Assembly of Lead-Free Bumped Flip-Chip With No-Flow Underfills

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***Abstract*—Lead-free solder reflow process has presented challenges to no-flow underfill material and assembly. The currently available no-flow underfill materials are mainly designed for eutectic Sn–Pb solders. This paper presents the assembly of lead-free bumped flip-chip with developed no-flow underfill materials. Epoxy resin/HMPA/metal AcAc/Flux G system is developed as no-flow underfills for Sn/Ag/Cu alloy bumped flip-chips. The solder wetting test is conducted to demonstrate the fluxing capability of the underfills for lead-free solders. A 100% solder joint yield has been achieved using Sn/Ag/Cu bumped flip-chips in a no-flow process. A scanning acoustic microscope is used to observe the underfill voiding. The out-gassing of HMPA at high curing temperatures causes severe voiding inside the package. A differential scanning calorimeter (DSC) is used to study the curing degree of the underfill after reflow with or without post-cure. The post-curing profiles indicate that the out-gassing of HMPA would destroy the stoichiometric balance between the epoxy and hardener, and result in a need for high temperature post-cure. The material properties of the underfills are characterized and the influence of underfill out-gassing on the assembly and material properties is investigated. The impact of lead-free reflow on the material design and process conditions of no-flow underfill is discussed.**

***Index Terms*—Flip-chip, lead-free solder, material properties, no-flow underfill, solder joint yield, underfill out-gassing.**

I. INTRODUCTION

THE TREND in electronics of today is not only smaller products with more functions, better performance and lower price, but also greener, more environmentally friendly products. Lead, a major component in solder, has long been recognized as a health threat to human beings. The common clinical types of lead poisoning may be classified according to their clinical picture as

a) alimentary;
b) neuromotor;
c) encephalic [1].

The major concern about lead in electronics is that the lead from disposed landfill could leach out into underground water and eventually into drinking water [2]. The attempt to ban lead from electronic solder was initiated in the U.S. Congress in 1990. However, the lead-free movement has advanced much more rapidly in Japan and Europe [3]. The leading Japanese Original Equipment Manufacturers (OEMs) have introduced products that contain no lead in the interconnects. Although the WEEE (Waste from Electrical and Electronic Equipment) Directive has put European Union's lead-free legislation back to 2008 [4], many companies such as Ericsson, Nokia, and Philips have already implemented "green" marketing strategies into their new products. The difference in lead-free progress has triggered great concerns of lead-containing solder users about maintaining business opportunity, therefore further expediting the advancement of lead-free soldering programs. Hence, lead-free electronics is not only perceived as a health issue, but a result of government and commercial drivers as well [5].

TABLE I
POSSIBLE LEAD-FREE ALLOYS

Alloy	Melting Point
Sn96.5/Ag3.5	221 °C
Sn99.3/Cu0.7	227 °C
Sn/Ag/Cu	217 °C (Ternary eutectic)
Sn/Ag/Cu/X(Sb, In)	Ranging according to compositions, usually above 210 °C
Sn/Ag/Bi	Ranging according to compositions, usually above 200 °C
Sn95/Sb5	232 – 240 °C
Sn91/Zn9	199 °C
Bi58/Sn42	138°C

Possible lead-free candidates and their melting points are listed in Table I [6]. Among these alloys, only Sn/Bi has a melting point lower than that of the eutectic Sn/Pb alloy (183 °C), which is commonly used in electronic interconnects. However, the concern that an eutectic Bi/Pb/Sn (96 °C) formed on the Pb surface finishes makes bismuth incompatible with the currently existing infrastructure for the transition stage. Much attention has been focused on tin alloys with silver and/or copper. Several sources have reported good thermal fatigue properties for these alloys as compared to lead-tin solder [7]. The ternary Sn/Ag/Cu has demonstrated to be the most promising for lead-free applications. The optimal composition 95.4 Sn/3.1Ag/1.5Cu has provided a combination of good strength, fatigue resistance and plasticity [8]. In addition, the alloy has sufficient supply and adequate wetting characteristics. It appears to be the most promising lead-free candidate in the industry.

