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Testing Intermetallic Fragility on Enig upon Addition of Limitless Cu

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ABSTRACT

As reliability requirements increase, especially for defense and aerospace applications, the need to characterize components used in electronic assembly also increases. OEM and EMS companies look to perform characterizations as early as possible in the process to be able to limit quality related issues and improve both assembly yields and ultimate device reliability. In terms of BGA devices, higher stress conditions, RoHS compatible materials and increased package densities tend to cause premature failures in intermetallic layers. Therefore it is necessary to have a quantitative and qualitative test methodology to address these interfaces.

Typically, solder ball shear or pull testing is employed to measure the interfacial strength, sometimes requiring very high speeds to do so. While there is no current industry accepted specification on proper test speeds, strength or energy metrics, procedures do exist which allow for relevant comparisons. These tests are always run on unassembled BGA devices, so the interaction with the PCB is completely removed. While the data is useful for the component manufacturer, the risk is that the test does not fully represent the final assembly in terms of metallurgical condition. Specifically when BGA components using a Nickel-Gold surface finish are soldered to PCBs with a Cu-based pad (ie, Cu-OSP, ImmAg, ImmSn or HASL), there will be additional Cu dissolved into the solder joint. The addition of this copper can have an important effect on the intermetallic structure at the ENIG pad. Current mechanical solder ball testing procedures on unassembled BGA devices do not accurately duplicate the condition of this intermetallic structure. The test results on ENIG pads will then not necessarily correlate to actual manufacturing reliability.

From this research we have determined that generating an intermetallic morphology that is similar to a standard mass reflow surface mount process is not straight forward. The method used to add Cu to the ENIG pad and lead-free solder system will affect the morphologies at the electroless Ni substrate and therefore the mechanical properties of the intermetallic. Data is presented on the intermetallic strengths and failure modes of two bond pull test methods. Specifically Hot Bump Pull (HBP) and Cold Bump Pull (CBP) testing are compared where Cu is added by the copper pins of the HBP tester or by Cu power in a second reflow followed by CBP testing.

Key words: Limitless Cu, ENIG, intermetallic morphology, fragility, lead-free

INTRODUCTION

In this study we present results using a thermo-mechanical test technique which develops a similar intermetallic condition as seen during second level assembly. A careful study was conducted which examines the influence of solder alloy, reflow condition and test technique on the interfacial behavior for the most accurate replication of 2nd level attach without actually performing the attachment process. The above variables are used to qualitatively vary both the Cu and Ni concentrations within the solder joint, and the interaction between the formation of Ni_3Sn_4 and Cu_6Sn_5. Microstructural analysis was conducted and shows a difference in intermetallic morphology as a function of the additional copper. The testing results show that when we simulate 2nd level reflow onto a Cu-based board, the failure mode and ultimate interfacial strength are significantly affected. The consequence of this work suggests a more rigorous testing approach can be employed for specific condition.

BACKGROUND

Soldering of ENIG components to Cu substrates generates a condition of elevated Cu concentrations with the solder joint during surface mount assembly. Typical ENIG intermetallic composition consists of primarily Cu_6Sn_5 due to the high concentration of Cu in the solder system. However although composition is known the intermetallic morphology is often not. Morphology is dependent on the concentrations of the various elements in the system, diffusion and dissolution rates of the pad metallurgy, and reflow soldering profile. These morphologies will have varying mechanical strengths and therefore may be more susceptible to failure during manufacturing and reliability testing. Typical intermetallic morphologies seen on the ENIG surface can be seen in Figure 1. Where areas of thick Cu_6Sn_5 scalloped structures are adjacent to thin areas of intermetallic that may be Ni_3Sn_4 or Cu_6Sn_5. Typically in these mixed systems Ni and Cu atoms can substitute for one another with the matrix making the more accurate description of the intermetallic formed $(Cu,Ni)_6Sn_5$ and $(Ni,Cu)_3Sn_4$. For simplicity within this paper the former will be referred to in all future discussions.

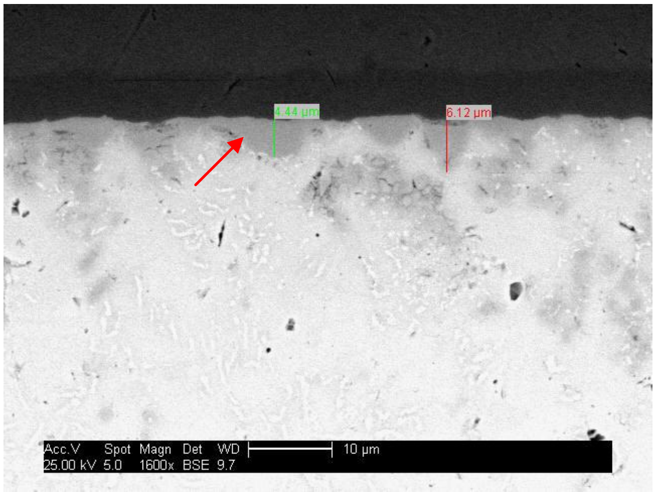


Figure 1: SEM cross-sectional micrograph of typical lead-free solder joint Cu_6Sn_5 formation at ENIG surface following assembly to Cu OSP board.

