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Research and Evaluate the Chemical Composition and Microstructure Characterization of the Ni-B Plating Process on the Surface of Plexiglas®

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Abstract:*.This present research article deals with the study of the chemical composition of the Ni-B plating process on the surface of Plexiglas®. The Ni-B coating is deposite on the Plexiglass (PMMA). The chemical composition of the Ni-B is evaluated with the ASTM by modern technologies such as XPS. EDX. SEM. Adhesion behavior of layer coating after different plating periods is evaluated with the ASTM standard's help. The microstructure characterization and the coating thickness are also an important influence on adhesion properties tested through SEM, EDX, XPS analysis. The Ni-B coating exhibits good adhesion on insulation with adequate time*

Keywords: *Electroless plating, Ni-B coatings, adhesion behavior, coating thickness, ASTM standard.*

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I. INTRODUCTION

Plexiglas (polymethylmethacrylate or PMMA) is one of the most widely used polymers in areas, from daily life to advanced technologies such as clinical medicine [1], drug delivery [2], dental materials [3], design and fabrication of newmicro/nano-systems [4,5] etc. The extensive use of Plexiglas® has been made possible by its fairly low glass transition temperature (Tg), excellent transparency in frequencies ranging from ultraviolet (UV) to near infrared (NIR), and high dimensional stability [5,6]. Plexiglas® is also low-cost and conformable to a variety of fabrication methods, and thus suitable for both prototyping and large scale replica molding [5]. The metallization of Plexiglas® is indispensable from the perspective of its utilization in a variety of application, and in particular for micro/nano-electromechanical systems (MEMS/NEMS)[6,7]. However, in general, the metallization of polymers is very challenging [7]. The metallization of an insulating polymer can be achieved through physical-based deposition technologies (physical or chemical vapor deposition techniques, sputtering) or chemical electroless deposition [7]. While the conventional physical deposition techniques in the field of MEMS/NEMS rely on high vacuum or high temperature equipment requiring very expensive and complex instrumentation, we have previously reported that chemical electroless plating can be an efficient alternative approach for metallizing the surface of insulating substrates [7].

Platings Ni-B have special properties to increase the working ability of machine parts in worn and corrosive conditions. The coating thickness available can reach a few µm to a few hundred µm depending on the plating time. Currently, Ni nanocomposite coating is receiving a lot of research interest, some publications about high quality Ni nanocomposite coatings, applied in the field of anti-wear and corrosion protection for products. made of metal and non-metallic materials. [8]. Adhesion of the coating is the essential goal of any plating process. When adhesion is not achieved, any attempt to improve other technical factors will be pointless. The coating's adhesion is also very much related to the thickness of the layer on the insulating substrate [9]. The plating thickness is too thin, which causes poor adhesion on the substrate, while the thickness of the plating is too thick will cause excessive stress, and the coating will also peel off. Therefore, the coating thickness is not only related to the product quality but also affects the economics of the production process. In general, the adhesion capacity of Ni composite coatings depends on many coating factors. The present study evaluates the adhesion of the coating through ATSD criteria, thereby drawing each relationship between plating time and bonding. In the last, a confirmation test is conducted to validate the test results. The surface morphology and composition of Ni- B coatings have been learned with the support of scanning electron microscopy (SEM), energy dispersed X- ray analysis (EDX), X-ray Photoelectron Spectroscopy (XPS) [8-10].

II. EXPERIMENTAL PROCEDURE

The experiment was done with circular samples (with a diameter of 15cm) made from a Plexiglas® (PMMA) sheet. A Wire EDM machine cut these samples. Besides, they were repeatedly cleaned with deionized water and isopropanol and then dried with nitrogen gas.

Surface pretreatment

In this study, the Plexiglas® surfaces were pretreated by the following solution: 37% HCl solution; Hydrogen peroxide H_2O_2 50%; and distilled water at a ratio of 1: 1: 5 (this solution is called SC_2 solution). This process takes about 8-12 minutes. Dip the substrate submerged in the solution, adjusting the pH in the solution to approximately 1,0. The cleaning process is done at 60^0C, then washed with distilled water, and then dried with nitrogen gas stream. As is well known, nitrogen is an inert gas. The nitrogen drying not only makes the surface cleaner, but also creates a tighter adhesion to the organic coating in the future.

