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Interfacial Reaction Studies on Lead (Pb)-Free Solder Alloys

S. K. Kang, D. Y. Shih, K. Fogel, P. Lauro, M. J. Yim, G. Advocate*, M. Griffin*, C. Goldsmith*, D. W. Henderson, T. Gosselin**, D. King**, J. Konrad**, A. Sarkhel**, K. J. Puttlitz*****

IBM Research Division
Thomas J. Watson Research Center
P.O. Box 218
Yorktown Heights, NY 10598

*IBM Microelectronics
Hopewell Junction, NY 12533

**IBM Microelectronics
1701 North Street
Endicott, NY 13760

***IBM Corporate Staff
Hopewell Junction, NY 12533

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***IBM Microelectronics, Hopewell Junction, NY, 12533**
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Abstract

Recently, the research and development activities for replacing Pb-containing solders with Pb-free solders have been intensified due to both competitive market pressures and environmental issues. As a result of these activities, a few promising candidate solder alloys have been identified, mainly, Sn-based alloys. A key issue affecting the integrity and reliability of solder joints is the interfacial reactions between a molten solder and surface finishes in the solder joint structures. In this paper, a fundamental study of the interfacial reactions between several Pb-free candidate solders and surface finishes commonly used in printed-circuit cards is reported. The Pb-free solders investigated include Sn-3.5Ag, Sn-3.8Ag-0.7Cu, and Sn-3.5Ag-3.0Bi. The surface finishes investigated include Cu, Au/Ni(P), Au/Pd/Ni(P), and Au/Ni (electroplated). The reaction kinetics of the dissolution of surface finishes and intermetallic compound growth have been measured as a function of reflow temperature and time. The intermetallic compounds formed during reflow reactions have been identified by SEM with Energy Dispersive X-ray Spectroscopy.

Introduction

The principal motivation of the search for Pb-free solder alloys in the microelectronic applications has been twofold; an environmental concern of Pb-containing materials and the development of an advanced soldering technology in terms of low temperature process and fine pitch connection (1-5). Lately, one more motivation has been added, which is the competitive market pressure coming from consumers desiring "green" electronic products. This pressure seems to be much stronger than the legislative pressure in the past, since it would have seriously impact on the market share of certain consumer products. Many consumer electronics manufacturers, especially in Japan, have announced their road maps to replace Pb-containing solder connections with Pb-free ones. In US, several Pb-free programs have been initiated by professional societies, such as EIA, IPC and NEMI to integrate the industry-wide efforts to implement Pb-free solutions in a timely fashion. An acceptable Pb-free solder solution must satisfy both process requirements and reliability objectives. Ideally, it should be suitable for mass production applications of both SMT and PTH soldering. Furthermore, the process technologies currently in use for Pb-Sn eutectic or other Pb-containing solders must be adaptable to the new solder without major changes in manufacturing processes, or significant capital investments. The wettability of the new solder should be better or equivalent to that of the Pb-Sn solder, and yielding a better or equivalent defect rate in the assembly line. The selected Pb-free solder should be usable with water-soluble or no-clean fluxes.

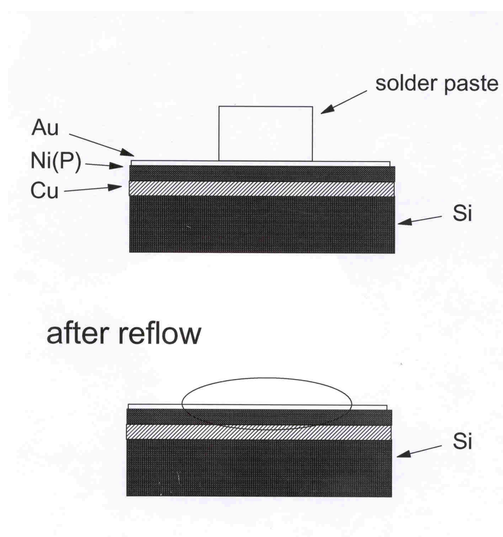

Fig. 1 Schematic diagram showing a reflow coupon sample with the surface finish Au/Ni(P) of a known thickness.

