Characterization of $BaO-B_2O_3-SiO_2$ glass for the application to PDP: Effect of BaO/B_2O_3 ratio

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Abstract

For the development of Pb-free low temperature sintering glass frits, $BaO-B_2O_3-SiO_2$ system was examined. The content of BaO and B_2O_3 was changed when the content of SiO_2 was fixed to 10 mol%. When the content of BaO was more than 60 mol% devitrification was observed. In the sintering temperature range between 520~620 ℃, the optimum sintering temperature decreased as the content of BaO increased. When BaO ≥ 45 mol%, the glasses were crystallized after sintering. Candidate compositions are suggested in $BaO-B_2O_3-SiO_2$ system, which can replace the PbO containing glass system.

1. Introduction

Recent great development of various display devices needs new various glasses which satisfies the specific requirements. Therefore, precise control of thermal and electrical properties of glasses is getting more important.

PbO containing glass system has been popular which is sinterable at low temperature. However, recent nature protection issues restrict the wide use of PbO system, so the development of materials, which can replace the PbO system, is urgent.[1]

PDP device is made up of many kinds of glasses such as frond and rear substrate glasses, barrier rib, dielectric layers, etc. Particularly, $PbO-B_2O_3-SiO_2$ glass system has been widely used for barrier rib and transparent dielectric layers.[2]

Required characteristics for barrier rib material are low sintering temperature below 600℃, dielectric constant below 15 and its thermal expansion coefficient is $7\sim9\times10^{-6}$/℃ which is almost the same with that of the glass substrate. [3,4]

In this study, $BaO-B_2O_3-SiO_2$ system, which is sinterable at low temperature, was investigated for the application to Pb-free paste as well as transparent dielectric layers in PDP. The effect of the content of the modifier (BaO) and the network former (B_2O_3) on the characteristics of the glasses was examined.

2. Experimental procedure

High purity chemicals of $BaCO_3$(99.6%), H_3BO_3(99.9%), and SiO_2 (99.9%) were used for starting raw materials. The required amounts of raw materials were weighed to the compositions with different molar ratios as shown in Table 1 and melted for 1 h at 1200℃ after mixing. The molten glass was quenched on a platinum plate. The glass powder was prepared by dry-crushing with a planetary ball mill for 2 h at 400 rpm. The density of powder was measured using a gas-pycnometer. The crystallization of the glass was analyzed with an X-ray diffractometer (Material Analysis and Characterization, M03XHF, Japan). Glass powders with different compositions were cold-isostatic-pressed (CIPed) under the pressure of 100MPa for 3 min. in order to form pellets. The pellets were sintered at 520℃ ~ 620℃. The density of pellet was measured by the Archimedes method. Microstructure of the sintered pellets was observed by using a SEM (JEOL, JSM-5400, Japan).

3. Result and discussion

Table 1 shows a list of compositions employed in this experiment. The content of SiO_2 was fixed to 10 mol% and the ratio of modifier (BaO) vs. network former (B_2O_3) was varied. After quenching the glass frits were X-ray analyzed and the results are presented in Fig. 1. From the X-ray diffraction patterns most of the compositions are in glass state, while several sharp diffraction peaks were appeared in B60 composition which implies that some extent of crystallization occurred.

The powder density which was measured by a gas pychnometer is presented as a function of BaO content. A linear increase of the density proportional to the content of BaO was observed.

Table 1 Employed compositions of BaO-B_2O_3-SiO_2 system (in mole%) and the state of glasses after quenching

	BaO	B_2O_3	SiO_2	State
B30	30	60	10	Glassy
B35	35	55	10	Glassy
B40	40	50	10	Glassy
B45	45	45	10	Glassy
B50	50	40	10	Glassy
B55	55	35	10	Glassy
B60	60	30	10	Partially crystallized

