

# Characterization of Fluxing and Hybrid Underfills with Micro-encapsulated Catalyst for Long Pot Life

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**For the fine-pitch application of flip-chip bonding with semiconductor packaging, fluxing and hybrid underfills were developed. A micro-encapsulated catalyst was adopted to control the chemical reaction at room and processing temperatures. From the experiments with a differential scanning calorimetry and viscometer, the chemical reaction and viscosity changes were quantitatively characterized, and the optimum type and amount of micro-encapsulated catalyst were determined to obtain the best pot life from a commercial viewpoint. It is expected that fluxing and hybrid underfills will be applied to fine-pitch flip-chip bonding processes and be highly reliable.**

**Keywords: Flip chip, underfill, micro-encapsulated catalyst, MEC, latent, pot life.**

## I. Introduction

Since IBM invented the controlled collapse chip connection, which was known as the C4 process in the 1960s, many applications based on flip-chip bonding technologies have been developed [1]. However, flip-chip bonding was carefully used for mounting onto silicon and ceramic substrates because of the issue of thermal-mechanical fatigue; caused by a coefficient of thermal expansion (CTE) mismatch between a semiconductor chip with 2.5 ppm/°C of silicon CTE and substrates with 4 ppm/°C to 10 ppm/°C of ceramics CTE. In particular, the serious thermal stress between a silicon chip and an organic FR4 substrate with a CTE of 18 ppm/°C to 24 ppm/°C, initiated cracks in the solder bumps during temperature cycling.

The innovative underfill process was first demonstrated by Hitachi, to reduce the solder fatigue phenomena with the use of filled resin between the chip and substrate. In addition, micro-sized silica filler was used to enhance the material properties of cured underfill; for example, one could achieve a low CTE, a high modulus, and a low moisture uptake. Figure 1 shows a conventional flip-chip bonding with surface mounting technology (SMT) and underfill processes. For the SMT process, the flux material used to remove the oxide on the surfaces of the solder bump and metal pads is usually dispensed on the substrate. In addition, the chip is placed with an alignment process and reflowed at a higher temperature than the melting temperature of the solder. The residual flux is then usually removed by solvents. The underfill resin is dispensed next to the device and dragged into the gap between the device and the substrate by a capillary force. However, there are a lot of problems with this method such as incomplete filling, voiding, and a lengthy filling time when the pitch of the solder

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Manuscript received June 14, 2013; revised Oct. 16, 2013; accepted Nov. 21, 2013.

This work was supported by KEIT, IT R&D program of Daeduck Innoplis Foundation (Grant No. A2012DD002, The industrialization of package electrode ESP materials for smart phone), the IT R&D program of MKE/KEIT (Grant No. 10041416, the core technology development of light and space adaptable new mode display for energy saving on 7 inch and 2 W), the R&D program of ISTK (Development of an image-based, real-time inspection and isolation system for hyperfine faults), and Electronics and Telecommunications Research Institute (ETRI).

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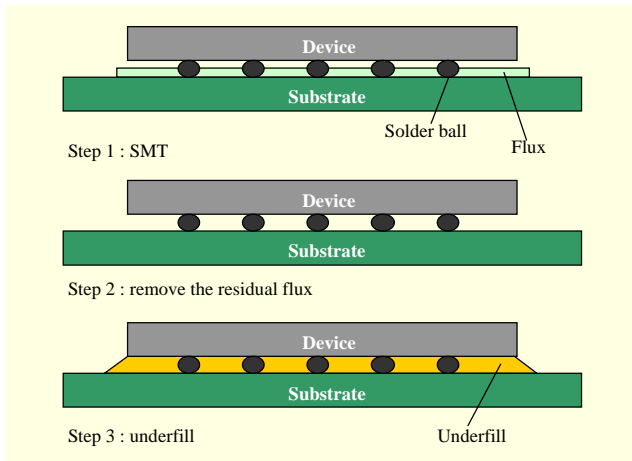


Fig. 1. Conventional flip-chip bonding with SMT and underfill processes.

