Electroless Plating of Moisture-Curable Polyurethane Undercoating Films

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ABSTRACT: The compatibility of using moisture-curable polyurethane (MCPU) system as a thin undercoating layer with electroless plating process was evaluated. The characteristics of the MCPU before and after chemical etching treatment were analyzed using atomic force microscope (AFM), scanning electrode microscope (SEM), contact-angle measurements, and (Fourier-Transform Infrared) FTIR spectroscopy. We found that surface morphology and roughness of the MCPU is affected by curing period and etching times. A proper combination of curing period and etching times are critical for obtaining a fully metallized surface. All MCPU samples that were etched for 15 min show poor plating performance due to surface damage caused by mild etching treatment. A standard pull-off testing method (ASTM 4541) was used to evaluate the adhesion strength of nickel–MCPU. Only samples that were postcured for 4 days show influence of surface roughness on adhesion strength. On average, samples that were postcured for 7 days before electroless plating showed better adhesion of nickel–MCPU compared with samples that were postcured for 2 or 4 days. The results show that MCPU system can be used as a thin undercoating layer for electroless plating. It also offers smooth metal–polymer interface and therefore has the potential to be exploited for use in many electroless plating applications including in the decorative such as ornaments and display items and also in electronic industries. J Appl Polym Sci 103: 1554–1565, 2007

Key words: moisture-curable polyurethane; electroless plating; surface roughness; interface

INTRODUCTION

The morphology of metal–polymer interface is critical, as it can not only provide information concerning the binding behavior of metal plating with polymer surface through a lock and key mechanism, but can also helps determining for which applications this interface is suitable.[1,2] In the chip manufacturing processes, thin film techniques are dominant for a variety of technical reasons, as these processes generally produce smooth interfaces between metal and dielectric substrates. In contrast, in the printed circuit board and chip packaging fields, the adhesion is based on anchoring the metal into cavities on a roughened polymer surface. This adhesion mechanism is not only prominent in wet chemical processes, but also in laminated copper foils that show rough interfaces.[2] In the case of electroless plating, adhesion depends on the surface morphology of polymeric substrates. The deposited metal layer becomes anchored due to the surface roughness and, therefore, roughening the surface will typically improve adhesion.[3]

Polyurethane (PU) has becoming increasingly important in electroless plating, especially those related to biomaterial devices, due to their stability, excellent mechanical properties, and biocompatibility.[4] Maeda et al.'s report (1995) on the fabrication of the microsensor with a successful metallization of metal line on a PU catheter[5] has drawn much attention to this process. This line is made for supplying electricity to the microsensor at the end of catheter tube. PU thin film possesses several outstanding characteristics with regard to electromechanical and dielectric properties, and thus is suitable for use in electromechanical applications such as sensors, actuators, and transducers.[6] In general, a rough metal–polymer interface is more conducive for wear resistance applications, while smoother interface is needed in electronic applications such as high-frequency broadband wireless communication.[2,7]

In the present work, PU layer was formed from a mixture of isocyanate and polyol that consists of divinyl ether functional groups and was used to adhere nickel coating on a rigid substrate through electroless plating procedures. The effect of curing period and etching times on the morphology of PU undercoating layer were investigated. Subsequently, the performances of the nickel coating and its behavior or property with the PU layer were determined.

EXPERIMENTAL

Preparation of prepolymer

Poly(ethylene) glycol divinyl ether 200 (polyol) and diphenylmethane-4,4′-diisocyanate (MDI) were obtained from Merck-Schuchardt, Germany, and used without further purification. The prepolymer solution was pre-

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pared by mixing the polyol and MDI at a 1 : 1 ratio in an amber bottle at room temperature. MDI was added to the polyol and gently stirred with a clean glass rod until the mixture was homogeneous. The final product was a clear yellowish liquid. The bottle was then sealed to prevent contact with moisture in the air and was allowed to stabilize at room temperature for at least 3 h before use.

Sample preparation, mild etching treatment, and characterization

Two sets of moisture-curable polyurethane (MCPU) were prepared: (i) MCPU coated on acrylonitrile-butadiene-styrene (ABS) substrates and (ii) MCPU film. A thin MCPU coating was obtained by dipping an ABS plate (25 mm $\times$55 mm $\times$ 2 mm) into the prepolymer solution for $\sim$30 s and gently resuming it. The excess polymer, which flowed to the bottom tip of the slide due to gravity, was removed with a soft brush. The wet polymer coating was allowed to cure at room temperature for 24 h. The MCPU polymer coating obtained was uniform in appearance and did not peel off compared with other types of polymer coatings use for example polyvinyl chloride and acrylate nitrile that show flaking and chipping property without subjecting to any physical test. Samples were then aged (postcure) in an oven at 70°C for 2, 4, or 7 days. The samples were then etched at 60–63°C in an acidic permanganate solution (mild etchant) containing 0.17 mol/L of $KMnO_4$, 1.24 mol/L of HNO_3 that has been mixed with 1.5 mL Enplate Wetter 62 solution (a commercial wetting agent supplied by Enthone (M) Sdn. Bhd.). Samples were treated at varying etching times of between 1 and 15 min. After etching, the samples were neutralized in a neutralizing solution containing 70 g/L sodium tartrate dihydrate, 25 g/L NaOH, and 25 mL/L hydrazine hydrate at room temperature for 45 s. Finally, the samples were rinsed with distilled water.

The effect of mild etching treatment was analyzed with three techniques. Changes in the morphology were examined with the atomic force microscopy (AFM) and scanning electron microscopy (SEM) methods. A Digital Instrument SIS Scancontrol equipped with Zeiss optical microscope was used to obtain the AFM images from the samples. Roughness was measured on three different locations of each sample and was presented as average. For the SEM images, a Leica Cambridge S360 scanning electron microscopy was used. The contact angle measurements using H_2O as a probe liquid have been used as indication of the degree of polarity of the surface. Therefore, contact angle measurement was also used to characterize the effect of curing period and etching time on treated MCPU samples. Measurement of contact angle was performed using Kyowa Kaimenkagaku Face Contact Angle Meter Type CA-P.

Finally, FTIR spectroscopy was used to examine the chemical changes within the treated MCPU. This was conducted by observing the disappearance and appearance or changes in the intensity of any peaks attributed to changes in the functional group. MCPU thin film (4–5 μm) was prepared by dipping a clean microscope glass slide (Sail Brand, CN: 7101, China) into the prepolymer solution with the same process applied for ABS plates. This was followed by similar curing steps. The polymer was then detached from the microscope slide by slashing its edge and using an alcohol-cleaned razor blade to remove it. The untreated MCPU film was placed on a PVC cardboard window and then mounted on a cell holder in the FTIR chamber of a PerkinElmer Spectrometer System 2000. This enabled the FTIR spectrometer to measure changes that had occurred on the same location of the film. Initially, untreated MCPU samples were scanned through the FTIR and the spectra refer to as a pure or virgin MCPU spectrum. Samples were etched using the procedure described earlier. Samples were dried in the air for 10 min and then rescanned as an etched-MCPU spectrum.