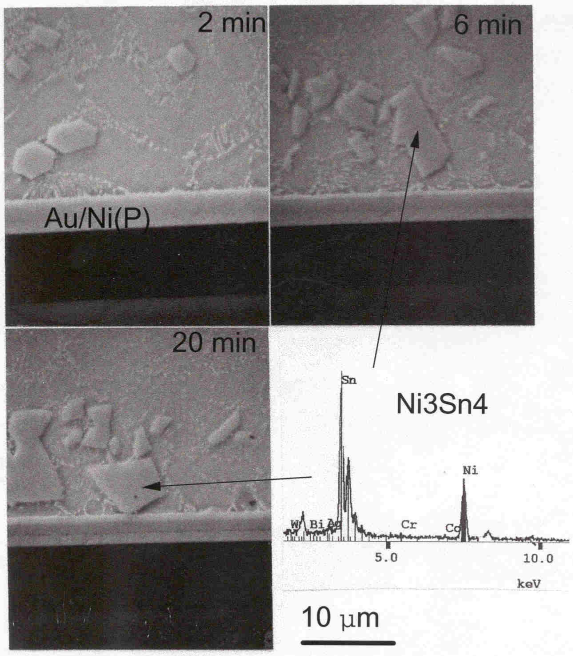

Fig. 2 An interfacial reaction series with Sn-3.5%Ag on Au/Ni(P) at 250 ºC, for 2, 6, and 20 min. An X-ray analysis identifies the Ni-Sn intermetallics.

The melting temperature of the new solder is desired to be lower than those of the current solders in order to reduce the magnitude of thermal stresses experienced during soldering. The new solder must be able to produce solder joints with acceptable joint strength, and at the same time, also be able to withstand thermal fatigue over the projected operating life of the soldered assembly and meet other reliability requirements, such as adequate corrosion, oxidation or electromigration resistance. Lastly, the material cost of the new solder should not be so high as to overwhelm the assembly cost. With the above guidelines in mind, a number of Pb-free solders has recently been revisted or newly developed (3-12). Some of the examples include Sn-3.5%Ag, Sn-3.5%Ag-0.7%Cu, Sn-3.5%Ag-4.8%Bi, Sn-0.7%Cu, Sn-5%Sb, Sn-20%In-3%Ag, Sn-8%Zn-3%Bi, Sn-10%In-3%Ag-1%Cu among others. Most recently, the list has been further shrunk to three or four promising candidates as a result of accelerated development efforts in the microelectronics industry (13, 14). This short list now consists of the solder alloys such as Sn-3.5%Ag, Sn-3.5%Ag-0.7%Cu, Sn-3.5%Ag-4.8%Bi or Sn-0.7%Cu (with slight variations in the composition, all in weight percent), which are all high Sn-based alloys. To further narrow down the list of solder candidates, one key determining factor can be the interfacial reactions between the molten solder and surface finishes in solder joints, because they may seriously affect the integrity and reliability of solder joints. In an early investigation, the interfacial reactions during soldering with several Sn-rich alloys were studied in terms of the dissolution of the base metals, Cu and Ni, in the molten solders and the growth of the intermetallic compounds at the solder/metal interface (15). It was found that for the Sn-rich solders, a thin Ni layer is not a good reaction barrier to survive extensive soldering reaction. A strong need to develop a good reaction barrier metal to control the interfacial reactions has been recognized for Sn-rich solders (2).

In the present investigation, we report the interfacial reactions between the recently selected Pb-free solder candidates and the surface finishes commonly used in printed circuit boards.

