Adhesion Quality Testing of Gold Plating on Aluminum Alloy

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Abstract: Gold plating is carried out on various microwave modules mainly to impart engineering properties. It is very important to study adhesion of gold plating to its substrate for smooth functioning during entire lifetime of product. In this work various adhesion tests such as heat-quench, burnishing, bending and plate breaking and heat resistance test are carried out. The gold plating on aluminum has given evidence of satisfactory adhesion during qualitative tests. The specific surface of gold obtained using this process is stable in large temperature gradients without deterioration in its physical properties.

1. Introduction

Adhesion is defined by ASTM as: "condition in which two surfaces are held together by either valence forces or by mechanical anchoring or by both together". Adhesion refers to chemical or physical bond amongst two materials. It depends on three factors: fracture mechanism, interfacial region bonding and type of interfacial region. Adhesion failure may be due to mechanism by which material is being fractured. In thin films chemical bonding adhesion is high but its intrinsic stress may cause adhesion failure. Plating should be strongly adhered to its base metal for its use in specific functional areas. Plating adhesion depends on many factors such as its thickness, purity, atomic bonding structure and internal stresses [1]. In this experiment, we have focused on controlled plating thickness only. Here gold is plated on aluminum alloy 6061 T6 which is having naturally occurring thin oxide films on it. It reforms immediately upon exposing to air. So, pickling is also not effective to remove this oxide layer, it is being regenerated before part is dipped in plating bath. So, specific pre-treatment is designed to prepare this alloy for plating. After cleaning first step of zincating on aluminum is a widely used process step that activates the parts for following electroless nickel deposition. This zinc layer provides better adhesion between aluminum base and electroless nickel deposit. Zincating occurs by electrochemical exchange reaction between aluminum metal and zinc complexes in solution. It deposits zinc and aluminum is getting dissolved in solution, etching occurs on part. Several methods are available in literature to improve the adhesion between the electroless nickel film and the aluminum part by changing of zincating bath chemistry and using a multiple zincate process [2]–[6]. These methods suggested that the zincating step control is important to increase the adhesion. Electroless nickel undercoat is given for better protection of base metal. Top coat nano gold plating is done by using pulse reverse current method. Pulse plating has emerged as a new technique for deposition of metals and alloys. In the conventional direct current electrodeposition, there is only one parameter, namely the current density, which can be varied. But in pulse methods we have three variables that are of primary importance. They are peak current density, current 'on' time and current 'off' time. The sum of 'on' and 'off' times constitute one pulse cycle. Duty cycles may vary from 1 to 100% and on-off times from microseconds to milliseconds. So, in pulse plating we have an unlimited number of combinations of different pulse current densities for obtaining a given average current density. Simple square wave pulses are generally used for plating operations, though a variety of combinations of square wave pulses are also available [7]. Pulse deposition techniques are used mostly to improve the distribution of the deposit (current distribution), the levelling and the brightness of the deposition [8].

In pulse electrodeposition (PED) the potential or current is alternated swiftly between two different values. This results in a series of pulses of equal amplitude, duration and polarity, separated by zero current. Each pulse consists of an ON-time (T_{ON}) during which potential and/current is applied and an OFF-time (T_{OFF}) during which zero current is applied. It is possible to control the deposited film composition and thickness in an atomic order by regulating the pulse amplitude and width. They favour the initiation of grain nuclei and greatly increase the number of grains per unit area resulting in finer grained deposit with better properties than conventionally plated coatings. Process and circuitry for generating current pulses for electrolytic metal deposition are dealt in literature [9].

