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Publisher's version / Version de l'éditeur:
https://doi.org/10.1016/j.surfcoat.2004.07.083
Surface & Coatings Technology, 192, 2-3, pp. 323-330, 2005-03-21

Residual stress analysis – an important consideration for coating of stereolithography polymers

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Abstract

Residual stress is a very significant issue related to the mechanical, physical and chemical performance of coatings. This communication reports our recent progress in the understanding of the effects of residual stresses on the coating of stereolithography polymers. X-ray diffraction and hole-drilling methods were used to analyze the residual stresses of metal coatings and SL5195 polymer, respectively. This analysis provides an insight into the engineering consideration when applying hard coatings on stereolithography polymers. A solution for successfully coating SL5195 polymer with favorable residual stress is also provided.

Key words: residual stress, XRD, hole-drilling, coating, stereolithography polymer

1. Introduction

Stereolithography Apparatus (SLA) has become an important tool for rapid prototyping (RP) [1-4]. This technology uses a UV laser to selectively solidify successive thin layers of a photocurable resin to form a 3D object. The rapid prototype is beneficial to both the design laboratory and the modeling process [5]. The technology allows designers to verify their product design at an early stage by using 3D representations for design review, marketing and production [6-8]. While the SLA part could also be used for many

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other applications, it is somewhat limited to form-and-fit analysis. However, applications that require withstanding of high stress/load and wear, high moisture/corrosion and high temperature are not without challenges and limitations. In order to enhance the functionality of the SLA product, encapsulation of the SLA part with a metallic coating appears to be an effective approach[1,2].

One way to enhance the high stress/load and wear performance of an SLA polymeric component is to apply a layer of nickel or nickel alloy on the surface. Electroless deposition is a logical choice due to the maturity of the technology and its unique characteristics such as uniform deposition and low capital investment and manufacturing cost. However, challenges were encountered when electroless Ni-P deposit was applied directly on the surface of stereolithography polymeric components. Blistering, cracking and delamination of the coating were observed. Significant substrate distortion was also observed when acidic electroless deposition process was applied. These phenomena are indicative of a high level of residual stress in the Ni-P coating. Even though the nature of the internal stresses developed during the electroless deposition process has not yet been fully explained, it is believed that the internal stresses might originate from the mismatch between Ni and P, and the evolution/incorporation of hydrogen during the deposition process.

This paper presents a successful solution to the challenge of coating SL5195 polymer along with an investigation on the effect of residual stress and the mechanism of metal layering.

2. Experimental

2.1 Materials & chemicals

All the chemicals of Reagent Grade (RA) for coating of SLA polymer in this study were supplied by Sigma Aldrich. The temperature for all the processes was controlled using a NESLAB GP200 water bath with a temperature variation of less than 1°C. Agitation of the solution was carried out throughout the processes to ensure a homogeneous temperature distribution in the bath.

2.2 Coating of SL5195 polymeric parts

The basic coating process flow is shown below.

Fabrication of SLA specimens
↓
Surface preparation of SLA specimens
↓
Surface Activation
↓
Electroless Cu deposition
↓
Cu electrodeposition
↓
Electroless deposition of Ni-P

2.2.1 Fabrication of SLA samples

Simple stereolithography RP samples were fabricated using the SL5195 resin provided by 3D Systems. The resin is widely used in rapid prototyping but the chemical composition of the polymer product is proprietary. According to the information

revealed by the manufacturer, the SL5195 resin is a six component mixture containing cycloaliphatic epoxy resin, aliphatic glycidyl ether, polyols, modified acrylate ester, acrylate ester and photocuring agent. The SLA parts for this study were designed on a Pro-Engineer with Version 18.0 software provided by Parametric Technologies. Model SLA 5000 stereolithography apparatus supplied by 3D Systems was used to make the samples. The SLA parts are characterized of a layered structure. The parts after photo-curing are drained and subsequently rinsed in a RAMCO tank containing TPM solvent with mechanical agitation, and subsequently thoroughly rinsed again with water to ensure the removal of any remaining liquid resin from the surface of the SLA parts. The cleaned parts are then subjected to a four-hour post-curing process in an UV irradiation chamber to complete the entire photo-curing process.