The use of Sn/Ag/Cu solder presents two major challenges on the process. First, the melting point of the alloy is more than 30 °C higher than that of the eutectic Sn/Pb alloy, so the process temperature is raised by 30–40 °C. The high process temperature has a great impact on the substrate since the conventional FR-4 material has a Tg at around 125 °C. Higher warpage is introduced when the board is subjected to higher temperature reflow. There has been considerable work in high Tg substrate for lead-free process, especially in Japan. The second challenge comes from the flux chemistry. The current fluxes in use are

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usually designed for eutectic Sn/Pb solder, so they either do not have high enough activity or do not possess sufficient thermal stability at higher temperatures. Generally, the wetting behavior of the lead-free solders is not as good as that of the eutectic Sn/Pb solder [9], [10].

As a result of the advances in IC packaging technology, flip-chip on organic substrates has been developed and practiced for more than ten years. Underfill technology is one of the keys to high reliability since it has addressed the problem of thermo-mechanical stress caused by the CTE mismatch between the silicon chip and the organic substrate [11], [12]. However, conventional underfill technology has relied on capillary flow, which needs separate flux dispensing, flux cleaning, solder bump reflow, underfill dispensing and flow, and off-line underfill curing procedures [13]. As such, the conventional underfilling process is tedious, expensive, and not transparent to the surface mount technology (SMT) facilities. Recent development in the no-flow underfill has integrated the flux into underfill, eliminated the capillary flow of underfill, and combined underfill cure together with solder reflow process [14], [15]. A successful no-flow underfill material should have high enough curing latency so that the underfill would not gel before the solder joints are connected in a reflow process. Also the material needs self-fluxing capability to eliminate the oxides on the surface of solder bumps to facilitate the wetting of solder on the contact pads. The first successful no-flow underfill material was developed and patented by Wong and Shi [16]. Many flip-chip assemblies have been built and successfully demonstrated using no-flow assembling process [17], [18].

The impacts of the lead-free process on the no-flow underfill are two-fold. The underfill material needs even higher latency in terms of curing. Currently available no-flow underfills are usually designed for eutectic Sn/Pb solder reflow process. Under high temperature during lead-free solder reflow, these underfills start to gel before the melting point of the lead-free solder is reached so that the solder is restricted and cannot wet the surface of the contact pad when it melts. On the other hand, the fluxing agent incorporated into the no-flow underfill might not have enough fluxing capability for lead-free solder. Research on the no-flow process for lead-free bumped flip-chip is not as common as those conducted on the eutectic Sn/Pb solders. Pioneered by Ericsson Mobile Communications, a joint project between Georgia Tech, Ericsson, and Flip Chip Technologies has focused on using lead-free solders and halogen-free boards for low cost flip-chip applications. Based on the early evaluations, the basic wettability tests showed that no no-flow underfill currently on the market is compatible with the chosen Sn/Ag/Cu solder [19]. So there is a need for a new no-flow underfill chemistry for lead-free solder bumped flip-chip applications.

The previous studies conducted by the authors have demonstrated that epoxy/anhydride/metal acetylacetonate (AcAc) system has the potential as a no-flow underfill for lead-free solder bumped flip-chip applications [20]. Several formulations have high curing peak temperatures and allow lead-free solder to wet on the copper board. This paper will present the process of lead-free bumped flip-chip using no-flow underfills.

TABLE II
COMPOSITION OF THE DEVELOPED NO-FLOW UNDERFILLS

	Epoxy	Hardener	Catalyst	Fluxing Agent
NF-A	EPON 8281 (100)	HMPA (76.4)	Be (II) AcAc (0.71)	Flux G (4.4)
NF-B	EPON 8281 (100)	HMPA (76.4)	Be (II) AcAc (0.71)	Flux G (7.06)
NF-C	EPON 8281 (100)	HMPA (76.4)	Co (II) AcAc (0.71)	Flux G (4.4)
NF-D	EPON 8281 (100)	HMPA (76.4)	Co (II) AcAc (0.71)	Flux G (7.06)

II. EXPERIMENTAL

A. Materials

Based on the previous study [20] of the curing behavior of epoxy/anhydride/metal AcAc/flux G system, four no-flow underfill formulations were developed for assembly and characterization. The four formulations were named as NF-A, NF-B, NF-C, and NF-D. Their compositions are listed in Table II and the parts based on weight are listed in the parenthesis.