After plating, baking is done at 150°C for one hour to improve adhesion and improving bond strengths. Adhesion tests can be classified in two groups: qualitative and quantitative. Most simple test is scotch tape test whereas some complicated test is flyer plate which requires specific testing facility. In this experiment we have checked adhesion qualitatively with in-house available resources. Surface preparation of aluminum alloys for electroplating is discussed in literature [10]. Procedure for deposition of gold on Al alloy specimen by high speed cyanide based electroplating bath is discussed in literature [11]. Electrodeposited nano gold thin films were studied using a confocal laser scanning microscope [12]. Adhesion test such as burnishing test, adhesive tape test, thermal shock test and bend test are described in IS 3266-1982. This standard provides requirements for electrodeposited gold coatings on brass, copper, zinc as well as its alloys, nickel, steel, silver, aluminium and its alloys [13]. C. N. Panagopoulos et al studied the adhesion of nano-crystalline Ni–W alloy coatings on copper, with the help of a scratch testing apparatus and found that the coatings suffer from both tensile failure and minor delamination [14]. Based upon the Pourbaix diagrams, M. A. M. Bakker has recognized conditions to be fulfilled to confirm good adhesion of an electrodeposited gold to its substrate [15]. There is no literature available about adhesion testing for pulse plated gold plating. Our current investigation is focused on adhesion quality testing of pulse plated gold plating on aluminum alloy 6061 T6.

2. Experimental

Nano gold plating was applied on 13 aluminum samples (size 25mm X 100mm X 1mm). Standard guideline as per ASTM B 253-87 was followed for pre-treatment of aluminium alloy 6061 T6 [16]. Pulsed reverse current (PRC) plating was used during final gold plating step for producing nano gold deposits. The coatings were produced in an electrolyte based on $K[Au(CN)_2]$ and citric acid/ sodium citrate buffer. Different types of current such as direct current (DC), pulse current (PC) and reverse pulse current (PRC) was introduced for gold deposition. DC and PC plating was done for 30 minutes time at average current density of 2 ampere per square feet to get gold coating thickness of 2 ± 0.5 μm. For PRC plating thickness was 1 ± 0.2 μm. Adhesion is having direct relation with plating thickness. So, plating thickness measurement was carried out using two methods. Adhesion testing for DC, PC and PRC plated samples was done.

Plating Thickness Measurement

The thickness of the plating was determined according to ASTM B568 using X-ray fluorescence spectroscope equipment [17]. This is a non-destructive test. Thickness was calculated as the average of six readings taken at different locations. The plating thickness was measured by couloscope on the selective specimens. It is destructive method. Instrument series using the coulometric method according to EN ISO 2177[18]. Coulometric method is one of the simplest methods for plating thickness measurement but it is destructive method.

3. Adhesion Testing

The plating should be inspected before and after the tests to ensure that there is no alteration in the physical appearance. Adhesion testing was carried out as per ASTM B571 [19]. Specimens were observed under a microscope at 10X magnification before testing to rule out any defects after plating. Various qualitative adhesion tests were performed as described below. After tests the region was examined at low magnification of 10X.

3.1. Heat-quench test

The sample was heated in the oven for 2 hour after reaching a temperature of 220 °C. Then the sample was submerged in room temperature water. Layer flaking or peeling if any, is evidence of unsatisfactory adhesion. During heating there is a chance of blisters and water may penetrate surface pits or holes that were bound by deposits when the sample was immersed in it. The adhesive layer can be separated from the parent metal because of deterioration due to expansion and subsequent shrinkage of metals by reducing heat with room temperature water. If the plating cannot be peeled or lifted off the substrate adhesion is good. The photographs of the specimen placed in oven and immersed in water is shown in Fig. 1.



Fig. 1 Heat-quench test

3.2. Burnishing test

A coated area of about 5 cm was rubbed with a steel rod. The end of rod was hemispherical in diameter 6 mm. So much pressure was given that each stroke was enough to burn the plating but not so much that it will etch into it. The plating should not blister or peel off. The burnishing test of the sample was carried out as per ASTM B571 [19] as shown in Fig. 2.

