

# High-adhesive electroless copper plating on polyethylene surface modified with primer

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**Abstract** In this work, the region-selective electroless copper plating on the surface of calcium carbonate (CaCO<sub>3</sub>)-filled polyethylene (PE) (CFP) films have been established based on printing primer by screen printing in combination with electroless copper plating. Results showed only the printed areas formed amine groups on the surface of CFP sheets, which served as cores for adsorbing Pd<sup>2+</sup> ions. Thus, the pattern on the screen was transferred to the surface of CFP sheets after electroless plating. When the plated time increased from 5 to 120 min, the thickness of plated coating was enhanced from 0.12 to 10 μm and the electrical resistivity was decreased from 12,544 to 0.4 Ω cm. The blown PE films mixed with foam powders and CaCO<sub>3</sub> powders could form the coarse

surface which can provide a large area for mechanical anchor between the primer and the plated coating, resulting in a better adhesion of the plated coating. In addition, the copper patterning possessed excellent selectivity, compactness, and flexibility. The EMI-SE of plated CFP films is above 30 dB.

**Keywords** Electroless plating · Primer · Copper · Region-selective

**Highlights** • Region-selective electroless copper plating on the calcium carbonate-filled polyethylene films can be achieved when the substrates were printed primer with a pattern.  
• The print areas formed higher density of amine groups.  
• The plated copper patterning possesses high adhesion.

## Introduction

Selective electroless deposition is the preferred method for the region-selective metalization of polymer surface due to its inexpensive and lower operating temperature [1–3]. Pretreatment, activation or seeding, and then plating are the most common process for the electroless plating [4–6]. Pretreatment can enhance the interfacial adhesion between metal nano-particle catalysts and substrate [7–9]. Catalysts (such as Au, Ag, and Pd) serve as seeds to catalyze the electroless plating [10, 11]. Thus, selective pretreatment is key to the subsequent region-selective metalization of polymer surface.

The region-selective modification of the polymer surface was reported to include selective deactivation [12] and selective activation [13–16]. The selective deactivation requires that the substrate was first all-region activation and then region-selective passivation treatment. Cao et al. reported that poly(ethylene terephthalate) (PET) sheets were first treated with ultraviolet light and 3-amino-propyltriethoxysilane (KH550), then modified by vacuum ultraviolet irradiation through a mask [12]. Copper coating plated on the regions covered with mask after electroless plating. The selective activation was reported to include flame etching [13], wet-chemical pretreatment [14], and photo etching [15–17] through a mask, and then formed active groups on the exposed

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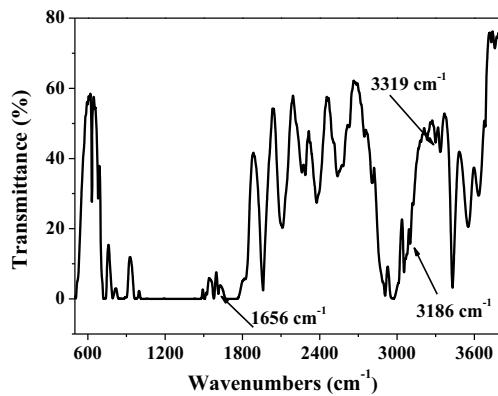
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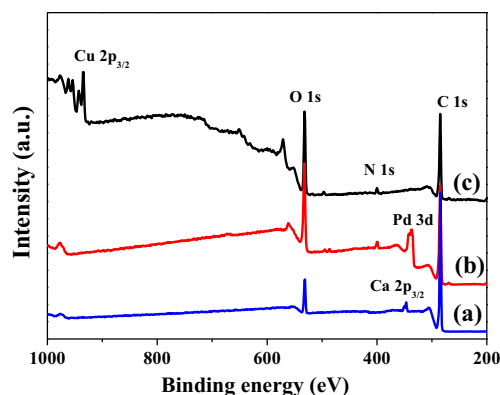
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**Fig. 1** FT-IR spectrum of the primer after drying into membranes

region. Chen et al. reported that selective exposure of polymer to UV light generated active groups on the target area of PET [17], poly-carbonate (PC) [16], and polystyrene (PS) [15] surface. In their work, the region-selective metalization on the pretreated polymer surface was achieved after electroless plating. Chen et al. also reported a method using laser-induced site-selective activation of  $\text{Ag}^+$ -doped polyimide (PI) sheets for selective electroless copper plating [18].

