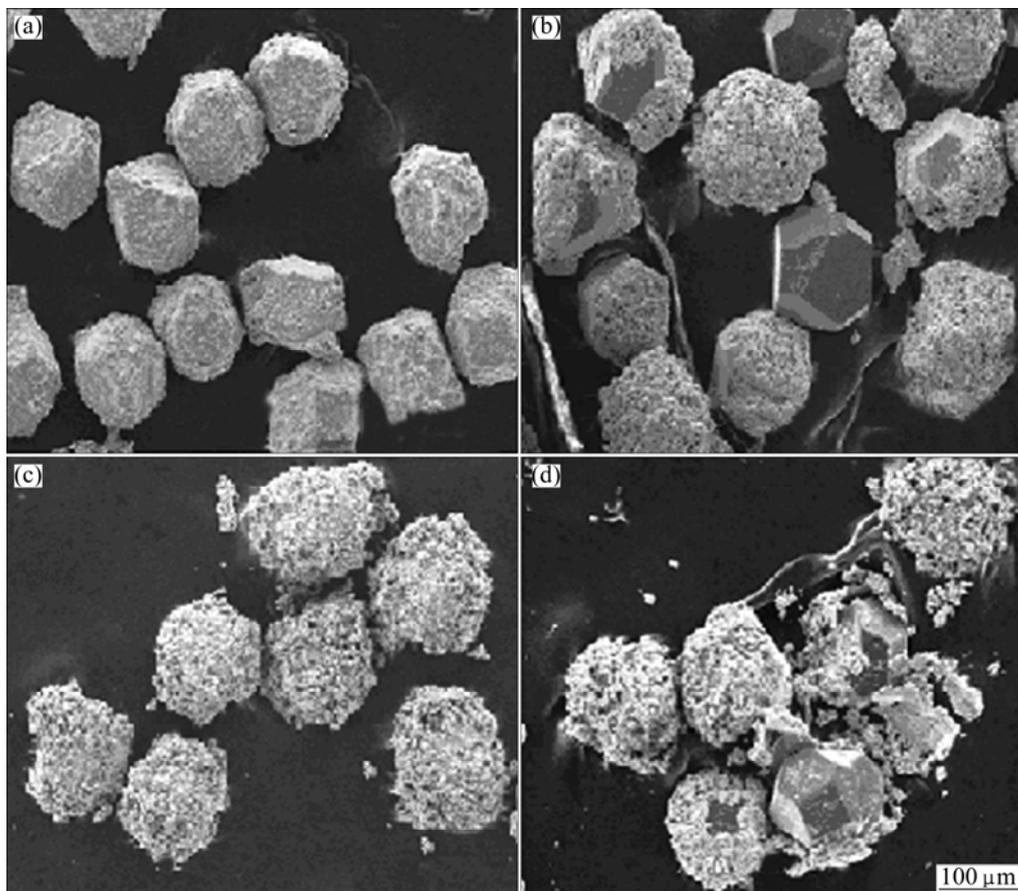


Fig. 3 SEM images showing concentration effect of activation solution on plating: (a) 0.25 g/L $\text{PdCl}_2 + 10$ mL/L HCl; (b) 0.5 g/L $\text{PdCl}_2 + 20$ mL/L HCl; (c) 0.75 g/L $\text{PdCl}_2 + 30$ mL/L HCl; (d) 1.0 g/L $\text{PdCl}_2 + 40$ mL/L HCl

complexing agents on plating process is as follows: EDTA, TEA, $\text{KNaC}_4\text{H}_4\text{O}_6$, citric acid.

EDTA forms strong complexes with cupric ions during plating, making bath highly stable and resultantly the deposition rate is very low. TEA based electroless copper deposition has a very high deposition rate because it forms weaker complexes with cupric ions, but it occurs only after palladium surface is covered with fresh layer of copper; otherwise, TEA adsorption could poison the catalytic oxidation of formaldehyde on active palladium surface, slowing down the deposition rate [21]. EDTA and $\text{KNaC}_4\text{H}_4\text{O}_6$ are most widely used complexing agents in electroless copper solution bath with HCHO as reducing agent due to their availability and low cost [22,23].