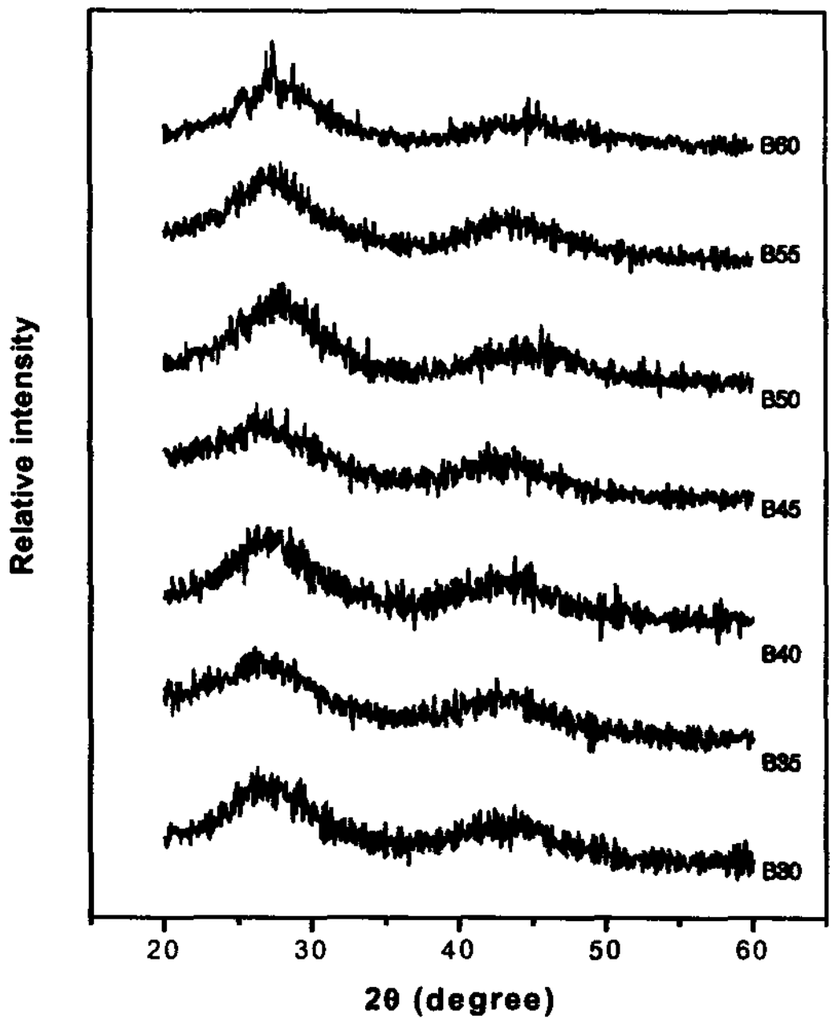

Fig 1. X-ray diffraction profiles of glasses after quenching

Fig. 3 shows the density of pellets sintered at the temperature range of 520℃~620℃. In the case of the B30 and 35, the density increased as the sintering temperature increased and showed the highest density over 600℃. However, B40 marked the highest density at 580℃ then decreased as the sintering temperature increased, which is thought due to the over firing phenomena.[5] While, B45~B55 pellets revealed a good densification through out the temperature range over 540℃. From this result, it is obvious that the optimum densification temperature, where the highest density achieved, decreased as the BaO content increased.