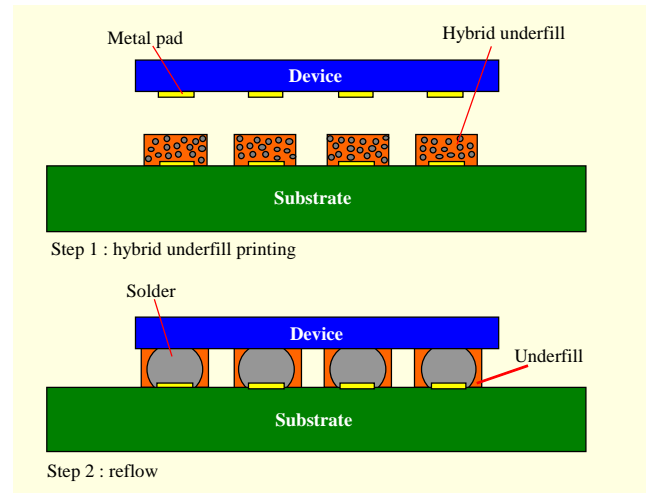


Fig. 3. Flip-chip bonding process with hybrid underfill.

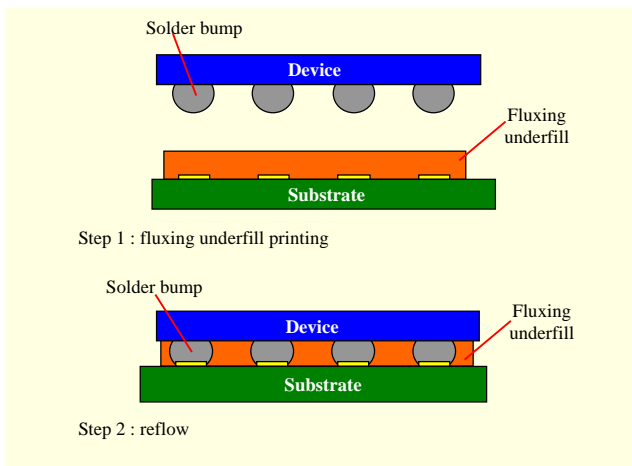


Fig. 2. Flip-chip bonding process with fluxing underfill.

bumps closes and the gap between the device and substrate narrows.

To overcome the problems observed in a conventional flip-chip bonding process, fluxing and hybrid underfill materials were developed in previous researches [2]–[6]. In the previous researches, a low-volume solder-on-pad technology was also developed to create solder bumps that had a fine pitch without a metal mask [7]–[11]. The fluxing underfill composed of an epoxy resin, curing agent, reductant, and solvent-free additives that remove oxide formed on the surface of solder and metal pads during the flip-chip bonding process. The hybrid underfill, which is a paste formed from the fluxing underfill and solder powder, was specially developed to obtain an electrical interconnection and to be more reliable after the flip-chip bonding process. Figures 2 and 3 show schematics of the flip-chip bonding process with fluxing and hybrid underfills, respectively.

To simplify and overcome problems in the conventional flip-

chip bonding process, including SMT and the underfill processes shown in Fig. 1, the flip-chip bonding processes with fluxing and hybrid underfills were introduced, as shown in Figs. 2 and 3, respectively. For the flip-chip bonding process, the fluxing and hybrid underfills are printed or dispensed on the substrates, and the devices are placed onto substrates with a precise alignment process. The temperature increases up to the melting temperature of the solder, and the oxide layers on the surface of the solder bump in Fig. 2 and solder powder in Fig. 3 are removed by the fluxing underfill without a void occurring from the resin itself. While the solder bump and powder are melted to make an electrical interconnection between the device and substrates, the fluxing underfill and resin matrix in the hybrid underfill are still in a liquid state. After that, the fluxing underfill and resin matrix in the hybrid underfill are changed to a solid state through a chemical reaction without any void generation. In the beginning of this research, a catalyst with a liquid state was used to control the speed of the chemical reaction for both the fluxing and hybrid underfills. However, it was observed that the viscosities of the fluxing and hybrid underfills slowly increased over time at room temperature owing to the use of a normal catalyst (NC); thus, resulting in a short pot life.

Many researches have tried to use a latent curing agent with liquid and powder states [12]–[15]. Shin and others, in particular tried to make a latent curing agent with micro-encapsulated powder based on imidazole for their epoxy system. The authors observed that the pot life was extended by up to 10 days at room temperature. In the present research, instead of the NC used in previous researches [2]–[6], latent catalysts were used to control the pot life at room temperature and the speed of the chemical reaction during the flip-chip bonding process. For a long pot life, several kinds of commercially available micro-encapsulated catalysts (MEC)

were selected and characterized for their compatibility with the fluxing and hybrid underfills.

