Prediction of mechanical properties of a minute electronic device with porous ceramic

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It is difficult to measure the mechanical properties of the barrier rib materials in PDP (plasma display panel), which is a composite material reinforced by a glass matrix with rigid fillers. The purpose of this paper is to describe by using indentation technology, the method of evaluating the mechanical properties of two types of tiny bulk–dense, porous materials. In this work, the elastic constants and hardness of two types of barrier ribs were measured by Berkovich nanoindentation. As a result, cracks appeared around the load of 1345mN for the dense type of rib, while porous ribs endured up to 2427mN without any crack formation. Since the mechanical properties of rib' composites are closely related to porosity, an empirical relationship of hardness, elastic constant and strength versus porosity would review the mechanical properties of commercial ribs for a certain range of index, provided the porosity of the material is known. Thus, we suggest that the mechanical properties obtained by a nanoindenter can be used to evaluate the reliability of minute electronic devices.

Keywords: Nanoindentation; mechanical properties; elastic constant; hardness; porosity.

Es difícil medir las propiedades mecánicas de los materiales con nervadura en PDP (plasma display panel), que son materiales compuestos reforzados por una matriz de vidrio con rellenos rígidos. El propósito de este artículo es describir, usando tecnología de indentación, el método de evaluación de las propiedades mecánicas de dos tipo de materiales diminutos: compactos y porosos. En este trabajo, las constantes elásticas y dureza de dos tipos de nervaduras fueron medidas por nanoindentación de Berkovich. Como resultado, aparecieron cuarteaduras alrededor de la carga de 1345 mN para el tipo de nervaduras compactas, mientras las nervaduras porosas resistieron hasta 2427 mN sin formarse ninguna cuarteadora. Puesto que las propiedades mecánicas de los compuestos con nervadura están íntimamente relacionadas con la porosidad, una relación empírica de dureza, constante elástica y fuerza versus porosidad reexaminaría las propiedades mecánicas de nervaduras comerciales para un cierto nivel de índice, suponiendo conocida la porosidad del material. Así, nosotros sugerimos que las propiedades mecánicas obtenidas por un nanoindentador pueden ser usadas para evaluar la confiabilidad de dispositivos electrónicos diminutos.

Descriptores: Nanoindentación; propiedades mecánicas; constante elástica; dureza; porosidad.

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1. Introduction

Recently, micro electronic devices have been especially developed for high performance, small size, high quality, low cost, and simple process [1]. Therefore, it is important to evaluate the mechanical properties such as strength, hardness, stiffness, and ductility, which are correlated with the deformation and fracture of micro-electronic devices. There are many evaluation methods of mechanical properties for monolithic materials in industry [2-3]. However, it is difficult to evaluate the mechanical properties of composite materials because of many factors such as size and quantity of pores and crystallinity and size of crystalline phases in the matrix [4-5].

In the display industry, the mechanical properties of barrier ribs in PDP (Plasma Display Panel) are crucial for the improvement of the reliability of the panel because barrier ribs (cross section area: 80×120-150um) might fracture during fabrication processes such as the assembling and sealing of the parts. The functions of barrier ribs are to prevent the mixing of phosphors (RGB), to make a small space for discharge, and to sustain the front plate in PDP [6-7]. The processes of barrier ribs have been transited for forming a minute pattern by using screen printing, sand blasting and dry film for etching and photosensitivity. Generally, in the display industry, the processes for forming barrier ribs prefer the formation of minute patterns. As a result, the most popular dry film method is one that is capable of forming micro-pitches for etching and photosensitivity. The materials used for the formation of barrier ribs are mostly glass composites such as glass-ceramic matrix composite, glass matrix composite reinforced with fillers [6-7], and the barrier ribs are manufactured by a sintering process. The sintering process consolidates powder and evaporates gases from additives. Consequently, various shapes and sizes of pores are formed in the barrier ribs with sintering parameters, and porosity significantly affects the thermal, electrical, and mechanical properties of materials [8]. With respect to porosity, the barrier ribs are classified as dense and porous, and are manufactured according to microstructural parameters.

In this work, we evaluate and compare two types of barrier ribs (dense and porous) in PDP, which are composite materials with a tiny size bulk, based on mechanical properties (elastic modulus and hardness) versus porosity. Our experimental results suggest that the mechanical properties are strongly correlated to porosity, and that the reliability of a minute electronic device can be measured by nano indentation.

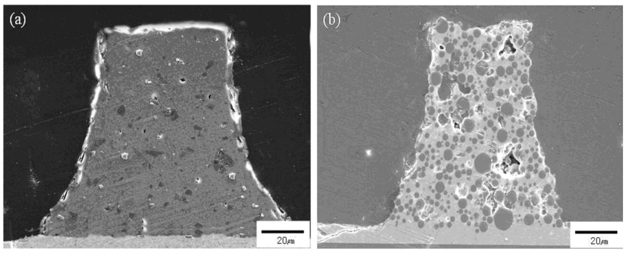

FIGURE 1. Surface morphologies of mounting samples: (a) dense and (b) porous barrier rib.

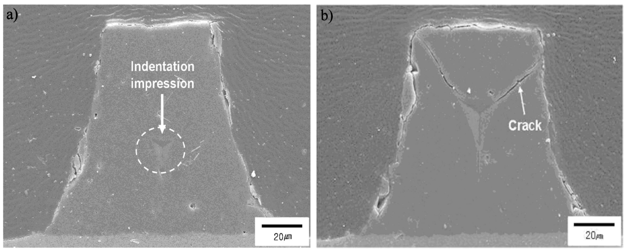

FIGURE 2. Indent images and crack propagation in D1 under different loads: (a) 50gf and (b) 150gf.

