



Mechanical properties and microstructure of Au–Ni–TiO₂ nano-composite coatings

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ABSTRACT

Two different kinds of Au–Ni–TiO₂ nano-composite coatings were electrodeposited by powder enhanced method and sol-enhanced technology. A comparative study on the mechanical properties and microstructure of Au–Ni–TiO₂ coatings was conducted. Due to the high surface energy of nano-particles, agglomeration of TiO₂ powders can be seen clearly in the solid particle enhanced Au–Ni–TiO₂ coating. Correspondingly, TiO₂ nano-particles with a size ranging from 3–12 nm were highly dispersed in the sol-enhanced Au–Ni–TiO₂ coating matrix, resulting in a significant improvement of nano-hardness.

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1. Introduction

Due to the beautiful/shining color, excellent corrosion resistance, good solderability and bondability, high electrical and thermal conductivity, gold (Au) and gold alloy have been widely used for various applications such as jewelry, craftwork, dentistry and electronic devices [1–5]. According to a recent survey, gold usage in electronics was achieved to 320 tons in 2011 [6]. The largest use of Au in electronics is the electroplated coatings for connectors and contacts [7–9]. Comparing with Cu and Ag which are also very good electric conductors, Au possesses better resistance to tarnishing and corrosion [10,11]. Electrodeposition are preferred techniques for Au and Au alloy coating due to the simple device, easy operation, low cost and relatively high deposition rate compared to the other methods such as E-beam evaporation, magnetron sputtering, and chemical vapor deposition (CVD) [4,12–14].

Hard Au, which Ni or Co were co-deposited as alloy elements with Au, is used as the contact material and circuit boards where high electrical conductivity, good wear resistance and chemical inertness are required. To satisfy long-term use, the most challenging research is to improve the mechanical properties without sacrificing electrical conductivity [15,16]. This is hard to achieve by alloy strengthening as the

alloying elements will cause a severe lattice distortion which deteriorates electrical properties.

Nano-composite coating fabricated by electroplating and electroless plating is an important topic since these techniques can provide good hardenability and wear resistance of working parts. During the conventional nano-composite plating process, a small amount of solid nano-particles were added into the plating bath and then co-deposited into the coating matrix as the second phase. The coating mechanical property was improved mainly by dispersion strengthening mechanism. This method does not result in significant decrease of electrical conductivity for the small addition and it does not change the solubility of alloy [17–19]. However, there are still problems that need to be solved. One of the problems is the agglomeration of nano-particles as it is difficult to achieve good suspension of nano-particles in electrolyte, which largely reduces the strengthening effect of nanoparticles.

Recently, we have developed a novel method which combines the sol-gel process with electrochemical plating processes to fabricate nano-composite coatings [20–22]. By virtue of this novel method, the mechanical properties of sol-enhanced Au–Ni–TiO₂ coatings were increased by ~25% comparing to the traditional Au–Ni coatings and ~10% comparing to the solid powder enhanced Au–Ni–TiO₂ coatings, while the electrical resistivity of the sol-enhanced coatings keeps a same level as the pure Au–Ni coatings.

Considering the broad application prospects and great market value of Au coatings, it is essential to understand the microstructure and strengthening mechanism of Au–Ni–TiO₂ nano-composite coating. In the present work, we fabricate the Au–Ni–TiO₂ nano-composite

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coatings by utilizing sol technology and traditional solid powder method respectively. A comparative study on the mechanical properties and microstructure for two types of Au–Ni–TiO₂ composite coatings was carried out.

2. Experimental details

2.1. Sample pretreatment

Both solid particle enhanced Au–Ni–TiO₂ and sol-enhanced Au–Ni–TiO₂ composite coatings were electroplated onto brass plates substrates (20 × 15 × 0.6 mm³). The composition of brass is 64 wt.%Cu and 36%Zn. A bright Ni layer of ~7 μm thick was deposited onto the substrate surface to prevent the inter-diffusion between Cu substrate and Au. Before electroplating, the specimens were pretreated in alkaline soap solution (50 g/L NaOH and 10 g/L NaH₂PO₄–H₂O) at 80 °C for 30 s under 10 mA/cm² current density, then electro-activated in HF containing solution at room temperature for 20 s under 20 mA/cm². These specimens were electroplated after rinse thoroughly by distilled water.

2.2. Electrodeposition of nano-composite coatings

Both solid powder enhanced Au–Ni–TiO₂ nano-composite coatings were electroplated based on a potassium dicyanoaurate contained solution. The bath was prepared using Sigma analytical grade reagents and contained 2 g/L KAu(CN)₂, 2 g/L NiSO₄, 10 g/L C₆H₈O₇, and 13 g/L K₃C₆H₅O₇–H₂O. The pH value of bath was adjusted at 3.8.

The solid powder enhanced Au–Ni–TiO₂ nano-composite coatings were produced by using the solid particle mixing method with a concentration of TiO₂ particles of 1.25 g/L. The TiO₂ powders have a crystalline anatase structure with an average diameter of ~25 nm. Transparent TiO₂ sol was prepared as reported in our previous research [9]. The sol-enhanced Au–Ni–TiO₂ nano-composite coatings were fabricated by our newly developed method with a concentration of TiO₂ sol of 6.25 mmol/L. 1.25 g/L and 6.25 mmol/L were the optimum additive quantity of each method according to our preliminary results of mechanical properties [10]. All the electroplating process was conducted for 10 min under a constant current density of 10 mA/cm² with an agitation speed of 200 rpm.