B. Solder Wetting Test

A solder wetting test was designed to evaluate whether the no-flow underfills possess desirable curing behavior and can provide sufficient fluxing capability. The lead-free solder spheres used for the wetting test were composed of 95.5Sn/3.5Ag/1.0Cu. A copper foil laminated FR-4 board was used as the substrate in the wetting test. The boards were cleaned by the following procedure: 5 min immersion in acetone, 5 min immersion in methanol, then 20 s immersion in 50/50 HCl/H_2O solution, followed by DI water rinse and clean air-jet drying.

In the solder wetting test, the solder spheres were placed on the copper foil. Then a drop of the underfill was applied to immerse the spheres. The test vehicle then went through a seven-zone forced convention BTU International Paragon 98N reflow oven. The spreading of the solder melt on the copper board was observed by the optical microscope.

C. Assembly

The lead-free solder bumped chip used was a 200 μm pitch periphery bumped (PB8) daisy chained chip. The chip had a silicon nitride passivation layer and a Under Bumping Metallurgy (UBM) of Al/Ni–V/Cu. The chips were supplied by Flip Chip Technologies. The lead-free alloy composition was 95.5Sn/3.5Ag/1.0Cu. The solder bumps were approximately 115 μm in diameter and 100 μm in height. Two different size chips were used for the assembly. The smaller one was 5 $\times$ 5 mm^2 and had 88 bumps around the perimeter. The larger one was 10 $\times$ 10 mm^2 and had 352 bumps, as shown in Fig. 1.

The substrates used for assembly were supplied by Ericsson, Sweden. The board trace metallization was copper with electroless Ni–Au. The boards were designed to allow electrical continuity testing of each chip as a complete circuit.

All flip chips were placed using a high speed Siemens SIPLACE F5-DCA placement machine. The substrates used for assembly were baked at 125 °C for two hours prior to the dispensing of no-flow underfill to eliminate the moisture absorbed by the substrate material.

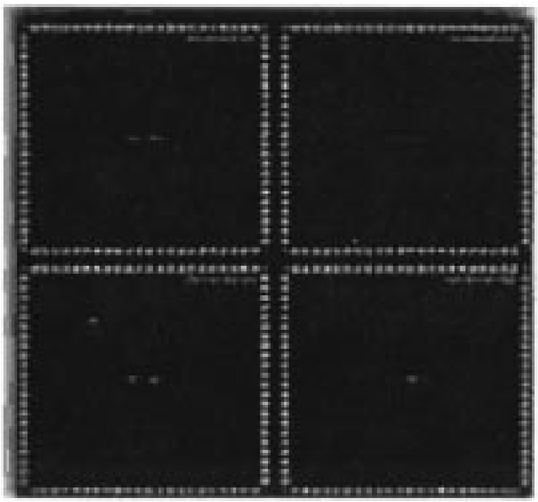

Fig. 1. Picture of 10 mm PB8 test die.

D. Characterizations

C-Mode Scanning Acoustic Microscope (C-SAM): After the assembly of the flip-chip, a Sonoscan D6000 scanning acoustic microscope was used in C-mode to observe the underfill-die passivation interface to investigate the underfill voiding, outgassing and interface delamination.

Underfill Curing Degree: In order to study the curing degree of the underfill after reflow or/and postcure, a drop of the underfill was placed on the copper board and went through the reflow oven. The underfill was then post-cured at 160 °C for 45 min. Then a piece of the underfill sample was cut and placed into a differential scanning calorimeter (DSC) furnace and was heated from room temperature to 350 °C at a heating rate of 5 °C/min under N_2 purge. The DSC heat flow was used to determine the residual curing latent heat. Compared with the original curing latent heat of this underfill, the curing degree can be calculated as follows:

$$\%C = \frac{\Delta H_a - \Delta H_p}{\Delta H_a} \times 100\%$$

where ΔH_a is the overall curing latent heat of the sample; ΔH_p is the curing latent heat of the sample after reflow or/and after post-curing.