It is well known that selective electroless plating on surface of polymer requires that functional groups were grafted onto the target area. At present, the technologies are tedious which raise the discharging cost [15–18]. To our knowledge, the region-selective electroless copper plating on polymer surface modified with primer by screen printing is still not well studied. This work was focused on the selectively induced active groups by screen-printing primer. The surface of printed areas could chemisorb  $\text{Pd}^{2+}$ , which would contribute to electroless plating. Correspondingly, a relatively simple electroless method for the region-selective metalization on the surface of polymer was developed. In addition, hydrogen is generated in the plated reaction, which will congregate on the surface of plated coating and degrade the adhesion, even causing the drop of the plated coating [19]. However, the coarse surface can anchor the plated coating tightly, resulting in a better adhesion [19]. In this



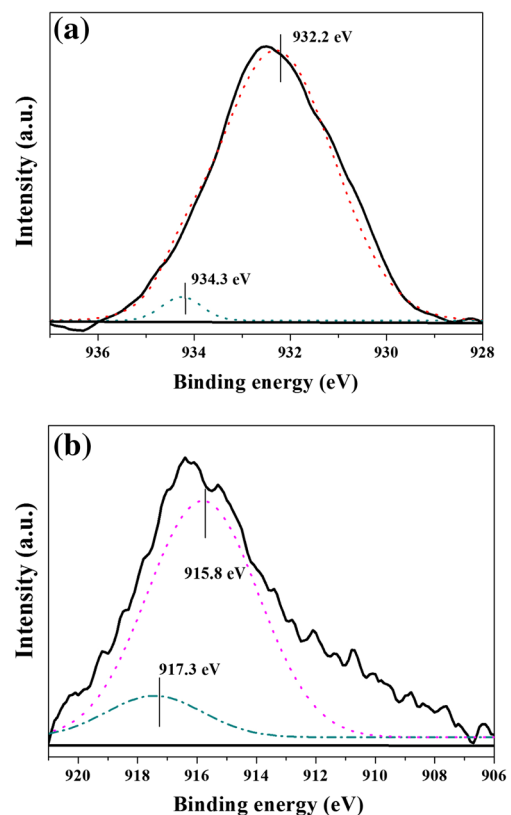
**Fig. 2** XPS spectra of activated pristine CFP films (a), activated primer-printed CFP films (b), and plated CFP films (c)

work, the polyethylene films with higher surface roughness were prepared and the high-adhesive and region-selective electroless copper plating on the surface have been established.

