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Interface reaction between an electroless Ni–Co–P metallization and Sn–3.5Ag lead-free solder with improved joint reliability

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Abstract

To address the reliability challenges brought about by the accelerated reaction with the implementation of lead-free solders, an electrolessly plated Ni–Co–P alloy (3–4 wt.% P and 9–12 wt.% Co) was developed as the solder metallization in this study. Three compounds layers, $(Ni,Co)_3Sn_4$, $(Ni,Co)_3P$ and $(Ni,Co)_{12}P_5$, are formed at the reaction interface. Nano-sized voids are visible in the $(Ni,Co)_3P$ layer under transmission electron microscopy, but no large voids are found under scanning electron microscopy. This is an indication of effective diffusion barrier performance by the Ni–Co–P metallization compared with the binary Ni–P metallization. The influence of interfacial reaction on the solder joint reliability was reported through the evaluation of the tensile strength of solder micro-joints. Upon aging at 180 °C for 600 h, the tensile strength of Ni–Co–P/Sn–3.5Ag solder joint remains high, and the failure is caused by the bulk solder necking and collapse. As a comparison, the tensile strength of the Ni–P/Sn–3.5Ag solder joint drops significantly after aging for 400 h at 180 °C, and the fracture mode has shifted from ductile failure in the bulk solder to brittle failure at the solder joint interface. The Ni–Co–P metallization, having a much slower consumption rate and improved resistance to joint strength degradation during long-term aging treatment, is a potential candidate for future microelectronic solder metallization materials.
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1. Introduction

Cu was previously the most popular choice as the metallization for solder joining due to its excellent conductivity and good wetting performance with liquid solders [1]. However, with the implementation of lead-free solders since 1 July 2006 following the EU Directive 2002/95/EC of the European Parliament and of the Council on the restriction of the use of certain hazardous substances in electrical and electronic equipment, new challenges for the long-term reliability of solder joints have been encountered by the industry, as commonly used lead-free solders react much faster with Cu metallization than eutectic Sn–Pb solder. This is because lead-free solders have a higher Sn content and higher melting temperatures than eutectic Sn–Pb solder. Thus the reaction between Cu and lead-free solders is much faster than that between Cu and eutectic Sn–Pb solder. Cu will be completely consumed by lead-free solders quickly, leaving only intermetallic compounds (IMCs) at the interface. This would result in the degradation of mechanical strength of the solder joint [2–4]. Therefore it is well understood that Cu-based metallizations are not suitable for lead-free soldering.

Ni and Ni alloys were reported to have a much slower reaction rate with Sn as compared with Cu and Cu alloys [1,5]. Thus over the years, Ni-based metallizations have received great attention in lead-free soldering. Among Ni-based metallizations, electrolessly plated NiiP has been widely used for the past several decades because of its

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excellent uniformity, mask-less selective process, low residual stress, good solderability and low processing cost. Accordingly, the interfacial reactions between electroless Ni–P and Sn-containing solders, along with the implication to long-term solder joint reliability, have been well studied [6–15]. During soldering with electroless Ni–P, the reaction between Sn and Ni activates the transformation of amorphous Ni–P metallization into a layer of crystalline Ni_3P [16]. Since this layer has a fine columnar structure [6,16,17], the grain boundaries of this layer become the fast diffusion path for Ni diffusion, leading to an accelerated interfacial reaction. As a result of the fast reaction, micro-sized voids are observed in the Ni_3P layer after prolonged reaction [6,13,17], contributing to the weakened interface and degraded reliability of the solder joint. Therefore, in order to slow down the interfacial reaction with lead-free solders, it is necessary to suppress the growth of the fast diffusion path, which is the void-containing Ni_3P layer formed during soldering reaction with Ni–P metallization.

