Evaluation of Mechanical Properties for Barrier Rib Using Micro-Tip Indenter

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Abstract

The mechanical properties of barrier ribs in PDP require quantification in order to control the defects and to increase the yield in the process. Several different types of rib materials were tested for hardness (H) and Young's modulus (E) with a micro-tip indenter (Berkovich type). For the assessment of fracture toughness of the rib, a macro Vikers indenter was used. The materials with 30wt% of filler were fired at between 490 ℃ and 570 ℃. As a result, the composite became fully densified at 520 ℃, which is near the T s (Littleton softening point) of glass frit. As the filler content increased, the fracture toughness also (K_{IC}) increased in the range of 0.60 to 2.63 $MPa \cdot m^{0.5}$ *after sintering at 550 ℃. The results suggest that the application of a nano-indenter would be useful for testing the mechanical properties of barrier ribs.*

1. Introduction

Of the new, promising flat panel display devices, PDP (plasma display panels) consist of various parts as shown in Fig. 1, including a dielectric layer, phosphors and barrier ribs. Among these components, the ribs which are made of composites of a large portion of glass and ceramic fillers (Al_2O_3 and TiO_2) create a discharging area for high luminance, and also form a sub-pixel of the PDP by preventing electrical and optical cross-talking between neighboring cells [1-2].

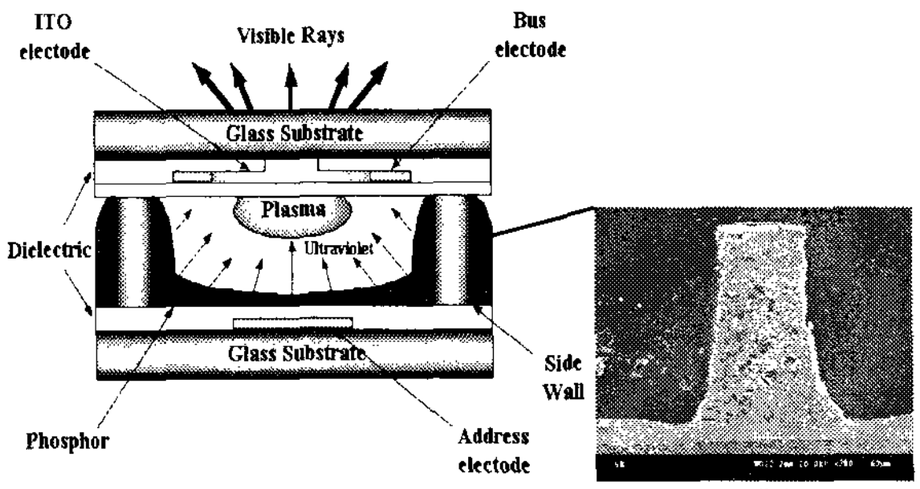

Fig. 1 Schematic diagram of the PDP panel (a) and SEM photo of barrier rib (b)

The many technological studies regarding PDP barrier ribs can generally be grouped into three categories; the development of environmentally friendly materials; more effective manufacturing processes; and finally, employment of an adjustable evaluation method for reducing trial errors (defect rate).

The present study is mainly focused on the third factor, particularly the evaluation of mechanical properties of the ribs. Testing the mechanical properties of barrier ribs is essential for reliable and economic production, however conventional methods have simply relied on methods such as dropping a steel ball, micro Vickers and erosion tests with SiC powder. However, these methods are often limited by the size of the ribs and the complicated experimental procedure. For this reason, a new method using a nano-indenter was developed in this study. Also, the improvement of the mechanical properties of rib materials from glass matrix to filler-contained composites will be determined in terms of Young's modulus, hardness and fracture toughness.

Consequently, the results suggest that using a nano indenter will be feasible for the measurement of mechanical properties of barrier ribs in PDP.

