



# Silver nanoparticle catalyst for electroless Ni deposition and the promotion of its adsorption onto PET substrate

Yinxiang Lu <sup>\*</sup>, Longlong Xue, Feng Li

Department of Materials Science, Fudan University, Shanghai 200433, China

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## ABSTRACT

Silver nanoparticle catalyst was prepared to replace Pd catalyst for electroless Ni plating. The adsorption of Ag nanoparticle onto the poly(ethylene terephthalate) (PET) was promoted by conditioning the substrate with 3-aminopropyltrimethoxysilane and 3-mercaptopropyl-triethoxysilane. Electroless Ni deposition was started at the PET substrates catalyzed with Ag nanoparticle linking to the substrate via Ag–S bond. The composition and topography of nickel plating PET films were characterized by scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD) and energy dispersive X-ray (EDX) analysis, respectively. Peel adhesion strength, more than 50 N/cm, was achieved for planting nickel layer to the Ag activated PET substrate; however, a relative low value as 10.2 N/cm was obtained for the sample with Pd catalyst by the same measurement. Silver catalyst together with silane modification has the significant merits for electroless deposition of Ni on PET film.

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## 1. Introduction

Poly(ethylene terephthalate) (PET) plastic is an important engineering material due to its high thermal stability, excellent mechanical strength, and high resistance to chemical reagents. However, its application is limited because it is non-conducting and easily fretted. Metalized PET can be widely used in many fields due to its outstanding properties of engineering plastic and metal. Nickel metallization of PET is of major importance in many fields of industry including automotive, aerospace, microelectronics, decoration and luxury packaging industries [1–3].

Electroless deposition of Ni has the significant merit of lower cost, fast deposition, and formation of good membranes at low temperature. Electroless nickel plating is a heterogeneous catalytic electron transfer reaction, in which electrons are transferred from a reducing agent to nickel ions at an interface. Therefore, a noncatalytic substrate, such as PET plastic, has to be catalyzed to produce catalytic nuclei before electroless deposition is performed. Good catalysis is the key to the electroless plating process. Pd colloid is commercially used as a catalyst in electroless plating due to its high activity [4–6]. Because there are no chemical bonding forces between the colloid particles and the substrate, a very small amount of Pd colloid particles drops off from the substrate and solves into a plating bath, which will result in the self decomposition of the plating solution. Although ways of keeping Pd catalyst stable and available for use for a long period have come into reality, Pd compound

is so expensive that the cost of the plating is high. Performing the electroless nickel of the PET substrate without seeding their surface with Pd catalytic clusters is an interesting challenge which aims at reducing the cost of the metallization process [7–9].

Another key to the performance and reliability of the metal/plastic system is the adhesion between the deposited Ni and PET substrate. Achieving a smooth metal–dielectric interface is especially challenging because the metal-to-dielectric adhesion is achieved through a combination of chemical bonding and mechanical anchoring. Enhancing adhesion while minimizing roughness requires advances in chemical bonding and reduced dependence on mechanical anchoring. In the case of traditional polymer materials and electroless nickel metallization, it has been shown that physical anchoring accounts for the majority of adhesion. Improving adhesion with these materials is typically achieved through modification of the dielectric surface. This modification occurs through a multistep swell and etch process that generates root mean squared roughness up to 500 nm. One method for reducing the need for large pores and the mechanical anchoring they provide is to increase chemical bonding, which has the ability to enhance adhesion without creating roughness on the surface [10,11].

The objective of the present study was to develop a nickel coated PET by using a silver catalyst electroless plating process without activation by Pd colloid. The principle of this work is to utilize chemical bonding to enhance adhesion while minimizing the necessary surface roughness of the substrate [12]. The following are adopted to investigate the structures and morphologies: Raman spectroscopy, X-ray diffraction spectroscopy (XRD), X-ray photoelectron spectroscopy (XPS), four-point probe conductivity measurements, scanning electron microscopy (SEM), energy dispersive X-ray

<sup>\*</sup> Corresponding author. Tel./fax: +86 21 65642871.

