[Technical Paper]

Multilayered Sn/Ag₃Sn Electroplating on Cu Alloys for High Reliable Electronic/Electric Materials

Song-Zhu Kure-Chu, Tohru Ogasawara, Hitoshi Yashiro, Rongbin Ye, Takuya Hosokai, Michimasa Uchida, Eiichi Suzuki, and Tomoyuki Naito

Faculty of Engineering, Iwate University, Morioka, Iwate 020-8551, Japan

(Received July 9, 2014; accepted November 13, 2014)

Abstract

We report a multilayered Sn/Ag₃Sn film electroplated on Cu alloys with high reliability as promising coating materials for LED lead-frames and for electric connectors. The multilayered Sn/Ag films consisted of an Ag-Sn alloy film with 30–80 nm-thick as Ag₃Sn phase covered on a 1 μ m-thick Sn film on Cu alloy substrates. The as-deposited Sn/Ag₃Sn films exhibited a high specular reflectivity around 70% at the wavelengths of 480 nm, which was lower than a commercial bright Ag film with total reflectivity of 90% but much higher than a commercial bright Au film in 24% at the same wavelength. It was found through an accelerated sulfuring test that, the reflectivity of the Sn/Ag₃Sn films was almost unchanged even after immersing in a 0.2% (NH₄)₂S_x solution for 60 min, compared to the abrupt decrease to 15% for the conventional Ag film even after immersing for 10 min. The excellent sulfuring resistance and durability of the multilayered Sn/Ag₃Sn films can be attributed to the existence of a stable SnO₂ film existed on the surface of Ag₃Sn alloy, which was revealed by a UPS analysis in a vacuum chamber by heating at 473–673 K, thus protecting underneath Ag₃Sn alloy film and inhibiting the chemical reaction of Ag and S²⁻ ions.

Keywords: Ag₃Sn, Sn, Multilayer, Electrodeposition, Reflectivity

1. Introduction

Light emitting diode (LED) is well known as an electroluminescence component that converts electrical energy into luminous energy. LED light sources have been widely used in various fields like television display, room lighting, and automotive headlight, due to their high energy-saving capacity, low voltage requirement, small size, long life, etc.[1] Generally, LED electroluminescence components in the package are set on a lead frame, which is required to provide excellent electrical and thermal conductivity and necessary light reflective performance. Ag-coated Cu alloy plates are mostly used as LED lead frame, because Ag layer has the highest reflectivity of 86–99% in broad wavelength[2] and Cu alloy plates possess high electrical/thermal conductivity and satisfied mechanical properties.[3] However, it is known that chemically active Ag films react readily with sulfur around and form a silver sulfide film on the surface, thus lowering their reflective performance and luminous efficiency of LED packages. Moreover, since Ag is a noble metal with increasing price of Ag nowadays, much effort has been conducted so far in searching low-

cost coating materials to replace costive Ag film on the lead frame.[4–6]

In our previous studies, we had reported a multilayered Sn/Ag film electroplated on Cu with excellent stability of electric contact resistance even after aging at 473 K for 3,000 h, satisfied fretting corrosion resistance, Sn whisker-free, and good solderability, which is expected as a promising coating material for various electric connectors and terminals.[7–10] The present study is aimed at exploring a new application of the multilayered Sn/Ag film on Cu alloy as potential LED reflective material with low cost and high durability. This paper focused on the evaluation of the sulfidizing resistance and the structure stability under high temperature circumstance of the multilayered Sn/Ag films, comparing with a commercial Ag film with 2- μ m thickness and a conventional reflowed Sn film with 1- μ m thickness.