2. Experimental Procedure

For glass preparation of the matrix, all compositions used chemically pure reagents: PbO, SiO_2, $BaCO_3$, H_3BO_3, Al_2O_3 and ZnO. A batch of each composition, consisting of high purity raw materials was well mixed with a mortar and pestle. The batches were melted in a Pt crucible at 1200-1300□ for 1 h and stirred several times. Each glass melt was quickly poured and quenched on a steel plate and then the glass was ground to a powder (d_{50}=2㎛). The glass powder and alumina powder (High Purity Chemicals, Saitama, Japan, 2.3㎛) were mixed by ball mill for 48h and then dried. Subsequently, pellets were made using the CIP method and then sintered at 490-570℃. For the preparation of bulk specimens, the glass melts from the furnace were poured into a graphite mold, and heated to the temperature of 10□ above Tg of each glass. The mold was then moved back into the furnace to anneal the glass for 1 hr and then cooled very slowly in the furnace. The glass was removed from the mold and polished to the required size (0.5cm×0.5cm×1㎝) in order to measure the coefficient of thermal expansion (CTE).

The CTE of the glasses were measured using a vertical type of thermal mechanical analyzer (Rhometric, UK, TMA) with a heating rate of 5℃/min. Glass fiber of 0.5～0.75 in diameter and 23.5㎝ in length was made for Ts. The glass transition temperature (Tg) and crystallization peak were determined using a differential thermal analyzer (DTA-TA 1600, USA). The sintered samples were tested for density using the Archimedes method. For mechanical properties (Young's modulus and hardness), the composites were tested using a nano-indenter (Nano Indenter XP, MTS, USA). K_{1C} was calculated by the equation

$$K_{1C}=0.0421\times P^{0.6}\times a^{0.8}\times E^{0.4}\times C^{-1.5}$$

3. Results and Discussion

The thermal properties of the glass powder are shown in Table 1. The glass transition temperature was detected at 435℃, and 455℃ for the dilatometer softening point. Ts, the critical point for the sintering of glass/ceramic composites was evidenced at 519℃. The thermal expansion coefficient was in the applicable range (for Soda lime, 7～8.5×10^{-6}/K).

Table 1 Thermal analysis of glass frit

	Tg[a](℃)	Td[b](℃)	Ts[c](℃)	CTE[d] (×10^{-6}/K)
Glass	435±3	455±3	519±3	8±0.5

*a: glass transition, b: dilatometer softening point,
c: Littleton softening point d: thermal expansion coefficient

3.1. Sintering temperature

Figure 2 shows that the relative and apparent density increased with the increased firing temperature, abruptly so at 520℃. As shown in the figure, the relative density (R.D.) at 520℃ reached over 95%, which means that the sintered body became fully densified at this temperature. The highest value for RD was shown at 550℃, thereafter the temperature RD slightly decreased.