It has been shown in this research that the test method one selects to test the intermetallic can have profound results on the results. In addition a method must be developed to generate morphologies that better represent the structures shown in Figure 1 above.

Solder Alloys

All solder balls were acquired from a single supplier and are 99.9% pure. The alloys used for this research were;

1. Sn/Ag(3.5wt.%) - (SnAg)
2. Sn/Ag(3.0wt.%)/Cu(0.4wt.%) - (SAC304)
3. Sn/Pb(37wt.%) - SnPb

The SAC304 alloy was selected due to the ternary Cu-Ni-Sn phase diagram. SAC304 was created combining a Sn/Ag3.0wt.%/Cu0.5wt.% (SAC305) solder ball with a calculated Sn/Ag3.5wt.% print volume knowing the solid content of the paste, density of the alloy, and calculated print transfer efficiencies for a square stencil aperture of a given thickness. A stencil was purchased with this aperture size and transfer efficiencies were measured to be ~95% with a Cyberoptics laser volume measurement tool. Exact Cu concentration is not critical in this case. However we wanted to have repeatable results with an alloy contain less than 0.5 wt.% Cu and greater than 0.3 wt.% Cu since this is what has been described in literature to be the limits for the formation of the Ni_3Sn_4 and Cu_6Sn_5 intermetallic formation at the Ni boundary[1,2].

Substrates

An electroless Ni/immersion Au (ENIG) test board that was used in this work is a 12-layer PCB with a nominal copper to copper thickness of 2.12mm (.083”). The board was constructed with Matsushita HF-FR4 (Tg 148C) laminate material and PSR-4000 BL01 solder mask. Pad openings were 22.7 mil diameter solder mask defined. Phosphorous concentration was determined to be 12.6 wt.% in the bulk Ni when evaluating cross-section by EDS. Figure 2 shows the condition of the test board as-received.

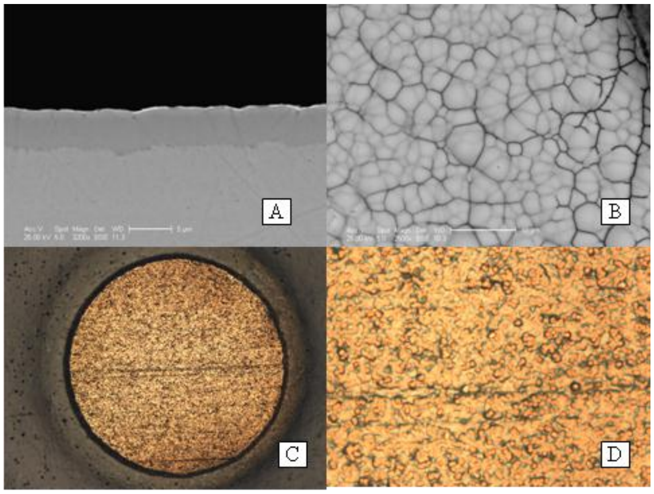


Figure 2: ENIG test board; A) SEM of cross-section, B) SEM of surface inspection ("mud flat" condition is typical for electroless Ni), C) optical image of entire pad, and D) 500x optical inspection of pad surface

In this study we compare our controlled experiment to a commercially available Intel SnPb device (Intel 845). The pad diameter for this device was measured to be 26.2 mil solder mask defined as compared to the 22.7 mils for the test board. Although these diameters are not exactly the same the bond testing performance should be comparable. We would expect the peak load to failure to be higher for this device due to the larger pad area.

Mechanical Test

CBP testing is a technique often used in electronics to test the mechanical properties of solder joints and laminates[3,4,5,6]. JESD22-B115A is the standard used for ball pull testing and the "A" revision has been released in August of 2010. Figure 3 and Figure 4 illustrate the pull testing apparatus and tweezer alignment respectively. A tweezer tool with a hollowed tip is used to grab the solder joint. Tweezers of similar diameter as the solder joint should be used in order to effectively distribute the gripping forces and minimize the deformation of the solder joint.