2. Experimental

2.1 Fabrication of multilayered Sn/Ag films on Cu alloys

The multilayered Sn/Ag₃Sn specimens were produced by consecutively electroplating Sn and Ag films on Cu alloys. Briefly, a Sn film with 1 μm thickness was first electrodeposited on Cu alloys using an industrial sulfuric bath, followed by a heat treatment at 505–593 K and resolidification to get a reflowed Sn film (denoted as Sn-RF). Here, the Sn-RF film were adopted to achieve a bright and smooth Sn film with high reflectivity, better soldering ability, and more importantly, to inhibit the growth of Sn whiskers. Then, silver electroplating was performed on the Sn-RF film by using a cyanide bath at 5–25 mA cm⁻² for 10–60 sec to obtain Ag films with different Ag thickness, followed by an alloying treatment below 373 K, to achieve a smooth Ag₃Sn alloy layer on the Sn-RF reflowed Sn film.

2.2 Sulfidization test

The corrosion resistance against sulfurization of the coating materials was evaluated by immersing specimens in 0.2 ml/L (NH₄)₂S_x solution at 298 K for 10–60 min, referring to Japan Industrial Standard (JIS-8652). For comparison, a commercial Ag film with 2 μm thickness and the conventional reflowed Sn film without Ag electroplating were used in the corrosion test as reference specimens.

2.3 Reflective characteristization

The reflectance, including total reflectivity (SCI) and diffusive reflectivity (SCE), of various multilayered Sn/Ag films before and after sulfuring tests was measured by a spectrophotometer (Konica Minolta – 2500d) at an incident angle of 10°, with a standard white plate as reference of 100% reflectivity.

2.4 Structural characterizations

The microstructures of multilayered Sn/Ag₃Sn films and reference specimens were investigated by means of field emission scanning electron microscopy (FE-SEM: JOEL-JSM-7001F) with x-ray energy-dispersive analysis (EDS), grazing incidence X-ray diffractometer (XRD: Rigaku-2000, 35 kV/ 30 mA, Cu K α).

The micro area roughness and topography of the surface of the multilayered Sn/Ag₃Sn films were measured by atomic force microscopy (AFM: Nanocute, SII).

2.5 Annealing test and surface composition analysis

The ultraviolet photoelectron spectroscopic (UPS) experiments were done at beamline 8 (bending magnet) (BL8B) of the Ultraviolet Synchrotron Orbital Radiation Facility (UVSOR) storage ring at the Institute for Molecu-

lar Science, where monochromatized SR by a plane-grating monochromator was available.[11]

All the metallic alloy samples were installed from a load-lock chamber and transferred into the preparation chamber (a base pressure: 7×10^{-8} Pa). The samples were initially cleaned by Ar ion sputtering at 500 eV for 1–3 h and were annealed consecutively at 473, 573, and 673 K for 1 h, respectively.

For each heating step, UPS measurements were conducted in situ at room temperature (around 296 K) in the analysis chamber (a base pressure: 3×10^{-8} Pa) using a high-angular-resolution analyzer with a multichannel detector system (VGARUPS10) and synchrotron light of $h\nu = 40$ or 100 eV. The angle of incident photons and the detected photoelectrons were 45° and 0° relative to the surface normal, respectively. The angular and energy resolutions were $\pm 0.2^\circ$ and about 150 meV, respectively.

3. Results and Discussion

3.1 Microstructures of multilayered Sn/Ag films on Cu alloys

Figure 1 shows the surface morphologies of multilayered Sn/Ag films formed by electroplating Ag on Sn-RF films for 10–60 sec, and a Sn-RF film before Ag electrodeposition and a conventional Ag film as reference samples. The EDS analysis results were also labeled in the Fig. 1. The Sn-RF film possessed a smooth surface due to the melting of Sn film (Fig. 1a). After electroplating Ag for 10 sec, a discontinuous Ag film was formed on the Sn-RF film (Fig. 1b). As the deposition time performed above 30 sec (Fig. 1c, d), uniform Ag films with smooth surface and fine grains around 20 nm across were obtained, thus remaining the smooth morphology of the underlying Sn-RF film. Whereas as Ag electroplating time extended over 60 sec, a coarse deposit with uneven grains was formed. Especially, some Ag grains in 0.5–1 μm across can be observed beside of granular Ag₃Sn, thus leading to a non-uniform Ag film (Fig. 1d). Nevertheless, in all cases, the Sn/Ag films exhibited smoother surface than a commercial bright Ag film, and gave a mirror-bright appearance due to the underneath Sn-RF film on Cu alloys.