## Experimental

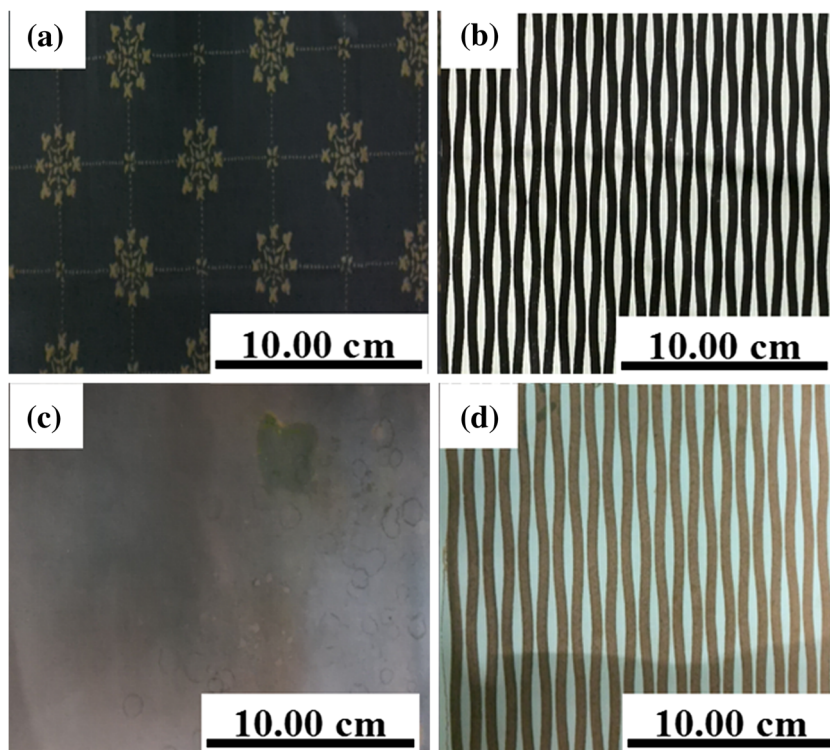
In a four-neck flask equipped with a thermometer, a condenser, a mechanical stirrer, and dropping funnels, an amount of potassium persulfate and water was added. A mixture of monomers consisting of methyl methacrylate (1.8%), butyl acrylate (2%), acrylic acid (8%), diacetone acrylamide (2%), and diacetone acrylamide (2%) was pre-emulsified by ultrasound and then dropped into the system at 350 K over a period of 4 h. The remaining potassium persulfate was then added twice at intervals of 1 h. The mixture was left to react for another 2 h. After reaching the standard of emulsion, the mixture was cooled to 330 K. Hydroxypropyl acrylate (0.5%) was added in the copolymer emulsion and stirred well. The primer was then obtained.

Polyethylene (PE) were used as carrier resin. The PE, foam powders,  $\text{CaCO}_3$  powders and stearic acid mixing is processed in Banbury mixer, manufactured Calcium carbonate ( $\text{CaCO}_3$ ) filled PE (CFP) films. The filling content of CFP films was between 60%. The particles of  $\text{CaCO}_3$  was 15  $\mu\text{m}$ . The CFP films were prepared using plastic blow molding process [20].