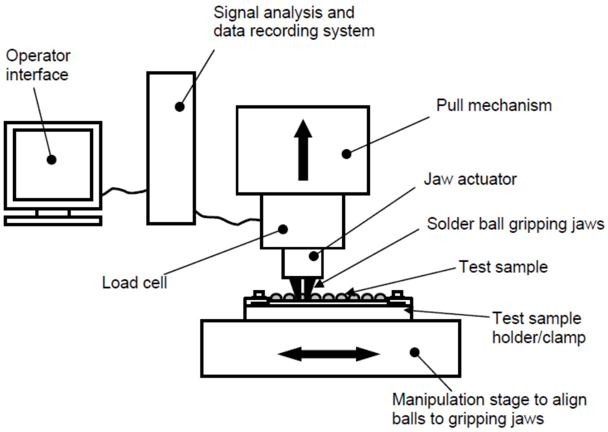


Figure 31: General solder ball pull apparatus[6]

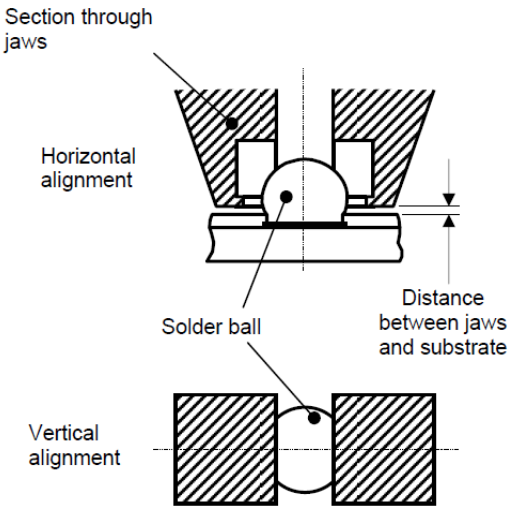


Figure 4: Tweezer alignment[6]

Solder joint deformation as well as tweezer alignment to the solder ball could affect the results of the test since the solder may not be placed under a uniform tensile stress across the pad. Tweezer alignment is described in the JEDEC standard (Figure 4) however it is very operator dependant and may not be performed properly. Any amount of torque or excessive deformation of the solder joint may result in changes in peak load to failure or failure mode. Therefore it is critical to test many solder joints of a given sample to accurately evaluate the repeatability of the data. It has been shown in this work that similar failure modes can be generated with a narrow distribution of peak loads to failure if proper tweezers are selected and care is taken in solder joint alignment.

HBP testing is an alternate technique for mechanical testing of solder joints in electronic devices. Historically the hot pin method is used in Japanese testing standards and has been adopted by the Military as acceptance criterion for laminate materials where a single unsupported land is repeatedly thermally stressed (Mil-P-5884D). Other standards also required thermal stressing of laminates and pads (IPC-6012, IPC-6013, and IPC-9708). This test method is significantly different than CBP testing in that heat is applied to the solder joint through a high purity Cu pin or a soldering iron Figure 5. Once the pin reaches a temperature above the melting point of the solder it is brought in contact with the solder at a pre-selected depth.

It is arguable that this process cannot be compared to CBP due to its affect on the solder joint intermetallic structures. The temperature gradient of this process and the addition of Cu from the pin to the solder system must be considered when testing solder joint intermetallics in this manner. Heating from a localized pin may also affect the Cu pad adhesion to the base dielectric and cohesion of the dielectric beneath the pad in the cases of pad cratering. This test is best suited for testing intermetallics of solder mask defined pads due to the additional strength of a larger pad defined by mask.

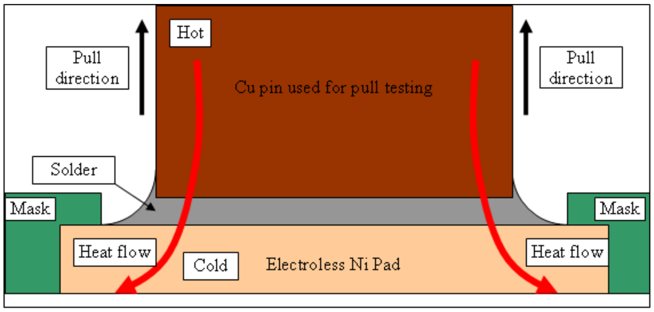


Figure 5: Illustration of pull testing procedure (not to scale) and heat flow direction

TEST METHOD

Intermetallic of various morphologies and compositions were created by soldering solder spheres of specific alloy to the ENIG substrate. These intermetallics were then either HBP tested or subjected to a second reflow with Cu powder and then CBP tested.

Ball Attach

All solder joints were reflowed in nitrogen environments using a Vitronics Soltec 10 zone convention reflow oven. For the first reflow process (ball attach) a short (~20 second) and long (~120 second) time above the reference temperature of 217ºC was used for the lead free solder alloys Sn/3.5Ag and SAC304. Similar times above the reference temperature 183ºC were used for the SnPb 25mil solder balls. A peak temperature was measured for the long profile to be 208ºC for SnPb and 236ºC for the lead free. For the short profiles peak temperatures were limited by duration above the reference temperature however for SnPb 185ºC was achieved and 226ºC for the lead-free alloys.