## II. Materials and Experiments

### 1. Materials

As introduced in previous research [2], the fluxing underfill and resin matrix of the hybrid underfill are composed of four components: a base resin, a curing agent, a fluxing agent to remove the oxide layer formed on the surface of the solder and Cu pads, and an MEC to control the speed of the chemical reaction at room temperature during the process. Figure 4 shows the schematic structure of a micro-encapsulated catalyst with a diameter range of 2  $\mu\text{m}$  to 15  $\mu\text{m}$  used for a latent catalyst. As shown in Fig. 4, the catalyst is safely covered by a capsule material at room temperature to prevent the chemical reaction of the resin matrix in fluxing and hybrid underfills. The MEC was designed such that the capsule will melt when the resin reaches the recommended curing temperature and the catalyst is exposed to the resin, as shown in Fig. 5. For the present research, five kinds of MEC — each having different curing temperatures ranging between 80°C and 165°C (recommended by the material supplier) — were selected and purchased as shown in Table 1.

The recommended curing temperature of the MEC increases with an increase in the alphabetical names of the products (MEC-A to MEC-E). For the control of the chemical reaction during the curing process, the amount of MEC used for the fluxing and hybrid underfills is 0.2 phr to 4.0 phr of the base resin. However, the thickness of the capsule that covers the

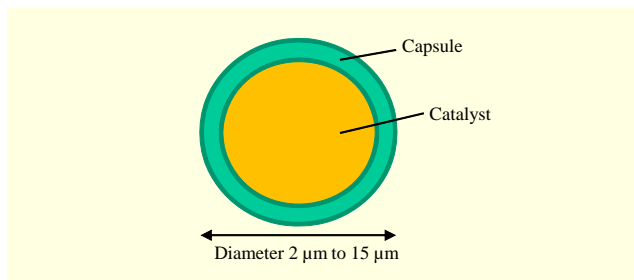


Fig. 4. Schematic structure of MEC.

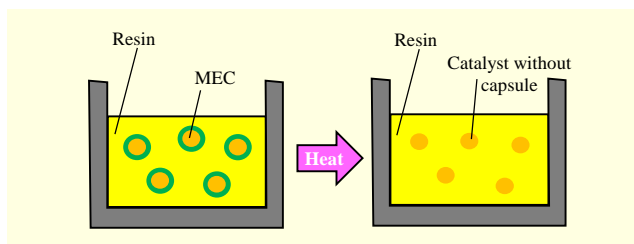


Fig. 5. Behavior of MEC after an increase in temperature.

Table 1. MEC used.

MEC	Diameter of particle ( $\mu\text{m}$ )	Recommended curing temp. ( $^{\circ}\text{C}$ )
MEC-A	10	> 80
MEC-B	6 – 15	> 130
MEC-C	2	> 130
MEC-D	2	> 145
MEC-E	2	> 165

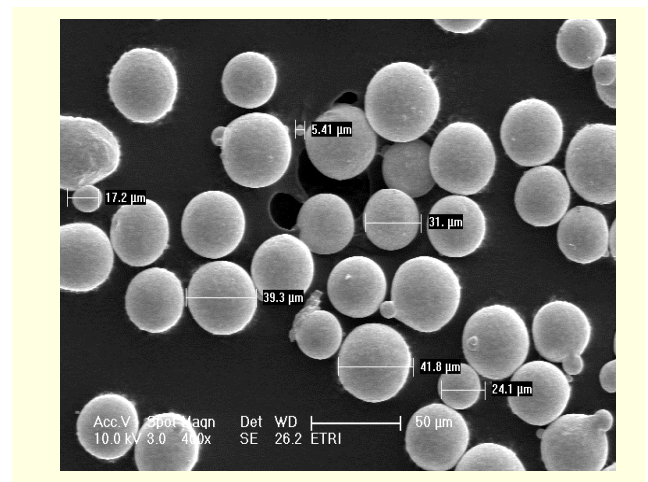


Fig. 6. SEM photograph of Sn/58Bi type 4 with a diameter of 20  $\mu\text{m}$  to 38  $\mu\text{m}$ .

catalyst is not disclosed by companies. To identify the latent effect of MECs, an NC that was used in [2] was also applied in this research.

The solder powder used for the hybrid underfill is Sn/58Bi type 4 with a diameter range of 20  $\mu\text{m}$  to 38  $\mu\text{m}$ , as shown in Fig. 6. For fabrication of the hybrid underfill, solder powder is mixed with resin at room temperature in the weight ratio of 80:20, respectively. For the fluxing underfill, an Sn/58Bi solder ball with a diameter of 0.15 mm was used to verify the ability to remove the oxide on the surface of the solder ball and Cu pad.