Figure 2 demonstrates the cross sections of multilayered Sn/Ag films with different thickness on Cu alloys, compared to the conventional Sn-RF and Ag films. The Sn-RF film exhibits an ideally smooth surface like water surface and a unique Cu-Sn alloy layer of Cu₆Sn₅ intermetallic compound, owing to the melting of electrodeposited Sn film at the high temperature above the Sn's melting point

Fig. 1 Surface FE-SEM images of (a) reflowed Sn, (b–e) multilayered Sn/Ag films formed by Ag electroplating for (b) 10, (c) 30, (d) 40, and (e) 60 sec, and (f) a conventional Ag film on Cu alloys.

Fig. 2 Cross-sectional FE-SEM images of (a) reflowed Sn, (b–e) multilayered Sn/Ag films formed by Ag electroplating for (b) 10, (c) 30, (d) 40, and (e) 60 sec, and (f) a conventional Ag film on Cu alloys.

(Fig. 2a). In contrast, the multilayered Sn/Ag₃Sn films, which were formed by electroplating thin Ag layers on Sn-RF films followed by a mild heat treatment, consist of a separate Ag-Sn alloy, a Sn layer, and a Cu₆Sn₅ layer similar to the Sn-RF films. It should be noticed that, the multilayered Sn/Ag₃Sn films reported in the present study are quite different from the conventional Sn-Ag alloy electroplating for soldering purposes[4] and Sn-Ag hot-dip coatings for automotive connectors,[12] in which the Ag component (the former) or the Ag₃Sn particles (the latter) distributed the whole thickness of coatings. Moreover, the Ag-Sn alloy layer composed of nano-grains 50–100 nm in across (Fig. 2b–2c), which are much smaller than the Ag grains of a commercial silver film (Fig. 2d) that are 200 nm–3 μ m across due to the well-known migration nature. This indicates that the multilayered Sn/Ag films can reserve the smooth surface of Sn-RF films through tailored fine plating method.

3.2 Surface roughness of multilayered Sn/Ag films on Cu

Figure 3 shows the effects of electrodeposition time on the average roughness of Sn/Ag films measured by AFM. The Sn-RF possesses a low roughness around 10 nm due to the smooth surface of molten Sn film. After Ag electroplating, the roughness of Sn/Ag films increased compared to the Sn-RF because of the deposition of granular Ag film (Fig. 1b–e). The roughness of the Sn/Ag film increased after Ag deposition at 10 sec, which can be ascribed to the insufficient Ag film thickness and the coagulation of Ag grains, consistent to the FE-SEM observation (Fig. 1a). As the Ag deposition time performed above 20 sec, a low roughness of 12 nm was obtained, because a continuous thin Ag film with nano-grains around 20 nm covered the

Fig. 3 Effects of Ag deposition time on the roughness of multilayered Sn/Ag films on Cu Alloys.

overall Sn-RF film uniformly. As the continuous Ag film formed, the roughness of multilayered Sn/Ag films changed in proportional to the Ag deposition time due to the increment of Ag particles and/or to the migration of Ag deposits.