Experimental Procedure

The interfacial reactions were investigated between three Pb-free solders (Sn-3.5%Ag(SA), Sn-3.8%Ag-0.7%Cu (SAC), Sn-3.5%Ag-3%Bi (SAB) and four surface finishes (Au/Ni(P), Au/Pd/Ni(P); all electroless plated, Au/Ni(electroplated) and Cu(electroplated). The experiment was performed by using a surface finish layer deposited on a Si wafer as a substrate. A Si wafer was sputter cleaned in an in-situ argon ion radio frequency plasma to remove contaminants. This was followed by deposition of a 30 nm Cr adhesion layer and a Cu layer of 1 μm thickness. Subsequently, an electroless Ni(P) layer of about 4 μm thickness was deposited, followed by deposition of either a thin layer of electroless Pd/Au or Au. In order to compare the interfacial reaction kinetics of the electroless Ni(P) with an electroplated Ni layer, the fourth surface finish was added to the experimental matrix, which has the thin film structure of TiW/CrCu/Cu/Ni/Au. The Ni layer of 4 μm was electroplated and a thin Au layer of 30 nm was immersion plated. Each wafer was diced into 15 mm x 15 mm coupons and cleaned in acetone and then in dilute sulfuric acid to remove any surface residue or oxide. One major advantage of using the Si coupon samples described here is to know precisely the initial thickness of a selected surface finish layer and thereby to measure accurately the amount of the dissolution of the surface finish layer. A schematic diagram of the experimental set up is shown in Figure 1, where a selected solder paste was dispensed through a metal mask with an opening of 5 mm diameter onto a clean Si coupon with a selected surface finish layer. Then each coupon was reflowed on a hot plate maintained at 250 ºC, covered with a nitrogen atmosphere for a selected reflow time (2, 6, or 20 min). After the reflow, the coupon was removed from the hot plate to a metal block to rapidly solidify the molten solder. The cooling rate was estimated to be about 10-20 ºC/sec. After reflow and cool down, the coupon samples were cross-sectioned using a diamond saw, mounted in room temperature curing epoxy and metallographically prepared to a final finish of 0.05 μm alumina slurry. To enhance the contrast under a scanning electron microscope (SEM), the polished samples were lightly etched with a 10% solution of HCl in methanol for a period of one to two minutes. The samples were then coated with a thin

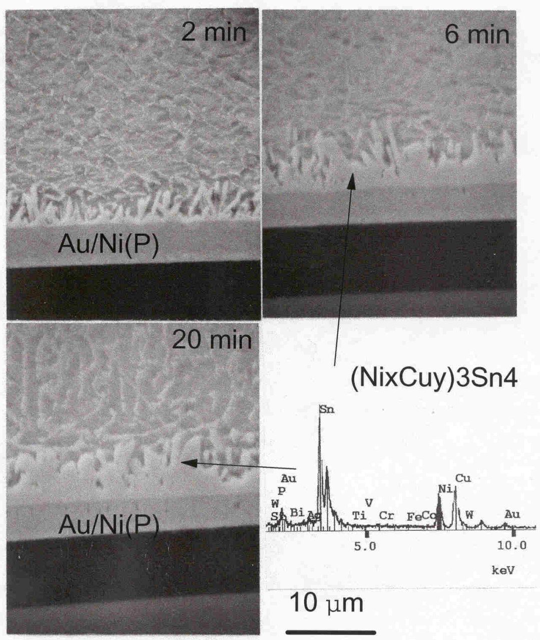

Fig. 3 An interfacial reaction series with Sn-3.8%Ag-0.7% Cu on Au/Ni(P) at 250 ºC, for 2, 6, and 20 min. EDX ananlysis identifies the Ni-Cu-Sn intermetallics.