The complexing ability of citric acid is very weak, which easily leads to the passivation of the surface, thereby reducing the copper plating process; however, it has also been found that citrate is an appropriate complexing agent for electroless copper plating when using hypophosphite as the reducing agent, leading to a substantial plating rate [24]. The effects of these complexing agents are shown in Fig. 4. Comparison of electroless plating using different complexing agents shows that $\text{KNaC}_4\text{H}_4\text{O}_6$ is an ideal complexing agent, which gives the highest deposition rate.

3.2.2 Effect of pH value

pH value is a very important factor during electroless copper plating process. By increasing the pH value, the reduction of formaldehyde (HCHO) increases and its value can be maintained or changed by the addition of 20% NaOH solution. At $\text{pH} > 12$, coatings are more homogenous and uniform and if pH is too lower than 8, the deposition of Cu does not take place [25]. Different pH values and their effects on the surface morphologies were investigated and are shown in Fig. 5. At $\text{pH} = 11$, there was very little plating. At $\text{pH} = 12$, the plating started. At $\text{pH} = 13$ and if all the other factors are unchanged, then the best coating was obtained. At $\text{pH} = 14$, the bath stability started to decline, decomposing HCHO, hindering the plating process. This phenomenon of first increase and then decrease in the plating rate was explained by FAANCIS and NUZZI [26] in the following two steps.

The initial increase in coating rate ($\text{pH} > 12$) was due to the fact that OH^- ions behaved as reactants as shown in Eq. (2). The decrease in the deposition rate at $\text{pH} > 13$ was due to the consumption of OH^- ions by hydrolysis of HCHO into methylene glycol anions and ultimately reduction to formate ions as shown in Eqs. (3) and (4):

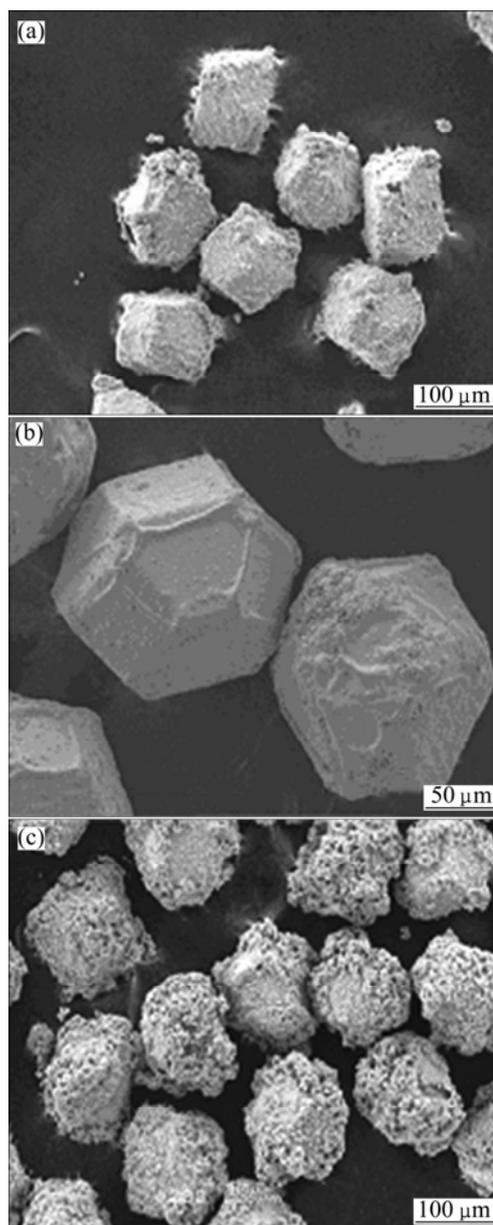


Fig. 4 SEM images showing effect of different complexing agents on pure diamond surface: (a) EDTA; (b) Citric acid; (c) Potassium sodium tartrate

3.2.3 Effect of temperature

Temperature being important factor was optimized to get the best plating results. The effect of temperature variation is shown in Fig. 6. No plating was observed at 30°C , while very small plating was observed at 40°C . The best plating result was achieved at 60°C . The plating started above 40°C and it was accelerated with the increase of temperature. If there was no change in the external conditions, then best plating was obtained at 60°C . In addition, if the temperature exceeded 60°C , the stability of bath declined. Due to rapid decomposition of HCHO (being volatile), the plating phenomena were reduced [16].

