Effect of Particle Size Distribution of Glass Frit on the Transparency of Transparent Dielectric Layer for Plasma Display Panel

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Abstract

We report the effect of the particle size (D50) and PSD (Particle Size Distribution) of glass frit on the transparency of transparent dielectric layer of PDP. The milling efficiency of wet milling with water was the best among the dry milling, wet milling with IPA and wet milling with water. The transparency increased with the reduction of particle size of glass frit as the milling time increased. Also the transparency changed by the PSD of glass frit. Glass frits of broad PSD showed high transparency compared with the glass frits of sharp PSD.

1. Introduction

Increasing the transparency of transparent dielectric layer is one of the most important issues for the high luminescent efficiency of PDP¹⁾. Many efforts are focused on the developing new glass compositions or application method such as green sheet technology for increasing the quality of transparent dielectric layer. But the processing factor for producing glass frits are also one of the key issues for increasing the transparency of dielectric layer.

The PSD of glass frit is most important factor for it used as a powder. But the research on PSD effect of the powder sintering was mainly focused on the ceramic sintering ²⁻³⁾.

We report the effect of the particle size and PSD of glass frit on the transparency of transparent dielectric layer of PDP.

2. Experimental

Table 1 Compositions of glass frit

| Contents | CaO | PbO | CuO | Al ₂ O ₃ | B ₂ O ₃ | SiO ₂ |
|----------|-----|----------|-----|--------------------------------|-------------------------------|------------------|
| Wt% | 0 | A | 0 | 0 | 0 | • |

O:0~10 ●:10~30 △:30~50 ▲:50~100

Table 1 shows the compositions of glass frit.

We investigated the effect of particle size and PSD on the transparency of transparent dielectric layer. For the PSD effect, we compared the glass frits having same particle size & different PSD. For that we make glass frits by three types of milling, Wet milling with water (M-W xx), wet milling with IPA (M-I xx) and dry milling (M-D xx). After that we compared the milling efficiency of milling methods and the effect of particle size and PSD. The mixture of raw materials was melted in a platinum crucible at 1100°C for 30min. And the melt was quenched into

stainless roller to make glass flakes.

The glass flakes was pulverized in a ball mill to obtain glass powder. Particle size of glass frit was measured by Mastersizer-s (Malvern). To measure the transparency, after mixing the glass frit, solvent and binder, coated onto soda lime silicate glass substrate by screen-printing method and heated at firing temperature for 30min. After that we measured transparent by UV-2401pc (Shimadzu)

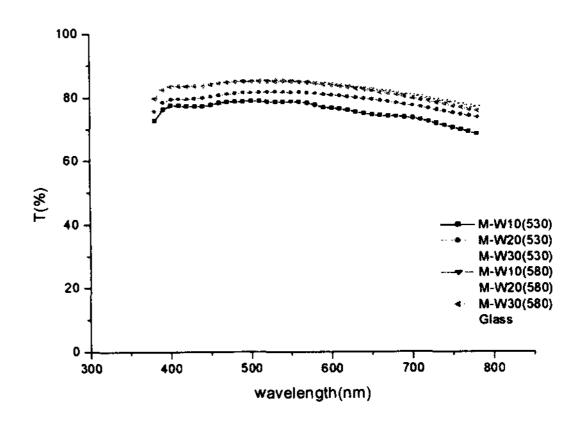


Figure 1 Transparency with milling time and method

Fig 1. shows the transparency with firing at 580° C and 530° C. The differences of the transmittance, firing at 580° C, was so small that we couldn't compare milling efficiency. So we compared results of 530° C.

3. Results and discussion

3.1 Milling efficiency

Table 2 Particle size(D50) changes with medium and milling times (Unit: \(\mu\max{m}\))

| TIME | M -10 | M-15 | M-20 | M-30 | M-10+30 |
|-------|--------------|------|------|------|---------|
| WATER | 1.54 | 1.3 | 1.17 | 1.07 | 1.3 |
| IPA | 3.18 | 2.49 | 2.24 | 1.58 | 2.46 |
| DRY | 6.33 | 4.71 | 3.91 | 3.30 | 4.76 |

Table 2 shows particle size of various milling conditions. M-10+30 is mixture of the frits milled 10hrs and 30hrs. Wet milling

with water showed the best milling efficiency among three milling methods. And the efficiency of dry milling was very low.

3.2 The effect of Particle Size

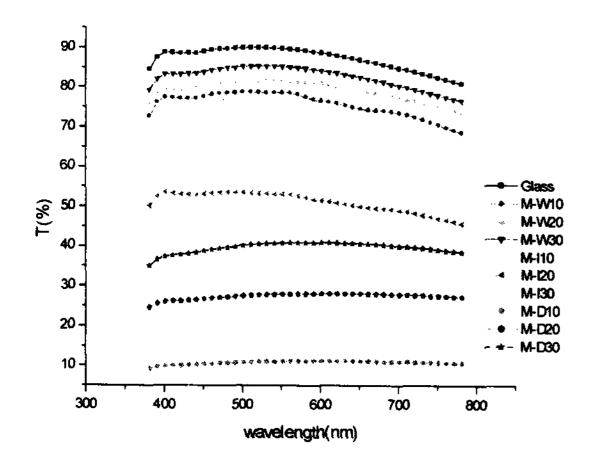
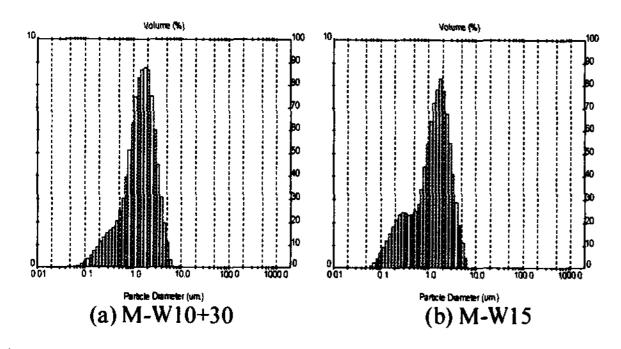