Grafting of vinyl pyridine seed layer

The grafting of vinyl pyridine seed layer process on Plexiglass is demonstrated in Fig. 1. According to the process, first 1.22 g of 4-nitrobenzenediazonium tetrafluoroborate (97%, Sigma Aldrich) was dissolved in 0.5 M HCl (Fisher), then 5.56 ml of 4-vinyl pyridine (97%, 0.975 g/ml, Sigma Aldrich) was put into the mixture. To adjust the volume of the solution until it reached 1000 ml, 0.5 M HCL was added, and thus, vinyl pyridine and iazonium cations had concentrations as 0.05 mol/l and 0.005 mol/l, respectively. The flask then received 250 ml of solution, and after that, 0.2 g of Fe powder (b10 m, Alfa Aesar). Special attention should be paid to adding Fe powder. If the iron powder content increases by a few percent, it can directly affect the coating quality.

The chemicals used for Ni – B composite plating are listed in Table 1.

Table 1. The chemicals used for Ni – Al_2O_3 composite plating

Clorua nikel ($NiCl_2.6H_2O$)	20 g/*l*
Hypophotphit natri ($NaH_2PO_2.H_2O$)	10-36 g/*l*
Natri axetat (CH_3COONa)	10 g/*l*
Suxinic Acid ($C_4H_6O_4$)	10 g/*l*
Palladi Clorua $PdCl_2$	0,05 g/*l*

Figure 1. *Grafting of vinylpyridine seed layer on Plexiglass*

Scanning electron microscopy (SEM) and energy dispersive X-ray (EDX)

Scanning electron microscopy and energy dispersive X-ray (EDX) machine (TESCAN, VEGA3) is used to observe the composite coating's surface morphology and thickness. The plating details are plated for 09 minutes, 18 minutes, …, 27 minutes. After plating, the sample is washed in running water and then ultrasonically rinsed in distilled water for about 10 minutes. The details are then cut on a metal cutting machine, then grinded on a polishing machine, polished with 1 µm diamond, then impregnated and thickness determined on a Scanning electron microscopy and energy dispersive X-ray (EDX) machine (TESCAN, VEGA3). The chemical composition of the Ni-B is evaluated by modern technologies such as XPS (AXIS Supra, KRATOS – SHIMADZU). It should be noted that polishing has a huge impact on the EDX evaluation process. During the polishing with diamond grinding powder, it is necessary to avoid the diamond powder being too wet, leading to stickiness on the surface of the sample. To achieve this it is necessary to carefully add distilled water during the polishing stage.

Adhesion tape test

The adhesion tape test was done based on the ASTM testing process. The test is expressed as follows: A set of 10× 10 cross-hatched scratches, with an inter-scratch distance of 1 mm and cross scratches at ~90°, were made on the film in completely dried condition. A pressure-sensitive tape was applied over the grid, smoothened, and rubbed over to ensure good contact. In addition, the tape was removed by seizing the free end and pulling it off rapidly back upon itself at as close to an angle of 180° as possible after gluing for 90 s. The adhesion was evaluated according to the ASTM standard. Also, three tests were conducted to evaluate the adhesion.

III. RESULTS AND DISCUSSION

The XPS survey spectrum of the pristine Plexiglas surface (Fig. 2, black) presents a typical behavior of PMMA with only two peaks, C 1s at 285 eV and O 1s at 531 eV. Thorough XPS characterization of the PMMA surface has already been reported elsewhere, and thus, is not repeated here. Here we will discuss the mechanism of functionalization of Plexiglas with vinylpyridine assisted through diazonium chemistry. According to the grafting procedure described in the experimental section, diazonium cations are reduced by Fe powder to generate 4-nitrophenyl radicals.

As anticipated, in addition to the C 1s and O 1s peaks, the XPS survey spectrumof themodified Plexiglas® also shows the presence of the N 1s peak (Fig. 21, red). The N 1s core level spectrumof the grafted layer on Plexiglas® (inset of Fig. 1) reveals a significant contribution fromN-containingmoieties present at 399.2 eV, which are mainly attributed to pyridine functionalities.