2.2.2 Surface preparation

Surface preparation is an essential step to create micro topographical features on the substrate surface to enhance the bonding between the coating and the substrate. This can be obtained using conventional glass beading method, namely, bombard the sample surface with glass beads. The effect of the glass beading can be optimized by adjusting the size of the beads and the bead pressure. The glass bead used in this study is Flex-o-Lite BT 10. The glass beaded samples were water rinsed and air dried alternately to prevent any contaminants from being transferred into the subsequent step. The readiness of the surface, a key factor that contributes to a satisfactory bonding, needs to be carefully assessed. It is well known that there exists an intimate link between the contact angle and wettability or hydrophilicity [9]. Contact angle of the polymer surface was therefore measured using a Kernco Cam Micro Contact Angle

Meter equipped with a microsyringe attachment. The measurement was conducted at four different points on both sides of each sample. The static contact angles on SL5195 polymer surfaces with and without glass beading were measured (five samples each) of 36^{o} and 63^{o}, respectively. The drastic decrease in contact angle of glass-beaded surface clearly demonstrates a significant improvement of the surface hydrophilicity in the presence of micro topographic features on the hydrophobic surface. In addition, these micro topographic features provide anchoring sites for metallic atoms during subsequent metal deposition process. It ought to be mentioned that the above contact angle data was not obtained from ideally smooth surfaces and is therefore not an equilibrium contact angle. The equilibrium contact angle θ_e corresponds to the lowest energy state for a system. On an ideally smooth and compositionally homogeneous surface, the equilibrium contact angle is the Young's angle θy. However, many real surfaces are rough or heterogeneous. A liquid drop resting on such a surface may reside in a metastable equilibrium (energy trough separated from neighboring states by energy barriers), exhibiting a metastable contact angle.

2.2.3 Surface activation

Electroless deposition proceeds only when the surface is catalytic. The catalytic property of a material originates from the existence of partially filled orbital in its atomic electron configuration. Surface activation is a critical step to render a non-catalytic polymer surface catalytic so as to induce subsequent electroless deposition. Once the surface is coated with a catalytic metal such as Cu, the deposition process proceeds continuously, which is normally referred to as 'auto-catalytic'.

A polymeric surface is non-catalytic and requires an activation process to make the surface catalytic. Of all the activation processes known, palladium based activation is the most commonly used. In this activation process, palladium ions are reduced to palladium metal by excess tin ions, which then adhere to the surface of the SLA polymer. Palladium metal is catalytic to electroless copper deposition, which imparts a subsequent continuous deposition once a catalytic layer of copper is generated on the SLA polymer surface. The activation bath and operating conditions used in this study are provided below.

$SnCl_2.2H_2O$: 80 g/L
$PdCl_2$: 1 g/L
HCl: 45 mL/L
Time: 7 minutes
Temperature: 25 °C

2.2.4 Electroless Cu deposition for conductivity enhancement

In order to render the SLA sample surface electrically conductive for the subsequent electrodeposition, a new electroless copper deposition process was studied. An optimized bath was developed as described below.

$[CuSO_45H_2O]$ = 10 g/L
$[KNaC_4H_4O_64H_2O]$ = 22 g/L
$[Na_2CO_3]$ = 2 g/L and 0.5 g/L
$[NiCl_26H_2O]$ = 2 g/L
[HCHO] = 70 mL
pH = 12.40
Temperature = 38 °C

2.2.5 Cu electrodeposition

Cu electrodeposition was conducted in a bath described below.

120 g/L $CuSO_4$
100 mL/L concentrated H_2SO_4
0.185 mL/L – 0.211 mL/L concentrated HCl (~70 – 80 ppm Cl^-)
0.00749 g/L dextrin
0.01123 g/L thiourea
Temperature: 25 ^0C
pH < 1.0
Current density: 100 A/m^2

2.2.6 Electroless deposition of Ni-P alloy

The bath composition and operating parameters are given below:

$NiSO_4.6H_2O$: 30 g/L
$NaH_2PO_2.H_2O.H_2O$: 20 g/L
CH_3COONa: 20 g/L
pH: 4.5
Temperature: 70 ~ 90 ^oC

2.3 Stress measurements

2.3.1 X-ray diffraction

X-ray diffraction technique was used to analyze the residual stress in the coatings. A Philips X'Pert X-ray Diffractometer (XRD) was used to measure the residual stress. The X-ray radiator was a graphite-monochromaticalized Cu tube with a wavelength of 0.1506 nm. The XRD system was set at 40 kV/45mA, and the scanning rate was

1/4°/min. The $\sin^2 \Psi$ method was applied for the residual stress evaluation[10]. For the copper coating, the diffracted line (331) or (420) was selected to determine the residual stress.