3.3 Crystalline structures of multilayered Sn/Ag films on Cu alloys

Figure 4 shows the XRD analysis results of multilayered Sn/Ag films after Ag electroplating for 10–60 sec and a Sn-RF film before Ag electroplating as reference. For Sn-RF film (Fig. 3a), Sn <200>, Sn <101>, and Cu₆Sn₅ <101> facets are detected, consistent to the FE-SEM observation and EDS analysis (Fig. 2a). As for multilayered Sn/Ag films, in case of Ag deposition time less than 30 sec, all of the peaks that related to Ag were indexed to be Ag-Sn intermetallic compound in form of ϵ -Ag₃Sn phase with a hexagonal close-packed structure ($a = 2.995$ Å, $c = 4.780$ Å, $c/a = 1.596$). This indicated that the Ag was completely alloyed with Sn by the alloying treatment followed by Ag electroplating. Whereas in case of the deposition time longer than 40 sec, a small peak corresponding to Ag <111> was detected out beside of ϵ -Ag₃Sn phase, which can be attributed to the surplus Ag deposits to stoichiometric Ag₃Sn alloy and/or insufficient Ag-Sn alloying treatment, thus leading to a multilayered Sn/Ag₃Sn/Ag film. Moreover, it was confirmed that the crystalline structure of Ag₃Sn phase was unchanged even after aging at 473 K for

Fig. 4 XRD patterns of (a) a reflowed Sn and multilayered Sn/Ag films after electroplating Ag on Sn-RF for (b) 30, (c) 40, (d) 50, and (e) 60 sec, respectively.

3,000 h in air,[9] indicating the excellent physical and chemical stability under the high temperature circumstance.

3.4 Reflectance of multilayered Sn/Ag films on Cu alloys

Figure 5 shows the reflectance of multilayered Sn/Ag films in forms of total reflectivity (SCI) and diffusive reflectivity (SCE). For references, a Sn-RF film before Ag electroplating and a commercial Ag electroplating film of 2 μm thick for LED lead frame were also given in Fig. 5. The Sn-RF film shows a high total reflectivity of 80% (480 nm; close to blue light wavelength) (Fig. 5a) and a low diffusive reflectivity near 0% (Fig. 5b) due to the smooth surface (refer to Fig. 1a). The reflectance of multilayered Sn/Ag films, on the other hand, is greatly affected by the deposition time or the corresponding Ag thickness on Sn-RF films. As the deposition time is less than 20 sec, the total reflectivity multilayered Sn/Ag films decreased to around 65% (480 nm) (Fig. 5a), which can be ascribed to variation of the surface roughness and Ag-Sn alloying extent. As the deposition time increases to 35 sec, the reflectivity increases to medium values of 65–75% (480 nm), though lower than that of Sn-RF film, which can be attributed to the increase of Ag content with high reflective nature. Whereas as the deposition time increases longer than 40 sec, the reflectivity of the specimens exhibited a higher reflectivity of around 86%, which close to the reflectivity of pure Ag film in 90% (refer to Fig. 6). This can be attributed to the surplus Ag particles on the Ag₃Sn alloy at prolonged Ag deposition, *i.e.*, possessing a multilayered Sn/Ag₃Sn/Ag structure, consistent to the XRD analysis results (Fig.

4). Moreover, it can be seen from Fig. 5b that the diffusive reflectivity of multilayered Sn/Ag₃Sn films increases with deposition time, which can be ascribed to the increment of surface roughness by Ag-Sn alloy particles as shown in Fig. 1e and Fig. 3.

3.5 Sulfidation resistance of multilayered Sn/Ag₃Sn films

Figure 6 shows the reflectivity of multilayered Sn/Ag₃Sn films with Ag film thickness of 50 and 80 nm and a commercial bright Ag film before and after immersing in 0.2 ml/L (NH₄)₂S_x solution for 10–60 min, and a commercial bright Au film for reference. It can be seen that the multilayered Sn/Ag₃Sn films exhibit a total reflectivity of 69% and 70% at wavelength of 480 nm for the films with Ag

Fig. 6 Reflectivity of multilayered Sn/Ag₃Sn films with different Ag thicknesses and Ag films before and after immersed in a 0.2 ml/L (NH₄)₂S_x solution for 10–60 min.