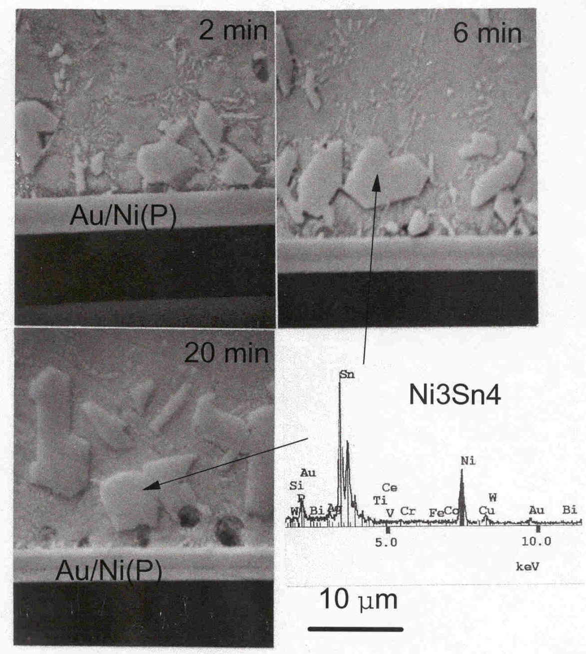

Fig. 4 An interfacial reaction series with Sn-3.5%Ag-3%Bi on Au/Ni(P) at 250 ºC, for 2, 6, and 20 min. The Ni-Sn intermetallics are identified by EDX.

layer of Au to prevent charging under the electron beam exposure. Extensive SEM observation was employed with each cross-sectional sample to record the dissolution of the surface finish layer(s) as well as the formation of the intermetallic phases. Energy dispersive X-ray (EDX) analysis was also performed to determine the elemental compositions of the intermetallic phases qualitatively.

Results

Representative SEM micrographs of the interfacial reactions of the Au/Ni(P) surface finish with three Pb-free solders, Sn-3.5%Ag (SA), Sn-3.8%Ag-0.7%Cu (SAC), and Sn-3.5%Ag-3.0%Bi (SAB), are shown in Fig. 2 to Fig. 4, in which the reaction periods of 2, 6, and 20 min at 250 ºC are shown for each solder alloy. One representative X-ray spectrum of a selected large intermetallic compound formed at (or near) the interface is also included in each Figure.

Fig. 2 exhibits the reaction sequence of Sn-3.5%Ag solder with Au/Ni(P) at 250 ºC. The majority of the intermetallic compounds formed are separated or spalling away from the interface, and only a thin layer of the intermetallics remains attached at the interface. The EDX spectrum identifies the Ni-Sn compound, Ni_3Sn_4, which has been widely reported (15). The morphology of the intermetallics is shown as blocky or faceted. The size and density of the intermetallics increases with increased reaction time.

Fig. 3 exhibits the reaction sequence of Sn-3.8%Ag-0.7%Cu with Au/Ni(P) at 250 ºC. Here, the intermetallics formed are mostly attached well to the interface, which is a quite different situation compared to those shown in Fig. 2. The morphology of the interfacial intermetallics is needle-like at the beginning and become more planar as the reaction proceeds longer. EDX reveals the intermetallics to have a ternary composition of Ni-Sn-Cu.

Fig. 4 exhibits the reaction sequence of Sn-3.5%Ag-3.0%Bi with Au/Ni(P) at 250 ºC. Here, again, the majority of the intermetallics formed is separated from the interface, similar to Fig. 2. Their morphology is again blocky or faceted. The X-ray analysis reveals the intermetallics to be the Ni-Sn binary, not containing any Bi in it (within the detection limit of EDX).

Table I summarizes the dissolution kinetics of various surface finish layers. The remaining thickness of each surface finish layer is listed as a function of reaction time and solder alloy composition. Each number represents an average number of three measurements from the middle portion of a cross-sectional sample for each reaction time. The initial thickness of a surface finish layer minus the remaining thickness after 20 min is estimated as the total amount of dissolution and listed in Table I.