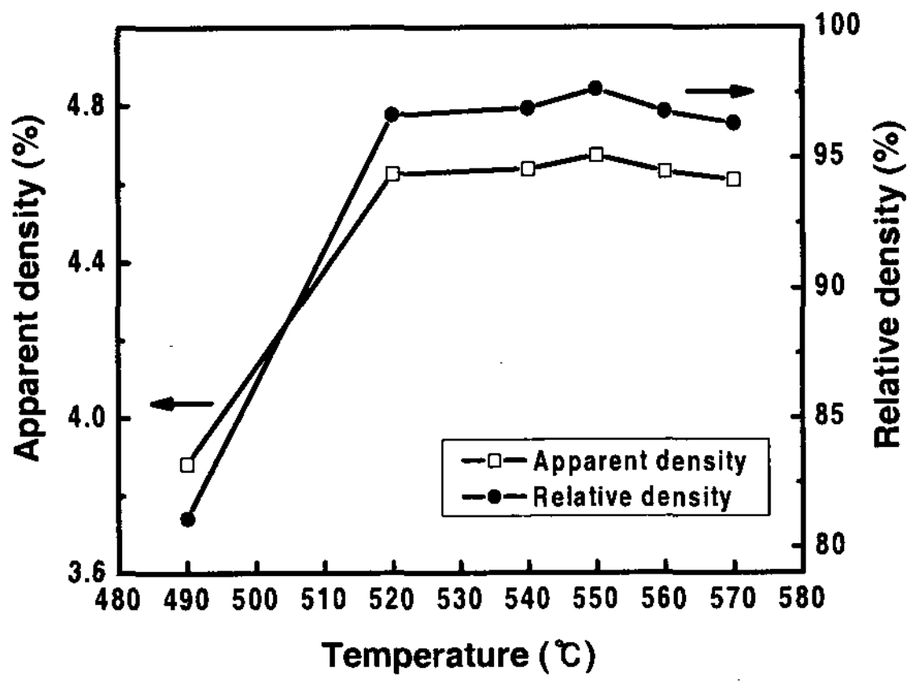

Fig. 2 Apparent and relative density as a function of sintering temperature: filler 20wt% fixed

Mechanical properties including Young's modulus (E), hardness (H) and fracture toughness (K_{1C}) are shown in Fig. 3(a) and (b). Young's modulus, hardness and fracture toughness increased as the sintering temperature increased. Similar to the density data, at 520℃, E and H abruptly increased to 83Gpa and 5.7GPa respectively and 1.62MPa·$m^{0.5}$ for K_{1C}. However, after this temperature there was no significant change in the value of E and H. The reason could be ascribed to the fact that sintering was already completed at 520℃, which is close to Ts of the glass frit, and a noticeable change did not occur until 560℃,

then slightly decreasing at 570℃ due to the formation of the closed pores in the matrix.

According to the previous studies involving commercial ribs [3], E and H under the same load condition (150gf) showed similar values after sintering (usually, near the Ts of each glass) although a slightly higher value for E was evidenced in this study. The reason may be due to a slightly larger amount of alumina than commercial composites in the matrix.

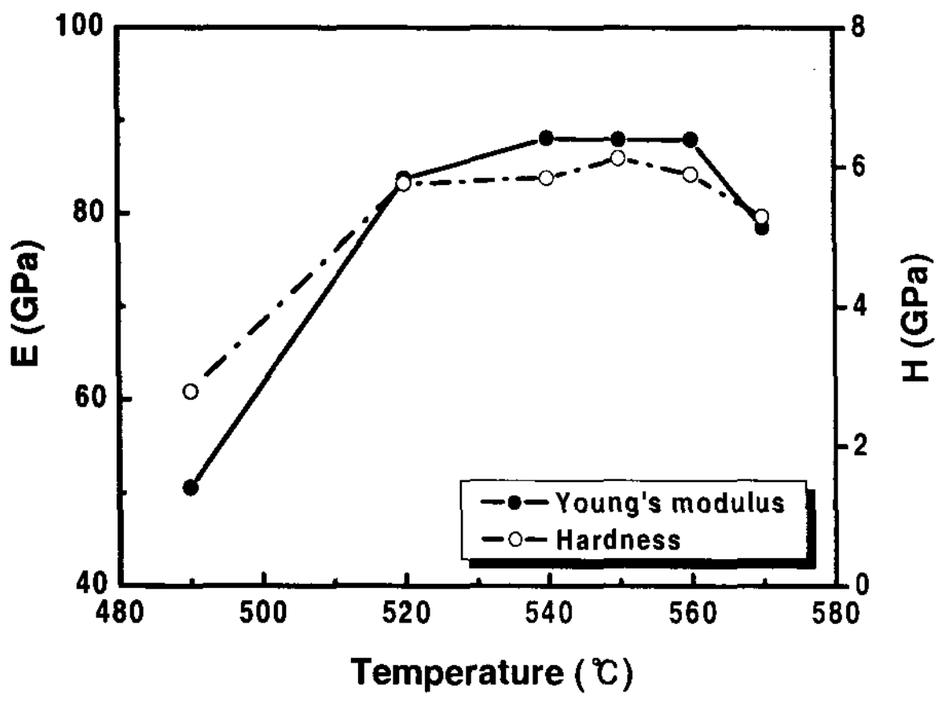

Fig. 3(a) Young's modulus and hardness as a function of temperature: filler 20wt% fixed, load=150gf