To measure the residual stress induced by an amorphous-like electroless Ni-P layer on an existing Cu-coating, the diffracted lines generated by the underlying Cu-layer were used. The result indirectly reflects the residual stress induced by subsequent Ni-P coating, where the underlying Cu layer is well-crystallized while the top Ni-P layer shows amorphous-like diffraction characteristics. This is applicable when the top Ni-P coating is reasonably thin, less than 20 microns thick, and the underlying Cu-coating will therefore exhibit strong diffracted lines because of the X-ray penetration effect.

2.3.2 Hole drilling

The residual stress in the SLA polymer substrates was assessed using a hole-drilling strain-gauge technique. The test was conducted as per ASTM E837-95[10] by using an RS-200 Milling Guide manufactured by Measurement Group Inc. A special strain gage rosette (TEA-06-062RK-120) was bonded at the point of the SLA sample where the residual stress was to be measured. A high-speed air turbine with a carbide cutter of 1.6 mm diameter was used to drill a hole at the centre of the rosette, while the relaxed strains around the hole were measured with the rosette and recorded by a data acquisition system. The residual stress was then deduced through ReStress software supplied with the system. It should be pointed out that the measured stress reflects the local average value across a depth of 0.4 to 0.6 mm, depending on the diameter of holes drilled.

3. Results and Discussion

3.1 Experimental observation on coating of SL5195 polymer

Severe cracking was observed in the coating when electroless deposition was applied directly on the SL5195 polymeric surface with a thin layer, about 1 μm, of electroless Cu coating, shown in Figures 1 & 2. As is readily apparent, the degree of cracking increases with increasing Ni-P coating thickness. However, the coating demonstrated no cracking when applied on the SL5195 polymer coated with a thin layer of electroless Cu followed by a thick layer of electrodeposited Cu of about 120 μm (Figures 3).

The above observations suggest the existence of a high level of residual stress in the Ni-P coating. A systematic residual stress analysis was therefore conducted to verify the hypothesis.

3.2 Residual stress analysis

Residual stresses, which are internal and therefore locked in, are contained in materials that are produced by nearly every mechanical, chemical, and thermal process, either alone or in combination[11]. Most coatings, metallics and ceramics, are in a state of internal stress. The stress can be either compressive or tensile. It is generally recognized that compressive stresses in coatings are more favorable than tensile stresses because they increase the resistance to fatigue failure. However, extremely high compressive stresses may cause either coating separation from the base metal or intra-coating spallation. Generally, if a tensile stress causes strain that exceeds the elastic limit of a brittle coating, then it will cause cracking in the coating perpendicular to the direction of the stress. Furthermore, residual stresses have significant influence on the mechanical and physical properties of the coatings, particularly electrical resistivity,

optical reflectance, fatigue, and corrosion. Therefore, understanding the residual stress in the coating is important to prevent the coating from peeling or cracking during service either by the elimination/reduction of the origins of stress (incorporation of gas atoms, grain size, microvoids, dislocation density, lattice misfit, surface tension, etc.) or by engineering design to counterbalance the detrimental effect of residual stress.

3.2.1. X-ray diffraction stress analysis for metal coatings

When a polycrystalline piece of metal is deformed elastically in such a manner that the strain is uniform over relatively large distances, the lattice plane spacings in the constituent grains change from their stress-free value to some new value corresponding to the magnitude of the applied stress. This new spacing is essentially constant from one grain to another for any particular set of planes. This uniform macro-strain causes a shift of the diffraction lines to new 2θ positions. From this shift the strain may be calculated and, knowing the strain, we can determine the stress present. X-ray diffraction can therefore be used as a method of "stress" measurement. Note, however, that stress is not measured directly by the x-ray method. It is the strain that is measured; the stress is determined indirectly by calculation.

The stress measurement results are provided in Table 1, with the XRD spectra acquired for stress analysis shown in Figure 4. The XRD results are described below.

(1) The XRD pattern of the copper coated specimen SLA1 reveals the presence of a single f.c.c. copper phase. No preferred orientation was observed.