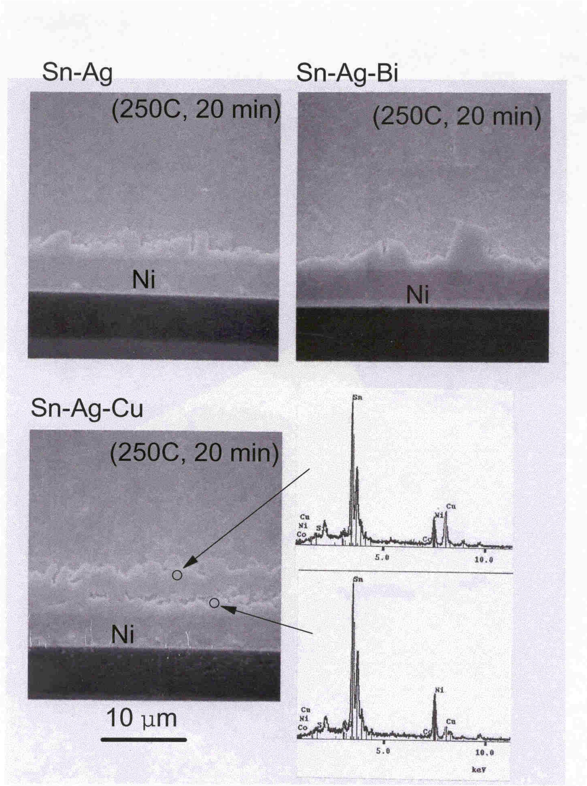

Fig.5 The interfacial reactions on electroplated Ni at 250 ºC, 20 min for Sn-Ag, Sn-Ag-Cu and Sn-Ag-Bi. Two different intermetallics are identified in SnAgCu.

Table II summarizes the growth kinetics of the intermetallic compounds as a function of reaction time, solder composition and surface finish. Since the intermetallic growth is not uniform or planar, the range of the intermetallic thickness is listed, rather than one average number. The largest intermetallic thickness observed for 20 min is regarded as the total amount of the intermetallics grown for each condition, as listed in the column of "Total intemetallics" in the Table.

Fig. 5 exhibits the intermetallic formation of three Pb-free solders on an electroplated Ni layer at 250 ºC, for 20 min. In contrast to the case of the electroless Ni(P), the intermetallic compounds formed in all three Pb-free solders are well adhered to the interface, not separated or spalling away from the interface. For Sn-Ag-Cu, the intermetallic compounds are grown much thicker than those observed with other two solders. In addition, the intermetallic compounds grown in Sn-Ag-Cu appear to consist of two layers, and the corresponding X-ray analysis has revealed the top layer having a higher Cu content than Ni, and the bottom layer attached to the Ni layer having a higher Ni content than Cu.

Table III and IV compare the dissolution and the intermetallic growth data, respectively, of the electroplated Ni layer with those of the electroless Ni(P) for three Pb-free solders at 250 ºC.

Discussion

Dissolution of Surface Finishes

The dissolution data in Table I is plotted in Fig. 6 for each surface finish with three different solders (SA, SAC, SAB). The dissolution kinetic behavior in all cases appears to be linear as a function of the reaction time, except for Cu. The Cu surface finish in Sn-3.5%Ag dissolves at a much faster rate at the beginning and slows down later, exhibiting a parabolic kinetic behavior. However, assuming all the dissolution kinetics to be linear, an average dissolution rate is calculated by dividing the total dissolution amount by 20 min, as listed in Table I. In general, the dissolution rate of Au/Ni(P) or Au/Pd/Ni(P) is about the half of that of Cu, which is in a good agreement with the previous work of electroless Ni(P) and electroplated Ni (15, 16). This reduced dissolution rate of Ni(P) over Cu is a strong benefit of using Ni(P) as a reaction barrier in a surface finish layer for Sn-rich solders. However, as a result of the dissolution and intermetallic compound formation, the well-known, brittle Ni-P intermetallic compound layer underneath the Ni-Sn compound is expected to form more easily with Pb-free solders compared with the Pb-Sn eutectic solder. This is due to the higher reflow temperature and the higher Sn content associated with the Pb-free solders, resulting in a faster rate of dissolution and intermetallic compound formation. Comparing the effect of the solder alloy composition on the dissolution rate of Au/Ni(P) or Au/Pd/Ni(P), Sn-3.8%Ag-0.7%Cu appears to produce the least dissolution rate among three solders. It seems the small amount of Cu in Sn-Ag-Cu reduces the dissolution rate of Au/Ni(P) or Au/Pd/Ni(P).

The dissolution rate of electroplated Ni in three Pb-free solders is again estimated by dividing the total dissolution amount by 20 min, being compared with that of the corresponding electroless Ni(P) in Table III. The dissolution rates of the electroplated Ni in Sn-Ag and Sn-Ag-Bi are much smaller than those of the electroless Ni(P), while they are similar to each other in Sn-Ag-Cu. The observed dissolution rate of the electroplated Ni in Sn-3.5%Ag agrees well with that reported previously for an equivalent condition (15).