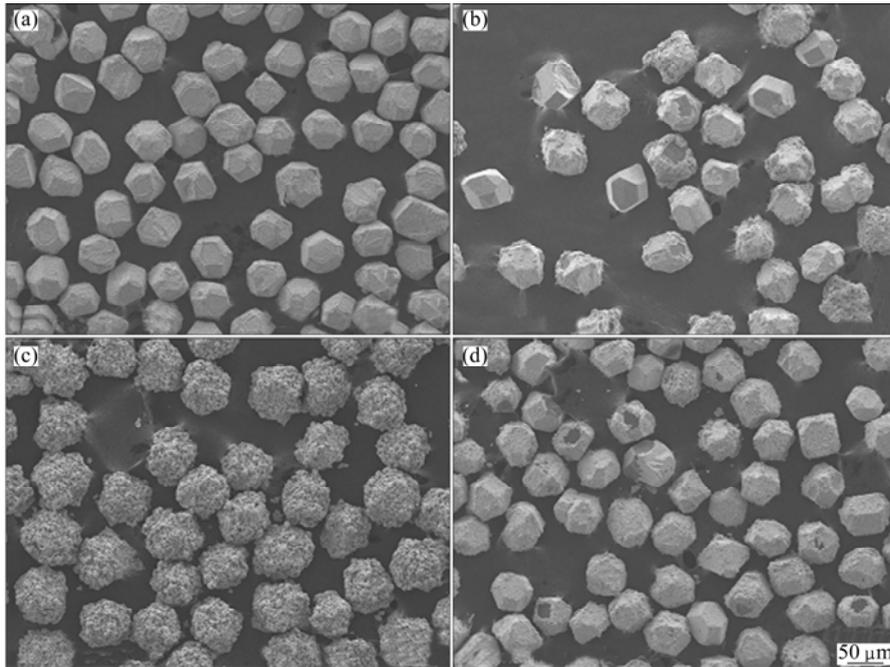


Fig. 5 SEM images showing effect of different pH values on surface morphology of Cr–diamond (110 μm): (a) pH 11; (b) pH 12; (c) pH 13; (d) pH 14

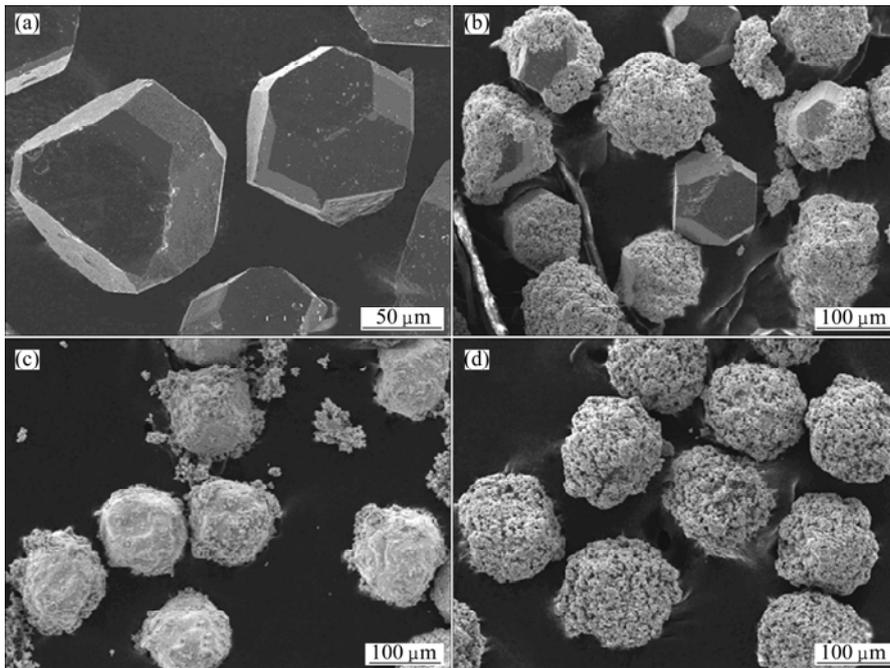


Fig. 6 SEM images showing effect of different temperatures on plating process: (a) 30 °C; (b) 40 °C; (c) 50 °C; (d) 60 °C