Underfill Material Properties: In order to investigate the thermo-mechanical properties of the underfill samples, a liquid formulation was heated in an aluminum pan to 250 °C in a convective oven at about 3 °C/min heating rate. The sample was continuously cured in the oven at 250 °C for additional 15 min. After that, the sample was taken from the oven and cooled to room temperature. The sample was then polished and cut, and tested using thermal mechanical analyzer (TMA) and dynamic mechanical analyzer (DMA). A square sample about 5 × 5 mm^2 in dimension and 1–2 mm in thickness was heated in a TMA (by TA Instruments, Model 2940) to about 250 °C at a heating rate of 5 °C/min. The coefficient of thermal expansion (CTE) was obtained from the thermal expansion displacement versus temperature. The inflection point of thermal expansion was defined as TMA Tg. The CTE before TMA Tg was defined as α_1 and that after Tg as α_2. The specimen for DMA testing is about 30 × 10 mm^2 in dimension and 1–2 mm in thickness. The measurement was performed in a DMA (by TA Instruments, Model 2980) under the single cantilever mode with 1 Hz sinusoidal strain loading from room temperature to 200 °C at a heating rate of 3 °C/min. Storage modulus (G′), loss modulus (G″), and loss angle ($\tan\delta$) were calculated by the installed software.

A Thermal Gravimetric Analyzer (TGA, by TA Instruments, Model 2050) was used to investigate the weight loss of the formulations during curing. A sample of ~40 mg of a liquid formulation was placed into a platinum TGA sample pan. The sample was then heated at 10 °C/min to about 280 °C under N_2 purging. The weight difference between 25 °C and 270 °C was taken as the weight loss percentage of the formulation during curing.

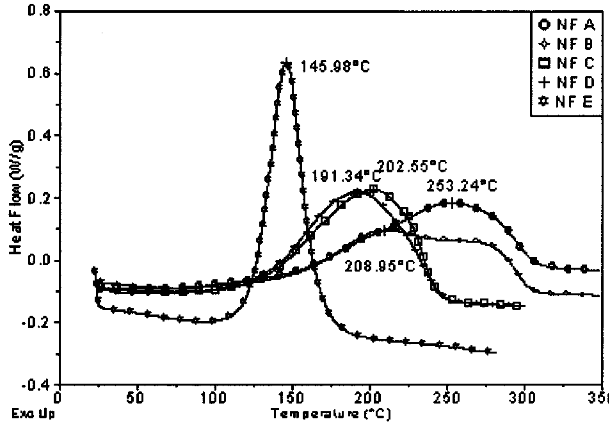

Fig. 2. DSC measured curing profiles of developed underfill formulations.

III. Results and Discussion

A. Solder Wetting Test

In the solder wetting test, the four no-flow underfills NF-A through NF-D were in-house developed for lead-free reflow applications. One commercially available no-flow underfill was used as the benchmark and named as NF-E. NF-E was designed for eutectic Sn/Pb solder reflow process. At the time of experiment, no commercial no-flow underfills designed for lead-free solder reflow were available.

The DSC heat flow of the five no-flow underfill materials (NF-A through NF-E) are shown in Fig. 2. NF-E had a curing peak temperature at around 145 °C and was significantly lower than those of the other four formulations. Within the four developed no-flow underfills, NF-A and NF-B had higher curing temperatures than NF-C and NF-D.

The five no-flow underfills were subjected to the solder wetting test using Sn/Ag/Cu solder balls and copper laminated FR4 board in a procedure described in the experimental session. The spreading of the Sn/Ag/Cu solder on the copper after reflow are shown in Fig. 3. It was observed that NF-A, B, and C allowed lead-free solders to wet on the copper board while NF-D and NF-E did not. Notice that NF-A, B, C, and D used the same fluxing agent and the concentration of fluxing agent in NF-B and NF-D was of a high level. So there should be sufficient fluxing capability in NF-D. However, since the curing temperature of NF-D was lower than the other three, NF-D cannot pass

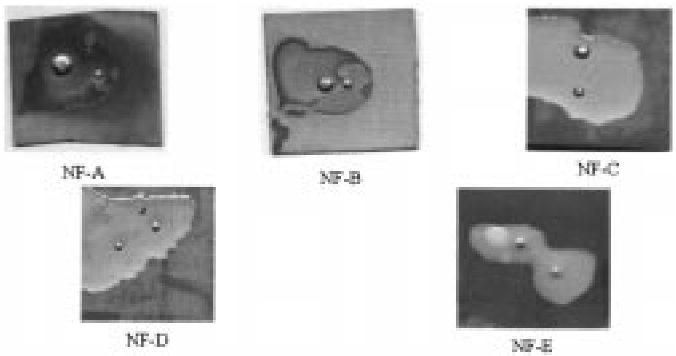

Fig. 3. Wetting of the lead-free solders on copper board.

