

An Experimental Study on Electroless Nickel Plating on Alumina Ceramic

R. K. Gupta^{1*}, Anjali Nihore², P. Ram Sankar¹, P. Ganesh¹, R. Kaul¹

¹Raja Ramanna Centre for Advanced Technology, Indore- 452013, MP, India

²Project Student SGSITS Indore, MP, India

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Abstract In this paper a low temperature metallization i.e. standardization of electroless Ni plating on alumina ceramic is summarized. It includes three main steps (i) pre-treatment, (ii) activation and (iii) Ni plating. Pre-treatment includes degreasing with non-silicate soap solution followed by ultrasonic cleaning in acetone and etching the alumina surface to generate roughness for making it suitable for adhesion. Etching has been obtained by immersion in 10% NaOH at 50°C for 10 minutes and 50% HF for 15 minutes at room temperature. Activation of the surface is the most important step before electroless plating on alumina ceramic. Activation has been achieved with Ginplate 442 (commercial chemical) by immersing the alumina specimen for 5 minutes at room temperature. Activated alumina samples have been used for electroless Ni plating in Ginplate 418 bath at 88°C. Uniform, densely compact coating has been achieved as revealed by SEM image. XRD plot confirms the presence of metallic nickel together with phases of Ni₂P, NiP, Ni₃P confirming the nickel phosphite based plating. Subsequently, tube to tube joining of these metallized alumina ceramic tubes has been conducted through vacuum brazing process with copper silver eutectic alloy (28Cu-72Ag) whose cross section showed good joint with excellent wettability of Cu-Ag with Ni plating.

Keywords Low Temperature Metallization, Electroless Nickel Plating, Pre-treatment, Activation

1. Introduction

Electroless nickel is a coating used for engineering purposes, normally used because of excellent corrosion and wear resistance. Electroless metal based plating is generally used to deposit the metal without applying potential or electric current. Hot acid hypophosphite based baths are mostly used to plate steel and other metals, whereas warm alkaline hypophosphite based baths are used for plating plastic material and non-metallic ceramics. Electroless nickel deposition is also used on aluminum substrates to provide a solderable surface and is used with molds and dies to improve lubricating properties. Due to these properties, electroless nickel plating got many important applications, including those in petrochemical, chemical application, ceramic plastics, optics purpose, printing, mining, aerospace industry, automotive industry, electronics use, computers/laptops, textiles industry, paper and food machinery parts [1]. Electroless Ni plating has many advantages like uniform plating, solder ability and braze ability with some limitations like less adherence, plating rate not so fast as compared to electroplating. Plastic material and ceramics insulator material need proper and suitable cleaning/pre-treatment for metallic deposition using electroless bath [2]. The microstructures and other properties of electroless Ni coating which is in the form of Ni-P (EN) depend on the phosphorous content alloyed in the deposit [3,4]. The mechanical and tribological properties of these coatings can be further tailored by adding hard particles [5], thermal treatment [3,6] and some dry lubricants [7,8]. High purity alumina

(Al₂O₃) material is an ultra-high vacuum (UHV) compatible ceramic which reduces eddy current losses. It shows high electrical resistance and reasonably good tensile strength & flexural strength with low out-gassing rate and high radiation resistance [9-10].

Joining of ceramics is often an essential part of the fabrication process for ceramic components. Vacuum brazing is the universally accepted joining process for ceramics [11,12]. The requirement of obtaining acceptable ceramic brazed joints is met through either by metallizing mating ceramic surfaces [13-16] or by using active brazes filler. Mo-Mn metallization is the conventional method which is often used to obtain wettable ceramic surface. The multi-step metallization process comprises of (i) coating of surface of the ceramic with metallization paint, (ii) drying and firing at 1723–1873 K in wet hydrogen atmosphere, (iii) nickel plating, followed by (iv) sinter firing at 1123–1173 K [17]. Major problems associated with Mo-Mn metallization are: (i) long and tedious process which requiring special skills and (ii) absence of qualified vendors. On the other hand, brazing with active braze filler is a simplified single step process [18-20] but involves formation brittle intermetallic compounds [21-23]. During brazing with active braze filler, the active metal (Ti or Zr) reacts with ceramic to produce an intermetallic reaction product which is wettable by the braze filler. The braze filler alloys, due to their proprietary, are quite expensive. This work standardizes a low temperature Ni metallization process for alumina ceramics and their subsequent joining through vacuum brazing with copper silver eutectic alloy (Cu-Ag). Generally plating on ceramics is a high temperature process. By using the commercial chemicals of Ginplate and required pre-treatment, a procedure is optimized for electroless Ni plating on alumina ceramics at comparatively low temperature. Electroless nickel plating is a simple, easy and better option than electroplating since we get uniform and thick nickel plating without applying any external current. This process may prove to be of great use in industrial applications.