Growth Kinetices of Intermetallics

The intermetallic growth behavior in three different Pb-free solders (SA, SAC, SAB) is shown in Fig. 7 by plotting the largest intermetallic thickness observed in each reflow condition. Except for Au/Pd/Ni(P) in Sn-3.5%Ag, the intermetallic growth appears to be linear with the reflow time. The growth rate is therefore approximated by dividing the maximum thickness observed by 20 min, as listed in Table II. For Au/Ni(P) or Au/Pd/Ni(P), the intermetallic growth rates among three Pb-free solders are ranked as SAB > SAC > SA. For Sn-3.5%Ag, the intermetallic growth rates associated with three surface finishes are ranked as Cu > Au/Ni(P) > Au/Pd/Ni(P). By comparing the intermetallic growth rate with the dissolution rate of each case, it is noted that the intermetallic growth rate is much larger (3-10 times) than that of the corresponding dissolution rate. It is also recognized that the intermetallic growth is much more affected by the solder composition than by the surface finish.

The intermetallic growth rate of the electroless Ni(P) is compared with that of the electroplated Ni for three different solders as listed in Table IV. It is found that the intermetallic growth rates on the electroplated Ni are two or three times smaller than those on the electroless Ni(P) for all three solders. The beneficial effect of an electroplated Ni over an electroless Ni(P) in terms of its interfacial reactions has been reported before with the Pb-Sn eutectic solder (17, 18). This beneficial effect is attributed to the difference in their microstructure and alloy composition between electroless Ni(P) and electroplated Ni.

Morphology of Intermetallic Growth

Fig. 2 to Fig. 4 exhibit the morphology of the intermetallic compounds grown on Au/Ni(P), indicating that solder composition plays a major role. The Cu-containing Sn-Ag-Cu solder yields an intermetallic compound which adhered well to the interface, while Sn-Ag and Sn-Ag-Bi produce an intermetallic compound which separated or spalled away from the interface. In addition, it is also observed that the intermetallics found in Sn-Ag-Cu have a ternary composition of $Ni_xCu_ySn_z$, while the intermetallics in Sn-Ag or Sn-Ag-Bi have the binary composition of Ni_3Sn_4. In addition, the parallel phenomena of the morphological dependency on the solder composition is observed with the surface finish of Au/Pd/Ni(P). A similar observation was reported from the morphological study of Pb-free solder balls reacted with the Ni-P under bump metallization (19), where the Cu-containing solder produced well adhering intermetallics.

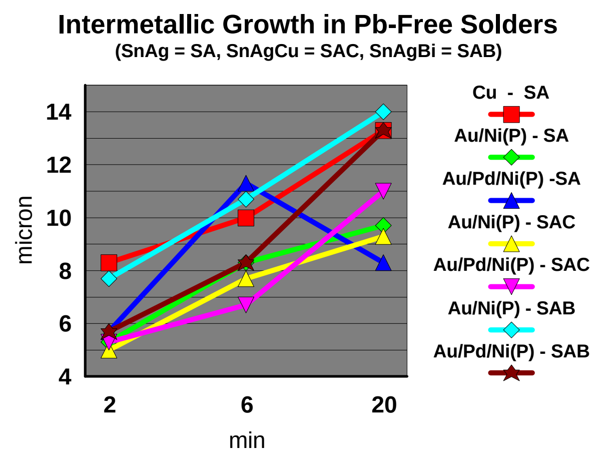

Fig. 7 Intermetallic Growth in Three Pb-Free Solders (Sn-Ag, Sn-Ag-Cu, Sn-Ag-Bi) at 250 °C.