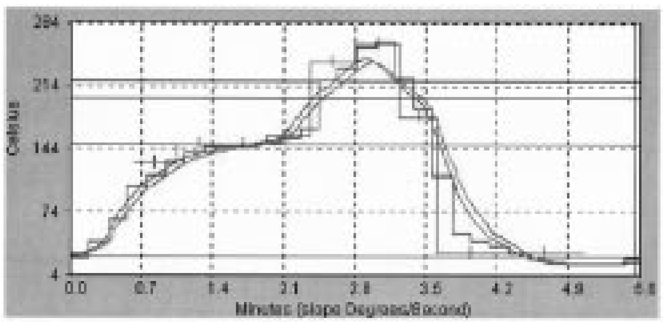

Fig. 4. Standard reflow profile.

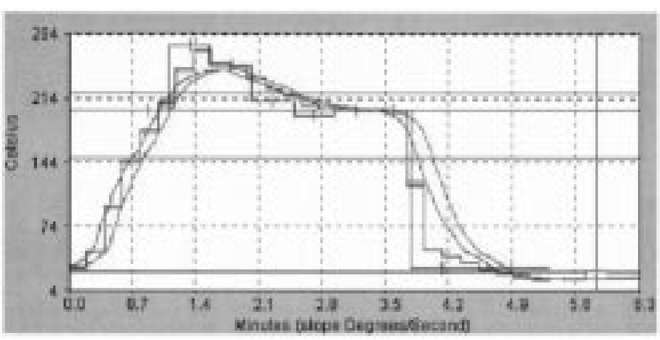

Fig. 5. Inversed reflow profile.

the solder wetting test under the reflow profile in this study. The result of the solder wetting test indicated that a high curing temperature was desired for lead-free reflow process. In this particular case, a curing peak temperature higher than 200 °C may be required to ensure the wetting of the lead-free solder ball on the copper board.

B. Assembly of Lead-Free Bumped Flip-Chip

The lead-free bumped flip-chip-on-board packages were built using NF-C. Two different reflow profiles were implemented. The two reflow profiles are as shown in Figs. 4 and 5. The profile in Fig. 4 was a standard reflow profile for surface mount applications, where there was a flux activation plateau at around 150 °C and a quick cooling down after reaching the solder melting. The profile in Fig. 5 was an inversed profile when the peak temperature was quickly reached followed by a prolonged post-curing period at around 200 °C. With both reflow profiles, 100% interconnection yields were achieved. Fig. 6 shows a picture of the assembly.

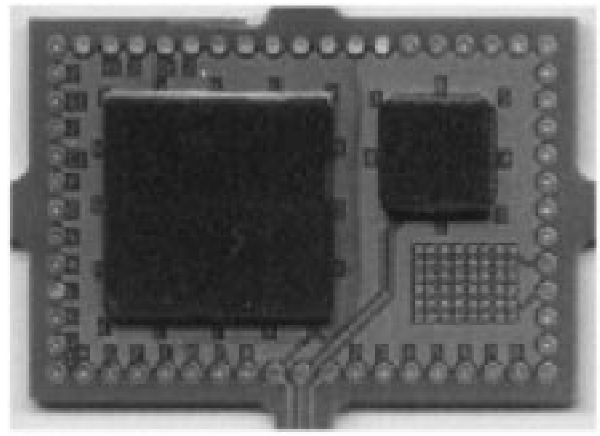

Fig. 6. Assembly of lead-free bumped flip-chip using developed no-flow underfill.