### 2. Experiments

Differential scanning calorimetry (DSC) was utilized to analyze the chemical reaction with a heating rate of 10°C/min from -60°C to 280°C. For the measurement of viscosity at room temperature, a Brookfield LVDV-II viscometer was used at 30 rpm with an SC4-25 spindle with a small sample adapter. In the present research, the pot life was defined as the time period to double the value of the initial viscosity — which is

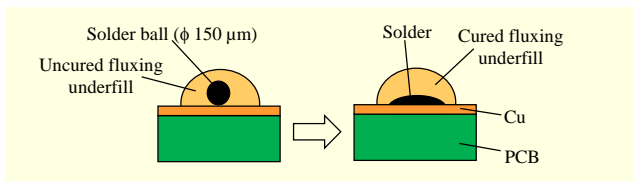


Fig. 7. Schematic of solder wettability with diameter of 0.15 mm on Cu pad for fluxing underfill.

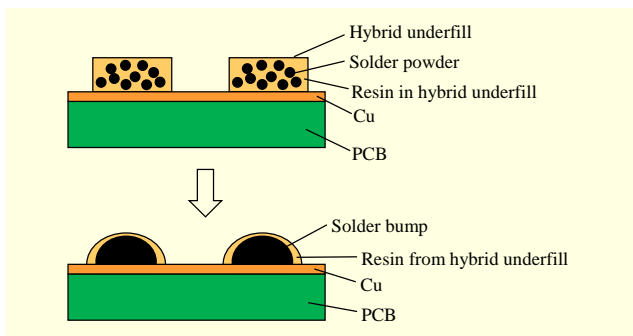


Fig. 8. Screen printing of hybrid underfill on Cu pad of PCB substrate and reflow processes.

commonly used in the industry. To observe the wettability of the solder ball in the fluxing underfill, a solder ball with a diameter of 0.15 mm is placed in the fluxing underfill on the Cu pad of the PCB, as shown in Fig. 7. Figure 8 shows the screen printing on the Cu pad of the PCB substrate and the reflow processes to observe the wettability of the solder powder in the hybrid underfill. A metal mask with a 10×10 hole array for the screen printing process was prepared with a 0.1 mm thickness, 0.5 mm hole diameter, and 1.0 mm pitch. The screen printing process was conducted on the Cu pad of the PCB using the paste of the hybrid underfill. For the reflow process, the temperature was increased up to 180°C with a heating rate of 2°C/sec and kept for two minutes and cooled to room temperature for both the fluxing and hybrid underfills, shown in Figs. 7 and 8. After the reflow process, a cross-section of the cured fluxing and hybrid underfill was observed using an optical microscope.

### III. Results and Discussion

#### 1. Fluxing Underfill

Dynamic-DSC thermograms of the fluxing underfills with 0.2 phr of NC in a liquid state and MEC-A in a powder state, were conducted to investigate the chemical reaction with a heating rate of 10°C/min, as shown in Fig. 9. With an increase in temperature, the glass-transition temperatures of both uncured fluxing underfills were observed at around -40°C. Endothermic peaks for both materials were continuously

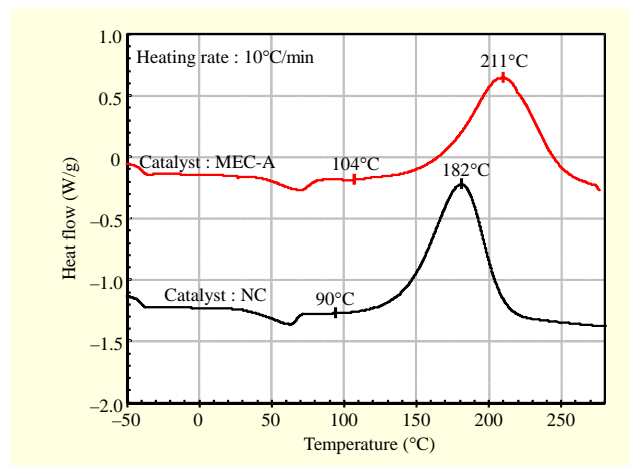


Fig. 9. Dynamic-DSC thermograms of fluxing underfill with NC and MEC-B.