E-mail address: [yxlu@fudan.edu.cn](mailto:yxlu@fudan.edu.cn) (Y. Lu).

spectroscopy (EDX) and a 180° peel test to determine adhesion between nickel and PET films.

## 2. Experimental

### 2.1. Materials

The PET film used in this study was purchased from Shanghai SPC Chemical Co. in rolls. 3-Aminopropyltrimethoxysilane, 3-mercaptopropyltriethoxysilane and dimethylammoniumborane (DMAB) were purchased from Sinopharm Chemical Reagent Co. Other reagents were of analytical grade and were used without further purification unless otherwise mentioned.

### 2.2. Pretreatment of PET films

PET films of 75 μm thickness were cut into 4 cm × 1.5 cm sections for treatment. Firstly, the films were washed ultrasonically in acetone and distilled water for 30 min, dried at 60 °C for an arbitrary length of time, and weighed. Secondly, the cleaned films were dip-coated with the acetone solution of 0.1 vol.% 3-aminopropyltrimethoxysilane and heated at 125 °C for 2 h to form a silica-like layer. Then the films were irradiated in air by UV-light for 1 h. The UV-light used in this work emits primarily at 185 nm. Finally, the films with the silica-like surface layer were immersed in an anhydrous xylene solution containing 0.1 vol.% 3-mercaptopropyltriethoxysilane for 2 h, then rinsed with xylene, ethanol and water, respectively. The resulting films were heated at 125 °C for 1 h in air to remove residual solvent, and weighed.

### 2.3. Silver activation

Pretreated PET films were immersed in an aqueous solution (50 mL) containing 3.5 g/L AgNO<sub>3</sub>, 3.5 mL/L NH<sub>3</sub>·H<sub>2</sub>O (28% by weight) and 8 g/L glucose at rt for 5 s. After activation, the samples were carefully rinsed with deionized water, ethanol and dried in an oven for 10 min. at 50 °C and weighed.

### 2.4. Electroless nickel plating

The silver activated PET films were immersed in the electroless nickel bath (100 mL) to form thin Ni layers for 40 min at rt. The plating bath was composed of 40 g/L nickel sulfate, 20 g/L tetrasodiumpyrophosphate and 2 g/L dimethylamineborane (DMAB) in deionized water. In alkaline and neutral solutions, the preceding chemical reaction of DMAB with OH<sup>-</sup> ions could represent as [13,14]: (CH<sub>3</sub>)<sub>2</sub>NH<sub>2</sub>BH<sub>3</sub> + OH<sup>-</sup> = (CH<sub>3</sub>)<sub>2</sub>NH + BH<sub>3</sub>OH<sup>-</sup>. DMAB has three active hydrogen atoms bonded to the boron, and theoretically can reduce three Ni<sup>2+</sup> metal ions for each BH<sub>3</sub>OH<sup>-</sup> ion: BH<sub>3</sub>OH<sup>-</sup> + 3Ni<sup>2+</sup> + 2H<sub>2</sub>O = 3Ni + B(OH)<sub>3</sub> + 5H<sup>+</sup>. After plating, the samples were carefully rinsed with deionized water, ethanol and dried in an oven for 1 h at 50 °C and weighed.