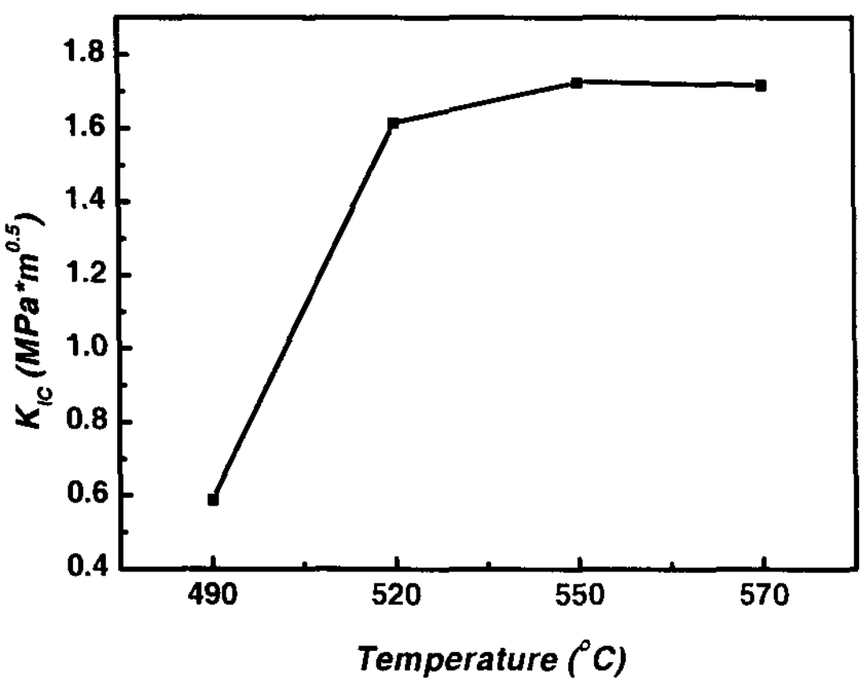

Fig. 3(b) Fracture toughness as a function of temperature: filler 20wt% fixed, load=2Kgf

With the SEM images below (Fig. 4), the improvement of the values (E, H) can be explained in part. At 490℃, the individual glass particles, which have just started to become viscous, are shown and there are unfilled spaces between the glass particles which are defined as open pores. At 570℃, round shaped, closed pores occurred.