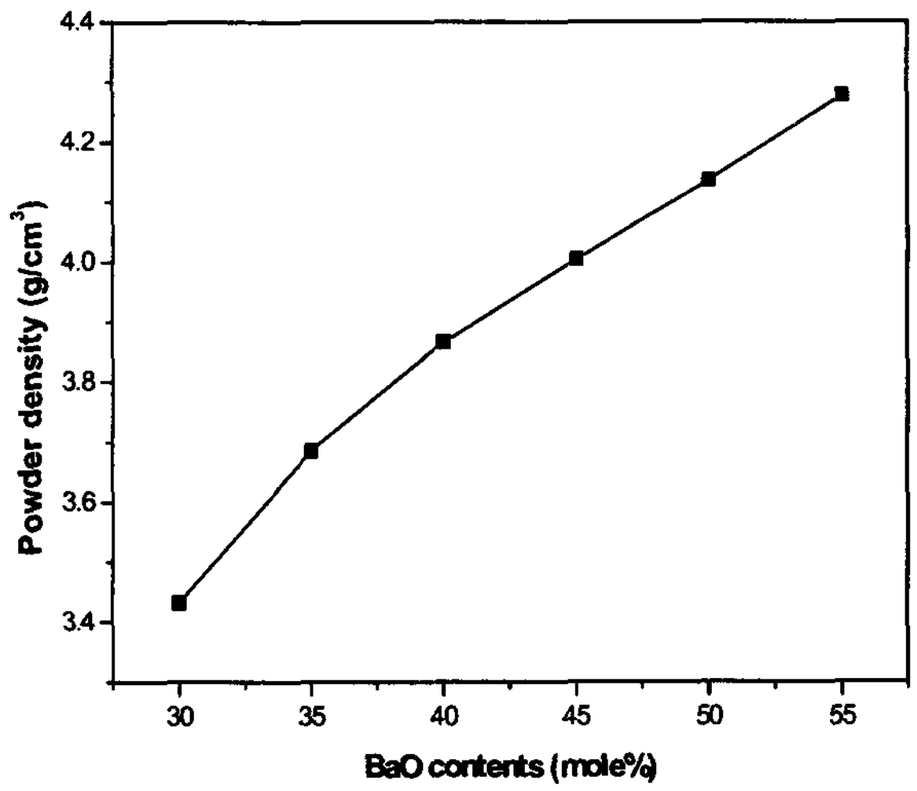

Fig. 2 Density of glass powders as a function of BaO content

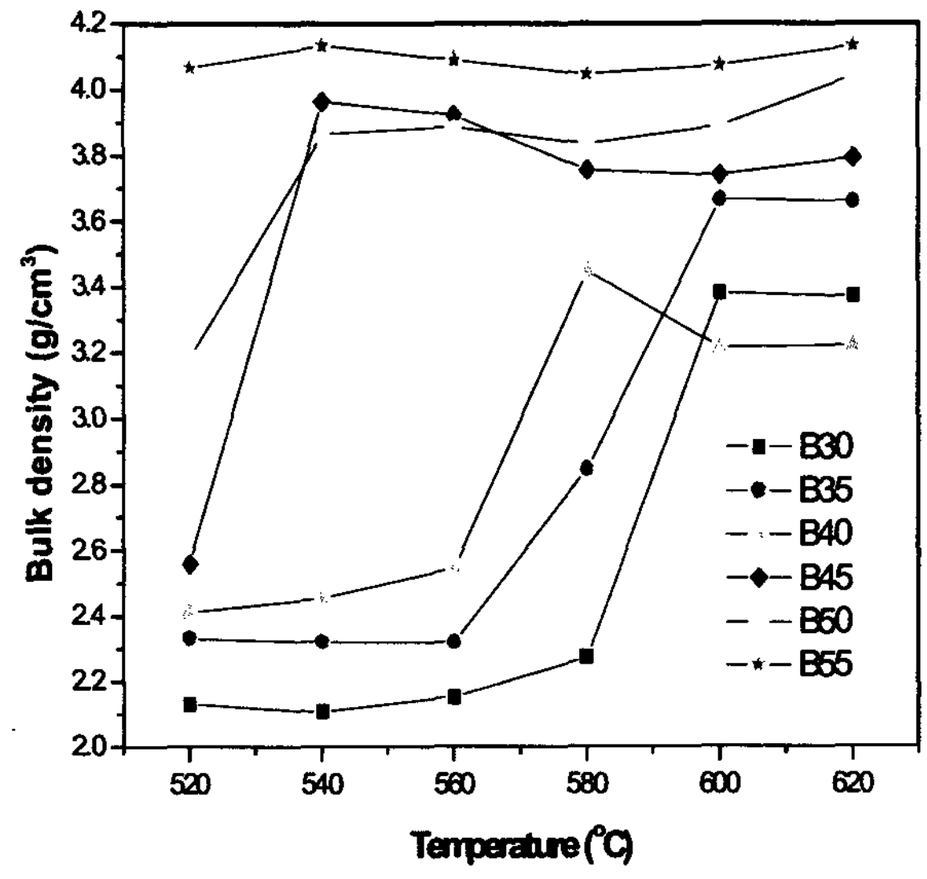

Fig. 3 Bulk density of B30~B55 pellets after sintering at various temperatures

Fig. 4(a) and 4(b) present X-ray diffraction profiles of the pellets after sintering at 600℃. It is clearly seen that the B30~B40 pellets are in glass state, while the B45~B55 pellets are in crystallized state after sintering. When the

content of BaO is ≥ 45 mol%, crystallization seems to occur simultaneously with densification.