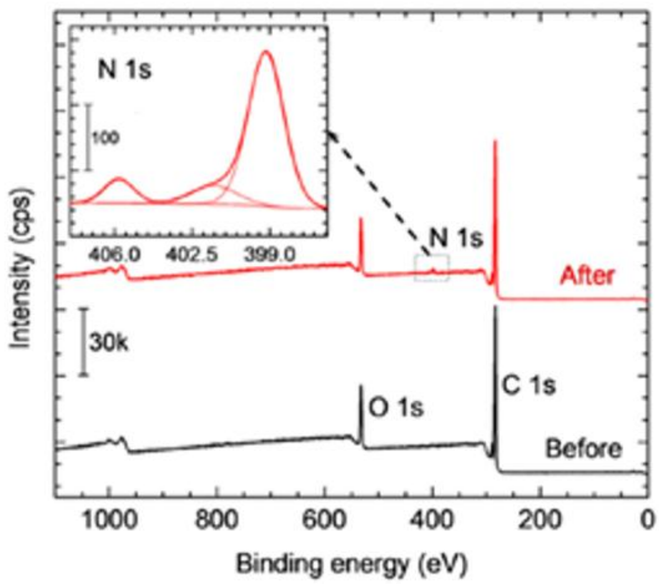

Figure 2. *XPS survey spectra of pristine Plexiglas® before (black) and after (red) functionalization through nitrophenyl-vinylpyridine organic layer with the inset showing high resolution N 1s spectrum of the functionalized Plexiglas.*

Effect of the plating time on the chemical composition and coating thickness

The plating composite samples (Ni-B) are plated in 09 minutes, 18 minutes, 27 minutes. After plating, the sample is rinsed in running water, then ultrasonic washed in distilled water for about 10 minutes, finally dried with a stream of nitrogen gas. To obtain a clear microscopic coating image, the coating sample is grinded on

800, 1000, and 1200 draft paper, polished on a Taiwan metal polishing machine with 1 µm diamond powder impregnated in 2% Nital solution for 1 minute.

The coating samples within 9 minutes

After 09 minutes of plating, the coating still adhesion well on the surface of the part, and the thickness of the coating reaches about 20-23,5 µm (Fig. 3). The main chemical composition is Ni (62.6%), O (5.9%), C (21.6%), Al (2.3%), Fe (2.6%); this shows that the coating composition has the full range of desirable chemicals to enhance the coating properties.

We can see that the adhesion of the composite coating to the substrate is quite good (without gaps - fig. 3).

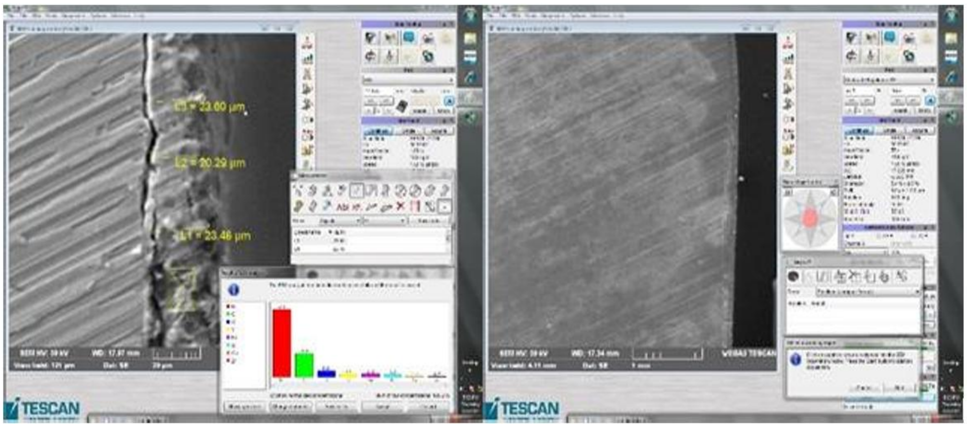

Figure 3. *SEM and EDX images of Ni-plated samples within 09 minutes*

The coating samples within 18 minutes

After 18 minutes of plating, the coating thickness is about reaches about 69-70 µm (Fig. 4), adhesion of coating layer is quite good. The main chemical composition is Ni (68.6%- increased), O (5.9%), C (10.6%), Al (1,5%); EDX analysis results in Fig. 4 show the participation of Al_2O_3 particles in the Ni coating very clearly. However, on the analytical chart, also appears element Fe is an impurity attached to the coating. This is a problem that needs to be overcome in the Ni-B composite plating process, removing Fe ions from the electrolyte solution altogether.