(2) XRD pattern of specimen SLA2 also reveals the co-existence of two mixed phases: a top layer of amorphous-like nickel phase plus an underlying f.c.c.

copper phase. As was mentioned in 2.3.1, the residual stress of Ni-P was measured indirectly from the underlying Cu layer. It is interesting to note that only the Cu (220) and (331) diffraction peaks are present while the rest of the peaks disappear. This might be ascribed to a more severe X-ray absorption by Ni-P layer at a low incident angle.

The calculation of elastic constants of Cu and Ni was based on both Voigt and Reuss models [12] in conjunction with Table 15.2 in the reference of "Smithells Metals Reference Book [13].

3.2.2 Hole-drilling stress analysis for SL5195 polymer

The measurement of residual stress for the cured SL5195, with a Young's Modulus of 2,090 MPa and a Poisson's ratio of 0.38, indicates that the residual stress is almost zero, see Table 2. The residual stresses, if any, could have been developed during the curing process of liquid monomers with laser radiation. However, such residual stresses cannot significantly accumulate to a high magnitude during the curing process because the semi-cured resin still behaves like a visco-elastic body, which readily relaxes the residual stresses. During the post-cure process, the process-induced residual stresses, if any, would be further reduced if no restriction is applied on the SLA part, which is the case of this study. It should be noted that few, if any, materials with a modulus of elasticity of less than 7,000 MPa are known to have the homogeneity, isotropy, and linearity required for the hole-drilling method. Plastic and composite materials in general do not possess these necessary properties. However, in the current case, the relieved strains during the hole-drilling measurement is not significant due to its extra low

Young's modulus. This is an indirect indication of very low residual stresses developed during the SLA fabrication process.

3.2.3. The effect of residual stress on coating of SL5195 polymer

The stress analysis indicates that the SL5195 polymeric substrate contributes little, if any, to the cracking of Ni-P coating directly applied on its surface. Discussion on the effect of residual stress on the cracking is therefore focused on the coatings.

Experimental results showed that an intermediate layer of copper coating applied between the Ni-P coating and the SLA substrate prevented cracking and separation of the Ni-P coating. Without the copper layer, the Ni-P coating tends to crack easily and to separate from the SLA substrate in the form of peeling or blistering. The beneficial effects of the intermediate copper layer can be understood from the mechanical properties of the coating materials and the substrate, the bonding strength at the interface, and the residual stresses in the coating layers.

The Ni-P coating formed by electroless plating is hard and brittle. For example, the tensile strength of the electroless Ni-P alloys varies from 400 MPa for alloys with 4.5%P to over 700 MPa for alloys with 7-10% P, but the elongation of the alloys is merely around 1% [14]. The low ductility and high strength of the Ni-P alloys indicate that the materials can be loaded to a highly stressed state, up to 400-700 MPa, and little plastic deformation takes place prior to crack growth and eventually materials fracture. In contrast, copper is a soft and ductile material with a yield strength of around 70 MPa and an elongation of 45% [15]. When the stress goes beyond the yield strength in

copper, it will be relaxed through plastic deformation. This determines that the maximum residual stress will be limited to approximately 70 MPa when under tension, because the work hardening can be neglected when the total amount of plastic deformation is small. The residual stress level can go higher when the material is confined and loaded compressively.

The stress analysis by X-ray diffraction revealed that the residual stress in the Cu layer is compressive in nature, and approximately 300 MPa in magnitude. The residual stress in copper prior to Ni-P deposition, on the other hand, is only 35 MPa. Because the coating layer is relatively thin and free from external constraints, the stress state in the coating layer can be treated as a plain stress condition. In other words, the residual stress is parallel to the surface of the sample and the stress is negligible in the direction normal to the sample surface. A compressive stress is desirable for maintaining the integrity/adhesion of the coatings, but a too high compressive stress can cause blistering. The force equilibrium requires that in any direction on a cross section through the sample, the resultant compressive force have to be balanced by a tensile force of equal magnitude. The existence of forces of opposite signs at different parallel planes will result in shearing and bending effects inside the material. Such a shear stress can cause interface separation if it surpasses the bonding strength of the interface. On the other hand, the difference in Young's modulus and/or thermal expansion coefficient under varying temperature conditions of different coating layers will result in bending moments. Detailed theoretical analysis of stress states in layered composite media is beyond the scope of this work. Interested readers can refer to reference [16, 17]. Given the brittle nature of the Ni-P coating, little flexural deformation

can be tolerated. Cracking of the Ni-P layer is expected if the bending moment in the materials is sufficiently high.