Electroless plating

Electroless plating was performed only on ABS plates (control) and ABS coated with MCPU samples. A conventional electroless plating process was applied on control specimens, in which chromosulphuric acid was used as etchant as described previously.[8] Two processes were used on ABS coated specimens; Process 1 involved without etching, followed by a prolong activation step in activator solution (1–3 h),[9] acceleration (2 min), and finally electroless plating (20–40 s). However, in process 2, it involved mild etching at various etching time then neutralized at 45 s, followed by 3 min activation and 2 min acceleration, and finally electroless plating process for 10 min.

A typical activator solution was used for both processes. It consisted of 1.8 g/L $PdCl_2$, 106 g/L $SnCl_2 \times 2H_2O$, 12.4 g/L $Na_2SnO_3 \times 2H_2O$, and 530 mL/L HCl (32%). The subsequent step (acceleration) that was applied for both processes comprised only the use of diluted HCl (pH $<$ 1) solution. Nickel was deposited on the samples by immersing the samples in Enplate® Ni-881 (a commercial electroless nickel bath) supplied by Enthone (Malaysia). The bath is of low phosphorus type and applicable for plating on plastics. The pH of the nickel bath was controlled at 6.5–7.5 and temperature at 60°C. After the electroless plating cycle was completed, samples were rinsed with distilled water and heat-treated in an oven at 70°C for 1 week.

Adhesion test

The pull-off test was performed as per ASTM D4541[10] using PosiTest AT-P adhesion tester. A schematic dia-

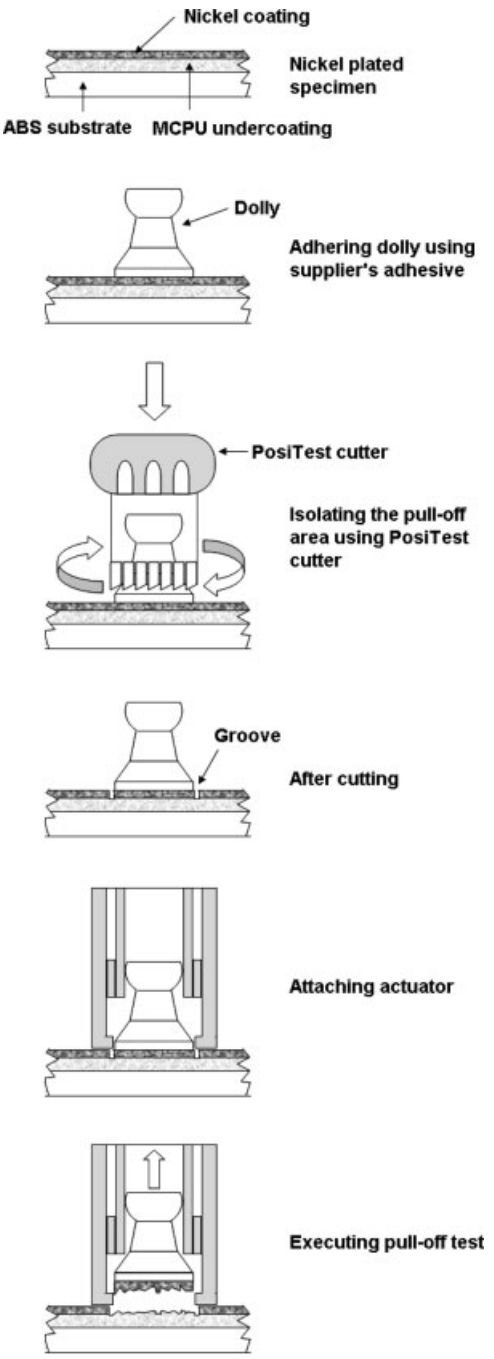

Figure 1 Schematic presentation of the preparation of the adhesion test specimen and the execution of the adhesion test with the pull-off equipment.

gram of the pull-off testing unit is illustrated in Figure 1. For each test, five replicate samples were used, and the average and standard deviation determined.

RESULTS AND DISCUSSION

Polymer derivatives

The IR spectra of the MCPU product is presented in Figure 2 and its spectral assignments are tabulated in Table I. The peak at 2278 cm^{-1} is indicative of the presence of isocyanate (NCO) groups that remained unreacted in the polymer. This constitutes one of the main characteristic of a moisture-cured system.[12] In addition, the absorption bands at 3334 and 1659 cm^{-1} attributed to NH and urea C=O groups respectively, are the yields of NCO reaction with moisture. Furthermore, there are also exist absorption bands at 1708 and 1108 cm^{-1}, which are attributable to urethane C=O and C—O—C stretch, respectively. This pattern suggests that NCO also reacted with divinyl ether polyol. The reaction between NCO and moisture and between NCO and alcohol has been evaluated elsewhere.[13,11] Preliminary studies of the formation of the functional groups are essential to monitor changes in functionality and polarity before and after surface modification by chemical etching.

Morphological characterization – AFM and SEM

AFM images of the untreated and the mildly etched MCPU coatings are shown in Figures 3–5. All untreated specimens regardless of its postcuring period exhibited a relatively smoother morphology compared with that of the mild etched specimens. In other words, the MCPU surface geometry before mild etching has a considerably long roughness wavelength with low peak-valley amplitude. The roughness values were 78, 62, and 44 nm respectively, for the untreated MCPU2, MCPU4, and MCPU7 specimens. Significant increase in roughness was observed once mild etching was introduced (Fig. 6). This can be correlated with changes in the MCPU surface ge-

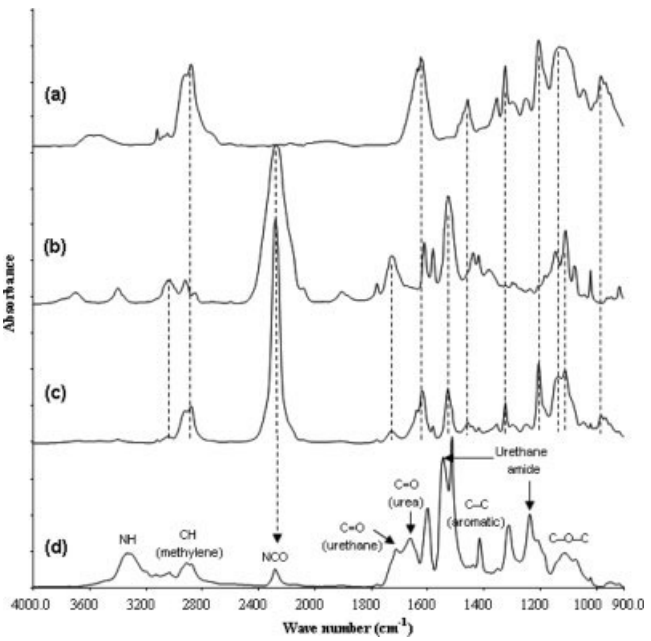

Figure 2 FTIR spectra of (a) polyethylene glycol divinyl ether, (b) MDI, (c) prepolymer, and (d) MCPU polymer derivative.