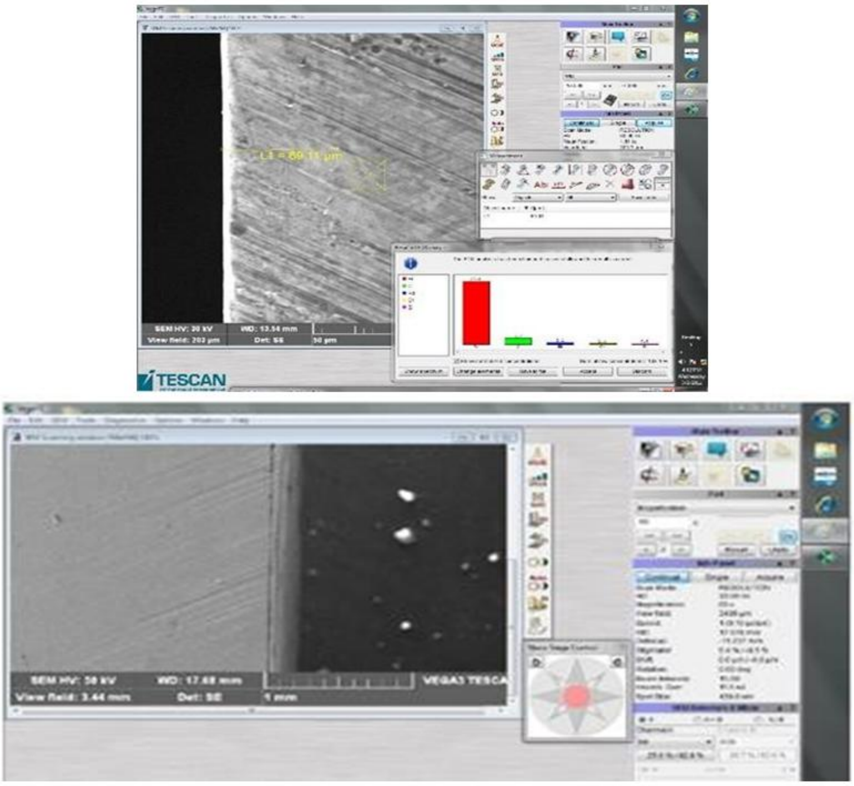

Figure 4. SEM and EDX images of Ni-plated samples within 18 minutes

The coating samples within 27 minutes

After 27 minutes of plating, the coating thickness reaches about 163-170 µm (fig. 5). Then, we can notice the separation of the coating from the substrate surface. The 75% increase in coating thickness in just a few minutes is explained by the fact that the coating surface becomes rough as it is completely covered with AL_2O_3. At this time, the neutral particles are deposited very quickly by deposition mechanism in arrays. This has negative consequences for the coating. This sudden increase in coating thickness will open up other research proposals after this topic. The main chemical composition is Ni (66.3%), O (8,2%), Al (5,4%-increased); However, at this time appears the stress concentration, the coating no longer has good adhesion on the substrate (fig. 5).