3.2.4 Effect of reducing agents

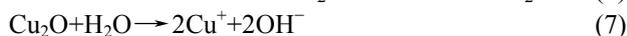
The available reducing agents are formaldehyde (HCHO), Na-hypophosphate, Na-borohydride and hydrazine. Formaldehyde is most widely used because of its low cost, nature friendly, being conveniently available, less amount of by-products and wide range of reaction temperatures. The oxidation of formaldehyde is independent of the complexing agents used, but can be accelerated by increasing pH value of the bath but at the

same time it is a volatile carcinogenic liquid, a serious hazard to health [19]. Na-hypophosphate is also widely used for electroless plating for Ni due to its lower cost and relative safety; however, it is not good for electroless copper plating because Cu is not as good as nickel as a catalyst for the oxidation of hypophosphite [2]. The basic reason is the redox reaction shown in Eq. (2). In addition, there are some of its side-effects.

Cannizzaro reaction:



Reduction of cuprous oxide:



Disproportionate reaction of Cu^{2+} :



When the amount of the HCHO was too high in bath, a large amount of copper was attached out of diamond surface, the bath was decomposed, and the plating rate dropped drastically. A certain fixed amount of HCHO increased the rate of copper plating [20,27,28]. The effect of different amounts of formaldehyde at different pH values on mass gain of copper deposition is shown in Fig. 7.

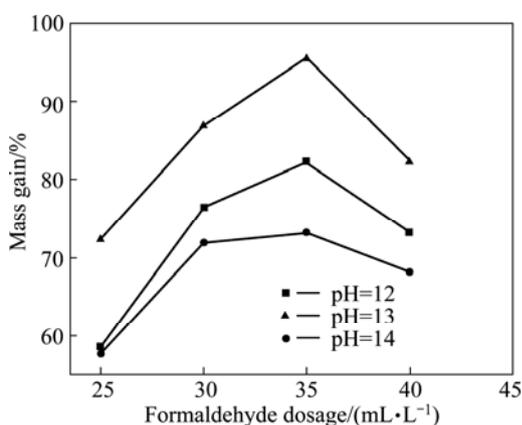


Fig. 7 Effect of formaldehyde concentration on deposition rate

3.2.5 Concentration of Cu^{2+} ions

The effect of Cu^{2+} ions was very significant as shown in Fig. 8. The plating rate increased from low to high when Cu^{2+} ions concentration rose from 0.01 mol/L to 0.06 mol/L in the bath. When the concentration of Cu^{2+} ions continued to rise, the mass gain reduced. At 0.1 mol/L of Cu ion, the deposition rate declined significantly because the stability of solution bath

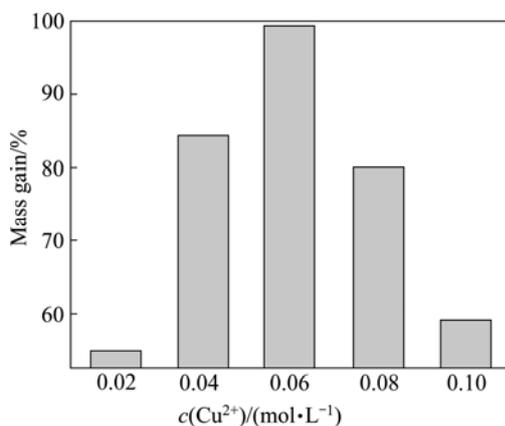


Fig. 8 Effect of copper ion concentrations on mass gain of products

decreased, minimizing the effect of complexing agents, leading to adverse reaction in solution, which resulted in electrolyte decomposition [20]. The best plating results with Cu^{2+} in bath were achieved at 0.06 mol/L.

3.2.6 Effect of mechanical stirring speed

The whole process was carried out under ultrasonication, which enhanced the deposition rate and adhesion, and contributed positive effects to the bath, such as heating and mechanical stirring. This led to an accelerated diffusion of reactants and increasing number of copper crystals nuclei in bath [29]. Experimental results revealed that if the stirring speed of the process was lower than 100 r/min, very small electroless plating occurred; if the speed was very high, i.e. higher than 450 r/min, also very small plating occurred. Hence, the optimized speed for the best coating results obtained was (350 ± 15) r/min. The SEM images of the plated particles at different stirring speeds are shown in Fig. 9.