TABLE I
A Typical FTIR Spectral Assignment of Untreated MCPU[11]

Wave number (cm^{-1})	Functional groups
3488	O—H (H-bonded)
3333	N—H (H-bonded)
3124, 3028	C—H (aromatic)
2908	C—H (methylene), ν_{as}
2870	C—H (methylene), ν_s
2278	N=C=O
1707	C=O (urethane, H-bonded)
1659	C=O (urea, H-bonded)
1598	C=C (aromatic ring)
1541	C—N stretch with N—H bending (amide)
1509	N—H bending
1412	C—C (aromatic ring)
1309	C—N and N—H
1234	C—N
1106	C—O—Cn

ometry, in which the shortening of roughness wavelength as well as an increase in the peak-valley amplitude were observed. Mathematically, shorter wavelength should give rougher interface.[14]

Roughness pattern may not necessarily increase as the etching time increases. Different curing periods generate different roughness profiles. Data indicates a significant decrease in roughness as the etching time increased for MCPU4 specimens. The phenomenon is supported by scanning electron micrographs that show a decrease in surface irregularities when etching time was increased to 15 min (Figs. 7 and 8). MCPU7, on the other hand, experienced slight increases in surface roughness with prolonged etching time. Its pitting as shown in scanning electron micrograph (Fig. 9) is smaller in size and fewer in number compared with MCPU2 and MCPU4. The differences in pit sizes for different postcured samples may be due to the amount

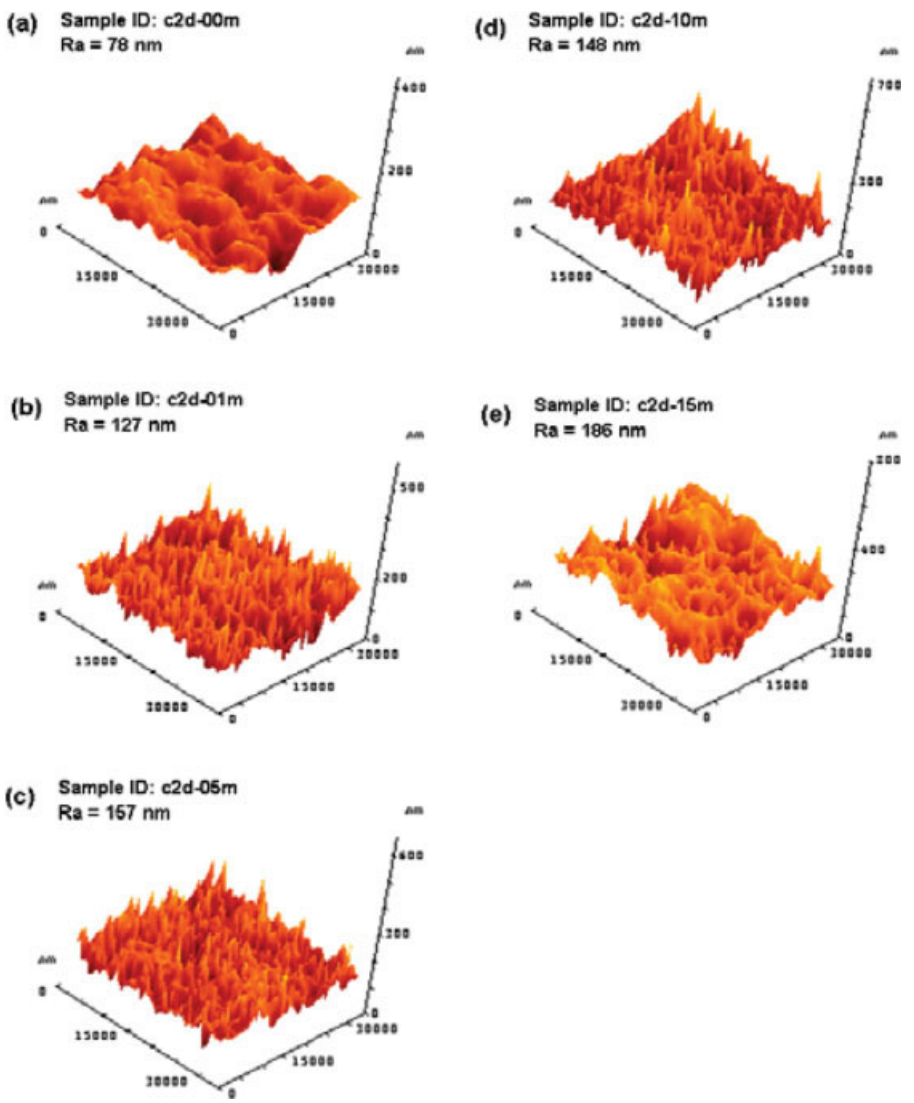

Figure 3 AFM images of MCPU2 (a) untreated, etched for (b) 1 min, (c) 5 min, (d) 10 min, and (e) 15 min. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

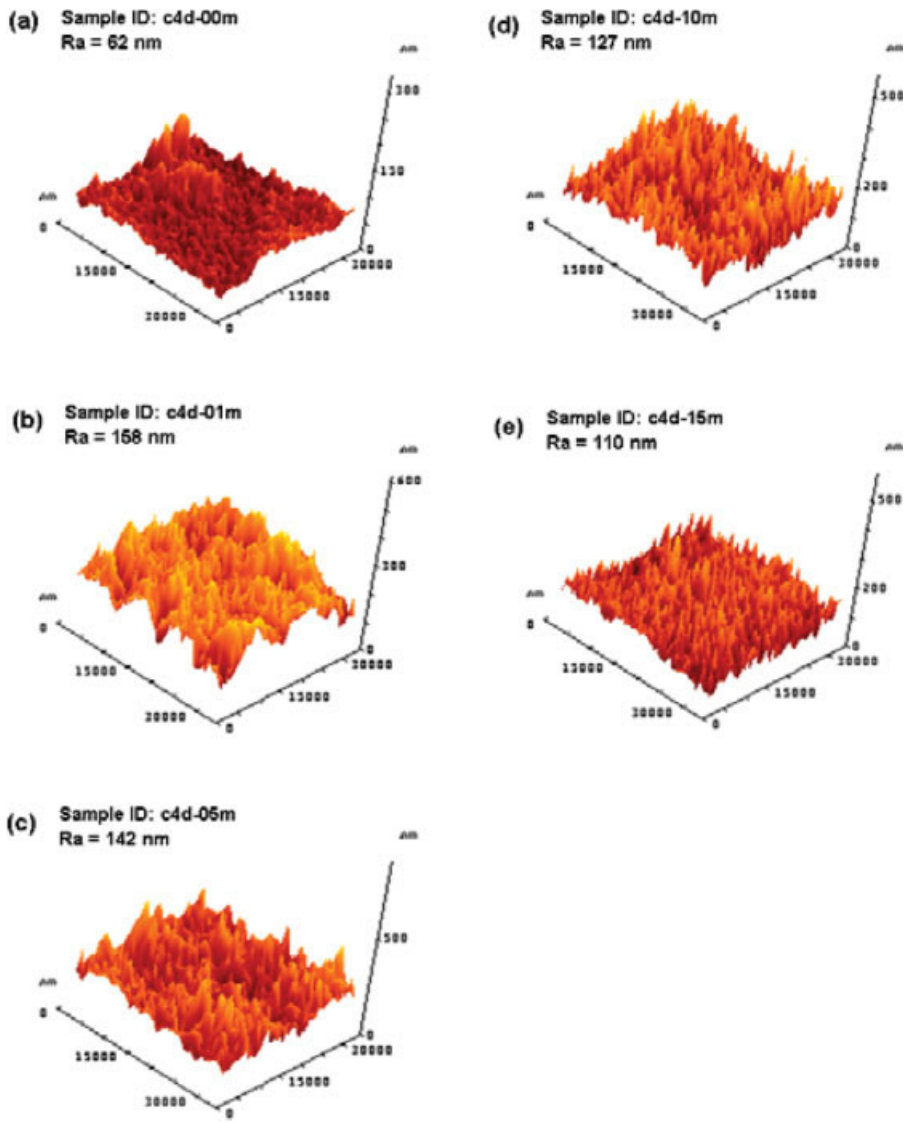

Figure 4 AFM images of MCPU4 (a) untreated, etched for (b) 1 min, (c) 5 min, (d) 10 min, and (e) 15 min. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