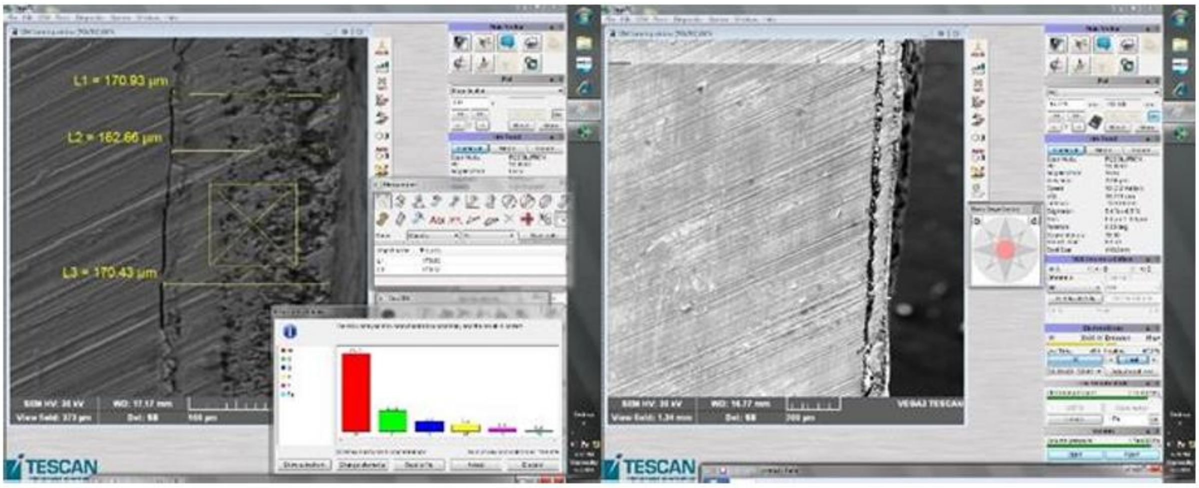

Figure 5. *SEM and EDX images of Ni-plated samples within 27 minutes*

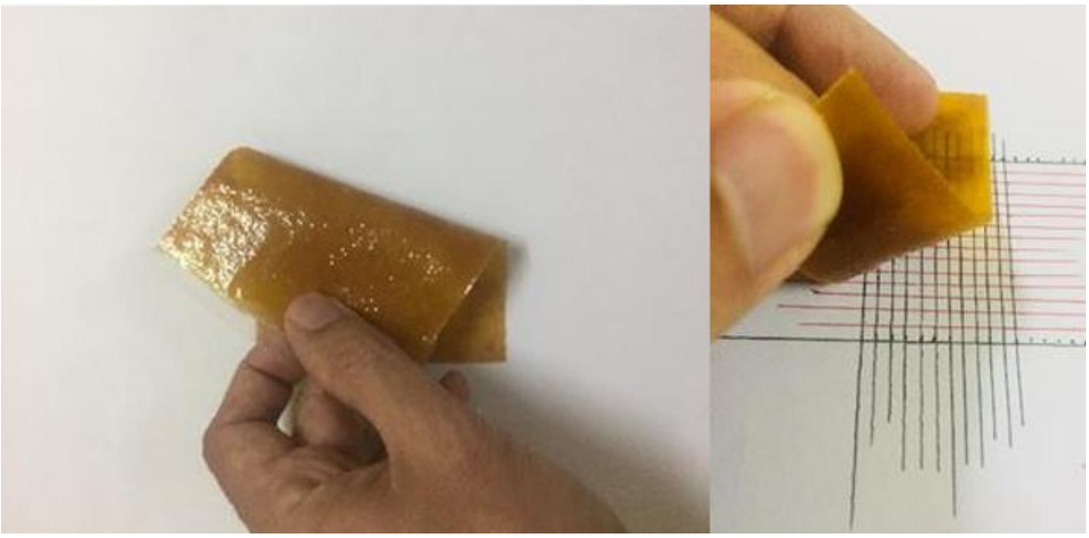

Figure 6. Adhesion test

Effect of the plating time on the adhesion of Ni-Al_2O_3 coating

In this work, an adhesion test is used to evaluate the adhesion of Ni-Al2O3 coating. The adhesion test has been used effectively in several studies [11,12]. In this method, the adhesion of Ni-Al_2O_3 coating on the Plexiglass® (PMMA) sheet is the essential factor. The adhesion of the coating is checked by a tape of ASTMD - US testing standards. Also, a diamond knife was used to cut several straight lines on the Ni-Al_2O_3 -coated Plexiglass® sheet (Fig. 6). These lines are separated by 1 mm, creating one of the scratches grid made 10 x 10 (mm^2). This test tape is applied to the cut Plexiglass® plastic sheet. According to the experiment, no squares are removed from the sheet after peeling off the adhesive tape. Clearly, according to the test on the Ni-Al_2O_3 coating, the required adhesion on the Plexiglass® plate was satisfied.

IV. CONCLUSION

From the above results, it is reported that the Al_2O_3-Ni composite coating has good adhesion ability on Plexiglass (PMMA). The reasonable time for plating is from 09-18 minutes for the plating process. When the plating time increases to more than 27 minutes, the coating thickness increases suddenly to 163-170 µm. There is a separation of the coating from the substrate surface, the next research issue is to optimize the plating time and also the thickness of the coating. Al_2O_3 particles are distributed relatively evenly according to the thickness of the coating. The bonding of the coating to the substrate at the corners is still good. American standard ASTMD adhesion test and demonstrate good adhesion between the substrate material and Al_2O_3-Ni coating. However, this is only a temporary test for adhesion by mechanical methods. In the near future, the author wishes to use more modern methods to test for adhesion, such as analysis by plasma, analysis by spectroscopy ....

Conflict of interest

There is no conflict to disclose.

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