However, the morphological dependency on the solder composition discussed above is not observed with electroplated Ni. Fig. 5 shows the intermetallics grown on an electroplated Ni layer at 250 °C, for 20 min for the three types of Pb-free solders. In all cases, the intermetallics are well adhered to the interface, even though there are some differences in the intermetallic thickness. Sn-Ag-Cu produces the largest thickness, then Sn-Ag-Bi, and then Sn-Ag. The intermetallic compounds shown with the Sn-Ag-Cu consist of two layers. The top layer is composed of Cu-rich, Sn-Cu-Ni ternary compound, and the bottom layer is composed of Ni-rich, Sn-Ni-Cu. It is postulated that the top layer is initially Cu_6Sn_5 substituting some of its Cu with Ni, while the bottom is initially Ni_3Sn_4 substituting some of Ni with Cu. This postulation can be confirmed by doing a X-ray micro-diffraction experiment on each layer to detect its crystal structure.

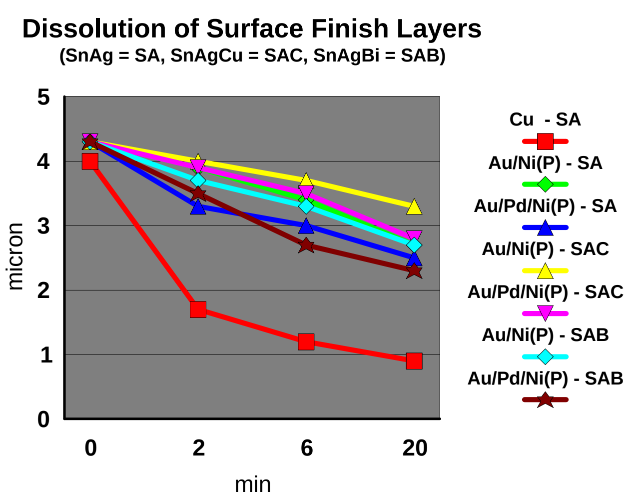

Fig. 6 Dissolution of Surface Finish in Pb-free Solders (Sn-Ag, Sn-Ag-Cu, Sn-Ag-Bi) at 250 °C

Conclusions

The interfacial reactions between three Pb-free solders (Sn-Ag, Sn-Ag-Cu, Sn-Ag-Bi) and several surface finishes commonly used in printed circuit boards were investigated in terms of the dissolution kinetics of surface finish layers, the intermetallic compound growth and the morphology of the interfacial intermetallic compounds. The following conclusions are drawn:

1. The dissolution rate of Au/Ni(P) or Au/Pd/Ni(P) is about one half of that of Cu at 250 °C.
2. Au/Ni(P) and Au/Pd/Ni(P) dissolve the least in Sn-Ag-Cu among three Pb-free solders at 250 °C.
3. Electroplated Ni dissolves much less in Sn-Ag and Sn-Ag-Bi than electroless Ni(P) does, but they dissolve similarly in Sn-Ag-Cu.
4. For Au/Ni(P) or Au/Pd/Ni(P), the intermetallic growth rates are ranked as SAB > SAC > SA.

5. In Sn-Ag, the intermetallic growth rates associated with three surface finishes are ranked as Cu > Au/Ni(P) > Au/Pd/Ni(P).
6. The intermetallic growth rates on electroplated Ni are two to three times smaller than those on electroless Ni(P) for all three Pb-free solders.
7. On Au/Ni(P) and Au/Pd/Ni(P), Sn-Ag-Cu produces Ni-Cu-Sn ternary intermetallic compounds which adhere well to the interface, while Sn-Ag and Sn-Ag-Bi yield Ni-Sn binary intermetallic compounds which separate or spall away from the interface.
8. In the electroplated Ni, all three Pb-free solders produce well adhered intermetallic compounds.

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Table I. Dissolution of Surface Finish Layers in Sn-Ag, Sn-Ag-Cu, Sn-Ag-Bi at 250 ºC