**Fig. 3**  $\text{Cu } 2p_{3/2}$  XPS and auger Cu LMM spectrum of plated CFP films (b)

**Fig. 4** Photographic images of primer-printed CFP films (a, b) and plated CFP films (c, d)

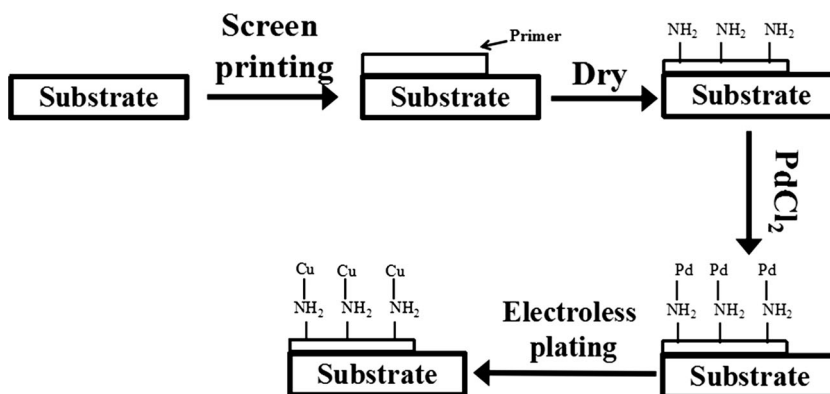


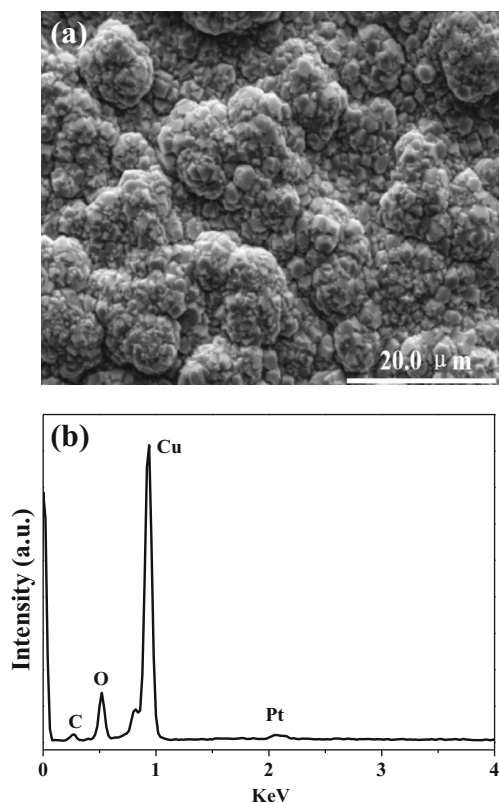
The highly filled and foamed CFP films were used as substrates. The CFP films were ultrasonically cleaned in ethanol, distilled and acetone water for 30 min, respectively, then dried in warm air stream. The CFP films were printed primer with a pattern by screen printing, and then forced dry at 373 K for 20 min.

The CFP films were activated by a mixture consisting of 0.18 g/l PdCl<sub>2</sub> (the pH value was 2) at 323 K for 10 min and rinsed with distilled water, and then the CFP films were added into the 2-g/l NaH<sub>2</sub>PO<sub>2</sub> solution at 313 K for 10 min. After that, the activated substrates were put into the electroless copper plating bath containing 16 g/l CuSO<sub>4</sub>·5H<sub>2</sub>O, 18.5 g/l Na<sub>2</sub>EDTA, 14 g/l NaKC<sub>4</sub>H<sub>4</sub>O<sub>6</sub>·2H<sub>2</sub>O, 14.5 g/l NaOH, and 15 ml/l HCHO [21–23]. The bath temperature was 333 K. The plated time was 5–120 min.

Vector network analyzer (Agilent 8720ET) was used for electromagnetic interference (EMI)—shielding effectiveness (SE) analysis. Surface morphology of the plated CFP sheets was observed by SEM (JEOL, JSM-5600LV). Links Systems energy dispersive spectrometer (EDS) was used for element analysis. Chemical structure of the samples were measured by XRD (Rigaku D/max-2550V), employing the Cu Kα wavelength and X-ray photoelectron spectroscopy (XPS; Shimadzu, AXIS ULTRADLD). X-ray diffraction (XRD) data was obtained in the 20–80° range, with a step interval of 0.03°. Surface functionality information of substrates were characterized by Fourier transform infrared spectroscopy (FT-IR; Thermo, NICOLET 6700). The primer after drying into membranes was used as substrates. 3D stereoscopic microscope

**Fig. 5** Schematic diagram of region-selective electroless copper plating on the surface of CFP films modified with primer



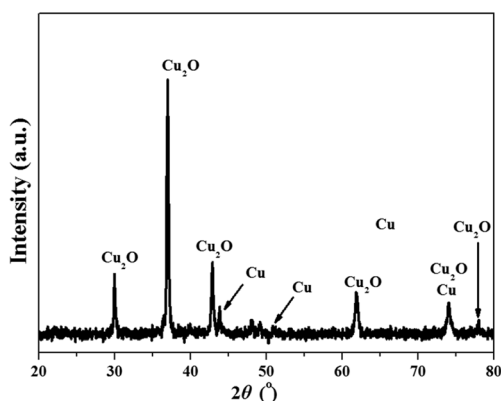


**Fig. 6** SEM images (a) and chemical compositions (b) of the plated CFP films modified with primer

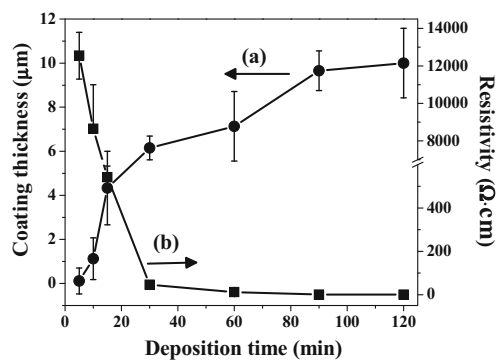
(Hirox, KH-7700, Japan) was used to study the surface topography of the plated coating. The Hall system (Nanometrics, HL5500PC) surface was used for electrical observation.