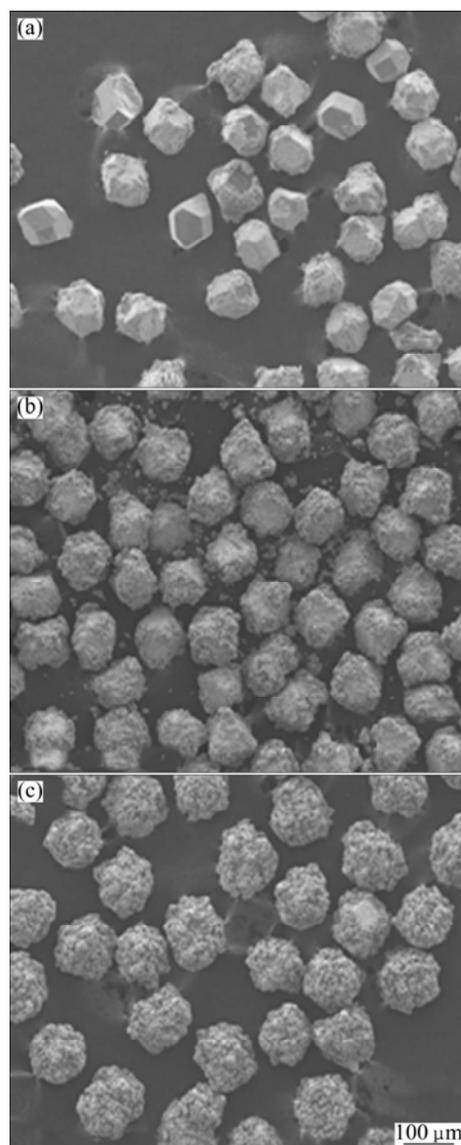


Fig. 9 SEM images showing effect of stirring speeds with ultrasonication: (a) 125 r/min; (b) 250 r/min; (c) 350 r/min

3.2.7 Time effect on copper plating

Plating time also plays an important role. Different mass gain was obtained at different plating time while keeping all other parameters the same. The best time for plating was 30 min. During this time, complete reduction of copper solution took place and color of the whole bath become colorless. The effect of plating time is shown in Fig. 10.

3.3 Optimized parameters and characterization of samples

A series of experiments were conducted to get optimized parameters, which gave the best coating

results for Cr-coated diamond particles are listed in Tables 1 and 2.

There are many other factors that also highly influence the plating phenomena, such as the cleaning of beakers, experimental setup conditions, and proper timing to add reducing agent in copper solution bath as well as maintaining the proper pH value by adding 20% NaOH in solution. Based on the above parameters, the electroless copper coating was carried out and the analyses of the samples were produced. The EDS, XRD, XPS and coating thickness measurements results are discussed as follows.