Solder & Surface finish	0 min (μm)	2 min (μm)	6 min (μm)	20 min (μm)	Total dissolution	Dissolution rate (μm/min)
Sn-3.5%Ag						
Cu (4 μm)	4.0	1.0-2.3	0.7-1.7	0 - 1.7	4.0	0.20
Au/Ni(P)/Cu	4.3	3.9	3.4	2.7	1.6	0.08
Au/Pd/Ni(P)/Cu	4.3	3.3	3.0	2.5	1.8	0.09
Sn-3.8%Ag-0.7%Cu						
Au/Ni(P)/Cu	4.3	4.0	3.7	3.3	1.0	0.05
Au/Pd/Ni(P)/Cu	4.3	3.9	3.5	2.8	1.5	0.08
Sn-3.5%Ag-3.0%Bi						
Au/Ni(P)/Cu	4.3	3.7	3.3	2.7	1.6	0.08
Au/Pd/Ni(P)	4.3	3.5	2.7	2.3	2.0	0.10

Table II. Intermetallic Growth in Sn-Ag, Sn-Ag-Cu, Sn-Ag-Bi at 250 ºC

Solder & Surface finish	2 min (μm)	6 min (μm)	20 min (μm)	Total intermetallics	Growth rate (μm/min)
Sn-3.5%Ag					
Cu (4 μm)	1.7-8.3	1.7-10.0	1.7-13.3	13.3	0.67
Au/Ni(P)/Cu	0.5-5.3	0.7-8.3	1.0-9.7	9.7	0.49
Au/Pd/Ni(P)/Cu	1.0-5.7	2.0-11.3	3.3-8.3	8.3	0.42
Sn-3.8%Ag-0.7%Cu					
Au/Ni(P)/Cu	1.7-5.0	3.0-7.7	5.0-9.3	9.3	0.47
Au/Pd/Ni(P)/Cu	2.3-5.3	3.3-6.7	5.0-11.0	11	0.55
Sn-3.5%Ag-3.0%Bi					
Au/Ni(P)/Cu	1.7-7.7	2.3-10.7	3.0-14.0	14	0.70
Au/Pd/Ni(P)	1.5-5.7	2.0-8.3	3.3-13.3	13.3	0.67

Table III. Dissolution of Ni Layers in Sn-Ag, Sn-Ag-Cu, Sn-Ag-Bi at 250 °C (electroless vs electroplated Ni)

Solder & Surface finish	0 min (μm)	2 min (μm)	6 min (μm)	20 min (μm)	Total dissolution	Dissolution rate (μm/min)
Sn-3.5%Ag						
Au/Ni(P)/Cu (electroless)	4.3	3.9	3.4	2.7	1.6	0.08
Au/Ni/Cu (electroplated))	4.7	4.3	3.8	3.6	1.1	0.055
Sn-3.8%Ag-0.7%Cu						
Au/Ni(P)/Cu (electroless)	4.3	4.0	3.7	3.3	1.0	0.05
Au/Ni/Cu (electroplated)	4.7	4.3	4.0	3.8	0.9	0.045
Sn-3.5%Ag-3.0%Bi						
Au/Ni(P)/Cu (electroless)	4.3	3.7	3.3	2.7	1.6	0.08
Au/Ni/Cu (electroplaed)	4.7	4.3	4.0	3.7	1.0	0.05

Table IV. Intermetallic Growth on Ni Layers in Sn-Ag, Sn-Ag-Cu, Sn-Ag-Bi at 250 °C (electroless vs electroplated Ni)

Solder & Surface finish	2 min (μm)	6 min (μm)	20 min (μm)	Total intermetallics	Growth rate (μm/min)
Sn-3.5%Ag					
Au/Ni(P)/Cu (electroless)	0.5-5.3	0.7-8.3	1.0-9.7	9.7	0.49
Au/Ni/Cu (electroplated)	0.3-1.0	1.0-2.3	1.2-3.3	3.3	0.17
Sn-3.8%Ag-0.7%Cu					
Au/Ni(P)/Cu (electroless)	1.7-5.0	3.0-7.7	5.0-9.3	9.3	0.47
Au/Ni/Cu (electroplated)	1.0-2.7	4.0-5.0	4.2-5.7	5.7	0.29
Sn-3.5%Ag-3.0%Bi					
Au/Ni(P)/Cu (electroless)	1.7-7.7	2.3-10.7	3.0-14.0	14.0	0.70
Au/Ni/Cu (electroplated)	0.7-1.7	1.3-2.3	2.0-5.0	5.0	0.25