## Results and discussion

Figure 1 shows FT-IR spectrum of the primer after drying into membrane. It was observed that the peaks of 1656, 3186, and 3319  $\text{cm}^{-1}$  appeared. These peaks are due to the amide stretching vibration ( $-\text{CONH}-$ ),  $-\text{N}-\text{H}$  ( $-\text{NH}-$ ) stretching



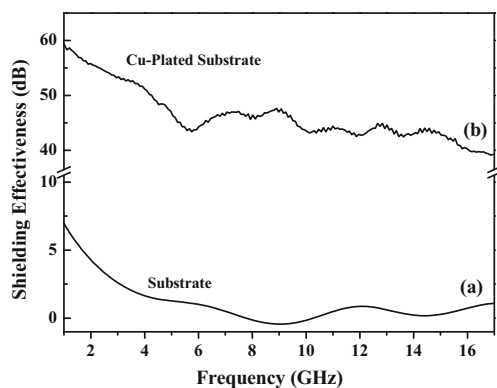
**Fig. 7** XRD pattern of the plated CFP films modified with primer



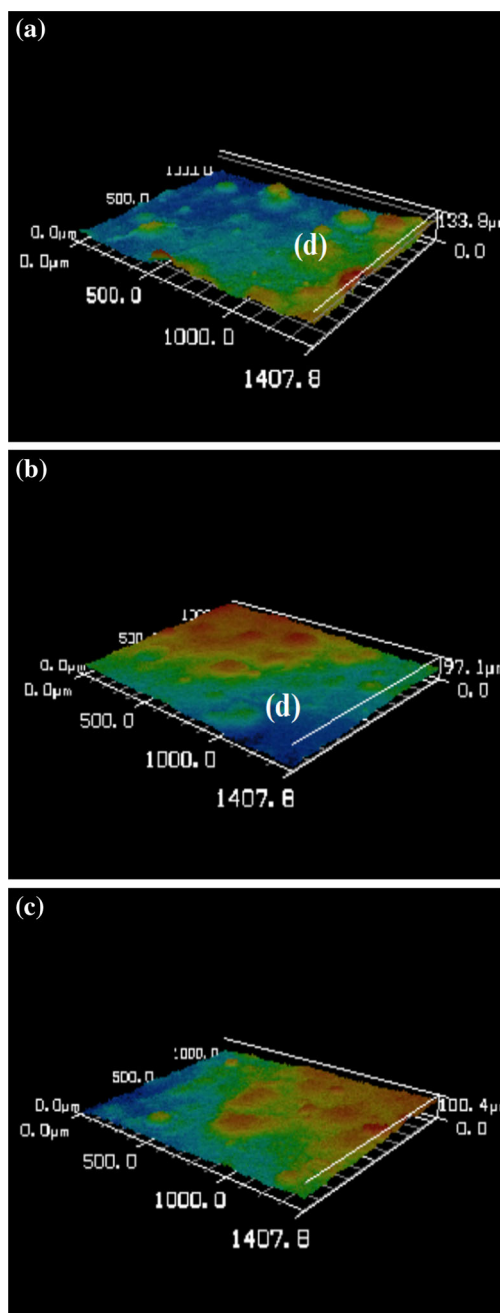
**Fig. 8** Coating thickness (a) and electrical resistivity (b) of copper-plated CFP films when the plated time increased from 5 to 120 min

vibration, and  $-\text{N}-\text{H}$  ( $-\text{NH}_2$ ) stretching vibration [16, 24]. It was indicated that the amine groups were formed on the surface of primer after drying into membrane.