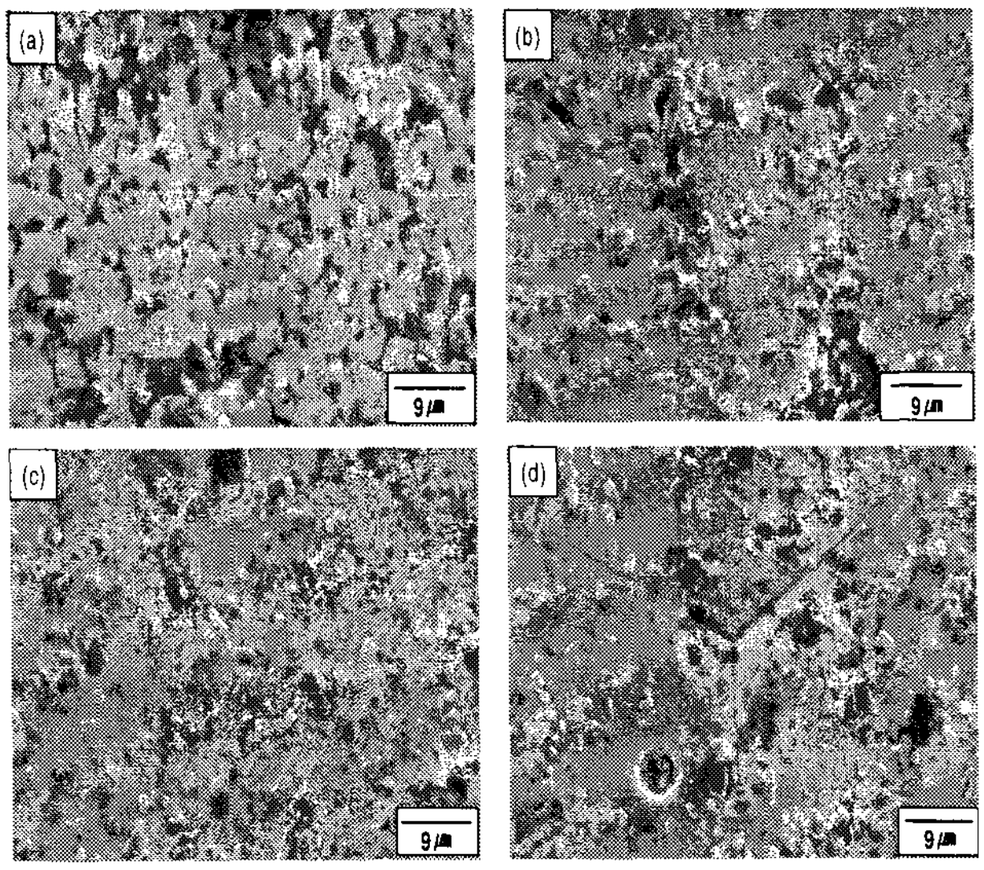

Fig. 4 Sintering images glass-alumina composites at various temperatures, (a) 490℃ (b) 520℃ (c) 550℃ (d) 570℃: alumina-20wt% fixed

3.2. Effect of Filler Content.

According to the density results (Fig. 2a), the sintering temperature of 550℃ was chosen because of the highest density characteristic at this temperature. At this temperature, the amount of alumina filler content varied from 0 to 30wt%. As shown in Fig 5(a), (b), mechanical properties improved from 53GPa (glass only) to 99GPa (containing 30wt% alumina) for Young's modulus, and from 5GPa to 5.5GPa for hardness. Similar trends were shown in K_{1C} results; the values represented from 0.60 to 2.63MPa·m$^{0.5}$.

These results can be explained in terms of the introduction of a compressive residual stress in the glass matrix [4-6] due to the mismatch in thermal expansion coefficients between the matrix (glass at 8.0) and alumina (8.9×10^{-6}/℃) fillers. These stresses originate from cooling. The processing temperature and the thermal residual stresses arising in composites are important because they can result in an improvement in the global fracture behavior of the material [7].