In previous studies, we have shown that the incorporation of a ternary element into Ni–P can be an effective way to slow down the interface reaction by either preventing the crystallization of Ni_3P by incorporating W [18,19] or completely avoiding the formation of Ni_3P by the addition of Sn into Ni–P [20]. In this study, we explore an electrolessly plated Ni–Co–P alloy as solder metallization. There have been some reports showing positive diffusion barrier performance using Co-containing metallizations for lead-free soldering [21–23]. Similar to Ni, Co is also reactive with Sn, and based on the Co–Sn phase diagram [24], a few IMCs may form between Co and Sn. However, the Ni–Co–P phase diagram is not available. In other studies, it was reported that Ni–Co–P alloys can be a better protective coating than Ni–P due to their improved corrosion resistance and thermal stability [25,26]. Ni–Co–P alloys were also explored as the plug contact on the silicide transistor gate [27]. In the early 1990s, Ni–Co–P alloy deposit was first investigated as a diffusion barrier between an Al pad and a high-lead solder bump (95Pb–5Sn) [28]. In recent years, the electrolessly plated Ni–Co–P alloys were also investigated as the diffusion barrier layer in ultra-large-scale integration (ULSI) technology [26,29,30]. Interfacial reaction between lead-free solder and Ni–Co–P plating was reported by Daito et al. [31]. However, their two-page report mainly looked into wetting spread area comparison among Ni–P. Co–P and Ni–Co–P. No information on the chemical composition of the Ni–Co–P plating was available; neither was there any IMC phase identification or solder joint reliability.

The objective of this work is to study the interfacial reaction and tensile strength of solder joints between lead-free Sn–3.5Ag solder and electroless Ni–Co–P metallization after reflow and prolonged aging. Attention is focused on the interfacial IMC formation mechanism and how the reaction affects the solder joint strength. To reveal the advantage of such a ternary alloy, a commercially available binary electroless Ni–P alloy was also investigated for comparison. Fractographic analysis of both types of failed solder joints was conducted to assist the understanding of the joint strength degradation.

2. Experimental procedures

2.1. Metallization preparation

Cu plates (99.98 wt.%) with a thickness of 6 mm were used as substrate for electroless plating of both Ni–P and Ni–Co–P. Prior to plating, the Cu plates were mechanically polished and then chemically etched with 35 vol.% nitric acid for 30 s. After that, the Cu surface was activated using a commercial ruthenium-based pre-initiator. Electroless Ni–P plating was conducted in a commercial acidic sodium hypophosphite bath (from MacDermid) with a pH level of 5.3 at 88 ± 2 °C for 30 min. The obtained Ni–P coating contains 6–7 wt.% P and has a thickness of 9.7 μm. On the other hand, the electroless Ni–Co–P plating was carried out in an alkaline bath with a pH level of 10.0 at 88 ± 2 °C for 1 h. As listed in Table 1, this alkaline bath contained nickel chloride and cobalt chloride as nickel and cobalt sources, respectively. Sodium hypophosphite was used as a reducing agent along with sodium citrate, lactic acid and ammonium chloride as the complexing agents. The obtained Ni–Co–P coating contains 3–4 wt.% P and 9–12 wt.% Co, with a thickness of 9.9 μm.

2.2. Reflow and aging process

The electrolessly coated Cu plates were joined by Sn–3.5Ag solder to form Ni–P/Sn–3.5Ag and Ni–Co–P/Sn–3.5Ag solder joints. A thin layer of no-clean paste flux was first applied on top of the plated surface of Cu to remove the surface oxides, and then small pieces of Sn–3.5Ag solder wires with flux in core were arranged in an orderly fashion on the plated surface. Alumina spacers with a thickness of ~650 μm were used to maintain a fixed and uniform thickness of solder layer in between the two joined Cu plates. The reflow soldering process was conducted in an infrared reflow oven involving an initial preheating at 190 °C for 100 s, followed by reflowing at a peak temperature of 260 °C for 60 s, before finally cooling down to 120 °C. After that, both types of as-reflowed solder joints were aged at 180 °C for up to 600 h to study their interfacial reactions and tensile strength upon aging.

Table 1
Composition of the plating bath for electroless Ni–Co–P.

Components of plating bath	Concentration
$NiCl_2$	10 g l^{-1}
$CoCl_2$	4 g l^{-1}
$NaH_2PO_2 \cdot H_2O$	16 g l^{-1}
$Na_3C_6H_5O_7 \cdot 2H_2O$	40 g l^{-1}
NH_4Cl	30 g l^{-1}
Lactic acid	4.5 ml l^{-1}

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