Addition of Cu by Powder

Copper powder with a nominal diameter of 20 microns was purchased from Advanced Powder Products. This powder was mixed with Kester no-clean tacky flux (TST-6592LV) and printed on a silicon wafer using a glass slide and 0.007" feeler gauges as standoffs. Bumped test boards and the Intel 845 device were dipped into this powder/flux mixture and then reflowed in Nitrogen. Images of the power dipped Intel 845 device pre and post reflow are shown in Figure 6.

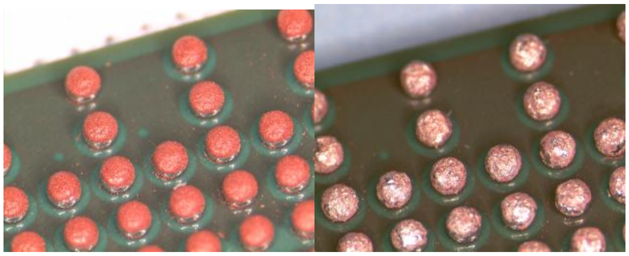


Figure 6: Device dipped in Cu power pre and post reflow respectively

In the second reflow the Cu powder was mixed with tacky flux and mixed with the solder joint providing a limitless source of Cu. A 60 second time above reference temperate was used since this is more representative of standard assembly processes used in electronics manufacturing. This second reflow has provided adequate time for the formation of Cu_6Sn_5 at the electroless Ni substrate. Peak temperatures for these profiles were 213ºC for SnPb and 242ºC for lead free.

Intermetallic formations following the addition of Cu were unique for the lead free cases. Morphological differences observed optically seemed to have evidence of precipitation of Cu_6Sn_5 above the pad surface for the long attach profile samples (Figure 7 C & D) and Cu_6Sn_5 intermetallic at the electroless Ni surface for short ball attach profiles (Figure 7 A &B).

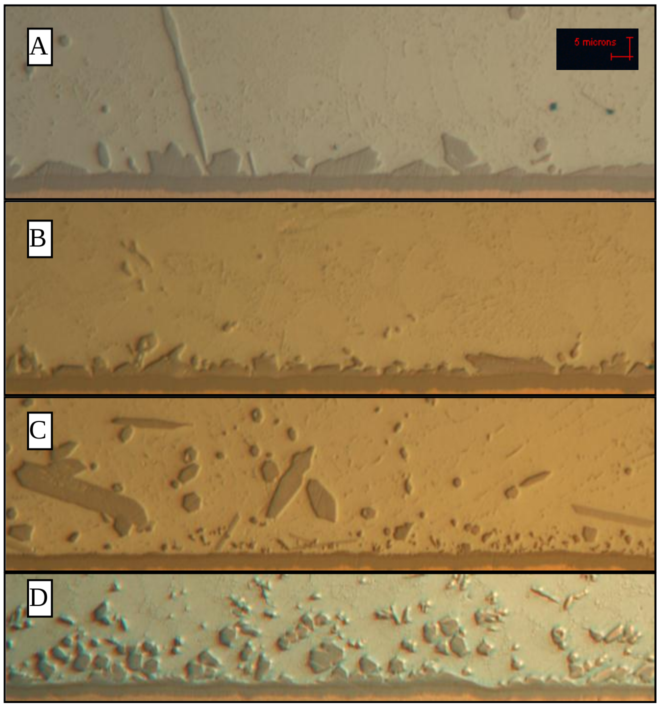


Figure 7: 500x optical inspection lead-free alloys following addition of Cu powder (A) SnAg Short (B) SAC304 Short (C) SnAg Long (D) SAC304 Long

SEM inspection of the lead free systems suggest that the SnAg Long (Figure 8C) had a unique morphology near the electroless Ni pad surface. Specifically a thinner intermetallic was observed in direct contact with the pad.

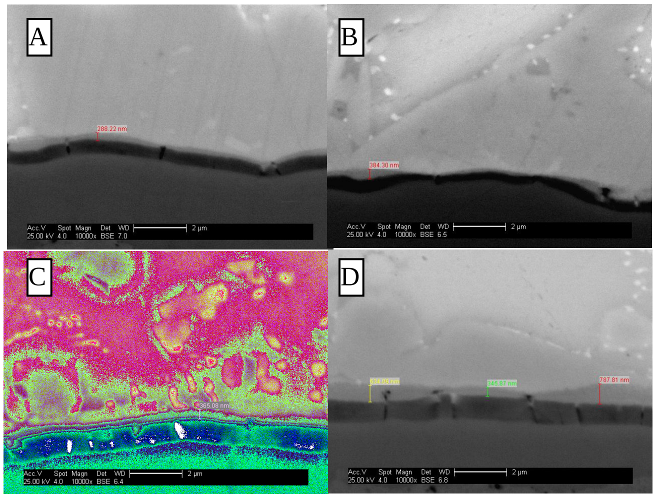


Figure 8: 10,000x SEM images of lead-free morphologies following addition of limitless Cu (A) SnAg Short (B) SAC304 Short (C) SnAg Long (D) SAC304 Long

Following addition of Cu powder the intermetallic variation for the SnPb samples was far less dramatic than the lead free samples. The long and short ball attach profile seemed to have little affect on the intermetallic morphology as determined by optical inspection (Figure 9) and SEM (Figure 10).