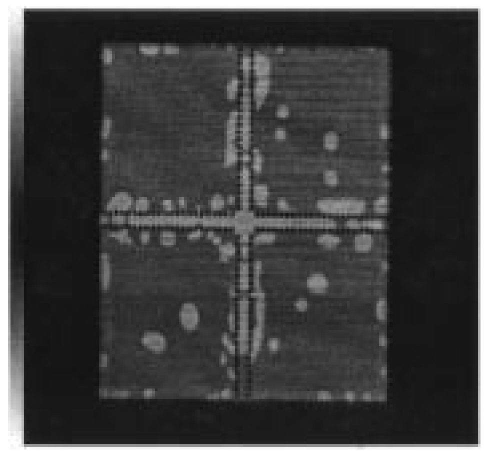

Fig. 7. C-SAM image of the assembly after standard reflow profile.

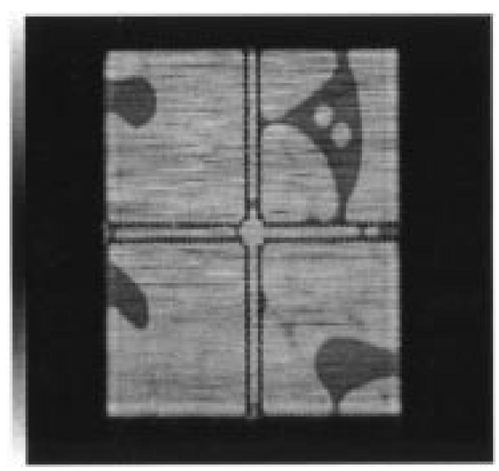

Fig. 8. C-SAM image of the assembly after inversed reflow profile.

After the chips were assembled, they were inspected using the C-SAM. Fig. 7 shows the C-SAM picture of flip chip assembly with the standard reflow profile and Fig. 8 shows the one with the inversed profile. Both assemblies experienced serious voiding. The voiding in the inversed reflow profile seemed to be even worse. The cause of the voiding was investigated in

TABLE III
CURING DEGREE OF UNDERFILLS WITH DIFFERENT PROCESSES

	NF-C	NF-D
After standard reflow	69.9%	67.2%
Standard reflow + post-cure @160°C for 45 min	86.4%	80.7%
After inversed reflow	87.7%	88.4%

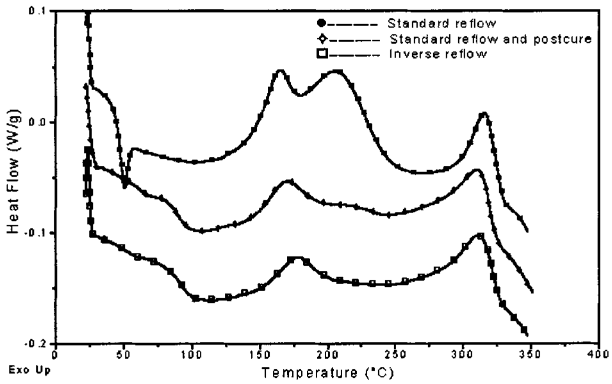

Fig. 9. DSC measured heat flow of the samples after different reflow and post-curing conditions.

the TGA study later and was illustrated to be a result of the out-gassing of the underfill during reflow.

C. Study of Curing Degree

One of the desirable features of no-flow underfill is the capability of combining solder reflow and underfill curing into one step to avoid the long off-line underfill curing which significantly limits the throughput. However, the design of no-flow underfill requires minimum curing before the solder melts. In addition, the epoxy curing reaction is usually a step-wise polymerization that becomes diffusion-controlled at the later stage. High viscosity prevents quick completion of the reaction. As a result, most no-flow underfill still needs post-cure after solder reflow. In this study, the inversed reflow profile was studied to increase the curing degree of no-flow underfill after solder reflow process.

Table III lists the curing degree determined by DSC in relation to the reflow profile and the post-cure conditions. These results indicate that two more minutes remaining at high temperature in the reflow oven can bring the curing degree of underfill up to the same curing degree when the underfill was post-cured for 45 min in addition to curing under a standard reflow profile.

Another phenomenon observed in the DSC residual latent heat is the multiple curing peaks shown in Fig. 9. In this figure, the residual curing diagrams of NF-C after the standard reflow, after post-curing in addition to standard reflow, and after the inversed reflow are illustrated. In addition to the curing peak that occurred at the normal temperature range indicated by Fig. 2, there was another high-temperature curing peak around 300 °C. The existence of this curing peak limited the curing degree of the underfill even after post cure, since post curing temperatures are usually around 150–200 °C. Post curing at higher temperature will lead to the secondary reflow of the solders, which is undesirable for the reliability of the package.