of crosslinking in the polymeric system. Prolong curing normally induces crosslinking,[13,11] which results in an increase in hardness and chemical resistance. As a result, MCPU7 became less accessible to oxidation.

Chemistry – FTIR

The typical FTIR spectra of the untreated and the mildly etched MCPU are presented in Figure 10. It was observed that all spectra regardless of their curing periods and etching times portrayed similar spectral patterns except for differences in absorbance intensity. To probe the changes in intensity resulting from mild etching treatment, positive subtraction as described in previous works was used.[15]

The characteristic subtraction profile obtained clearly reveals the depreciation pattern of CH (methylene), urethane C=O and ether peaks (Fig. 10). It can be elucidated that these functional groups underwent an oxidation reaction with acidic permanganate solution (mild etchant). The decrease of 2907 and 2873 cm^{-1} peaks intensity were attributed to the heterolytic C—H bond cleavage from the methylene group,[16] while the decrease of 1110 and 1707 cm^{-1} peaks intensity can be attributed to the oxidation reaction of C—O—C and urethane C=O species that yielded esters and carboxylic acids respectively, as reaction byproducts.[17] The increase of 3490 cm^{-1} peak intensity (appeared as shoulder in the spectrum) attributed to the OH (H-bonded) species can probably be linked to the formation of a carboxylic acid byproduct. The reaction byproducts are normally of low-molecular weight and exist as extractable moieties.[15,18] The removal of the reagents and reaction byproducts by rinsing step resulted in the formation of microscopic cavities as observed through AFM and SEM.

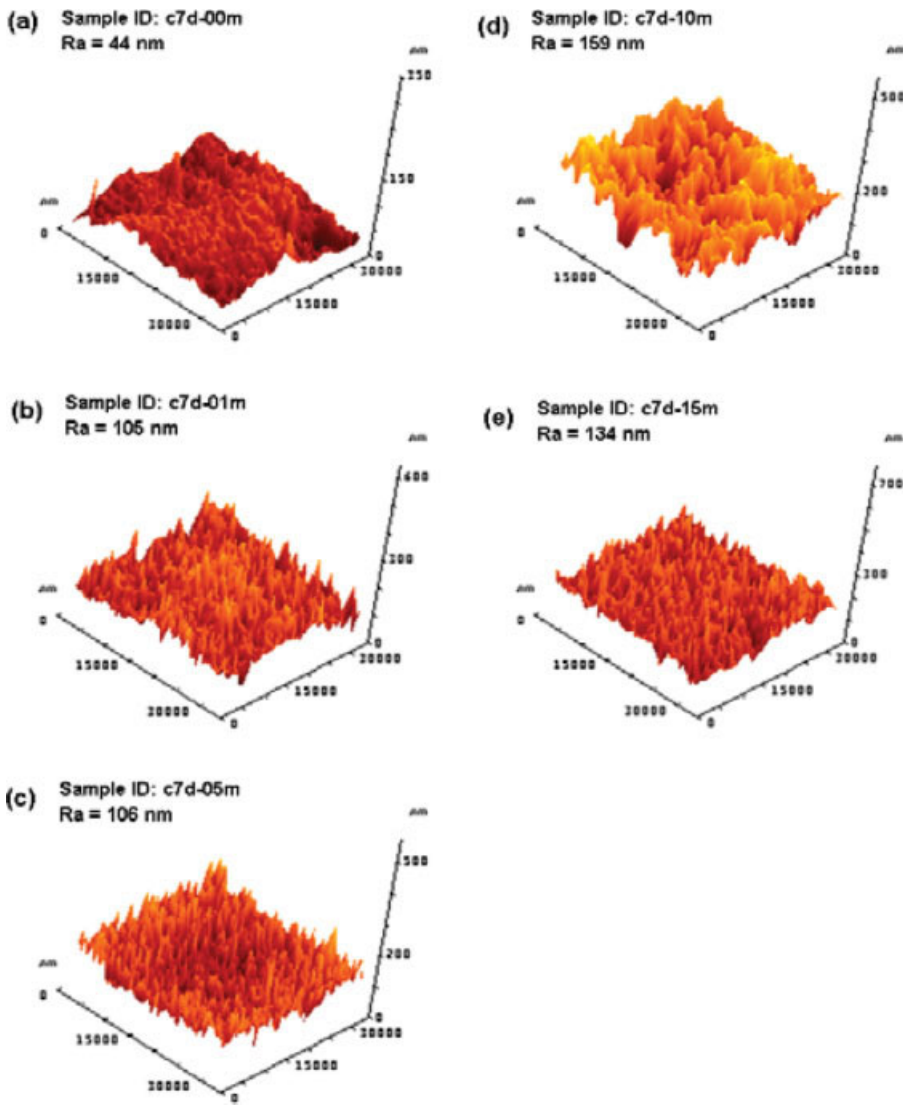

Figure 5 AFM images of MCPU7 (a) untreated, etched for (b) 1 min, (c) 5 min, (d) 10 min, and (e) 15 min. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Three peaks denoting significant increases in intensity after mild etching treatment were noted for urea C=O, NH, and OH (free), at 1659, 3334, and 3620 cm^{-1} respectively. The increase in the OH (free) peak is definitely linked to the improved wettability of polymer surfaces.[19] While, the increase in NH and urea C=O peaks are probably due to the reaction of balance unreacted isocyanates with water, which is the main component in the mild etching solution. It can be inferred that NH and urea C=O were formed and NCO was consumed during mild etching process. It was proven by the decrease of NCO peak intensity after mild etching treatment which confirms that NCO was consumed during the isocyanate reaction. This result is similar for all MCPU specimens.