Figure 2 shows XPS spectra of activated pristine CFP films (a), activated primer-printed CFP films (b), and plated CFP films (c). The peaks at about 286 eV, 338.4 eV, 346.6 eV, 398.8 eV, 534 eV, and 932 eV are due to the C 1s, Pd 3d, Ca 2p<sub>3/2</sub>, N 1s, O 1s, and Cu 2p<sub>3/2</sub> [12, 24], respectively. As shown in Fig. 2 (b), the characteristic peak of nitrogen was detected, which indicated that the amine groups were formed on the surface of primer-printed CFP films. Meanwhile, Pd 3d signal is also detected in the spectrum. It was found that the structure of primer-printed CFP films would facilitate the chemisorption of palladium ions. These results indicate that only the printed areas could adsorb the palladium ions on the surface of CFP films. The chemisorbed Pd atoms used as the catalyst for the electroless plating [8, 18, 19], as shown in Fig. 2 (c), Cu signal was detected in the spectrum. Two contributions are discerned at 934.3 and 932.2 eV, as shown in Fig. 3a. The higher peak at 934.3 eV is assigned to  $\text{Cu}^{2+}$  in the plated coating [12, 24, 25]. The lower peak at 932.2 eV suggests the presence of  $\text{Cu}^+$  or  $\text{Cu}^0$  [12, 24, 25]. Auger Cu LMM spectrum was used to confirm the presence of  $\text{Cu}^+$  at 915.8 eV and  $\text{Cu}^0$  at 917.3 eV, as shown in Fig. 3b [12, 24, 25]. It was



**Fig. 9** EMI-SE of primer-printed CFP films (a) and plated CFP films (b) in a frequency range of 1–17 GHz



**Fig. 10** Surface topography of pristine CFP films (a), primer-printed CFP films (b), and copper-coating plated CFP films

indicated that the Cu and Cu<sub>2</sub>O deposited on the surface of pretreated CFP films after electroless plating.

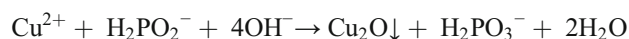
Figure 4 shows photographic images of primer-printed CFP films (Fig. 4a, b) and plated CFP films (Fig. 4c, d). As shown in Fig. 4a, b, the CFP films were printed primer with a pattern by screen printing. Black pigment and tints were added to achieve the desired visual effect. As shown in Fig. 4c, the region-wide copper films plated on the CFP films after electroless plating when the substrate all-region modified with primer. The results indicated that the copper coating could

grow on the surface of CFP films modified with primer. It was also found that the copper coating only deposited on the primer-printed region and nowhere else, resulting in copper patterning being formed on the surface of the CFP films, as shown in Fig. 4d. Thus, the pattern on the screen was transferred to the surface of the CFP films after electroless plating. It was indicated that selectivity in copper patterning was obtained successfully by electroless plating on the surface of the CFP films modified with primer.

Figure 5 shows a schematic diagram of region-selective electroless copper plating on the surface of the CFP films modified with primer. As shown in Fig. 5, the CFP films were printed primer with a pattern by screen printing, and then forced dry. During drying, the active groups were linked through dehydration condensation between the primer and substrate, thus the region-selective primer was formed. In addition, the hydroxypropyl acrylate would also facilitate the dehydration condensation between the primer and substrate, leading to high adhesion. Thus, only the printed areas formed higher density of amine groups on the surface of the CFP films. The amine groups serve as cores for chemisorption of Pd<sup>2+</sup> ions via complexation [15–17, 26]. The Pd<sup>2+</sup> ions were used as the catalyst for the electroless plating on the printed regions [8, 9]. The pattern on the screen was transferred to the surface of the CFP films after electroless plating.

Figure 6 shows SEM image (a) and chemical compositions (b) of the plated CFP films modified with primer. It was revealed that the uniform copper crystals were formed in the plated coating, as shown in Fig. 6a. Figure 6b shows the chemical compositions of copper-plated CFP films measured using EDS. The signals of chlorine, oxide, and copper were found. The samples contain oxygen element because the copper coating were oxidized. It was suggested that the copper and copper oxide was deposited on the surface of CFP films modified with primer after electroless plating.