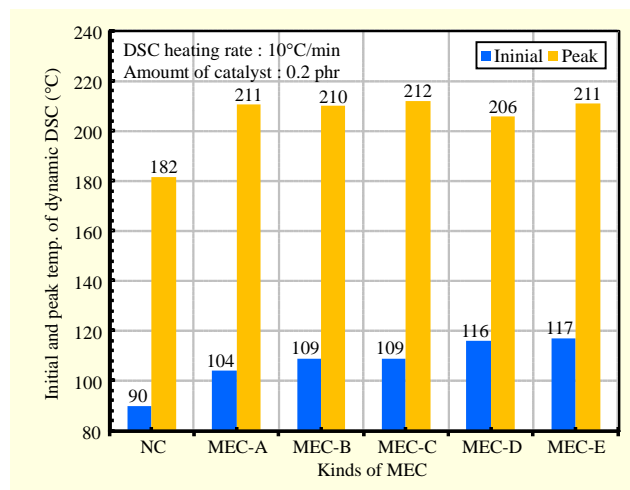


Fig. 10. Initial and peak temperatures of chemical reaction between fluxing underfill and several kinds of MECs from dynamic DSC thermograms.

detected at around 60°C. It was inferred that these endothermic peaks were caused by the melting of the fluxing agent indicated in [2].

The fluxing underfill was transparent when the resin was mixed at around 100°C, and the color was then changed to white with a decrease in temperature owing to some recrystallization of the fluxing agent. The initial and peak temperatures were 90°C for the chemical reactions for both materials, 182°C for a fluxing underfill with NC, and 104°C and 211°C for a fluxing underfill with MEC-A, respectively. It is believed that the differences in initial and peak temperatures between both materials were observed as 14°C and 29°C; owing to the latent effect of the MEC. Figure 10 shows the summarized initial- and peak-temperatures of the chemical reaction between fluxing underfills and several kinds of MECs; with 0.2 phr measured using dynamic DSC thermograms. The

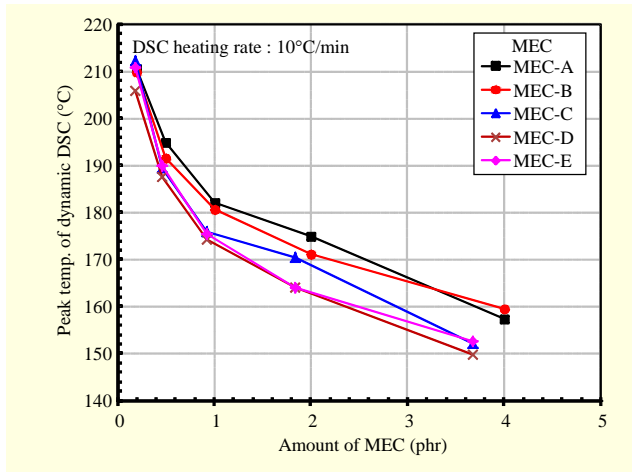


Fig. 11. Peak temperatures of chemical reaction for fluxing underfill with MECs from dynamic DSC thermograms.

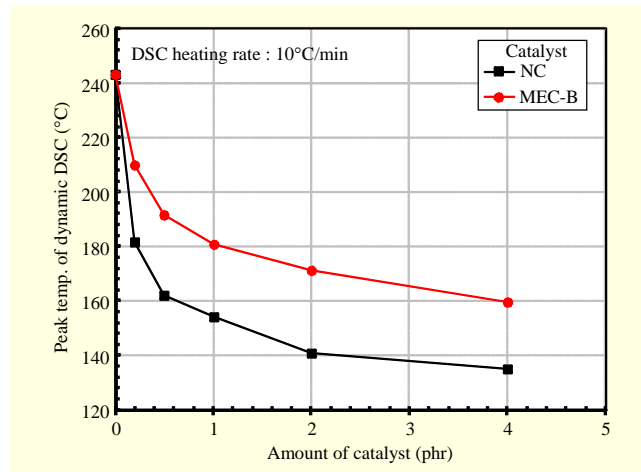


Fig. 13. Comparison of peak temperatures from dynamic DSC thermograms for fluxing underfills with NC and MEC-B.

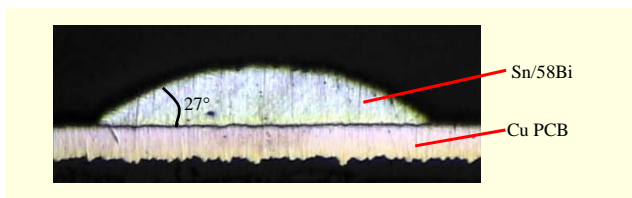


Fig. 12. Solder wetting on Cu pads in fluxing underfill with MEC-B.

initial- and peak-temperatures of the fluxing underfill with all of the MECs are clearly higher than that with an NC. It was also observed that the initial temperature of the chemical reaction increased with an increase of the recommended curing temperature, shown in Table 1, while the peak temperatures of the chemical reaction with all of the MECs were almost the same at around 210°C. With an increase in the processing temperature, it was inferred that the capsules from MEC-A to MEC-E were alphabetically melted, and thus their catalysts were also alphabetically exposed.