In this respect, present experimental study was taken up with an objective to evaluate electro-less nickel plating as an alternative route for ceramic metallization which is adaptable on the shop floor.

2. Experimental Details

Alumina (Al₂O₃) tubes with dimensions (Height=20mm; Diameter=20mm) were used as a substrate for electroless Ni plating for low temperature metallization as shown in the Fig.1.

2.1. Pre-treatment

Pre-treatment involves, degreasing with soap cleaning in non-silicate bath and ultrasonic cleaning in acetone for

10 minutes. Ultrasonic cleaned specimens were immersed in 10% NaOH solution at temperature 50°C for 10 minutes followed by rinsing with distilled water. These specimens were etched in 50% HF for 15 minutes followed by rinsing with distilled water.

2.2. Activation

Commercial chemicals Ginplate 442 [24] solution was used as an activator for activating the surface. It is an acid activator used to prepare the surface before plating for promoting uniform and adherent formation of electroless coating. Activator solution was prepared by mixing 12.5% Ginplate 442 and 12.5% conc. HCl in distilled water. Pre-treated Alumina specimens were immersed in above activating solution for 5 minutes at room temperature (RT).

2.3. Electroless Ni Plating

Electroless Ni plating bath was prepared with commercial Ginplate 418 [25] chemicals which consist of Ginplate 418A (a solution containing Ni ion) and Ginplate 418B (a solution containing reducing agent) in the ratio of 6:9 in distilled water. pH of the bath was monitored between 4.6-4.9 with the addition of dilute ammonia solution. 100 ml of the above solution was reduced to 80 ml by heating. Temperature of the bath was maintained at 80-90°C. Pre-treated and activated alumina specimens were immersed in above solution for 40 minutes for Ni deposition.



Figure 1. Alumina substrate (Al₂O₃) used for electroless Ni plating

2.4. Joining of Electroless Ni Plated Alumina Tubes Through Brazing

These Ni plated alumina tubes were subsequently

joined through brazing (a joining process) using copper-silver (72Ag/28Cu) filler alloy. The brazing cycle followed during brazing in the furnace is summarized as follows.

Brazing Cycle used for joining

- Heating from room temperature to 250 °C @3° /min;
- Soaking at 250 °C for 10 min;
- Heating from 250 °C to 550 °C @ 3° /min;
- Soaking at 550 °C for 15 min;
- Heating from 550 °C to 760 °C @ 3°/min;
- Soaking at 760 °C for 20 min,
- Heating from 760 °C to 800 °C @ 5°/min;
- Soaking at 800 °C for 1 min, followed by
- Furnace cooling.

2.5. X-ray Diffraction and Microstructure Analysis

X-ray diffraction (XRD) of Ni plated alumina specimen were performed to confirm the presence of Ni plating. SEM image was recorded to observe the plating uniformity and surface topology. Microstructure of cross section of braze joint was analyzed through OLYMPUS make optical microscope.

3. Results and Discussion

In the pre-treatment step, alumina tubular specimens were first cleaned with non-silicate soap solution having good detergency to remove the oil grease and fingerprints followed by ultrasonic cleaning with acetone to remove all the contaminating particles. Etching of the specimen by 10% NaOH and 50%HF generated rough surface and making it suitable for adhesion of the ions and developing active sites. These etched specimens were further exposed to Ginplate 442 activator where stannous and palladium salts may form a layer so that metallisation process is initiated to promote the formation of the electroless deposits with good conductivity (Fig 2).