Figure 2 Transparency with each milling times and methods (530°C)

Fig 2 shows the transparency of different milling time (10, 20, 30 hrs) and different milling medium. The transparency increased with the size reduction of glass frit. It caused by positive effect of small particle in diffusion controlled sintering of powder compaction. In this experiment, wet milling with water turned out to be the most effective milling method, even though it is well known that wet milling has some negative effect on milling of glass frit, for it caused chemical reaction of glass frit during milling⁴⁾. But it resulted in high transparency for it's high milling efficiency. The transparency of M-D30 was lower than that of M-110, even though their particle size (D50) was almost same. These gap can be explained by the existence of large particles over $10 \mu m$. It was reported that, less than $0.5\mu m$ and more than $10\mu m$ size of frit makes falling-off in quality4). When compared with other frit powder, M-D XXs have lots of more than 10 \(\mu\)m size powder. So we can see that the M-D XXs showed lower transparency than others. The transparency of M-I30 was lower than that of M-W10, even though their particle size (D50) was almost same. These gaps can be explained by carbon remains from IPA.

3.3 The effect of PSD



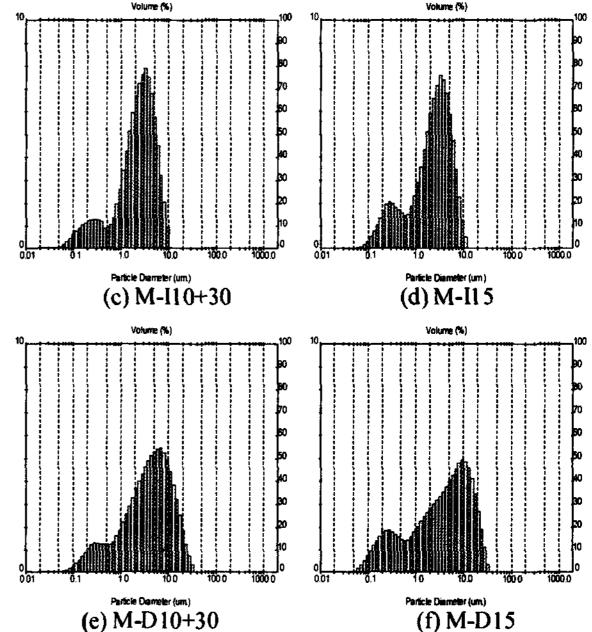


Figure 3 Particle size distributions

We compared the transparency of two kinds of glass frits having the same particle size and different PSD. M15 and M10+30 are selected because of the same particle size and different PSD.

Fig3 shows the particle size distributions for various milling conditions. M10+30 showed more sharp distribution than M15 for every milling medium. The distributions of M10, M30 was broad like M15. If we mix M10 and M30, the populations of middle size particles of M10+30 increased compared to smaller and bigger size particles, so the PSD of M10+30 was sharper than M15.

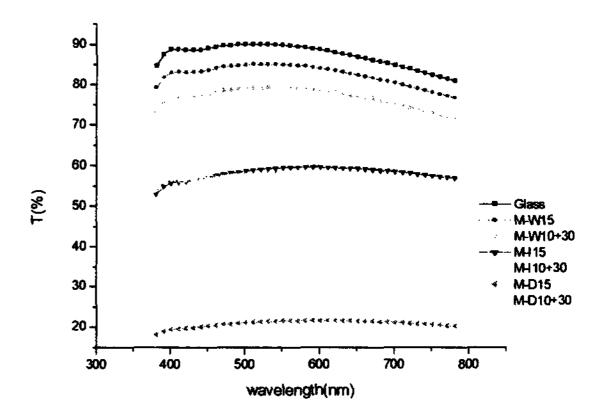


Figure 4 Transparency with same particle sizes and different PSD (530℃)

Fig. 4 shows that relationship between PSD and transparency. M15 showed higher transparency than M10+30. In this case, PSD showed an effect on the transparency and the glass frit of broad PSD showed higher transparency than the glass frits of sharp PSD. Because sintering efficiency of broad distribution is higher than that of sharp one²⁾, so the glass frits of broad PSD showed better transparency.

4. Conclusion

We proposed the method how to increase the transparency of transparent dielectric glass frit by controlling particle size and PSD. The efficiency of milling method turned out to be quite different even in the same wet milling according to the milling medium. We can easily reduce the size of glass frit with the wet milling with water and the small particle size makes better transparency. Regarding the effect of PSD, glass frit of broad PSD showed high transparency. Even though it is well known that wet milling has some negative effect on milling of glass frit, for it caused chemical reaction of glass frit during milling⁴⁾. But in this experiment, wet milling with water resulted in high transparency

for it's high milling efficiency.

5. References

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