Figure 11 indicates the peak temperatures of the chemical reaction of the fluxing underfill with an increasing amount of MECs. With all MECs, the peak temperatures are dramatically decreased up to 1.0 phr of the base resin and linearly reduced to 4.0 phr. Except for a long pot life, there are three design requirements of the fluxing and hybrid underfills with an MEC: the ability to effectively remove oxide on the surface of solder and metal pads, low MEC material cost, and the ability to change from a liquid to solid state through a chemical reaction during processing. Figure 12 shows the cross-section of solder (Sn/58Bi ball with a diameter of 150  $\mu\text{m}$ ) wetting on Cu PCB in the fluxing underfill with MEC-B after processing. With an increase of the processing temperature, the Sn/58Bi solder ball was melted and wetted on the Cu pads at around its melting temperature in the liquid state of the fluxing underfill.

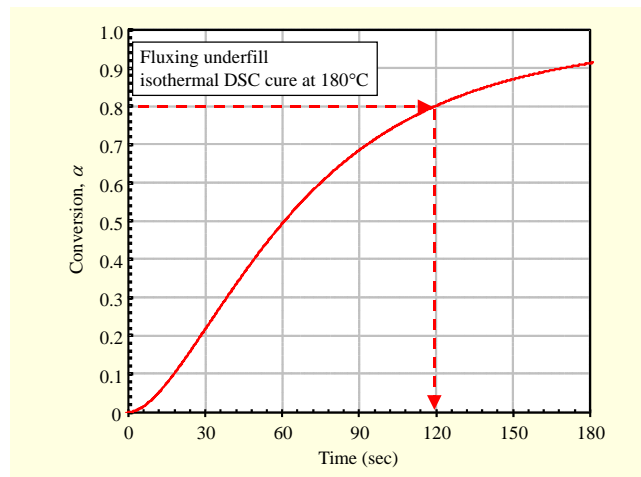


Fig. 14. Measured conversion of fluxing underfill with MEC-B by isothermal DSC at 180°C.

In addition, the fluxing underfill is cross-linked by a chemical reaction and changed to a solid state. It was proven that the fluxing underfill with MEC-B has the ability to remove the oxide on the surface of the solder ball and Cu pad, because the wetting angle was measured as 27 degrees. All fluxing underfills with all kinds of MECs showed an adequate solder-wetting effect on Cu pads, and changed from a liquid to solid state after processing. Finally, MEC-B as a latent catalyst, was decided based on a consideration of de-oxidation effect, a complete chemical reaction during the processing, and the lowest material cost.

Figure 13 shows a comparison of peak temperatures measured by dynamic DSC for the fluxing underfills with NC and MEC-B. With an increase of the catalyst NC and MEC-B, the peak temperatures are rapidly decreased to 1.0 phr. The difference in peak temperatures between NC and MEC-B were

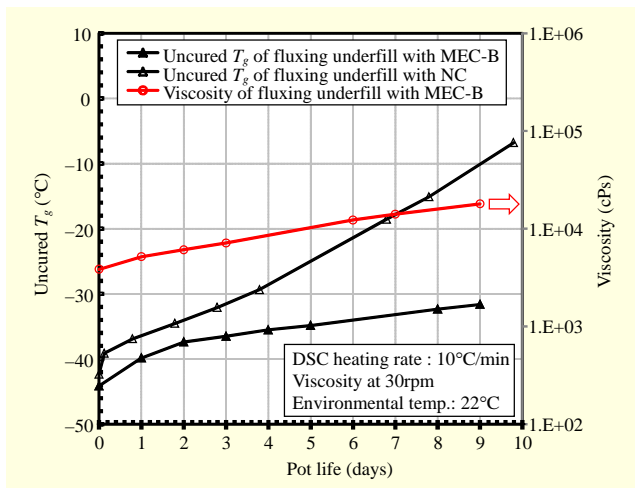


Fig. 15. Changes of uncured glass transition temperature and viscosity of fluxing underfill with MEC-B at 22°C.

25°C and 30°C for 1.0 phr and 2.0 phr, respectively. Figure 14 shows the measured conversion of the fluxing underfill with 2.0 phr MEC-B by isothermal DSC thermograms at 180°C. The amount of MEC-B for a fluxing underfill was determined as 2.0 phr to obtain a conversion of 0.8 within a curing time of two minutes.