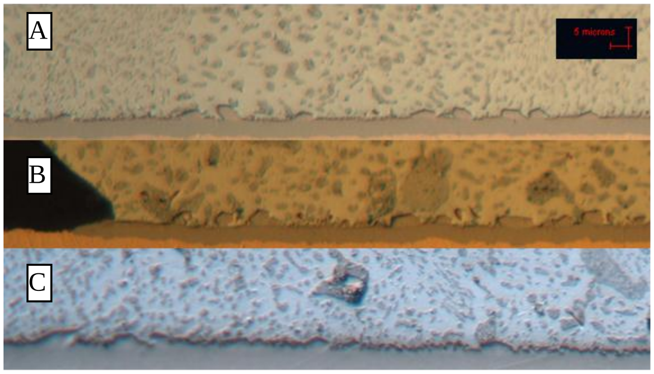


Figure 9: 500x optical images of SnPb morphologies following addition of Cu powder (A) SnPb Short (B) SnPb Long and (C) Intel 845

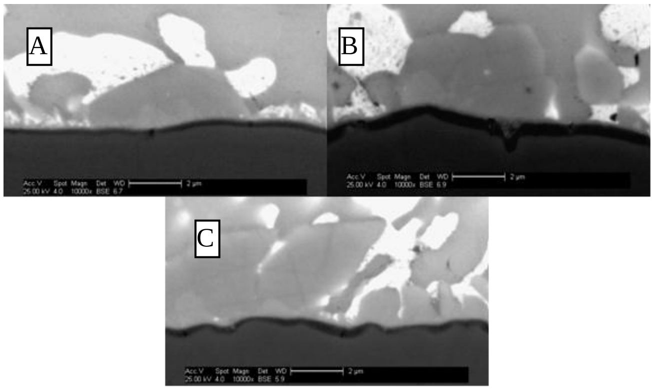


Figure 102: 10,000x SEM images of morphology IX (A) SnPb Short (B) SnPb Long and (C) Intel 845

Cold Bump Pull (CBP) Testing

CBP was performed on a Dage 4000 bond tester (Figure 11) using a tweezer of diameter 30 mils and a CBP5kg load cell. Pull rates were determined based upon the frequency of brittle failure modes observed. A speed was selected that generated nearly 100% brittle failure at the electroless Ni substrate.

Figure 11: Dage 4000 slow speed bond tester

Hot Bump Pull (HBP) Testing

HBP testing was also performed on the Dage 4000 bond tester however a 30mil diameter pin is heated and soldered to the solder joint using a HBP10kg lead cell. This load cell has an integrated heating element controlled by a temperature controller. Exact temperatures are difficult to quantify since the equipment thermocouple is attached to the heating element and not the tip of the Cu pin. A temperature was selected that induced reflow of the solder joint. The pin was brought in contact with the ENIG pad for approximately 10 seconds during the soldering process in order to ensure complete reflow and mixing of the solder joint.

Prior to performing the pull testing on a solder joint the heating element temperature was monitored to be less than 30ºC. Cooling of the pin is accomplished by a compressed air nozzle near the heating element. Pin and heating elements are identified in the image of the HBP10kg head image in Figure 12. Care was taken not to disturb the liquid solder joint during the cooling of the pin.

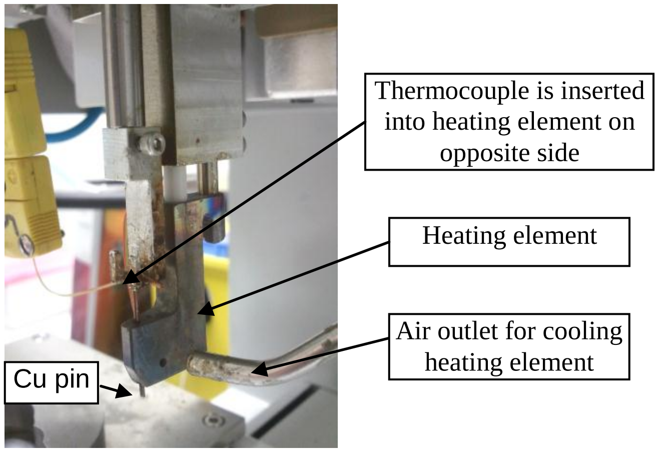


Figure 12: HBP10Kg load cell

Several solder joints were reflowed followed by pin removal from the liquid solder in order to cross-section the solder joint and inspect the intermetallic condition. Intermetallic structures following the reflow soldering of the HBP tester Cu pin appeared very similar in SEM analysis of the solder joints. Lead free solder joints all appeared to have similar thickness intermetallic at the electroless Ni boundary (Figure 13). A similar observation was also made of the SnPb solder samples (Figure 14).