The intermediate copper layer reduces the cracking and separation tendency of the Ni-P coating possibly through the following mechanisms.

First, the good metallurgical compatibility of copper with Ni-P and the low initial residual stress state in copper allow the high elastic energy (exemplified as residual stress) stored in Ni-P alloy to be relaxed through the deformation of copper. The bonding between nickel and copper is metallic in nature. The strong metallic bonding between copper and nickel enables the internal stress in Ni-P to be transferred across the Ni/Cu interface through shearing without breaking the interface. With a yield strength of as low as 70 MPa, copper can deform both elastically and plastically under high stress. In both cases, the high elastic energy stored in nickel is transferred to the copper layer, partially relaxed, and thus reducing the cracking tendency of Ni-P. It is of note that since copper is under compressive load and the deformation of copper is confined by the surrounding Ni-P coating, plastic deformation of copper could be limited. As such, the energy transferred from Ni-P will be mainly stored in an elastic form in copper, which is exemplified as an increased residual stress. In fact, the significant increase of residual stress from 35 MPa in copper without Ni-P coating to about 300 MPa in the copper beneath the Ni-P coating, sample SLA2, clearly demonstrates that copper has, indeed, been elastically compressed as a result of the electroless Ni-P coating. The increase in compressive stress in copper is evidently transferred from Ni-P coating and is in support of the stress redistribution mechanism. Apparently, for effective relaxation of the residual stress in the Ni-P coating, the low initial stress state in copper is critical.

In addition, a sufficient thickness of copper is necessary, about 50 μm from our experimental results, to prevent nickel from cracking according to our experimental results. Based on the stress redistribution mechanism, it can be inferred that the stress in Ni-P coating without a copper intermediate layer would be much greater than the measured numbers.

Secondly, the bonding between the metal coating and the SLA substrate is mechanical in nature and is expected to be relatively weak. When Ni-P is deposited directly onto SLA substrate, the high residual stress in the coating will translate into large shear stress at the Ni-P/SLA interface. This shear stress can cause separation of the coating from the SLA substrate if it is sufficiently high, particularly at locations where poor surface conditions, defects or stress concentration exists. The copper layer deposited between the Ni-P coating and SLA substrate has a much lower residual stress than the Ni-P coating, even after the stress or energy transfer. As a result, the shear stress at the metal/SLA interface will be reduced accordingly, thus preventing separation of coating at the interface.

Thirdly, the bending moment associated with shear stresses existing in the materials tends to produce flexural deformation in the materials. Due to the brittle nature of the Ni-P coating, bending of the specimen can lead to premature fracture of the coating at little flexural deformation. The ease of flexural deformation of a material depends on the geometry and the elastic modulus of the material. The SLA material has a Young's modulus of 2.1 GPa [18]. Such a substrate is very flexible and can be bent at a very low load. In comparison, the Young's modulus of copper is 115 GPa, approximately 55 times of that of SLA. A thin layer of copper coating on to the surface of SLA will

significantly increase the stiffness of the polymer specimen and therefore the resistance to flexural deformation under the bending moment generated by the internal stress. This increased stiffness by copper coating will minimize or completely eliminate cracking caused by bending deformation.

In summary, an intermediate layer of copper coating between Ni-P coating and the SLA substrate plays a critical role in reducing the cracking and separation tendency of the coating. Stress re-distribution, reduced interface stress, and stiffening of the substrate are believed to be the main mechanisms for the favorable effects resulted from the application of an intermediate copper coating.

4. Conclusions

- Direct electroless deposition of Ni-P on SL5195 polymer coated with a thin electroless Cu layer results in cracking and/or peeling of the coating.
- The cracking and peeling of direct Ni-P coating on SL5195 with only a thin electroless Cu deposit was found to be due to the high level of residual stress in the Ni-P coating.
- Electroless Ni-P deposition was found to proceed satisfactorily without cracking on Cu coated SL5195 polymer, with a minimum Cu coating thickness of about 50 μm.
- The intermediate Cu deposit provides stress relaxation possibly through a combination of the following mechanisms: energy transfer through elastic and plastic deformation of a lower stressed material (Cu); reduction of interfacial

shear stress between hard metal and soft plastics by using a lower stressed material as a buffer; and strengthening of the soft substrate to reduce its flexural deformation when a hard coating is applied.