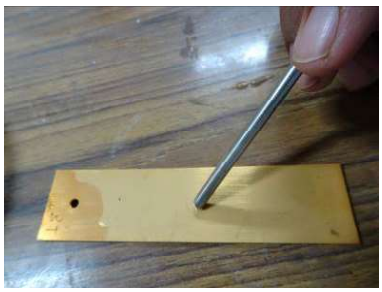


Fig. 2 Burnishing test

3.3. Bending and plate breaking Test

According to ASTM E290-09 [20] the ductility of the coated material and the base material was examined. The adhesion of the multi-layer build up was tested. Mandrel diameter of sample should be four times the thickness of specimen. One specimen of 1 mm thickness having coated surface was held tightly on mandrel in bend test jig at one end, and the other end was free. The part was folded away from one end until its two legs are parallel. Whereas the specimen was bent and one end was held, the middle part took the bend loop. The bend and tensile stress were applied on the inner side of the specimen and the outer curvature was subject to compression stress. It was indication of poor adhesion if plating fractures or blisters appear. An attempt was made to lift the layer by using a sharp blade. Cracking usually occurs in the bend area with hard or brittle plating. The part was repeatedly bent back and forth at an angle of 180° until the base metal fails, any lack of adhesion between the layers may result in non-adherence and the peeled off layers. The sample was turned on an 8 mm mandrel. Metal breakdown or the failure zone if any, was examined for chipping of the plating. The bend test of the sample is shown in Fig. 3.



Fig. 3 Bend test

3.4. Heat resistance test

Evaluation of plating performance at elevated operating temperatures was done using heat resistance test. The plating was tested at 190°C for 2 hours. This weak, non-adhesive plating may have spots or blisters when subjected to testing. The heat resistance test of the sample is carried out in our lab as shown in fig. 4.



Fig. 4 Samples after heat resistance test

3.5. Microstucture of the coating

The gold plated aluminium samples were mounted on a sample holder and SEM images were taken at a magnification of 75000X to analyse the microstructure.

4. Results and discussion

Visual observation of all specimens was done at 10X magnification for plating quality. The plating was bright yellow, uniform, and flawless in appearance. X-ray fluorescence spectroscopy and couloscopic measurements have shown that for the DC and PC, gold average thickness was 2±0.5 µm, for PRC gold average thickness was 1±0.2 µm and the average thickness of the electroless nickel plating was 10±2 µm. Adhesion testing for DC, PC and PRC plated samples passed which include heat-quench test, burnishing test, bend and plate breaking test. Blister or scratched layer was not observed after heat resistance test. Scanning electron microscope images were taken before and after heat-quench test as given in Figure 5. It is clearly visible in image that some grainy texture was observed on plated specimen after test, but it didn't cause delamination of gold layer from base metal.

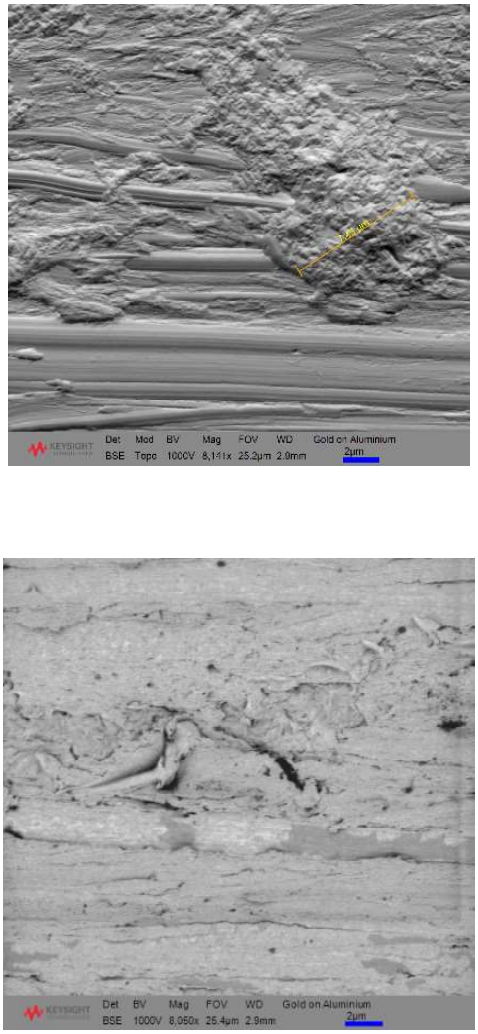


Fig. 5 SEM images of specimen before and after heat-quench test

SEM image (Fig. 6) was taken before and after burnishing test. It didn't show any degradation except scratch marks.