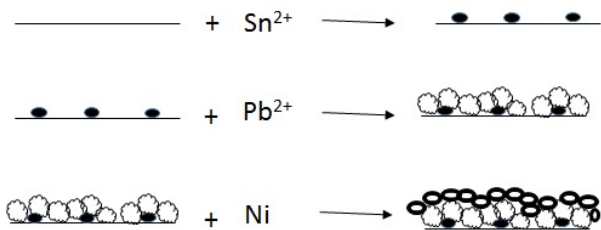


Figure 2. Schematic showing activation and Ni plating

After activation these alumina specimens were immersed into Ginplate 418 electroless Ni bath for 40 minutes at 88°C. Uniform plating was obtained. Fig 3 and Fig 4 show alumina tubes and buttons plated with above procedure and good repeatability was observed.

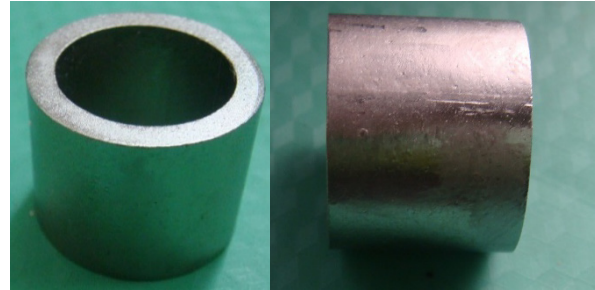


Figure 3. Electroless Ni plated Alumina tube specimen (Left) Vertical View; (Right) Horizontal view



Figure 4. Electroless Nickel plating on tubes and buttons

These electroless Ni plated alumina ceramic tubes were joined through brazing in vacuum furnace. Filler used was Cu-Ag eutectic alloy (CuSil) foil because it has got good wettability with Ni. Thus bond formation can take place to form the joint. Fig.5 shows tube to tube and tube to button brazed joint and excess filler flown around the joints can also be seen.



Figure 5. Brazed Joint of a Electroless Nickel Plated Tube to tube (left) and Tube to Button (right)

X-RAY diffraction analysis of these plated alumina ceramics clearly indicated the presence of metallic Ni peaks together with intermetallic phases of Ni_2P , NiP , Ni_3P confirming the nickel phosphite based plating. This phosphorous based plating was observed because of presence of phosphorous content in Ginplate 418 bath. Mostly Ni is plated as NiP with electroless means which is supported by XRD in the present case. Fig 6. shows the XRD plot of deposited Ni plating on alumina ceramic. Visual examination showed the shiny metallic coating on the alumina ceramics. The surface was conductive as examined by multimeter. Micro-structural SEM examination of electroless Ni deposition showed uniform deposition on the surface of alumina ceramics. Deposition is dumble shaped granular coating which covers whole

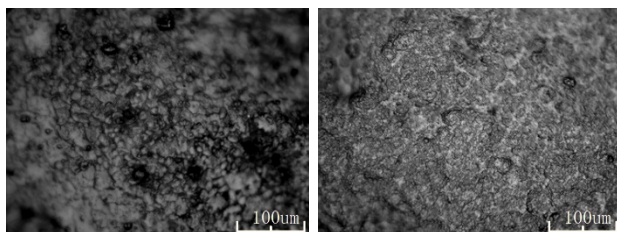


Figure 10. Optical microstructures of electroless Ni plating on alumina ceramic (left) before brazing and (right) after exposing brazing cycle

4. Conclusions

In this paper, an experimental study for low temperature metallization i.e. for electroless nickel plating on alumina ceramic is described. Pros and cons of the present study and its subsequent outcomes are presented. Plating process includes three main steps (i) pre-treatment, (ii) activation and (iii) Ni plating.

- (1) Pre-treatment includes degreasing with soap solution followed by ultrasonic cleaning in acetone and etching the alumina surface to generate roughness for making it suitable for adhesion. Etching has been carried out by immersion in 10% NaOH at 50°C for 10 minutes and 50% HF for 15 minutes at room temperature.
- (2) Activation has been achieved with Ginplate 442 (commercial chemical) by immersing the alumina specimen for 5 minutes at room temperature.
- (3) Electroless Ni plating was done in Ginplate 418 bath at 88°C.
- (4) Uniform, densely compact coating has been observed by SEM.
- (5) XRD plot confirm the presence of metallic nickel together with phases of Ni₂P, NiP, Ni₃P confirming the nickel phosphite based plating.
- (6) Joining of metallized alumina tubes, through brazing results good metallised alumina joint with excellent wettability of Cu-Ag with Ni plating. However, coating delamination was observed from the alumina ceramic surface due to blister formation after exposing to the brazing cycle.

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