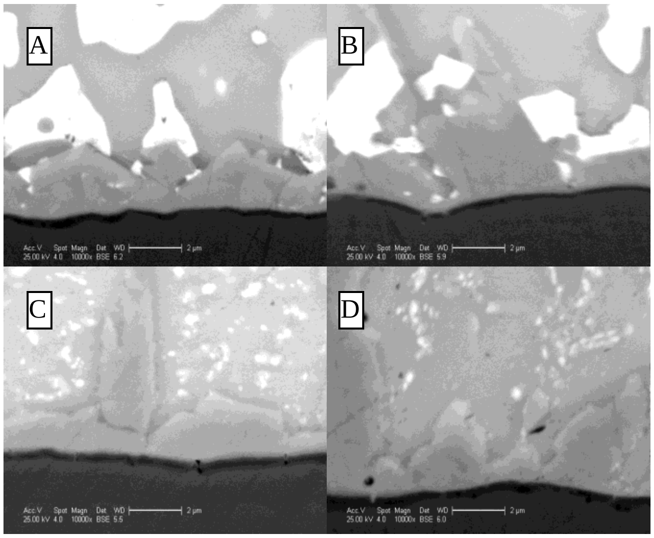


Figure 13: 10,000x SEM inspection of HBP intermetallic morphologies for Intel and Short ball attach profiles (A) Intel 845, (B) SnPb, (C) SnAg, (D) SAC304

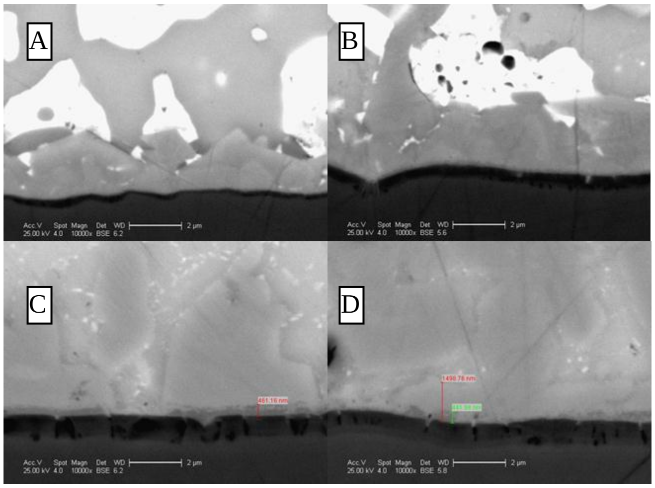


Figure 14: 10,000x SEM inspection of HBP intermetallic morphologies for Intel and Long ball attach profiles (A) Intel 845, (B) SnPb, (C) SnAg, (D) SAC304

EXPERIMENTAL RESULTS

In both HBP and CBP testing the failure mode produced were brittle failures within the intermetallic at the electroless Ni substrate (Figure 15). As an aggregate the average peak load to failure of the HBP test samples was far lower than the CBP test procedure. This suggests either a dramatic decrease in the intermetallic strength or a difference in the mechanical loading between these two tests.

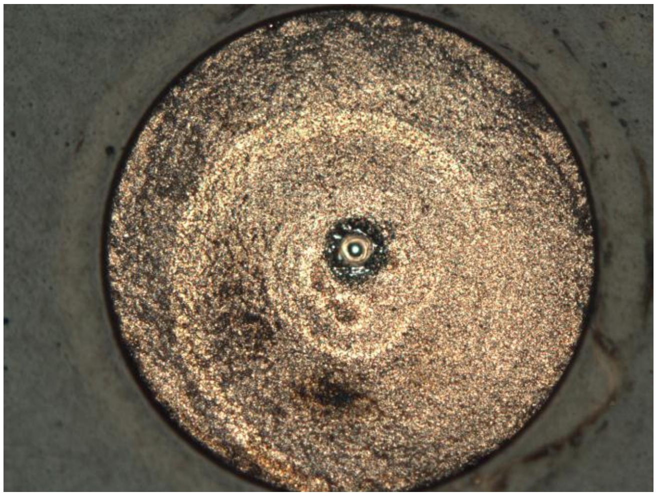


Figure 15: HBP brittle failure mode example

Peak load to failure and failure mode in HBP testing was nearly identical for all solder and ball attach variations. The HBP testing method developed for this research has mitigated the intermetallic morphology variability during ball attach and alloy selection as shown in Figure 16. Ultimately all solder joints failed in a brittle failure mode with similar fracture morphologies as shown in Figure 16 and Figure 17. Inspection of the fracture surfaces revealed that fractures for the test board were through the $(Cu,Ni)_6Sn_5$ intermetallic at the pad surface.