Acknowledgement: The authors are grateful to Dr. Jianyin Chen for the XRD stress analysis and the valuable discussion. Thanks are also given to Mr. Mike Meinert for the SEM work. Valuable suggestions from Mr. M. Islam are also acknowledged.

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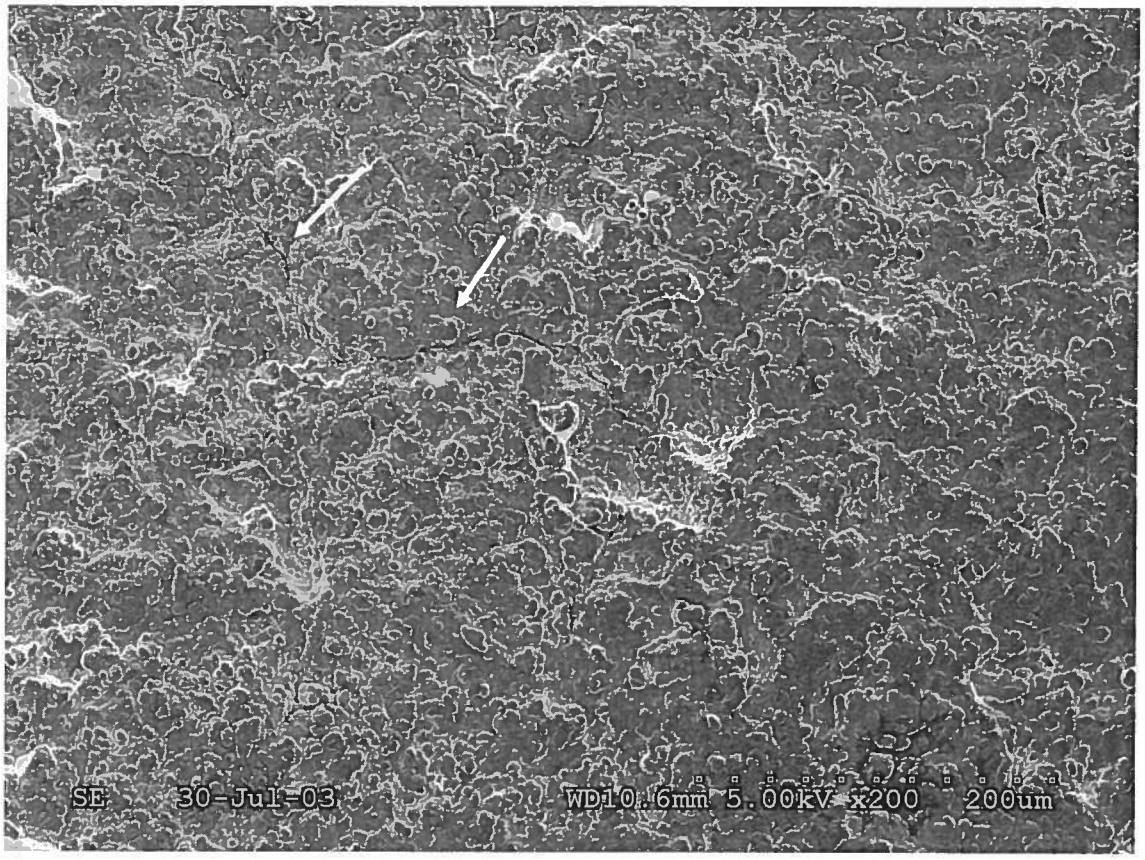

Figure 1. 4 μm of Electroless Ni-P deposited directly on electroless Cu showing the initiation of cracking