For the measurement of pot life for the fluxing underfill with MEC-B, the uncured glass-transition temperature  $T_g$  and viscosity were measured at 22°C, as shown in Fig. 15. The uncured  $T_g$  of the fluxing underfill with NC was also measured for a comparison with MEC-B. For the fluxing underfill of both NC and MEC-B, the uncured glass-transition temperatures increased rapidly for up to two days. After two days, the increasing rate of  $T_g$  for the fluxing underfill with MEC-B was clearly reduced, while that with NC was slightly increased up to ten days. Gillham and others [16], reported the concept of time-temperature-transformation cure diagram for the thermosetting materials. When the environmental temperature is in-between the uncured glass-transition temperature and the gelation temperature, the glass-transition temperature is very slowly increased and keeps a constant value at which the conversion is almost stopped at the given curing temperature. This limited glass-transition temperature, at a given environmental temperature, can be considered as an aging time of the thermosetting resin system. Therefore, it is believed that the aging time of the fluxing underfill with MEC-B, at room temperature, was 48 hours. The measured viscosities of the fluxing underfill with MEC-B after two and four days were 6,100 cps and 12,280 cps, respectively. Based on the definition of the pot life mentioned in the previous section, it was observed that the pot life and the aging time of the fluxing underfill with MEC-B was in both cases, 48 hours.

## 2. Hybrid Underfill

The hybrid underfill was composed of a fluxing underfill with MEC-B and Sn/58Bi type 4 solder powder of 80 wt.%. Figure 16 shows the measured conversion of the hybrid underfill with MEC-B of 0.1 phr based on the base resin using an isothermal DSC at 180°C. A curing time of ninety-two seconds for the hybrid underfill was required to reach the conversion of 0.8 at 180°C. To obtain a conversion of 0.8, the amount of MEC-B was optimized to 1.0 phr for the hybrid underfill with the solder powder. In [5], it was reported that the Sn/58Bi solder powder had catalytic activity in epoxy systems. Therefore, it was inferred that a conversion of 0.8 was obtained at ninety-two seconds owing to the catalytic activity of the solder powder, even though the amount of MEC-B was 1.0 phr, which was lower than that of the fluxing underfill with 2.0 phr.

Figure 17 shows optical photographs of the hybrid underfill with MEC-B 1.0 phr and 80 wt.% of the solder powder, before and after curing processes. The hybrid underfill was screen-printed onto the Cu pad of the PCB substrate with a 1.0 mm pitch and 0.5 mm pad diameter, as shown in Fig. 17(a). A cross-section of the cured hybrid underfill is shown in Fig. 18.

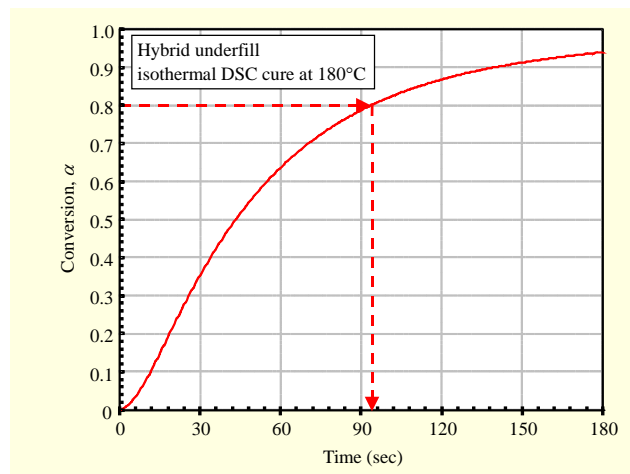


Fig. 16. Measured conversion of hybrid underfill with MEC-B by isothermal DSC at 180°C.

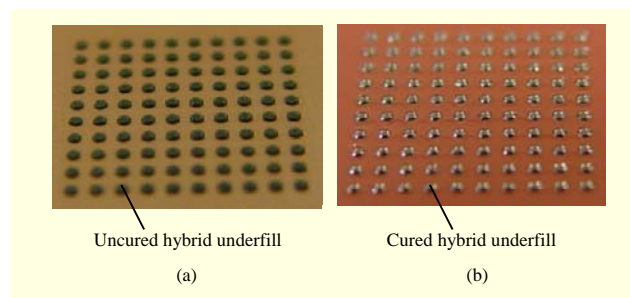


Fig. 17. Optical photographs of hybrid underfill on Cu pads of PCB substrate: (a) before and (b) after curing processes.