Subtraction spectra also reveal the effect of curing period on the spectral intensity. Under similar etch-

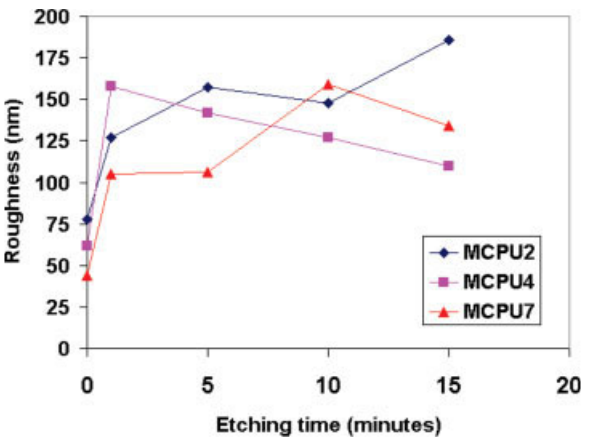

Figure 6 Roughness of MCPU as a function of etching time in the mild etching treatment process. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

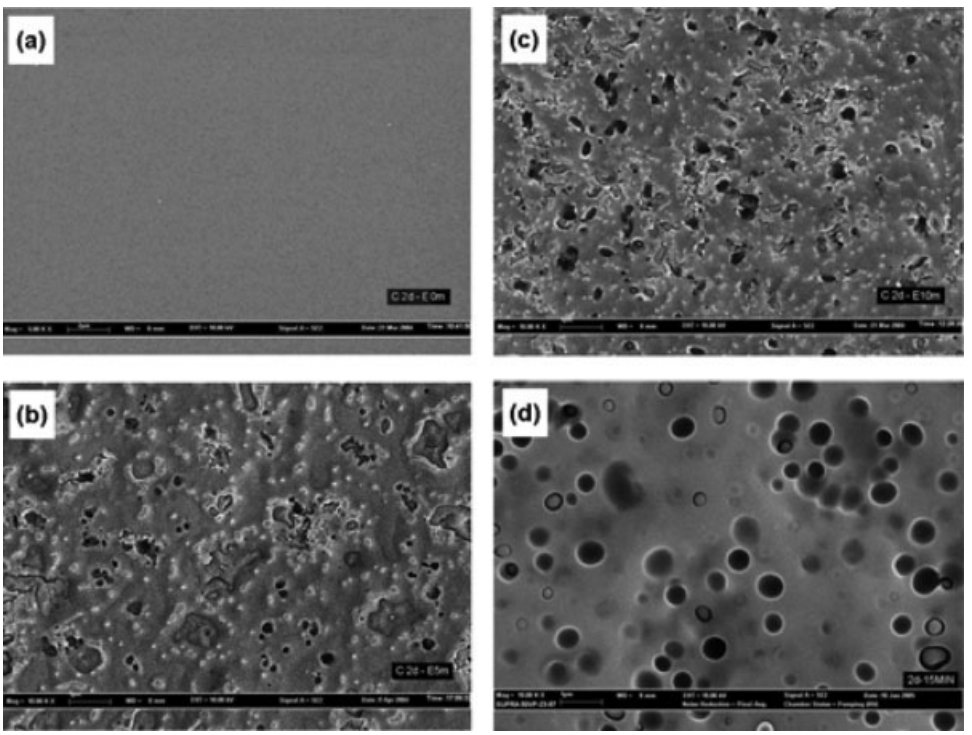

Figure 7 SEM morphology of MCPU2 (a) untreated, etched for (b) 5 min, (c) 10 min, and (d) 15 min.

ing conditions, MCPU7 shows the least intensity difference (Fig. 11). This finding suggests that MCPU7 underwent lower degree of oxidation than MCPU2 or MCPU4, and this finding is reflected by the small morphological change as observed in SEM.

FTIR subtraction spectra of MCPU2, MCPU4, and MCPU7 with respect to etching times are presented in Figure 12. All spectra of MCPU (regardless of curing period) after etching at 15 min show a significant decrease of ~1541, 1509, and ~1234 cm^{-1} peaks that

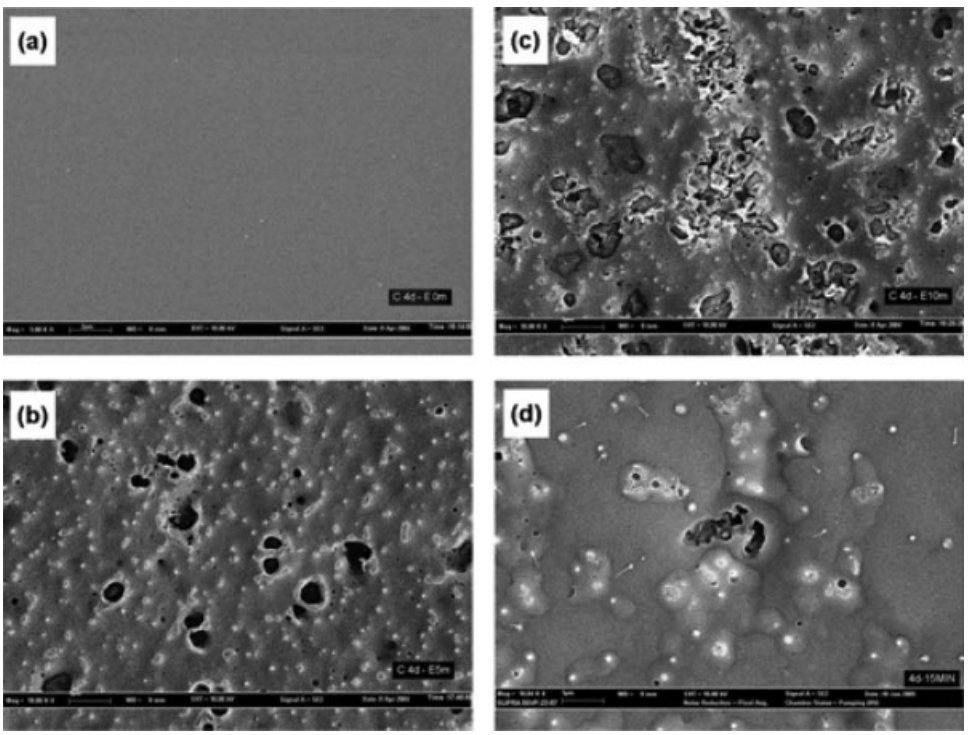

Figure 8 SEM morphology of MCPU4 (a) untreated, etched for (b) 5 min, (c) 10 min, and (d) 15 min.