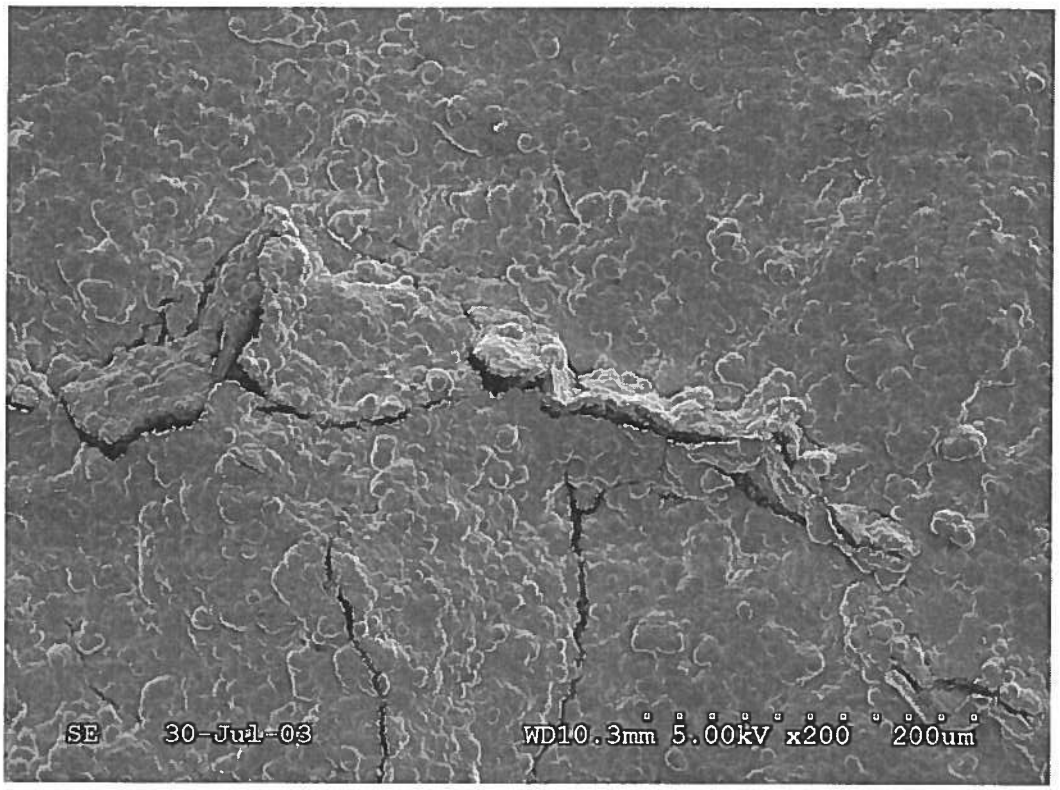

Figure 2. 8 μm of electroless Ni-P deposited directly on electroless Cu showing developed cracking