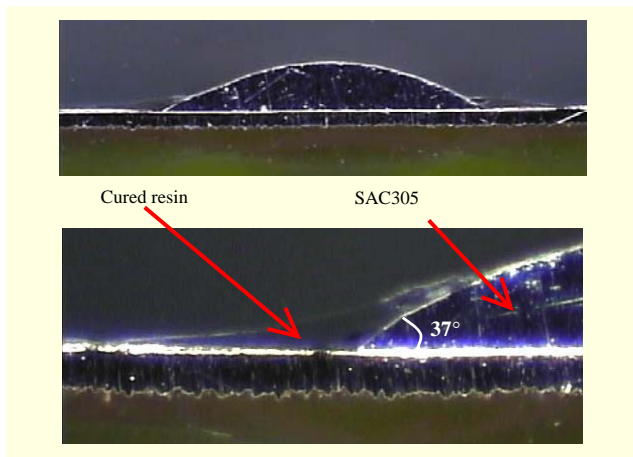


Fig. 18. Optical photographs of solder wetting on Cu pads of hybrid underfill with MEC-B and solder powder.

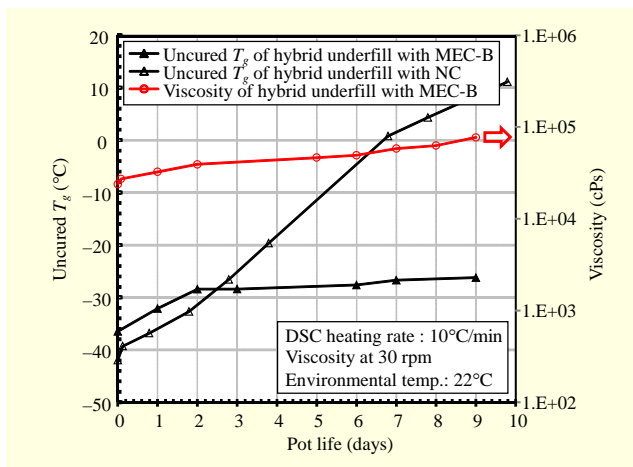


Fig. 19. Changes in uncured glass transition temperature and viscosity of hybrid underfill with MEC-B and solder powder at 22°C.

With an increase in temperature, the solder particle was melted and continuously wetted between adjacent solder particles and finally wetted on the Cu pad by the force of gravity. During the melting and wetting of the solder powder, it was inferred that the resin matrix between solder particles in the hybrid underfill was squeezed out and placed on the surface and edge of the solder because the resin matrix was still in a liquid state when the temperature was at around the melting temperature of the solder, as shown in Fig. 18. The measured wetting of solder from the hybrid underfill was 37°.

The uncured glass-transition temperature and viscosity of the hybrid underfill with 1.0 phr MEC-B and solder powder, were measured using a dynamic DSC and viscometer, as shown in Fig. 19. The glass-transition temperatures of the hybrid underfills with both NC and MEC-B were linearly increased for two days at 22°C. After two days,  $T_g$  of the hybrid underfill with MEC-B remained constant for up to nine days, while that

with NC was consistently increased up to about 10°C for ten days. The viscosity was also increased for two days, while the rate of increase was reduced slightly for nine days. It is believed that the aging time required to reach a stable viscosity of certain value was 48 hours for the hybrid underfill with MEC-B. The measured viscosities at two and nine days, stored at room temperature, were 39,000 cps and 77,000 cps, respectively. It was identified that the pot life of the hybrid underfill with MEC-B was seven days according to the definition mentioned in the previous section. In the near future, new research for the reliability of the fluxing and hybrid underfills will be investigated to analyze their mechanical and physical properties related to flip-chip bonding applications for semiconductor packaging.

#### IV. Conclusion

For a long pot life, fluxing and hybrid underfills with a micro-encapsulated catalyst were characterized. To obtain the latent effect of both underfills at room temperature, the micro-encapsulated catalysts were covered by capsules ranging in diameter from 2  $\mu\text{m}$  to 15  $\mu\text{m}$  and were prepared through commercial sources. A DSC and viscometer were used to characterize the chemical reaction and viscosity change at room temperature during processing. Based on these measurements, it was observed that the pot life of the fluxing and hybrid underfills were two and seven days, respectively, while the aging times were two days for both materials.

#### Acknowledgements

The authors would like to thank Sun-Woo Chu for his support in the sample preparation and measurement.

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