The EDS results revealed that the coating consisted

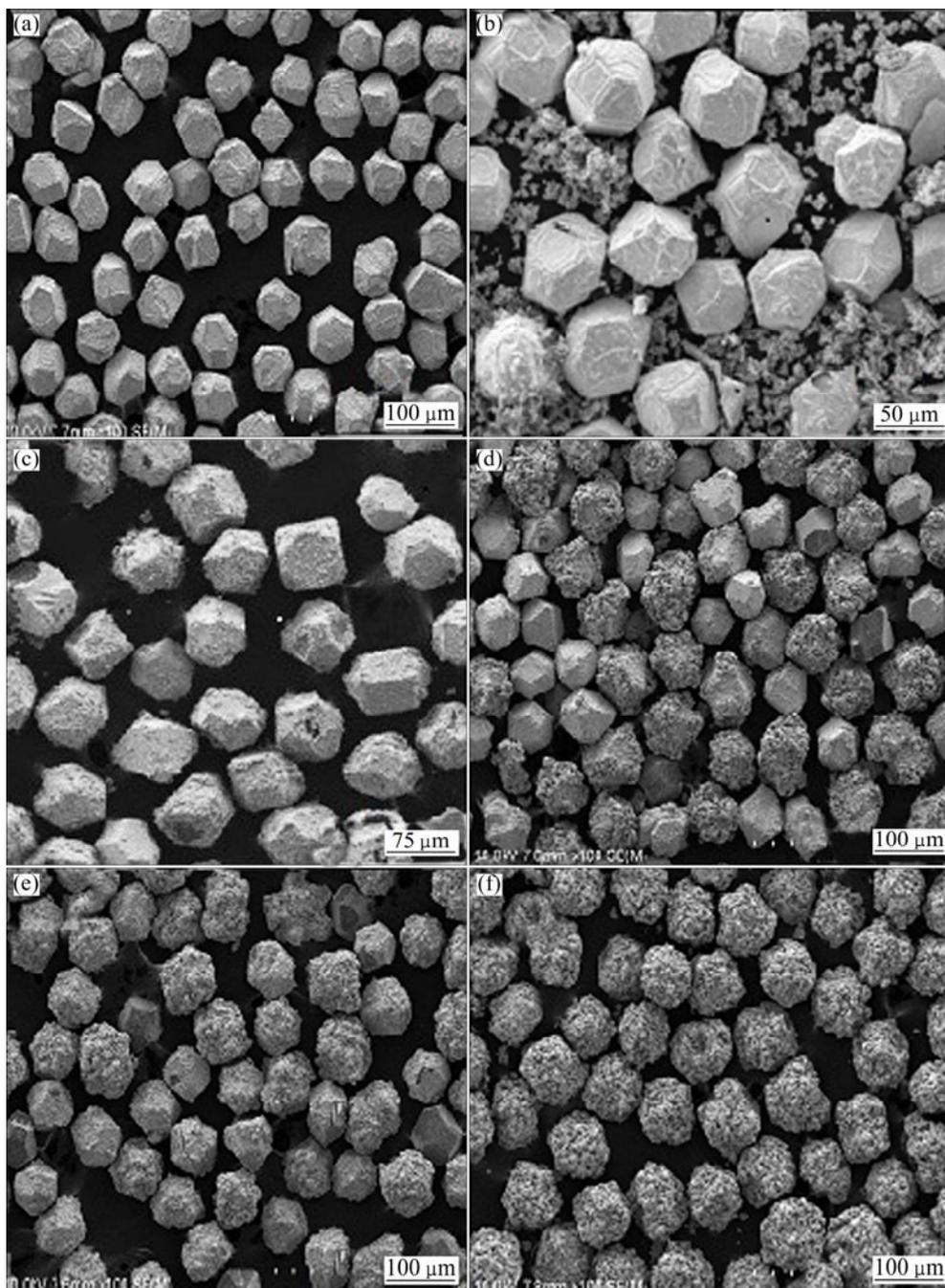


Fig. 10 SEM images showing effect of plating time: (a) 5 min; (b) 10 min; (c) 15 min; (d) 20 min; (e) 25 min; (f) 30 min

Table 1 Optimized parameters for Cr-coated diamond during pretreatment stage

Chemical	Application
NaOH washing	Washing for 30 min with 20% NaOH (5 mol/L)
Sensitization	Immersing in SnCl ₂ (20 g/L)+ HCl (20 mL/L) solution for 5 min
Activation	Immersing in PdCl ₂ (0.25 g/L)+ HCl (10 mL/L) solution for 20 min