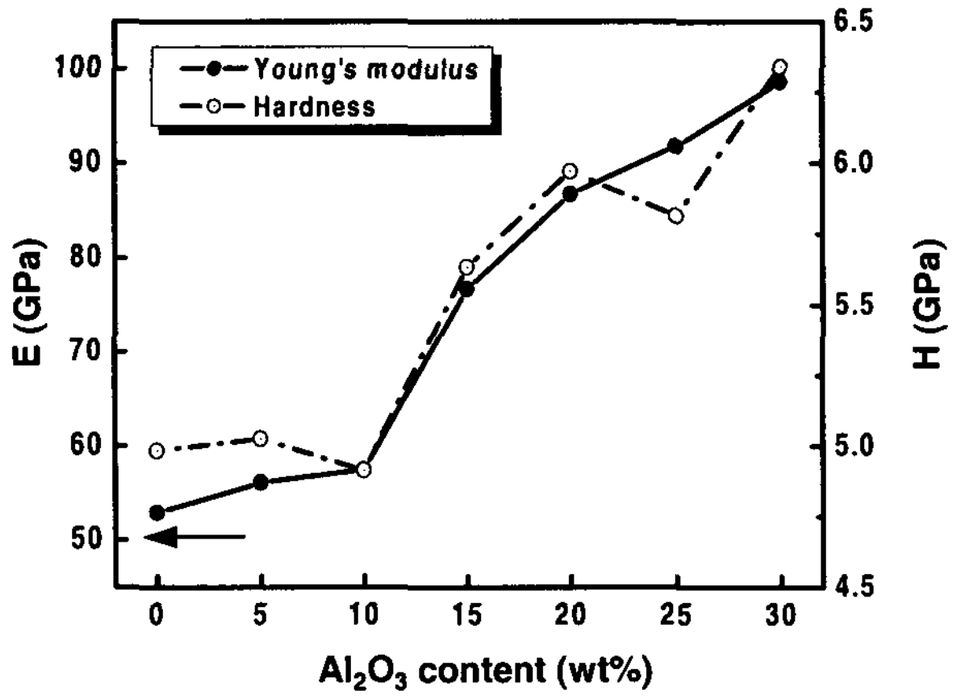

Fig. 5(a) Young's modulus and hardness as a function of filler (alumina) content: load=150gf at 550℃.

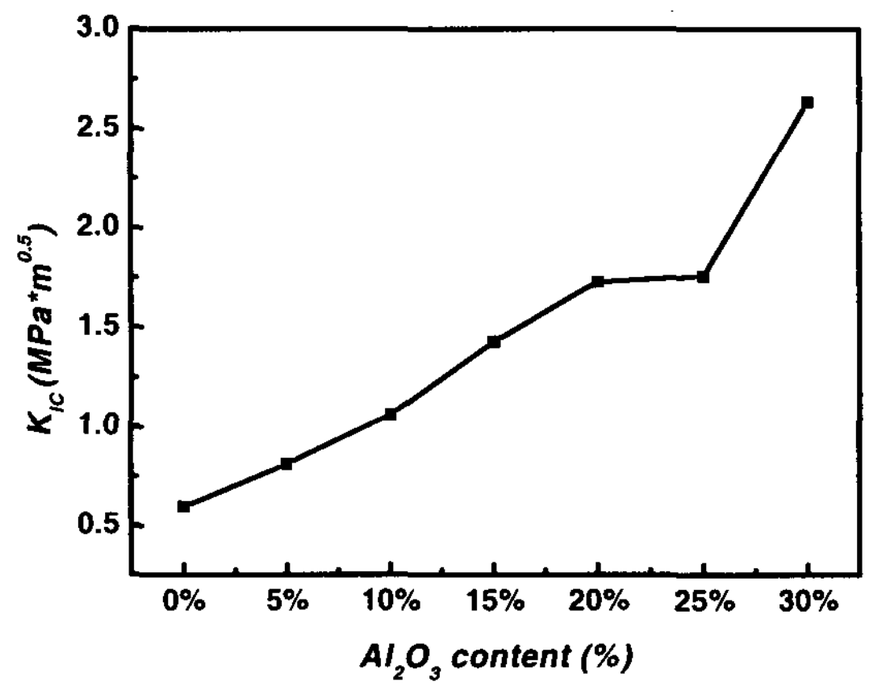

Fig. 5(b) Young's modulus and hardness as a function of filler (alumina) content: load=2Kgf at 550℃.

4. Conclusion

Evidence that the glass matrix was reinforced by ceramic fillers (Al_2O_3) was found in the present study investigating improvement of mechanical properties (E, H, K_{1C}) of barrier rib materials. For real application, other properties such as thermal expansion coefficient (CTE) and dielectric factor (ε) should be considered.

5. Acknowledgement

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6. References

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