The study on the epoxy/metal AcAc system suggested that metal AcAc's have the ability to cure epoxy resin without other

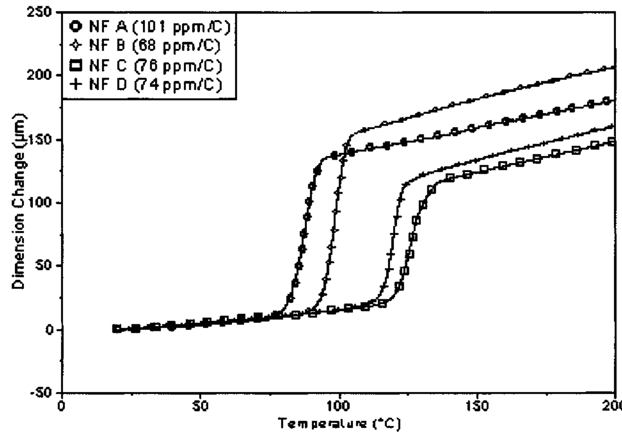

Fig. 10. TMA measured thermal expansion behavior of developed underfill materials.

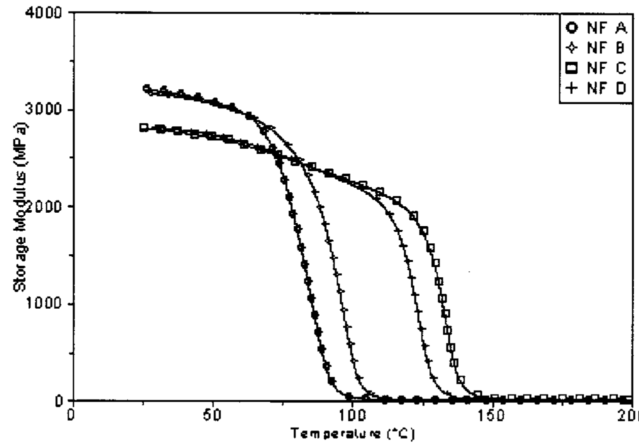

Fig. 11. DMA measured storage modulus change with teemperature of developed underfill materials.

hardeners and the curing temperatures of epoxy/metal AcAc system are above 250 °C. Also found in the study was that HMPA experienced significant weight losses during the reflow as suggested in the later investigation on the material properties. The stoichiometric balance between the epoxy and HMPA was altered due to the evaporation of HMPA and there was an excess of epoxide groups in the system after reflow. The origin of this high-temperature curing peak might be the curing reaction of unreacted epoxy under the catalytic effect of metal AcAc.

D. Characterization of Material Properties

The material properties of the developed no-flow underfills NF-A through NF-D were characterized using TMA, DMA, and TGA as specified in the experimental session. Fig. 10 shows the TMA measured thermal expansion behavior of the cured materials. The CTEs of the four materials before Tg (α_1) are listed in the legend of the Fig. 10. The DMA measured storage modulus changes with temperature are shown in Fig. 11. It can be concluded that the glass transition temperatures of NF-C and NF-D were higher than those of NF-A and NF-B.

The weight loss of the four formulations during curing is shown in Fig. 12. It can be seen that all the formulations experienced significant weight loss during curing, especially NF-A and NF-B. Fig. 13 shows the weight loss of HMPA when it

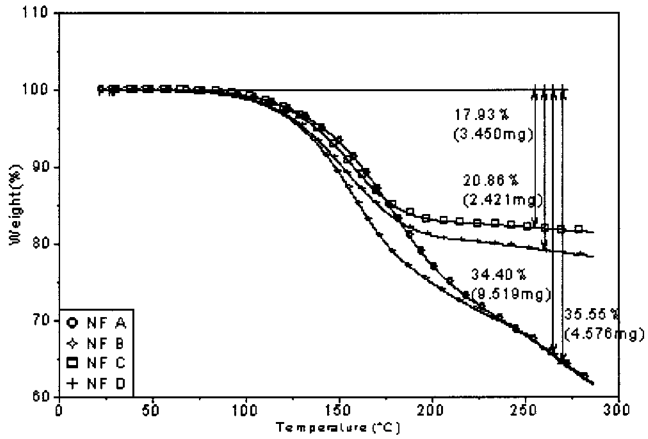

Fig. 12. Weight loss during curing of developed underfill formulations.