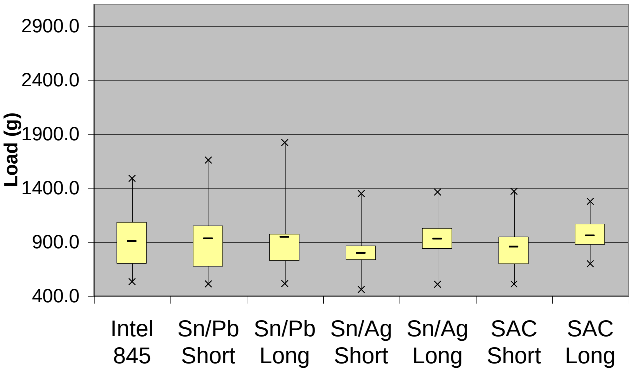
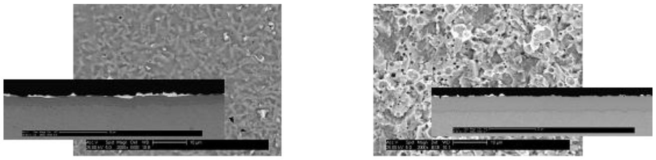


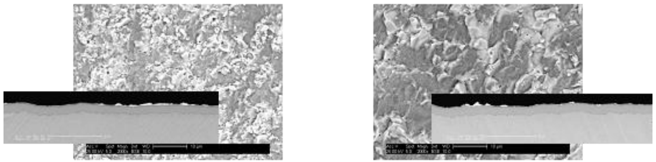
Figure 16: HBP test results between all alloy and process variations

Further research needs to be conducted of this test method in order to develop a process that produces variability with solder alloy and ball attach process as seen in the CBP testing.



Intel HBP

SnPb Short HBP



SnAg Short HBP

SAC304 Short HBP

Figure 17: HBP Short profile fracture morphology comparison to Intel 845

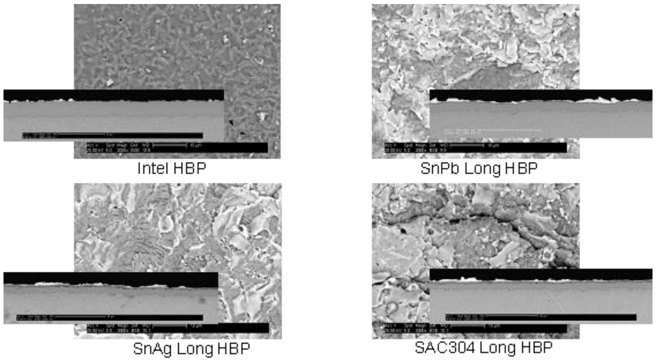


Figure 18: HBP Long profile fracture morphology comparison to Intel 845

In CBP testing peak load to failure distributions showed a marked difference between the various lead free morphologies formed during ball attached and subsequent reflow with copper powder. All but one lead-free case produced a large standard deviation in pull testing. Only the SnAg Long ball attach sample resulted in a narrow peak load distribution similar to the SnPb cases as shown in Figure 19. This suggests that the SnAg Long sample has more consistent intermetallic properties from ball to ball. The fracture surfaces were compared and the SnAg long ball attach process seemed to have slightly more intermetallic remaining at the pad surface than the other lead free samples as shown in Figure 20. Although brittle failures modes are not favorable in electronics packaging, having consistent result does improve an Engineer's ability to predict characteristic life of the device in mechanical accelerated life testing.

The SnPb samples by comparison also failed in a narrow distribution of peak load to failure. This has driven the average up and arguably reduces the possibility of infant mortalities in mechanical stress conditions. Failure modes were similar for the control sample however the Intel 845 device exhibited a different fracture characteristic with more intermetallic remaining at the electroless Ni pad surface following ball removal as shown in Figure 21.