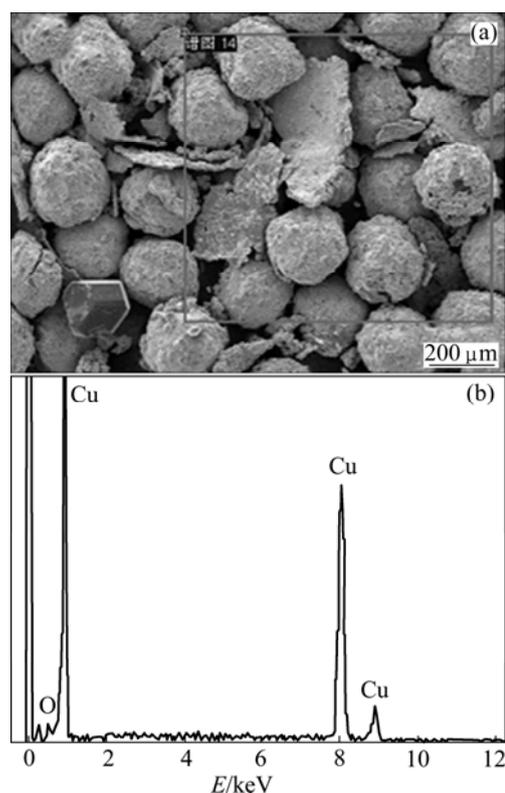
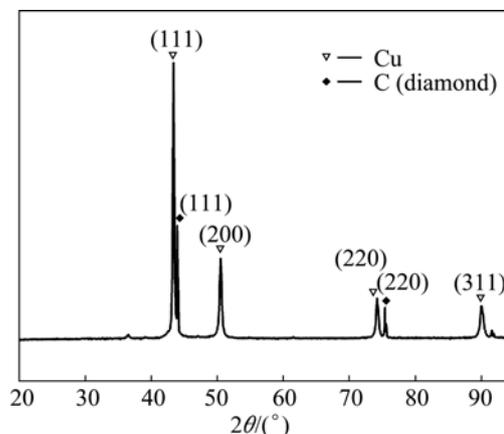
Table 2 Composition of Cu-solution bath and parameters for electroless copper plating

Composition or parameter	Value
CuSO ₄ ·5H ₂ O	16 g/L (0.06 mol/L Cu ²⁺)
NaKC ₄ H ₄ O ₆ ·4H ₂ O	23 g/L
Formaldehyde (HCHO)	35 mL/L
pH	13
Temperature	60 °C
Time	30 min
20% NaOH	According to requirement, pH value should not low below 13

of only Cu crystal layers; however, the presence of carbon peaks indicated that diamond was exposed, where the coating was not so thick and uniform. About 99% copper was electrolessly coated on the surface of diamond particles. Also no or trace amount of oxygen was detected on the deposit. It was confirmed that electroless copper coating consisted of only copper crystal structures and either no or very small amount of CuO present on the deposit as shown in Fig. 11.

XRD pattern of the electrolessly coated diamond particles was obtained by Philips X'Pert (high score) as shown Fig. 12. The sharp diffraction peaks were observed at $2\theta=43.9135^\circ$ and 75.3204° , corresponding to the characteristics peaks of diamond (111) and (220) reflection, respectively, with lattice parameter $a=3.5712 \text{ \AA}$, making a good agreement with the standard diamond peaks (JCPDS Card No.65-0537); while the diffraction peaks obtained at $2\theta=43.3017^\circ$, 50.4549° and 74.11105° , represented crystals planes of cubic metallic copper (111), (200) and (220), respectively, confirming the existence of copper in the form of Cu crystals, indexed to copper metal with lattice parameter, $a=3.6184 \text{ \AA}$, which was also in a very good agreement with the standard of copper peaks (JCPDS Card No.85-1326).

The surface analysis of the electroless plated diamond was carried out by X-ray photoelectron spectroscopy (XPS). The XPS spectrum for copper coating is shown in Fig. 13(a). The peaks for C, O and Cu were detected on the coated diamond particles. The

**Fig. 11** SEM image of copper coating (a) and EDX result of single copper crystals (b) based on optimized parameters**Fig. 12** XRD pattern of diamond particles

presence of C and O peaks was due to the fact that oxygen and carbon dioxide were present in air, which could easily be adsorbed on the coated surface. All the remaining peaks were related to copper. A narrow spectrum of Cu 2p is shown in Fig. 13(b). Two peaks corresponded to pure metallic Cu with $2p_{1/2}$ at 952.30 eV and $2p_{2/3}$ at 932.36 eV, confirming that coating consisted of only copper element.