Figure 7 shows XRD pattern of the plated CFP films modified with primer. The diffraction peaks centered around 43.3°, 50.4°, and 74.3° are very close to the expected Bragg peaks of the face-centered Cu (111), (200), and (220) [23, 27]. In addition, the remaining peaks centered around 29.7°, 36.5°, 42.4°, 61.54°, 73.7°, and 77.6° are very close to the expected Bragg peaks of Cu<sub>2</sub>O (110), (111), (200), (220), (311), and (222) [23, 27]. The Cu<sub>2</sub>O has good ability of shielding effect [1, 10]. It was suggested that the Cu and Cu<sub>2</sub>O was plated on the surface of CFP films. The chemical reaction process can be expressed as the following reaction [1, 10]:



The results indicated that the Cu and Cu<sub>2</sub>O deposited on the surface of pretreated CFP films after electroless plating.

**Fig. 11** The photographic images (a–c) and cross-section microscope image (d) of plated CFP films

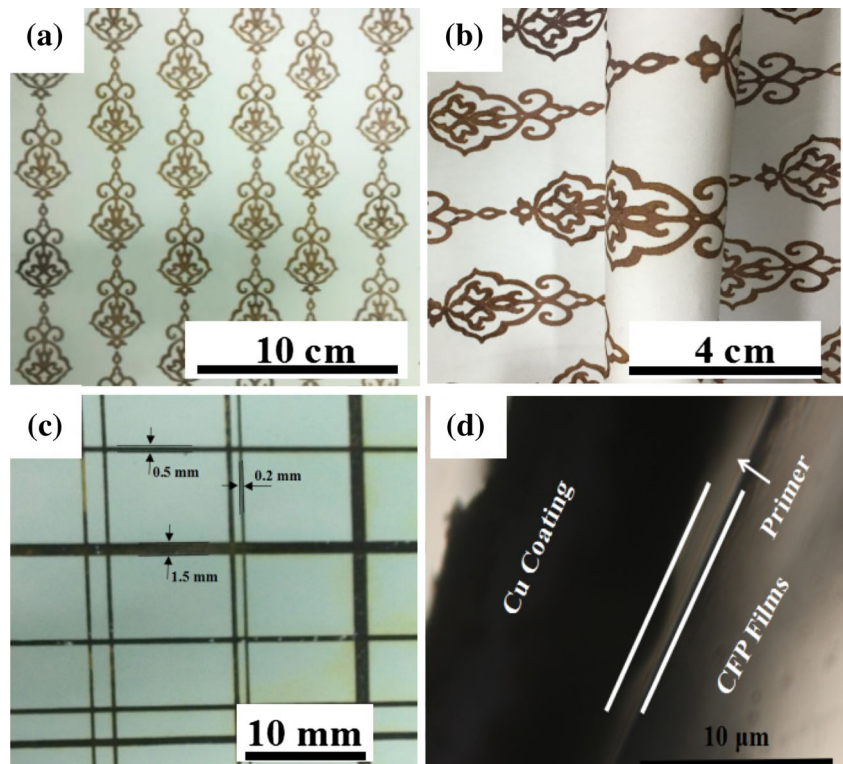


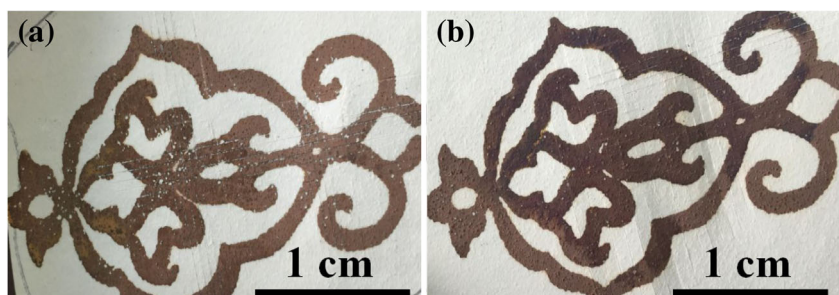
Figure 8 shows the coating thickness (a) and electrical resistivity (b) of plated CFP films when the plated time increased from 5 to 120 min. When the plated time increased from 5 to 120 min, the coating thickness was enhanced from 0.12 to 10  $\mu\text{m}$ , and the electrical resistivity was decreased from 12,544 to 0.4  $\Omega\text{ cm}$ . It probably resulted from the higher plated time which would facilitate electroless copper plating on the surface of CFP films. The electrical resistivity reduced with the increase of the thickness of copper layers.