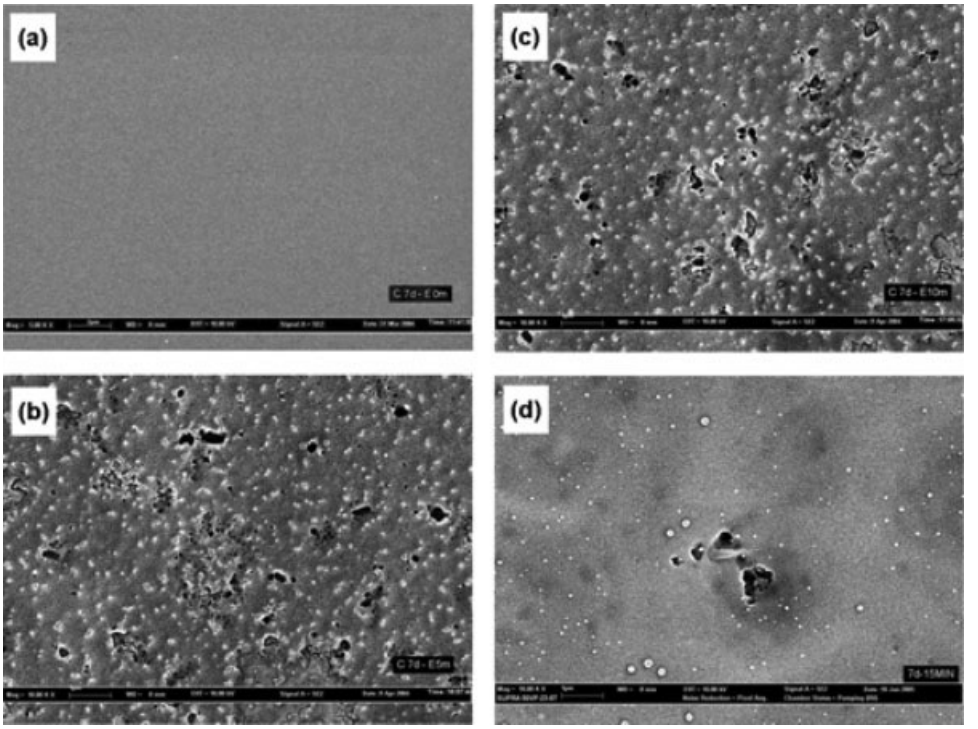

Figure 9 SEM morphology of MCPU7 (a) untreated, etched for (b) 5 min, (c) 10 min, and (d) 15 min.

assigned to urethane amide. Prolonging the etching time to 15 min seriously affected the urethane amide group, perhaps rendered in the collapse of its chemical structure. This observation is supported by SEM analysis that shows decrease in surface irregularities, which is actually can be referred as surface damage.

Wettability

Contact angles are especially useful in that they act as probes of functional group changes, which occur within 5 Å of the water–polymer interface.[19] There was a significant decrease in static contact angle values after mild etching was introduced for 1 min for all MCPU specimens (Table II). This outcome suggests that the surfaces had transformed from hydrophobic to hydrophilic. The increase in polarity was reflected by a broad peak at 3620 cm^{-1} that can be attributed to the presence free OH groups as noted in the FTIR spectrum (Fig. 10). It therefore can be concluded that there is no indication that increasing etching times reduced the contact angle.

Electroless plating and adhesion test

After performing electroless nickel plating according to the mentioned procedures, most of MCPU samples

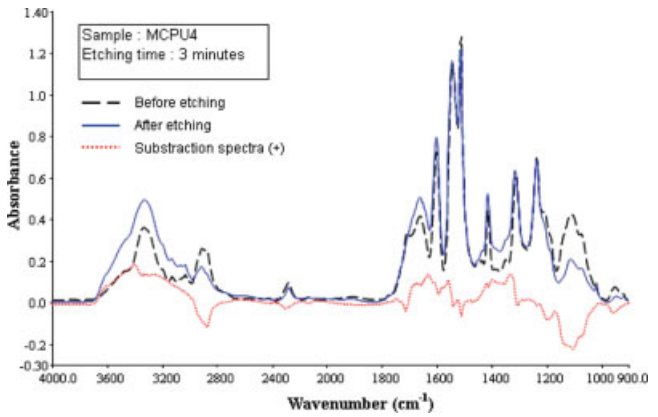

Figure 10 Typical FTIR spectra of untreated, treated and the subtraction spectrum of MCPU. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

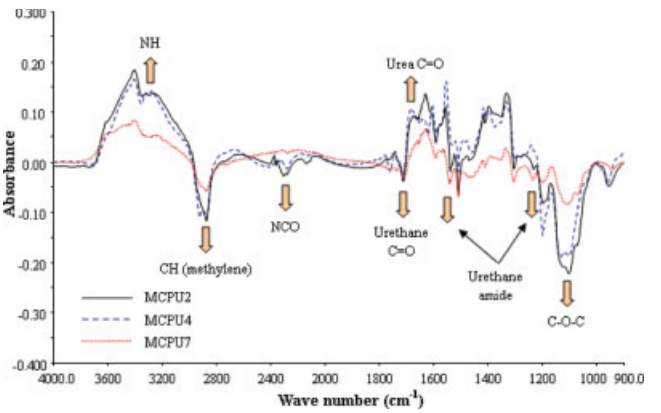

Figure 11 Subtraction spectra of MCPU after mild etching treatment for 5 min show that MCPU7 has the least intensity difference. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