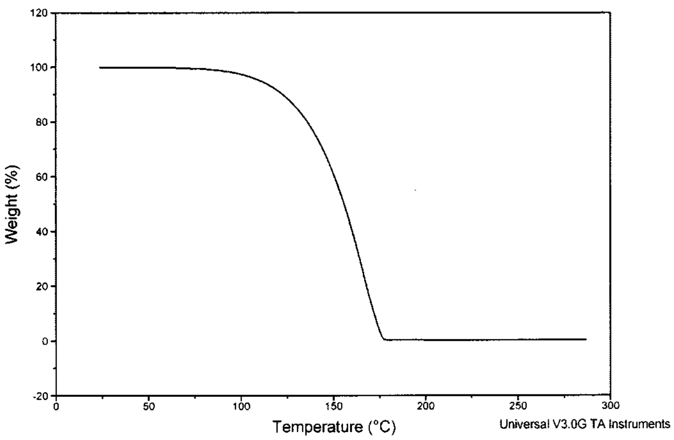

Fig. 13. Weight loss of HMPA upon heating.

was heated at 10 °C/min. The hardener HMPA started to lose weight at around 100 °C and totally evaporated at around 175 °C without curing reactions. Hence, the weight losses of the underfills were high because the catalysts involved in these formulations had high latency and there was no significant reaction at the temperature when HMPA started to evaporate. There were more significant weight losses in NF-A and NF-B whose curing temperatures were higher than those of NF-C and NF-D as indicated in Fig. 2. So the NF-A and NF-B cannot be fully cured at 250 °C, which caused their Tgs to be lower.

An important requirement in a reflow process is that the underfill material should avoid significant curing and remain at a low viscosity before the solder reaches its melting point. The DSC studies conducted at a heating rate of 5 °C/min suggest that the materials that had a curing peak temperature higher than 200 °C were desired for Sn/Ag/Cu solder with the melting point at 217 °C. However, as shown in the TGA study, the material would suffer from weight loss especially when the lead-free process has raised the reflow temperature. The out-gassing of the underfill during the reflow process would cause voids in the package and poor mechanical properties of the cured material. Although the inversed reflow profile increased the curing degree after reflow, the out-gassing of HMPA was more severe since the underfill was heated up to a high temperature so quickly that minimum curing took place while HMPA evaporated. The investigations on the DSC profile of post-cure showed that out-gassing of HMPA would destroy the stoichiometric balance between epoxy and hardener and result in a high temperature post curing that cannot be completed in the normal curing conditions. Therefore, there is a trade-off in between the material design of no-flow underfill and the process conditions. It becomes even more important in a lead-free reflow process when the reflow temperature is raised 30–40 °C compared with the eutectic Sn/Pb solder reflow process.

IV. Conclusions

Epoxy resin/HMPA/metal AcAc/Flux G system was investigated for the application of no-flow underfill for Sn/Ag/Cu alloy bumped flip-chips. The solder wetting test proved that the underfills developed have sufficient fluxing capability for lead-free solder to wet on the copper board during reflow process. Flip-chip on board packages were built using the no-flow process with lead-free reflow profiles. 100% solder joint yield was achieved using PB-8 daisy-chained chips. The developed underfill materials were characterized using DSC, TMA, DMA, and TGA. The DSC studies conducted at a heating rate of 5 °C/min suggested that materials that had a curing peak temperature around or higher than 200 °C were desired for Sn/Ag/Cu solder with the melting point at 217 °C. However, high curing temperatures would lead to large amount of out-gassing during curing, which would cause voids in the package as observed in C-SAM. The investigations on the curing degree of these no-flow underfills after different reflow profiles and post-cure condition showed that out-gassing of HMPA would destroy the stoichiometric balance between epoxy and hardener and result in a high temperature post curing that cannot be completed in the normal curing conditions. In a lead-free reflow process, both the material designs and the process conditions have to be considered when no-flow underfill is used.

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