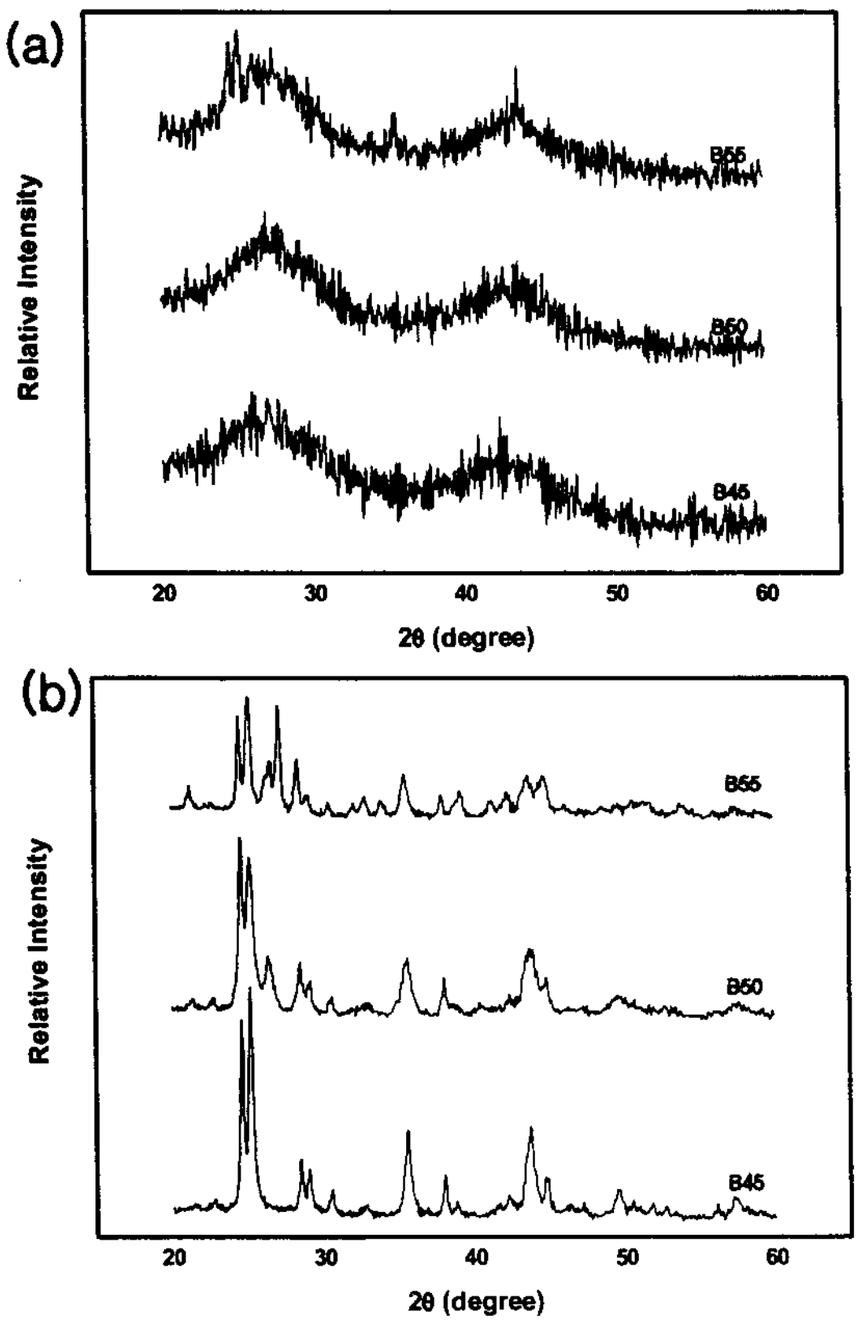

Fig. 4 X-ray diffraction profiles of B30~B55 pellets after sintering at 600℃

Fig. 5 shows the fracture images of the pellets after sintering at 580℃. B30 revealed a typical fracture image of glasses with a smooth surface, while B50 showed a rough fracture image due to the densification of grains after the crystallization. In the case of the B60, which showed poor densification, fine crystallized grains were observed.