2.3. Microstructure characterization

The crystal structure of the coatings was characterized by X-ray diffraction (XRD) with Cu Kα radiation (Bruker D2, V = 30 kV, I = 10 mA). Diffraction patterns were recorded in the 2θ range from 20 to 80° at a scanning step of 0.02°. Both surface morphology and cross-section microstructure of the Au–Ni–TiO₂ nano-composite coatings were studied by using a field emission scanning electron microscope (FESEM) with an energy dispersive spectroscope (EDS). The microstructure and

distributions of TiO₂ nanoparticles in the composite coating were studied by a FEI Tecnai F20-ST transmission electron microscope (TEM). This TEM instrument equipped with an energy dispersive X-ray (EDX) device, Gatan imaging filter (GIF) and owning scanning transmission electron microscopy (STEM) capabilities. TEM images were analyzed by using the Image-Pro Plus software.

2.4. Mechanical properties tests

The nano-indentation tests were conducted on a nanoindenter (TI950 TriboIndenter, Hyston, USA). Before tests, the surface roughness of coatings was determined to below the level of ~10 nm by using attached Scanning Probe Microscopy (SPM). As the thickness was controlled in a level of ~1.2 nm, 9 indents were performed on the original coating specimens to a peak depth of 50 nm for nano-hardness investigation with a Berkovich diamond tip.

3. Results and discussion

3.1. Mechanical properties of Au–Ni–TiO₂ nano-composite coatings

Fig. 1 shows the nano-hardness of Au–Ni and Au–Ni–TiO₂ nano-composite coatings. The nano-hardness of Au–Ni coating was 2.55 ± 0.13 GPa. After adding 1.25 g/L TiO₂ powder into the solution, the nano-hardness of powder enhanced nano-composite coating was increased to 2.91 ± 0.09 GPa. When 6.25 mmol/L TiO₂ sol was added into the electrolyte, the nano-hardness increased to 3.20 ± 0.15 GPa, ~25% improvement comparing to the traditional Au–Ni coatings and ~10% comparing to the solid powder enhanced Au–Ni–TiO₂ coatings. The enhancement of hardness can be attributed to the incorporation of TiO₂ nano-particles and microstructure change which we will further discuss in the following parts.

3.2. Microstructure of Au–Ni–TiO₂ nano-composite coatings

3.2.1. XRD patterns of Au–Ni–TiO₂ nano-composite coatings

Phase structure of coatings is shown by the XRD analysis in Fig. 2. Fig. 2 shows typical diffraction patterns of electrodeposited Au–Ni coatings with broadening diffraction peaks which contributed to the nanocrystalline phase. The peaks at 2θ = 38.22°, 44.41°, 64.70°, and 77.71° are assigned to the (111), (200), (220) and (311) lattice planes of the cubic Au, respectively. No TiO₂ peaks could be seen from the composite coatings (Fig. 2b and c), probably due to the low quantity of TiO₂ particles and high intensity of other diffraction peaks.

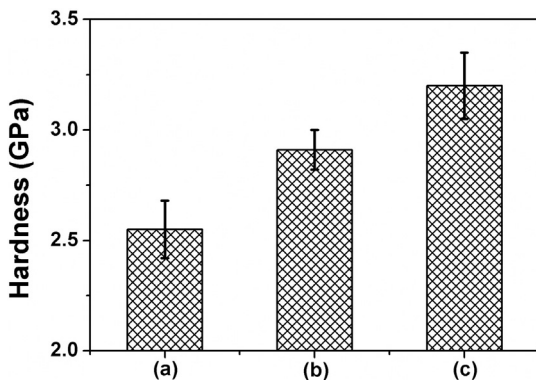


Fig. 1. Nano-hardness of: (a) Au–Ni coating, (b) powder enhanced Au–Ni–1.25 g/L TiO₂ coating and (c) sol-enhanced Au–Ni–6.25 mmol/L TiO₂ coating.

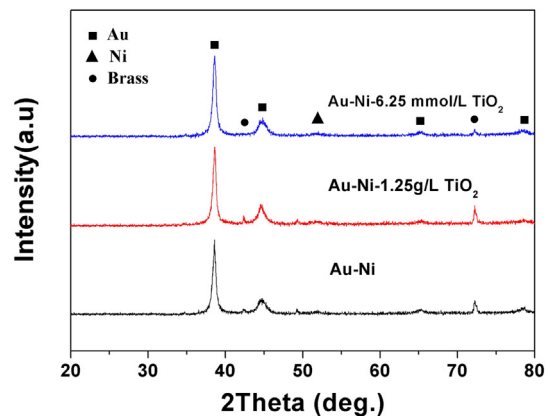


Fig. 2. XRD patterns of: (a) Au–Ni coating, (b) powder enhanced Au–Ni–1.25 g/L TiO₂ coating and (c) sol-enhanced Au–Ni–6.25 mmol/L TiO₂ coating.

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