Fig. 5 (a) Total reflectivity (SCI) and (b) diffusive reflectivity (SCE) of multilayered Sn/Ag films obtained by Ag electroplating for 10–60 sec and a Sn-RF film before Ag electroplating.

thickness of 50 and 80 nm, respectively, which is a little lower than that of bright Ag film of 90% (480 nm) but definitely higher than that of bright Au film of 24% (480 nm). Noticeably, the multilayered Sn/Ag₃Sn film exhibited excellent durability in the sulfidizing test, remaining a high reflectivity of 68% and 69.6% (480 nm) even after immersion for 60 min. Whereas for the conventional Ag film, the reflectivity changed from 90% to around 20% even after 10 min immersing, and decreased to about 5% after 60-min immersion. From the appearance of the films, the multilayered Sn/Ag films kept their mirror-like luster throughout the sulfidizing test, whereas the Ag films changed from silvery to yellow, brown, and dark bluish, lost the metallic luster even within short immersing time of 10 min. Moreover, it was found that a similar tarnish phenomenon to the Ag film was observed for specimens with Ag thickness more than 90 nm or formed at deposition time longer than 40 sec, which can be ascribed to the surplus Ag deposit on Ag₃Sn alloy layer (refer to Fig. 4). This means that the completion of Ag-Sn alloying treatment to form chemically stable Ag₃Sn layer is essential for application as high durable reflective coating materials.

3.6 Stability of multilayered Sn/Ag₃Sn films at high temperature circumstance

The variation of the surface composition of Sn/Ag₃Sn films at high temperature circumstance was investigated by a stepwise heat treatment in a vacuum chamber and measured in-situ with an ultraviolet photoelectron spectroscopy (UPS). Figure 7 shows the UPS spectra for a multilayered Sn/Ag₃Sn film with Ag film thickness of 50 nm before and after heating continuously at 473 K for 1 h, 573 K for 1 h, and 673 K for 1 h, respectively. For the as-deposited specimen, only a peak corresponding to SnO₂ binding energy can be detected out, indicating that the multilayered Sn/Ag₃Sn film was actually covered by a thin tin oxide film. This discloses the reason why the multilayered Sn/Ag₃Sn film exhibited the excellent anti-corrosion performance in the sulfurizing test (Fig.6). In other words, it is the chemically stable SnO₂ film on the surface of the multilayered Sn/Ag₃Sn film that protected the Ag element in Ag₃Sn alloy from the attack of S²⁻ ions. Here, the thickness of the SnO₂ film was estimated to be around 2–3 nm from the UPS results.

With increasing annealing temperature from 473 K to 573 K, peaks that corresponded to Sn and Ag metals emerged and intensified gradually, with the peak intensity of SnO₂ decreasing correspondingly. This revealed the Ag-Sn alloy beneath the SnO₂ film emerged on the film surface with aging at high temperature. The SnO₂ film was stable even after aging at 673 K, though the peak intensity or the SnO₂ film thickness decreased greatly, which may be ascribed to the partly thermal decomposition of tin oxide at high vacuum circumstance. In all cases, Cu element diffused from the substrate materials was not detected at the test range. This means that layered microstructure of Sn/Ag₃Sn film was stable at high temperature circumstance, which is required for practical applications as both LED lead-frames and electric connectors.

Figure 8 shows the XRD patterns of the multilayered Sn/Ag₃Sn film before and after aging at the conditions shown in Fig.7. The Sn layer disappeared after aging at high temperature due to the progress of Cu-Sn alloying

Fig. 7 Ultraviolet photoelectron spectroscopic (UPS) spectra of a multilayered Sn/Ag₃Sn film before and after heated at step-increased temperatures in a vacuum chamber of 7×10^{-8} Pa.