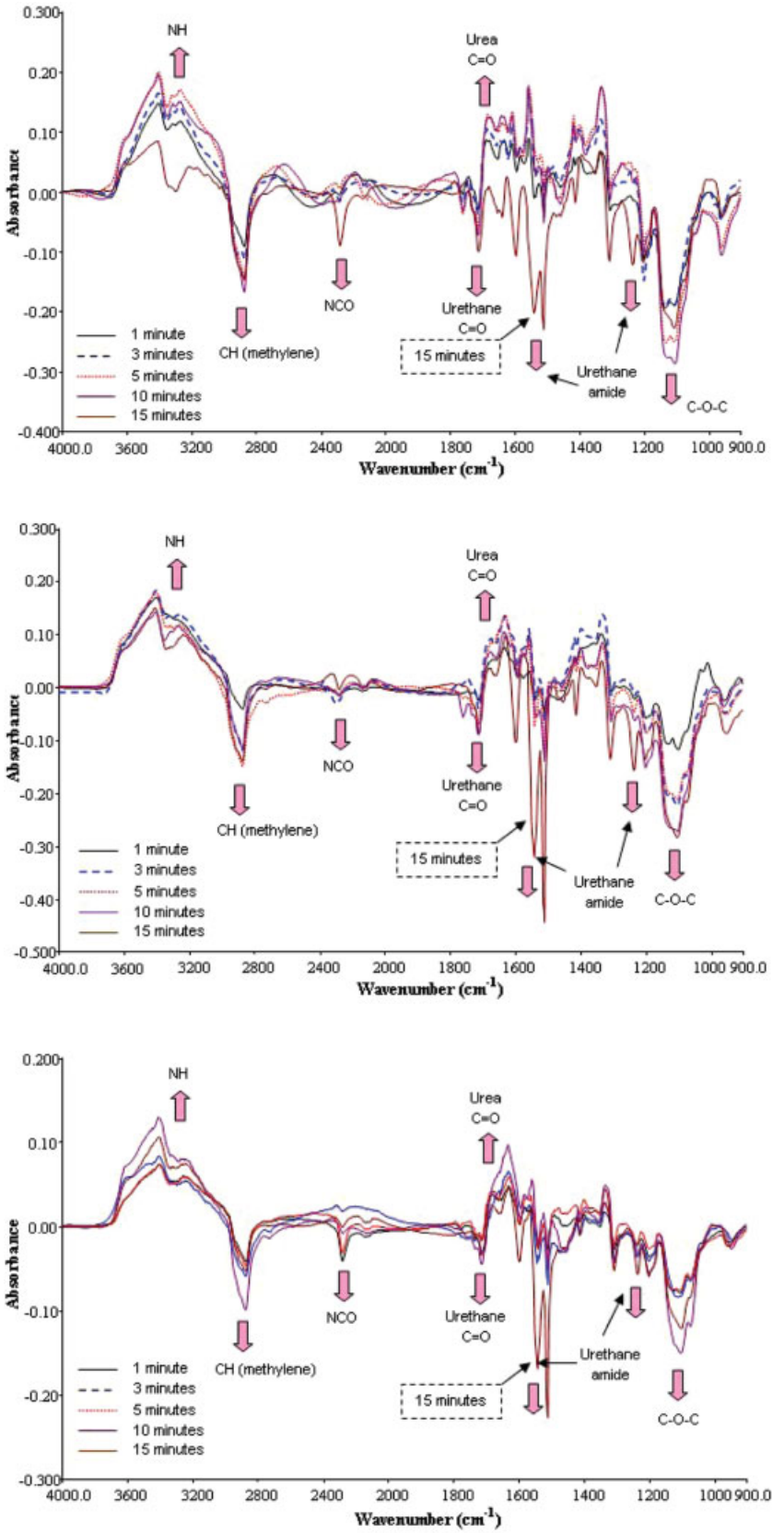

Figure 12 Subtraction spectra of MCPU after etching for 15 min show significant decrease of urethane amide peaks. From top to bottom: spectra of MCPU2, MCPU4, and MCPU7. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

TABLE II
Result of Contact-Angle Measurements of Untreated and Mild Etched MCPU at Varying Etching Times

Etching time (min)	Contact angle (°)		
	MCPU2	MCPU4	MCPU7
0	83 ± 4	74 ± 3	72 ± 2
1	52 ± 4	44 ± 4	44 ± 3
2	48 ± 3	46 ± 3	44 ± 3
3	50 ± 4	47 ± 3	42 ± 4
4	51 ± 3	47 ± 3	41 ± 3
5	50 ± 4	45 ± 4	44 ± 3
7	47 ± 3	47 ± 3	39 ± 4
10	48 ± 5	46 ± 4	42 ± 4
15	54 ± 6	52 ± 5	49 ± 6

that were mildly etched for 15 min were found to have undergone skip plating or poor plating coverage. As described previously,[15,20] extended treatment times can lead to the degradation of the polymer structure, often resulting in low adhesion and poor plating performance due to the formation of weak boundary layers. On the other hand, samples that were mildly etched at varying times between 1 and 10 min attained complete plating coverage. Full coverage of nickel coatings on these samples was obtained approximately after 30 s of immersion in the nickel bath.

The other MCPU samples, which were not subjected to mild etching, were immersed in the activator solution for 1–3 h,[10] and obtained full coverage of nickel coatings within 5 s. Nickel coatings derived by this technique produced better metallic shine. However, the morphology of the nickel deposits revealed cracks on its coating surface (Fig. 13). The adhesion performance of these samples as shown in Figure 14 was also very poor (almost zero).

Data also revealed that all samples that were subjected to mild etching before electroless plating had a better nickel to MCPU adhesion compared with samples without mild etching. The morphology of the nickel deposits also did not show any signs of cracking and fractures on its surface (Fig. 15). All of the samples had a spherical nodular structure and virtually uniform grain size. The nickel deposits constituted mainly of nickel–phosphorus alloys with the phosphorus content being ~2% as confirmed through EDX, which placed them in the low phosphorus content category.[21] Thus, the deposits had a crystalline appearance as there are not enough phosphorus atoms to distort the nickel lattice.[21]

SEM image of nickel–MCPU microsection is presented in Figure 16. The cross-sectional view shows the integrity of nickel deposits with MCPU surfaces. The ''peak and valley'' structure could not be clearly observed on the nickel–MCPU interface, indicating that acidic permanganate etching rendered virtually smooth metal–polymer interface.

In addition to metal deposition, the adhesion of the nickel deposits to MCPU is important. It is observed that different combination of curing period and etching time gave different adhesion performance. For instance, the pull-off test value of MCPU2 increased when mild etching time was increased. In contrast, MCPU4 showed a decrease in pull-off value as mild etching time increased, whereas for MCPU7, there is not much difference in pull-off test value in relation to extended etching time. Based on the surface roughness data with respect to etching time, there is no indication that etching time has a considerable influence on nickel–MCPU adhesion. This was due to the adhesion strength did not appear to increase linearly with the etching time. In addition, it can be seen that different combination of etching time and curing period resulted in different roughness values. In theory, adhesion can increase with increasing roughness.[3,7] This was due to increased surface roughness offers a larger surface area for forming mechanical interlocking between the nickel deposits and MCPU. In this work, only MCPU2 and MCPU4 showed a considerable

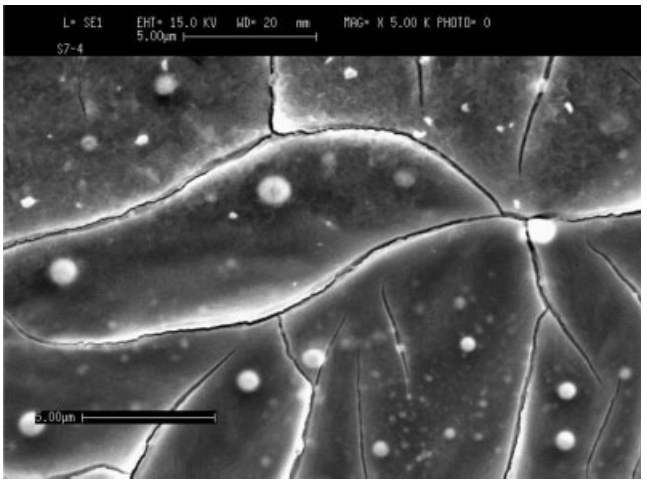

Figure 13 Obvious cracks can be observed on the morphology of nickel deposits obtained from Process 1.

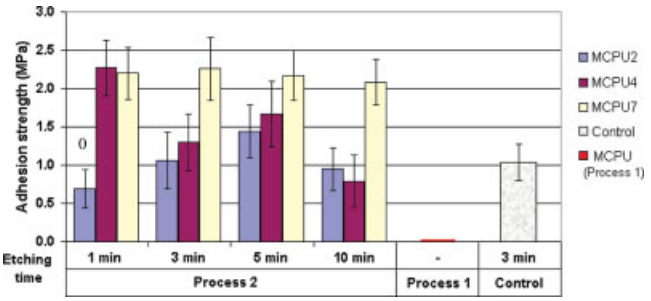

Figure 14 Result of pull-off adhesion test for 1-µm-thick nickel alloy plated on MCPU undercoat at varying mild etching times (Process 2) and compared with Process 1 and control specimens. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