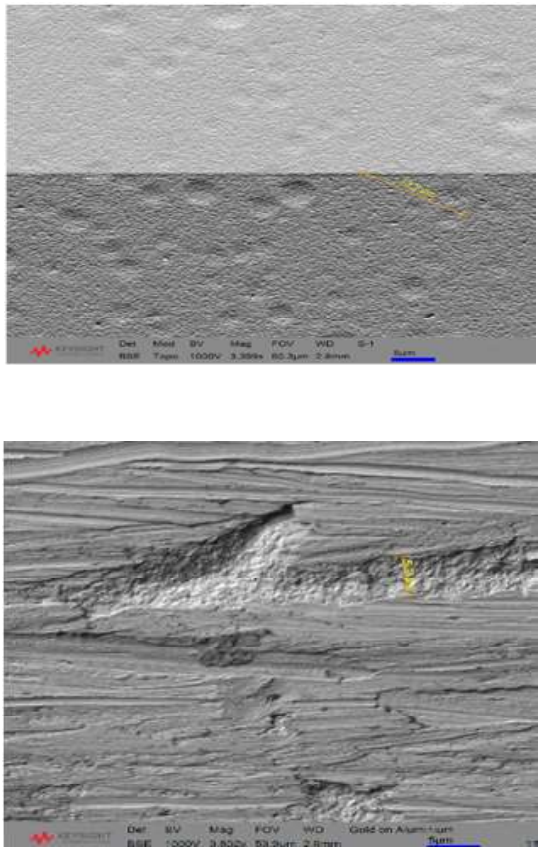
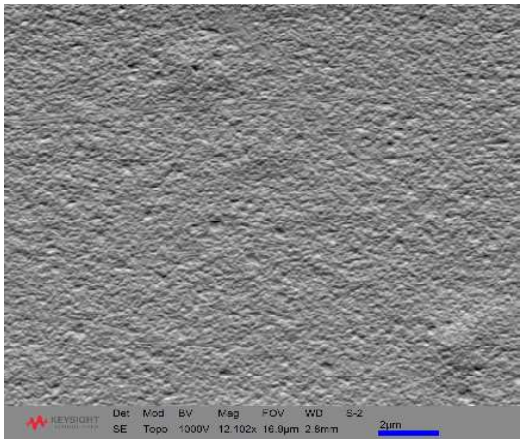


Fig. 6 SEM image of specimen before and after burnishing test

SEM image (Fig. 7) was taken before and after bending and plate breaking test. It didn't show any degradation except black spots. However, it didn't expose base metal.



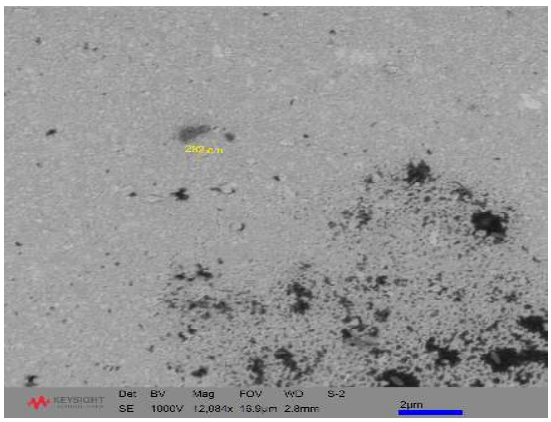


Fig. 7 SEM image of specimen before and after bending and plate breaking test

SEM image (Fig. 8) was taken before and after heat resistance test. It was observed that clear grain boundary was observed before test whereas grains were fused together after test and clear grain boundary was not visible. Smooth surface was observed.