Fig. 8 XRD patterns of a multilayered Sn/Ag-50 nm film before and after stepwise aging at 473 K for 1 h, 573 K for 1 h, and 674 K for 1 h consecutively.

reaction, and the Cu-Sn alloy layer transferred from Cu₆Sn₅ phase to Cu₃Sn phase because of the outward thermal diffusion of Cu element from the base Cu alloy material. Irrespective of the transition of underlying Sn and Cu-Sn alloy layers, the Ag₃Sn alloy with a preferential orientation of (002) facet remains on the surface of the specimen, indicating a stable crystalline structure under the high temperature circumstance.

4. Conclusions

We reported the fabrication and characterization of a multilayered Sn/Ag film electroplated on Cu alloys considering practical applications for LED lead-frames and for electric connectors. The multilayered Sn/Ag₃Sn films possessed a smooth surface, which inherited to underlying reflowed Sn film, and gave low roughness values of 12–33 nm, dependent on the electroplating conditions.

The multilayered Sn/Ag₃Sn films with Ag thickness of 30–80 nm formed by Ag electroplating for 10–30 sec on reflowed Sn films on Cu alloys exhibited a high reflectivity around 70% at the wavelengths of 480 nm (close to blue light wavelength) and excellent anti-tarnish performance in an accelerated sulfuring test referring to an industrial standard. Especially, the reflectivity of the Sn/Ag₃Sn films was almost unchanged even after immersing in a 0.2% (NH₄)₂S_x solution for 60 min, compared to the abrupt decrease to 15% for the conventional Ag film even after immersing for only 10 min. Moreover, generally, the lifetime of LED devices is calculated by the period when the reflectivity or the luminance decreases to the 70% of the

initial value, or in other word, to 63% (480 nm). The fact that the multilayered Sn/Ag₃Sn films can keep a stable reflectivity of around 70% (480 nm) in the accelerated sulfuring test revealed an excellent durability which is essential for practical application as promising LED reflective materials.

The excellent sulfuring resistance and durability of the multilayered Sn/Ag₃Sn films can be attributed to the existence of a stable SnO₂ film existed on the surface of Ag₃Sn alloy, which was revealed by a UPS analysis in a vacuum chamber by heating at 473–673 K, thus protecting underneath Ag₃Sn alloy film and inhibiting the chemical reaction of Ag and S²⁻ ions.

Acknowledgment

The authors thank Mr. Sasaki for assisting preparation of specimens for FE-SEM observation. This work is supported by the Grants-in-Aid for Science Research of Japan Society for the Promotion of Science (JSPS; No. 25420769) and the Adaptable and Seamless Technology Transfer Program through target driven R & D (A-STEP- JST; No. AS 251Z02572M).

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Song-Zhu Shochiku KURE-CHU received a M.E. in Applied Chemistry from North-eastern University in P. R. China and a Ph.D. in Molecular Chemistry from Hokkaido University in Japan. Her specialty mainly includes electroplating, electroless-plating, anodizing, sol-gel coating, and electro/chemical polishing. She had worked as a researcher in KRI, NIMS, and CRIEPI in the field of fabricating versatile functional nanomaterials for photocatalysis, perpendicular magnetic recording media, H_2 production, etc. In April 2012, she became a tenured associate Professor in the department of Chemistry and Bioengineering of Iwate University, after 6-year's working in Mitsubishi Shindoh Co. Ltd as a senior researcher in R & D division in charge of surface finishing of Cu alloy plates for automotive connectors and lead-frames of LED devices.

Tohru Ogasawara

Hitoshi Yashiro is a full professor in the Department of Chemistry and Bioengineering at Iwate University in Morioka, Japan. He earned his bachelor's (1982) and doctoral (1994) degrees from Tohoku University. He was an international fellow at SRI International in 1991–1992. His research covers variety of corrosion problems from that in lithium ion battery to nuclear power plants. He served as an editor-in-chief for Japan Society of Corrosion Engineering in 2009–2011.

Rongbin Ye

Takuya Hosokai

Michimasa Uchidate

Eiichi Suzuki

Tomoyuki Naito