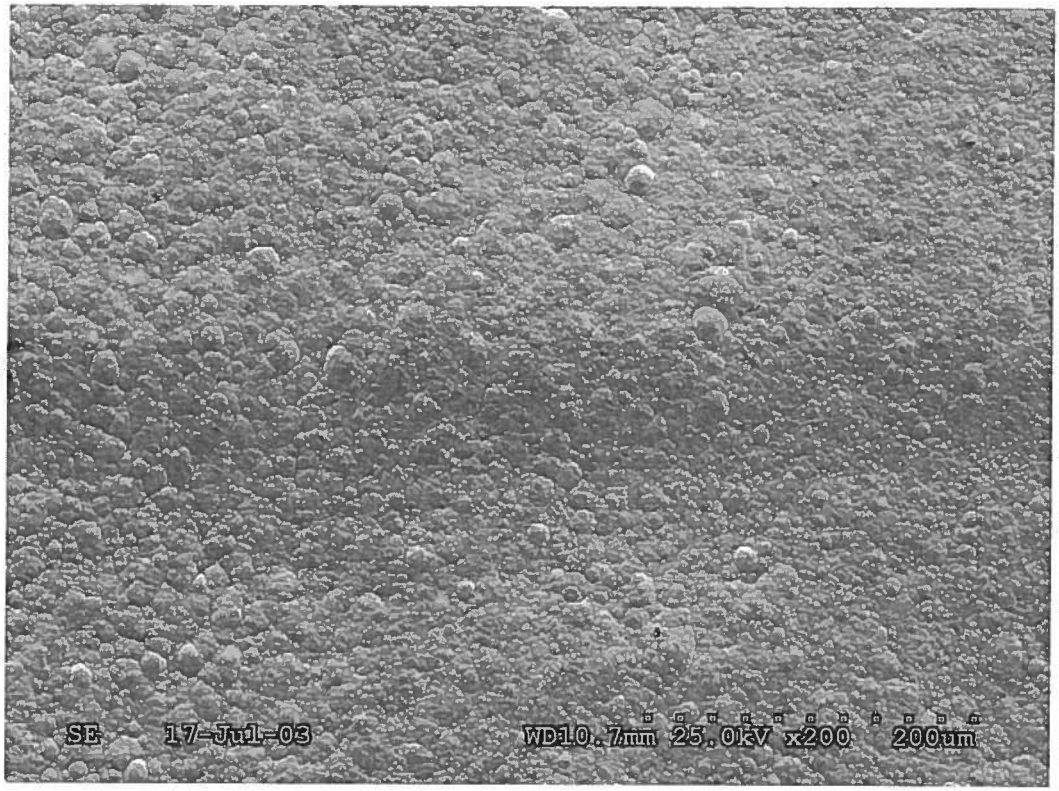

Figure 3. 20 μm Ni-P coating on 120 μm Cu, showing no cracking.

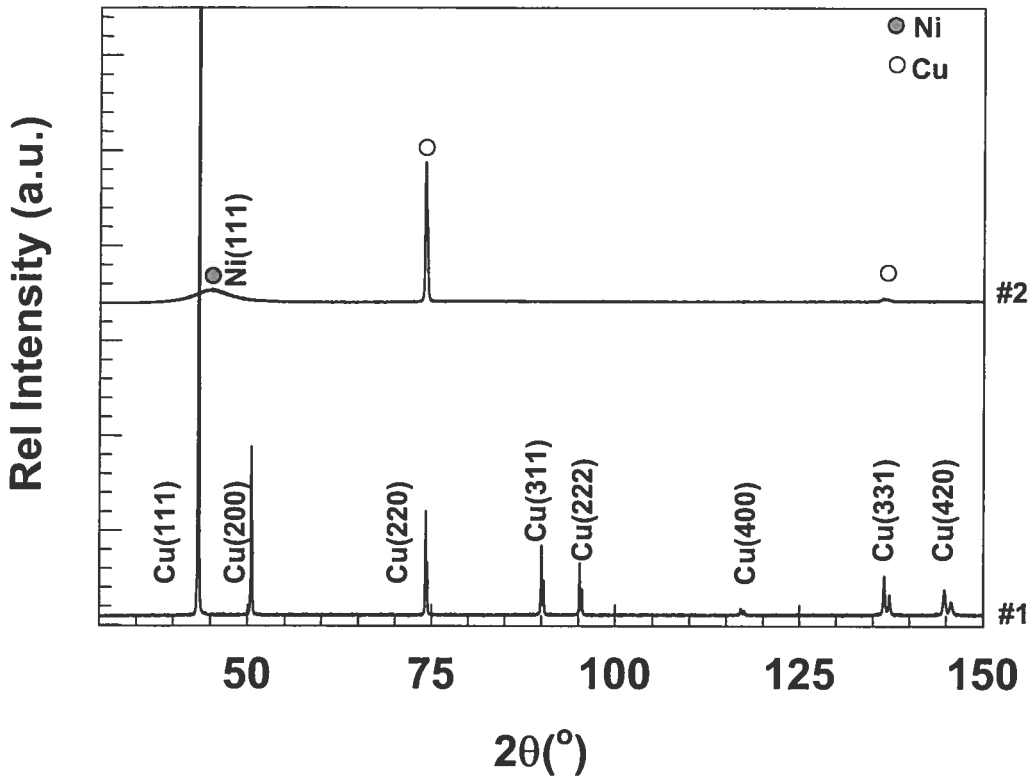

Figure 4. XRD spectra. #1: Cu coated SL5195 polymer; #2: Ni-P coating on Cu coated SL5195 polymer

Table 1. Results of residual stress analysis (All the coatings are on SL5195 substrate pre-coated with 1μm electroless Cu)

Sample	**Cu coating (under coat)**	**Ni-P coating**	**Stress (MPa)**
SLA1	137 μm	-	-35
SLA2	130 μm	20 μm	-298

Table 2. Hole-drilling stress analysis for SL5195 polymer.

Depth	Equiv. Uniform Stress (MPa)	
Z(mm)	Min.	Max.
0.1	-1	0
0.2	-1	1
0.3	1	3
0.4	-3	-1
0.5	-2	0
0.6	-1	0