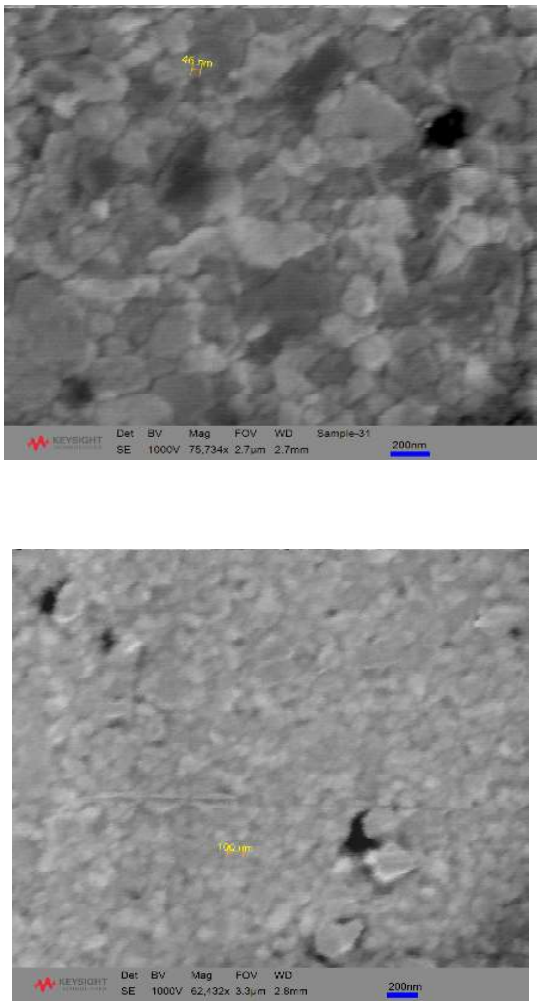


Fig. 8 Samples after heat resistance test

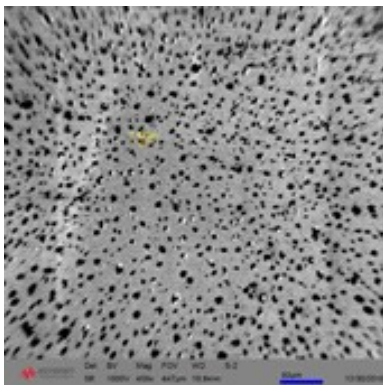
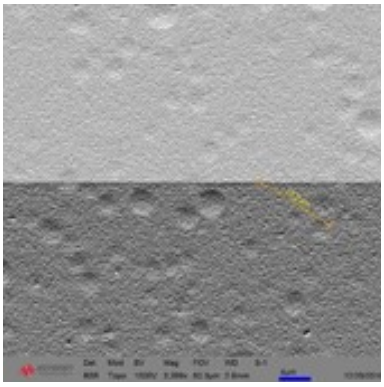
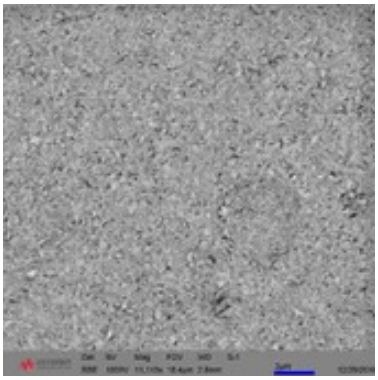
At 50 µm scale	At 5 µm scale	At 2 µm scale
		