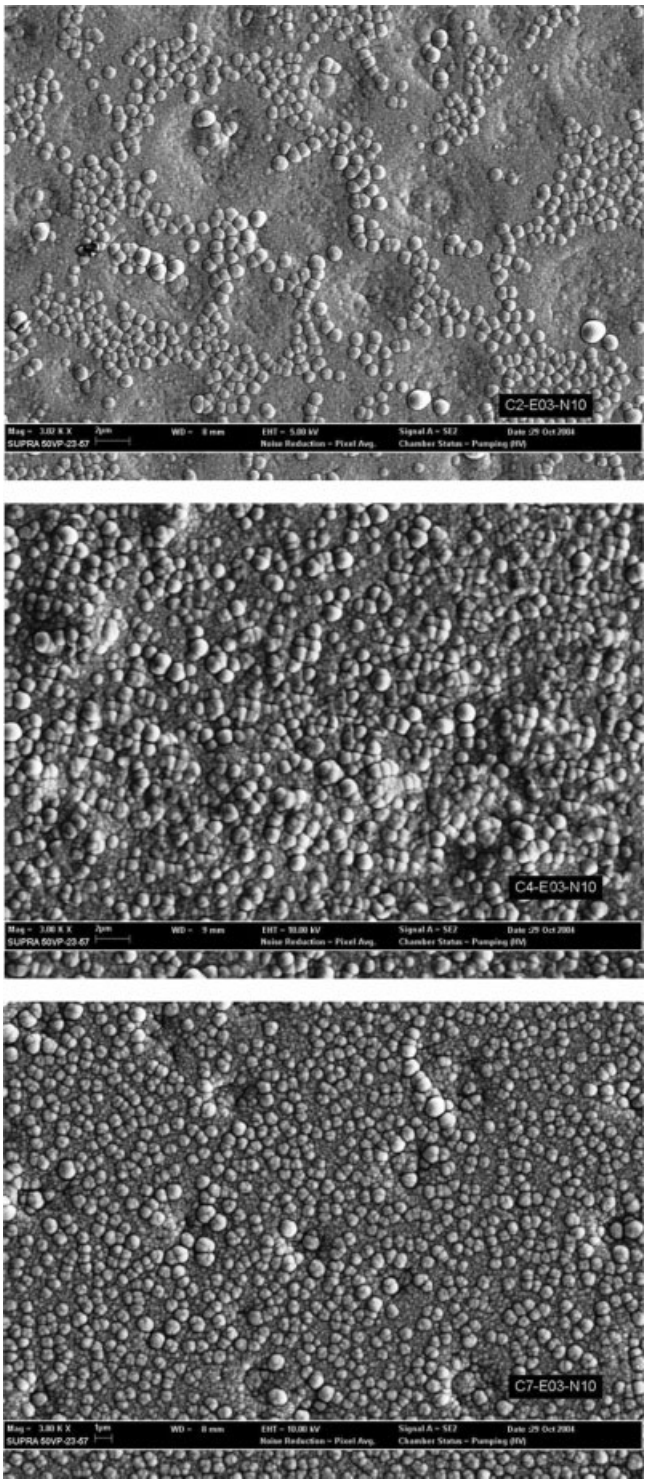

Figure 15 The morphology of nickel deposits obtained from Process 2; nickel is deposited on (a) MCPU2, (b) MCPU4, and (c) MCPU7, after mild etching for 3 min.

influence of surface roughness on adhesion strength. While for MCPU7, increase surface roughness did not result in improve adhesion. In fact its adhesion values are virtually comparable for all etching times although the corresponding roughness is different.

The contrast phenomenon of having virtually comparable adhesion values for nickel–MCPU7 at varying etching time might be due to the corresponding stresses at the nickel–MCPU7 interface were similar or within the same range during the execution of pull-off test. For this reason, increase surface area due to the increasing roughness did not seem to induce nickel–MCPU7 adhesion. Shahid and Hashim[22] concluded that the contrast finding might be due to the differences in joint geometry. Therefore, it is postulated that some MCPU7 surfaces with a relatively lower roughness may have a better nickel-to-MCPU anchor condition compared with other MCPU7 surfaces with higher roughness. As previously described, measured stress of an interface is strongly depends on the particular interface morphology.[14]

In addition, it is interesting to note that MCPU7 had a consistently higher adhesion strength when compared with MCPU2 and MCPU4. MCPU7 samples that were etched between 1 and 5 min had lower surface roughness but possessed higher adhesion strength compared with MCPU2 under similar etching conditions. The differences in adhesion performance between MCPU7 and MCPU2 or MCPU4 can be related to the differences in their joint geometries due to each system has a unique surface profile. Certain surface profiles can trap air and possibly weak boundary layers (WBL) hence resulting in poor filling of nickel deposits within pitting and crevices structures. As previously described,[15,18] weak boundary layers can impair the joint strength of two surfaces. It is believed that MCPU7 has the least interference from WBL compared with MCPU2 and MCPU4. This can be correlated to the finding in FTIR subtraction analysis that revealed MCPU7 has the least intensity difference after mild etching. The intensity difference in subtraction spectra has been described earlier as the degree of

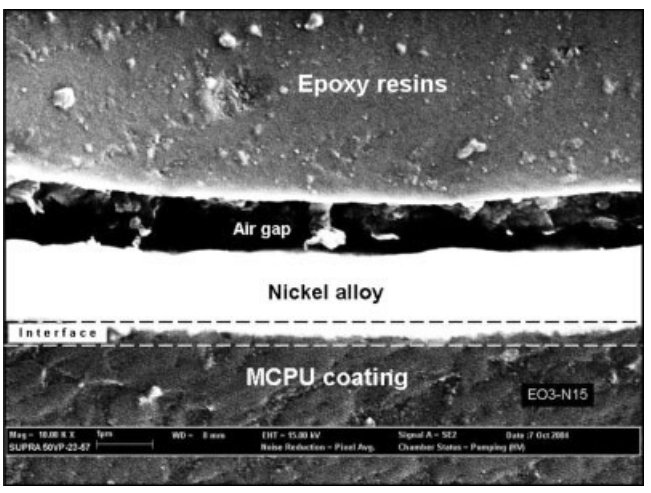

Figure 16 The microsection of nickel–MCPU interface from Process 2 (dash lines) reveals low surface roughness.

chemical reactions that have taken place on the MCPU specimens. More reactions possibly yield more by-products that later turned into WBL and *vice versa*.

Although all of the above-mentioned factors are important, surface profile may have a major effect on the interfacial strength. Improve adhesion due to increase surface roughness can be off-set by surface profile. This is due to surface profile can lead to the trap of WBL as well as providing differences in joint geometry. Without determining which factor is more dominant, pull-off testing can identify the best combinations of curing period and etching time to generate the best nickel to MCPU adhesion.

CONCLUSIONS

The urethane polymer derivative revealed the characteristics of moisture-curable system. In this system, the critical parameter, i.e., curing period directly affected the topography of MCPU after mild etching treatment. It was observed that mild etching treatment produced a low surface roughness (below 200 nm), regardless of curing period and etching time. Etching at 15 min caused severe damage of MCPU surface therefore is not compatible for electroless nickel plating. Although surface topography and roughness of MCPU were affected by curing period and etching times, there was no trend observed on the effect of curing period and etching times on adhesion performance except on MCPU4. On average, samples that were postcured for 7 days showed better adhesion performance. The results show that MCPU system can be used as a thin layer for electroless plating and it offers smooth metal–polymer interface. However, extra precaution during etching (i.e., time) need to be taken since the results show that the longer the oxidation reaction of the MCPU layer by an acidic permanganate solution causing more skip plating due to the less adhesion between nickel particle with the MCPU. Finally, the method developed from this investigation (i.e., Process 2) can be applied to produce decorative object or souvenirs made form nonplatable grade materials, which can enhance their appearances and value.

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