Figure 9 shows EMI-SE of primer-printed CFP films (a) and plated CFP films (b) in a frequency range of 1–17 GHz. The EMI-SE of copper-plated CFP films is closely related to its all-region conductivity [26]. Thus, the all-region metalization of the surface of CFP films can be estimated by investigating the EMI-SE [26]. It was found that the EMI-SE of plated CFP films modified with primer above 30 dB, and

the EMI-SE of plated CFP films higher than the one of primer-printed CFP films in the frequency range of 1–17 GHz. It was concluded that the tightly packed copper layers deposited on the surface of CFP films modified with primer after electroless plating.

Figure 10 shows surface topography of pristine CFP films (a), primer-printed CFP films (b), and copper coating plated CFP films, respectively. The average roughness value ( $R_a$ ) were measured to be 84  $\mu\text{m}$  for pristine CFP films, 49  $\mu\text{m}$  for primer-printed CFP films, and 53  $\mu\text{m}$  for plated CFP films, respectively. The blown PE films mixed with foam powders and  $\text{CaCO}_3$  powders could form the coarse surface, which could also enhance  $R_a$  of the surface of primer-printed CFP films. The rough surface of the substrates could provide a large area for chemical bond between primer and substrate and mechanical anchor between primer and plated copper.

**Fig. 12** Plated coating remained on plated pure PE films (a) and CFP films (b) after adhesion assessment



The increase in the  $R_a$  for the copper coating plated on the CFP films was obviously due to large copper particles deposited on the primer surface.

Figure 11 shows photographic images (a–c) and cross-section microscope image (d) of plated CFP films. As shown in Fig. 11a, b, a selective copper patterning was obtained successfully by electroless copper plating on the surface of the CFP films modified with primer, which had many advantages, such as high precision, no cracking, and good toughness. Figure 11c shows the copper lines of different widths on the surface of the CFP films. The minimum width of copper lines and spacing between copper lines are 0.2 and 0.6 mm, respectively. It is reasonable to expect that the feature size of plated patterning become more accurate with the increase of screen mesh. Referring to the cross-section analysis, the copper coating is deposited on the surface of the primer, as shown in Fig. 11d. The results indicated that the flexible and high-precision copper patterning on the surface of CFP films could be achieved based on printing primer with a pattern by screen printing in combination with electroless copper plating.

Figure 12 shows plated coating remained on the plated substrate after adhesion assessment. Selective electroless copper plating on pure PE films (a) and CFP films (b) were subjected to the Scotch tape test. The surface of plated coatings was carved with similar grids and was then analyzed with Scotch®-tape tests. The bubbling, corrugation, and abscission were found through tape test on plated pure PE films. The plated coatings on CFP films never failed, and 95% of dots passed the Scotch®-tape test ( $n = 3$  for all the tested levels). The results indicated that mechanical anchor between deposited copper and coarse primer was strong, leading to the good adhesion.

## Conclusions

In this work, the region-selective electroless copper plating on the surface of polyethylene films was developed. The main steps included blown highly filled and foamed CFP films, printed primer with a pattern by screen printing, ion-adsorption of  $\text{Pd}^{2+}$ , and electroless plating. It was found that the blown PE films mixed with foam powders and  $\text{CaCO}_3$  powders could form the coarse surface. The  $R_a$  of pristine CFP films and primer-printed CFP films were 84 and 49  $\mu\text{m}$ , respectively, resulting in a better adhesion of the primer and plated coating. The copper coating plated on the surface of primer and nowhere else after electroless plating. Thus, the pattern on the screen was transferred to the surface of the CFP films. When the plated time increased from 5 to 120 min, the coating thickness was enhanced from 0.12 to 10  $\mu\text{m}$  and the electrical resistivity was decreased from 12,544 to 0.4  $\Omega \text{ cm}$ . The EMI-SE of plated CFP films is above 30 dB. In addition, the copper patterning possessed excellent selectivity, adhesion, and flexibility.

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