### 2.5. Preparation of the surface-nickelized PET films with Pd catalyst

Electroless plating was carried out by a multistep process, which included: scouring, rinsing, etching, rinsing, sensitization, rinsing, activation, electroless plating, rinsing and drying [15]. The specimens were scoured in 10 g/L NaOH solution (100 mL) at 70 °C for 10 min prior to use. The samples were then rinsed in deionized water and etched in a mixture of 15 g/L KMnO<sub>4</sub> and 40 mL/L H<sub>2</sub>SO<sub>4</sub> solution (100 mL) for 10 min. Surface sensitization was conducted by immersion of the samples into an aqueous solution (100 mL) containing 10 g/L SnCl<sub>2</sub> and 40 mL/L 38% HCl at 50 °C for 10 min. The specimens were then rinsed in deionized water and immersed in an activator solution (100 mL) containing 0.5 g/L PdCl<sub>2</sub> and 20 mL/L

38% HCl at 50 °C for 10 min. The specimens were rinsed in a large volume of deionized water to prevent contamination of the plating bath, and then rinsed with ethanol, dried and weighed. The dried specimens were immersed in the electroless plating bath for 40 min. The composition of electroless bath and the operating conditions were the same as above. After plating, the samples were carefully rinsed with deionized water, ethanol and dried in an oven for 1 h at 50 °C and weighed again.

### 2.6. Characterization

Scanning electron micrographs (SEM) were obtained using Philips XL 30 electron microscope. The samples were deposited on a sample holder with a piece of adhesive carbon tape. The chemical composition of the film was determined using energy dispersive X-ray (EDX) analysis attached to the SEM. The topography of the catalyst layer was characterized by a Nanoscope AFM from Veeco Ins. An arithmetic mean of the surface roughness ( $R_{RMS}$ ) (area: 3 μm × 3 μm) was calculated from the roughness profile determined by AFM. X-ray diffraction (XRD) pattern (2θ ranges from 35 to 80°) was recorded at room temperature with a scanning speed of 0.15°/min using Cu Kα radiation (λ = 0.154 nm) from a 40 kV X-ray source (Rigaku D/max-γB) and diffracted beam monochromator, operated at 100 mA. Raman spectrums were characterized by Raman spectrometer (LabRam-1B, France, JY Co. Ltd.). X-ray photoelectron spectroscopy (XPS) measurements were performed on a PHI 5000C ESCA system with Mg Kα source at 14.0 KV and 25 mA. All the binding energies were referenced to the C 1s peak at 284.6 eV of the surface adventitious carbon. The surface resistance was measured by a four-point probe (BD-90), the four-point probe device was specified to have square fixed four probes made of solid tungsten carbide needles (diameter of 0.4 mm) with conical tips angled at 45°. The nominal radius of a probe tip was specified to be 100 μm. The distance of probe spacing was 1 mm and downward force was approximately of 0.2 kg per probe. To evaluate the adhesion of the nickel plating, a standard peel test (ASTM D 3330) was carried out at a rate of 12 cm/min at room temperature in air. For the above study, scotch tape with a width of 1.5 cm was stuck over a length of 4.0 cm on the nickel film. Care was taken to see that there were no air gaps or wrinkles and the film was kept under a pressure of 10 N for 10 min. A 180° peel test was carried out after fixing one end of the sample in one jaw and the scotch tape end with the piece of paper adhered to it in another jaw. For adhesion strength reported here, at least twenty sample measurements were performed.

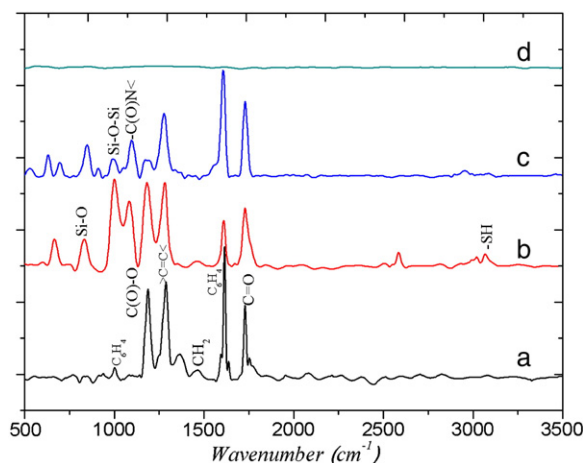


Fig. 1. Raman spectra of pristine (a), silane modified (b) followed by silver activated (c), and nickel coated (d) PET films.

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