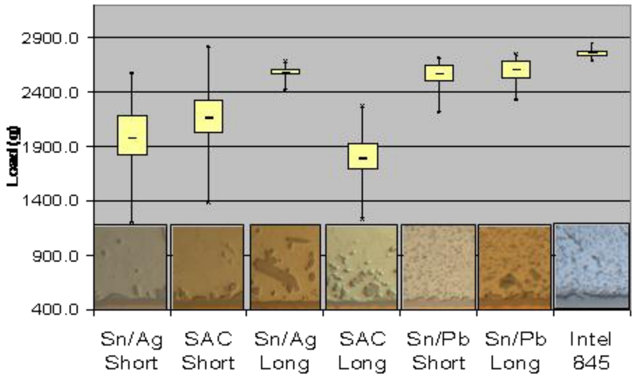


Figure 19: CBP load to failure distribution for various morphologies

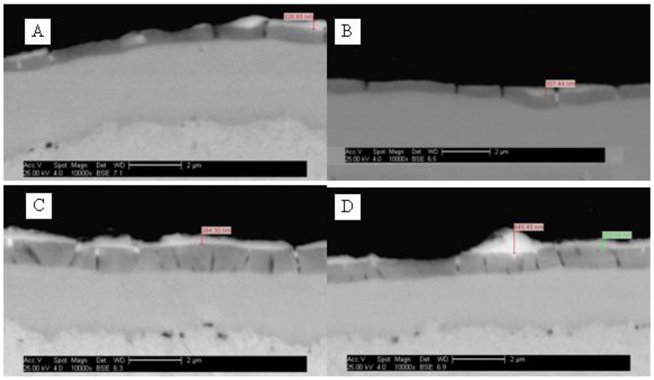


Figure 20: Cross-sectional SEM comparison of CBP fracture surfaces (A) SnAg Short (B) SAC304 Short (C) SnAg Long (D) SAC304 Long

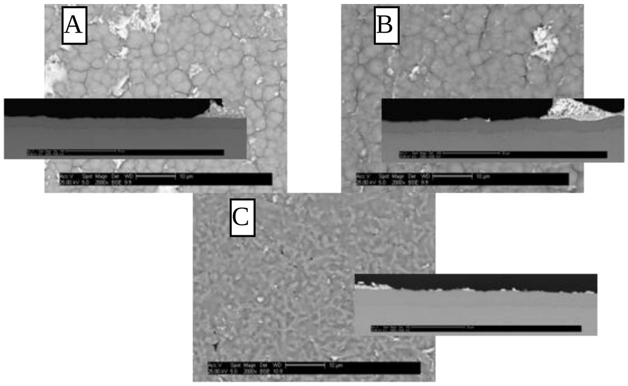


Figure 21: SEM SnPb fracture morphology comparison (A) SnPb Short (B) SnPb Long and (C) Intel 845

CONCLUSIONS

Intermetallic morphology affects the peak load to failure distribution in CBP testing and a possible narrow distribution can be created for SnAg using a long ball attach profile. This condition is favorable for reliability predictions and mitigation of infant mortalities for lead free product.

More research must be conducted to compare the intermetallic morphologies created in these tests with those observed in standard component attach. Since individual solder joints cannot be mechanical tested in a BGA the only comparison that can be made is through intermetallic morphological comparison.

An improved HBP test method may provide pull testing results that vary based upon ball attach method and solder alloy. Current equipment offerings can mimic pin temperatures that are indicative of a mass reflow as shown in Figure 22.

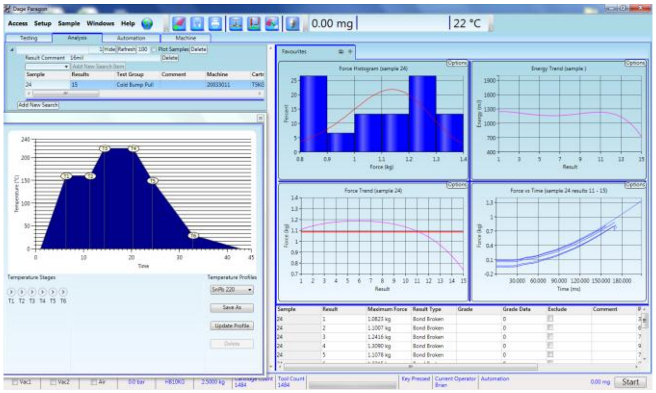


Figure 22: HBP reflow pin temperature profile used for current research into this test method

FUTURE RESEARCH

Further testing of the HBP test is required to better mimic a surface mount process. Research is being developed to test a standard ramp, soak, reflow soldering profile as well as pins of various alloys. Test methods for HBP testing may only be applicable for devices attached to circuit boards with Ni substrates since intermetallic formation rates are far slower and the system is less elementally complex.

ACKNOWLEDGEMENTS

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REFERENCES

[1] D.Q. Yu, C.M.L. Wu, D.P. He, N. Zhao, L. Wang, J.K.L. Lai,. “Effects of Cu contents in Sn–Cu solder on the composition and morphology of IMCs at a older/Ni interface.”, J. Mater. Res., Vol. 20, No. 8, Aug 2005

[2] R.C. Kao, “Cross-Interaction Between Cu and Ni in Lead-Free Solder Joints” APEX Presentation March 12th 2006.

[3] R. Pandher, and A. Pachamuthu, “Effect of Multiple Reflow Cycles On Solder Joint Formation And Reliability” SMTAi 2010 Conference Proceedings.

[4] B. Roggeman, and W. Jones, “Characterizing the Lead-Free Impact on PCB Pad Craters”, APEX 2010 Conference Proceedings.

[5] G. Milad, D. Gudeczauskas, G. O’Brien, and A. Gruenwald. “A Study of the ENEPIG IMC for Eutectic and LF Solders” SMTAi 2010 Conference Proceedings.

[6] JEDEC JESD22-B115A, “Solder Ball Pull” JEDEC Solid State Technology Association, August 2010