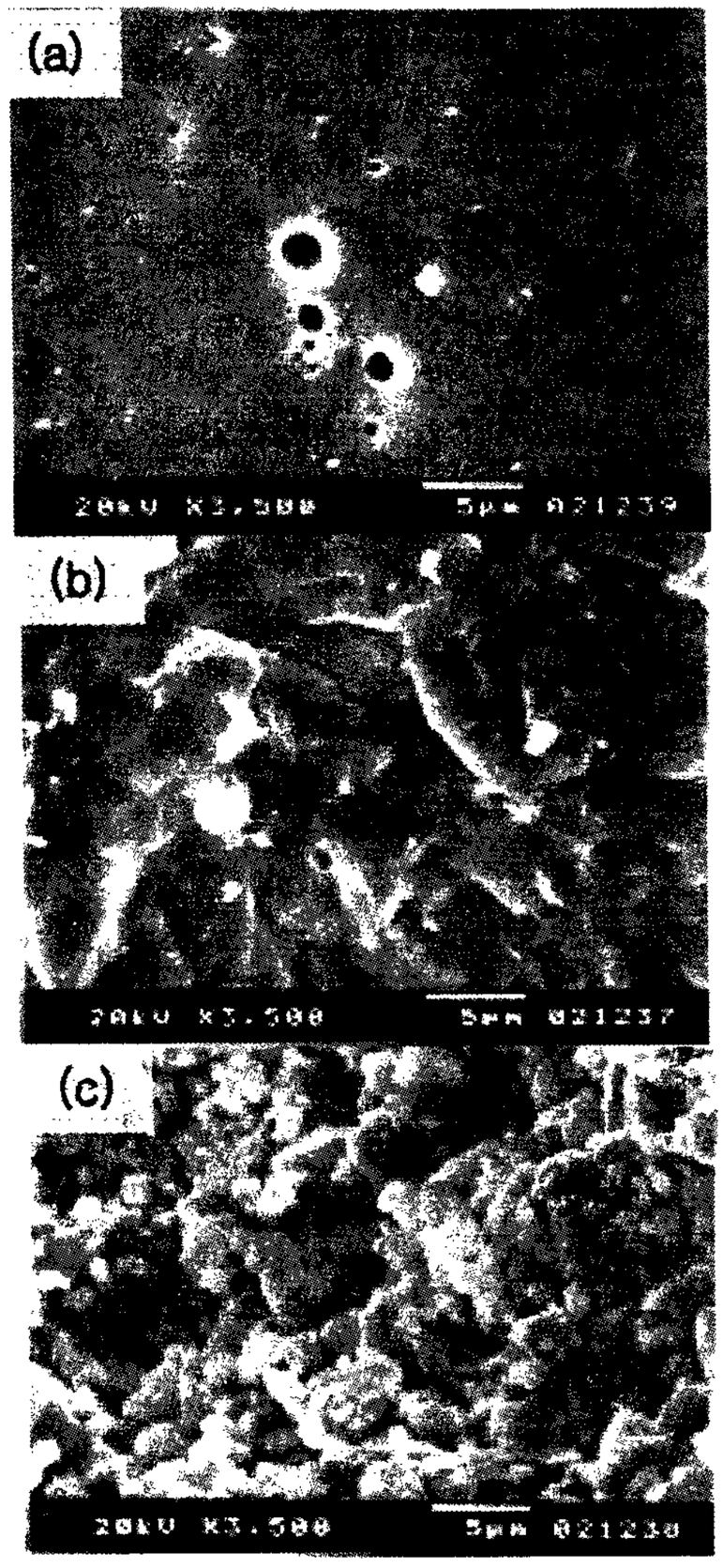

Fig. 5 Microstructures of (a) B30, (b) B50, (c) B60 pellets after sintering

3. Conclusion

1) Density of glass powders was increased as the content of BaO increased.
2) The optimum sintering temperature decreased as the content of BaO increased. When BaO ≥ 45 mol%, densification and crystallization proceeded at the same time.
3) Pb-free low temperature glasses were found in $BaO-B_2O_3-SiO_2$ system, which could be applicable to PDP.

4. Acknowledgement

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5. References

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