Copper coating thickness was measured using an optical microscope as shown in Fig. 14. It revealed that the diamond particles got good coating thickness. The average measured thickness for Cr-coated diamond

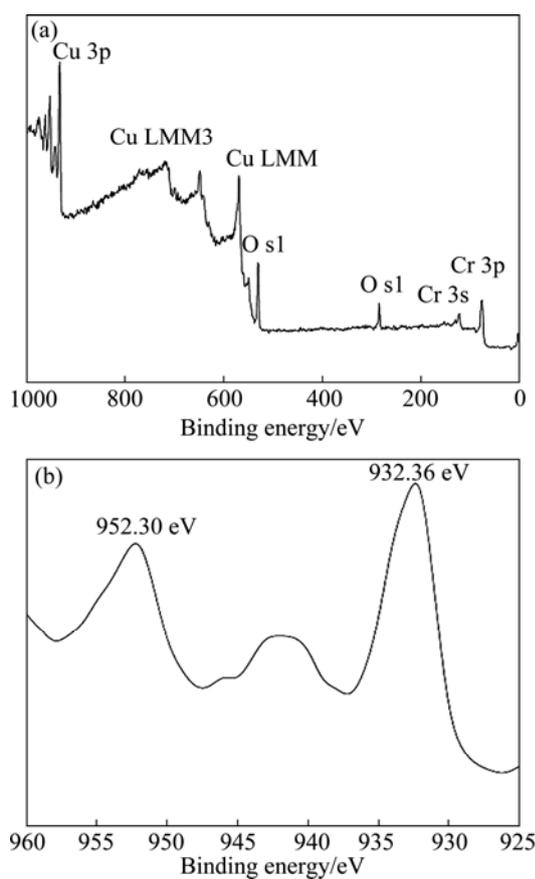


Fig. 13 XPS spectra of copper coating: (a) Wide range spectrum; (b) Spectrum of Cu 2p

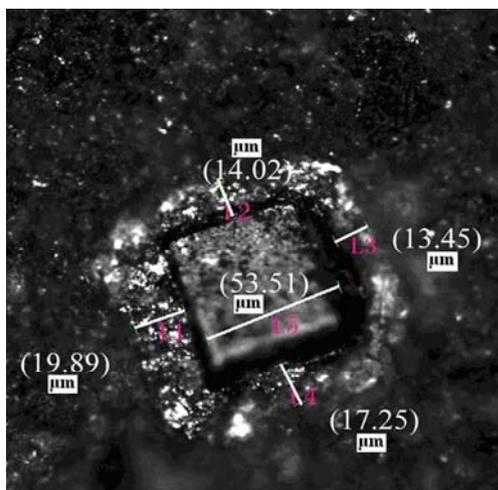


Fig. 14 Optical micrographs of Cr-coated diamond particles

particles was $16.0 \mu\text{m}$. The micrograph showed a core structure of diamond particles covered with a thick coating of copper deposits.

4 Conclusions

The diamond in nature is inert and incompatible to form any type of bonding or interaction to other elements, until it is pretreated. Hence, before electroless copper

plating diamond must be pretreated, so that the surface of diamond particles becomes catalyzed. The best optimized pretreatment conditions are etching with 20% NaOH for 30 min and sensitization and activation with SnCl_2 and PdCl_2 solutions for 5 and 20 min, respectively. The best optimization parameters after pretreatment in copper solution bath for electroless copper coating on diamond particles coated with 1% Cr are, the bath composition of 16 g/L $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, 35 mL/L HCHO and 23 g/L $\text{NaKC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$, at temperature $60 \text{ }^\circ\text{C}$, pH=13 and stirring speed of $(350 \pm 15) \text{ r/min}$ under ultrasonication. Combination of the best optimized pretreatment and electroless copper plating parameters can achieve a uniform and maximum coating thickness at a high deposition rate.

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金刚石表面化学镀铜工艺的优化

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摘要: 研究预镀 1%铬金刚石颗粒表面化学镀铜的预处理工艺、镀液成分和工艺参数对表面形貌、沉积效率、镀层均匀性等的影响, 优化出最佳工艺参数。结果表明, 采用 20% NaOH 溶液处理 30 min 后, 再在 SnCl₂ 溶液中进行 5 min 敏化和在 PbCl₂ 溶液中进行 20 min 活化, 能提高预镀 1%铬金刚石颗粒表面镀铜质量, 并获得较高的铜沉积率。化学镀铜最佳工艺条件为: 16 g/L CuSO₄·5H₂O, 35 mL/L 甲醛, 23 g/L 酒石酸钾钠, 温度 60 °C, pH=13, 辅助超声加(350±15) r/min 的机械搅拌。采用此工艺在预镀 1%铬金刚石颗粒表面获得了厚度均匀的纯铜层。

关键词: 化学镀铜; 预镀铬金刚石; 工艺优化

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