Particle size: 1200 - 1600 nm		

Fig. 9 SEM images of sample no. 1 gold plated using DC

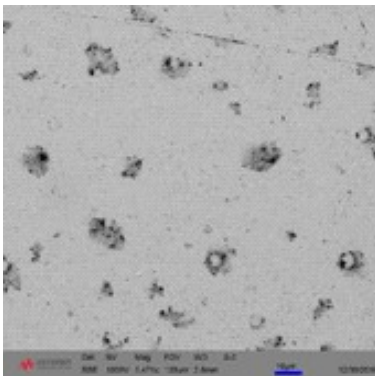
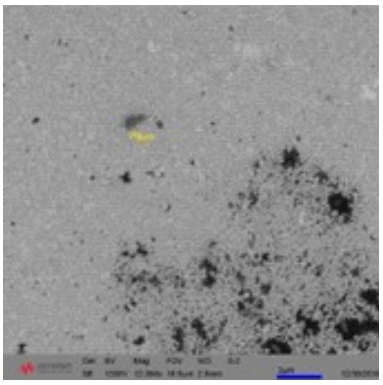
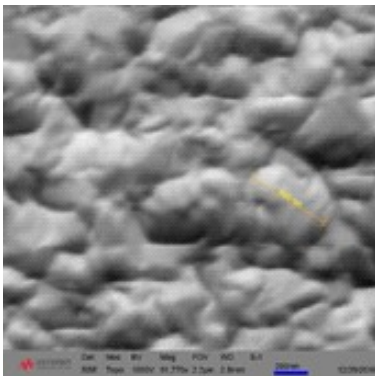
At 10 µm scale	At 2 µm scale	At 200 nm scale
		
Particle size: 200 - 500 nm		

Fig. 10 SEM images of sample no. 2 gold plated using PC

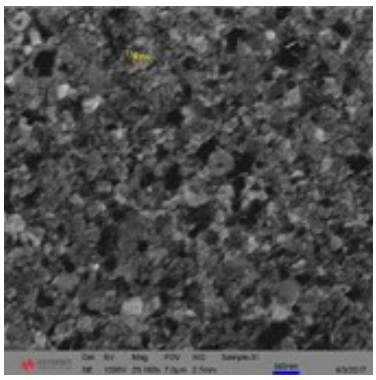
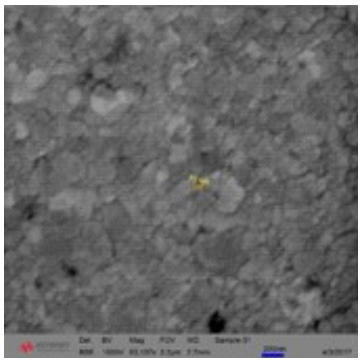
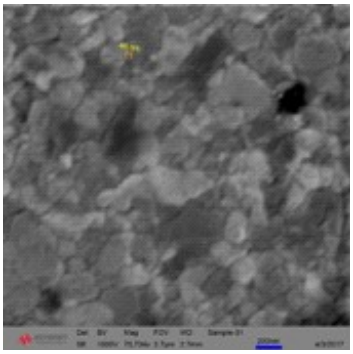
At 500 nm scale	At 200 nm scale	At 200 nm scale
		
Particle size: 40 - 60 nm		

Fig. 11 SEM images of sample no. 31 gold plated using PRC

For functional requirements adhesion is tested for satisfactory life cycle operation in extreme environments and without degradation. Process and repeatability of the performance has been confirmed.

The surface morphology and particle size of the DC, PC and PRC gold plated deposits formed on aluminium were studied by SEM analysis (Fig. 9, 10, 11).

5. Conclusion

The plating of nano gold on aluminum has indicated very good adhesion. The plating was stable in large temperature gradients of 25°C to 220°C (based on heat quench test) without deterioration in its physical properties. In burnishing test, the pressure was sufficient to burnish the coating at each stroke but not so great as to dig into it, only small scratches were observed at high magnification. Blisters, lifting, or peeling was not developed. During bending and plate breaking test specimen observed tensile and compression stresses but plated layer was enough ductile to withstand those stresses. During heat resistance test grains were merged together which formed uniform surface. Thus nano gold plating passed all four adhesion tests successfully. SEM microscope images revealed that PRC plated sample grains in the range of 40-60 nm, PC plated sample is having smaller grain size (in the range of 400 nm), as compared to DC plated sample (in the range of 1200 nm). Other adhesion tests such as draw test and Scribe-grid test can be done in future for further evaluation of plating.

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