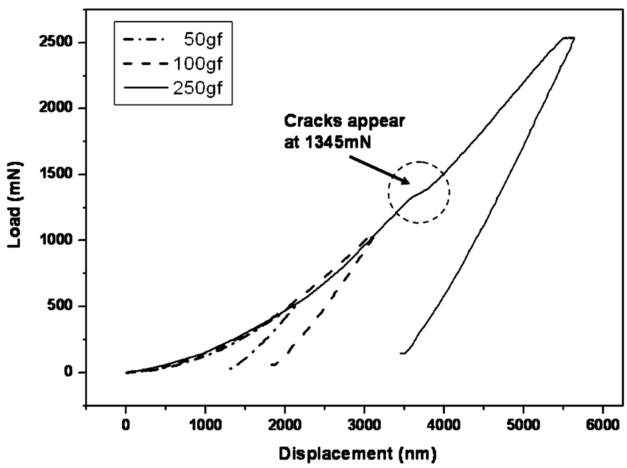

FIGURE 3. Comparison of the curves of load and displacement of D1 under different loads applied.

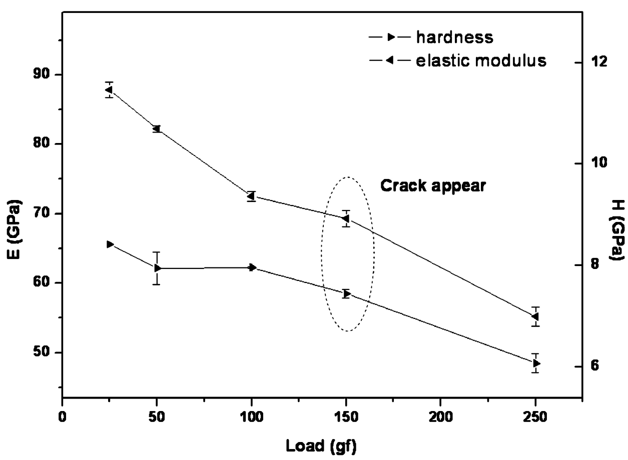

FIGURE 4. Mechanical properties (elastic modulus and hardness) of D1.

2. Experimental procedure

Two types of commercial barrier ribs (dense and porous) in PDP were used for samples. The barrier ribs were cut in the shape of a sample holder. The dimension of the barrier rib was 80 by 120§ (Fig. 1). The barrier ribs were vertically mounted with epoxy and polished with SiC paper and diamond paste (0.25§) on an auto polishing machine. Figure 1 shows the fine surfaces of polished samples.

The hardness and elastic modulus of the barrier rib were measured by nanoindentation (Nano Indenter XP, MTS, USA) [9]. The indenter of the nanoindentation was Berkovich, which has a pyramidal shape and is made of diamond (XPb1683, MTS, USA). When the tip was indented into the barrier rib in a sample, the optical microscopy was observed at the nanodindentation. The indentation for the barrier ribs was tested at least 5 times. The microstructure of the barrier rib was detected by FEG-SEM (Field Emission Gun-Scanning Electron Microscopy, S-4300SE, HIACHI, Japan) and EDX (Energy Dispersive X-Ray Micro-analysis System, Phoenix60, EDAX).

3. Results and discussion

The bulk samples used in this work are divided into dense (D1) and porous (P1) types in terms of the microstructure of the materials. They have a complex microstructure with fillers and pores in a matrix. As shown in Fig. 1, the P1 contains about 10§ the size of bimodal pores having approximately 10% for porosity. The EDX result showed that the P1, a glass-composite material, is composed of a PbO system of composition for the matrix and TiO_2, Al_2O_3 as a filler. On the other hand, the D1 composition for the matrix is a Pb-free glass system that is composed of various oxides such as Na_2O_3, SiO_2, Al_2O_3 and MgO for additive. The D1 is a glass matrix composite material, and its high density material means that the porosity is less than 2%.

The D1 barrier rib was indented with an increasing load (50-250gf) so that the cracks around the indent were found at 150gf in Fig. 2b. As D1 was indented with a load of 50gf, the indentation impression size was measured to be about 14§. As shown in Fig. 3, the occurrence of cracks accompanied a sudden change in displacement. It suggests that the formation of cracks could be predicted. Although an abrupt change of displacement does not occur at low loads (50, 100gf), the sudden non-smooth section appears at around 1345mN for high loads (250gf). This means that the formation of cracks appeared for a load smaller than 150gf (≒ 1500mN), which was found in Fig. 2b.

As shown in Fig. 4, the mechanical properties of D1 barrier rib decreased (from 89 to 58GPa in elastic modulus and from 8 to 6GPa in hardness) when an increasing load was applied. Before the cracks occurred around the indent, the hardness and elastic modulus decreased because of the indentation size effect (ISE). It has been reported that the hardness decreases with an increasing indentation depth. In other words, hardness is dependent on the load applied [10-12]. Hence, the true hardness of D1, which is known to be nearly constant in the relationship between indent size versus hardness, is around 8GPa. The results of load and displacement data (Fig. 3) show that the crack occurred at a load less than 150gf in D1 barrier rib, which means that the mechanical properties should be considered before the appearance of cracks.

TABLE I. Comparison of predicted and experimental mechanical properties.

Mechanical Properties	Prediction†		Experiment	
samples	D1	P1	D1	P1
Elastic modulus (GPa)	64.7	35.6	72.5 - 87.8	65.1 - 117.0
Hardness (GPa)	4.1	2.3	8.0 - 8.4	4.6 - 6.3
Strength (MPa)	36.25	24.84	-	-

†Calculated using the empirical formula with constants which was reported by Kim *et al.* [13].

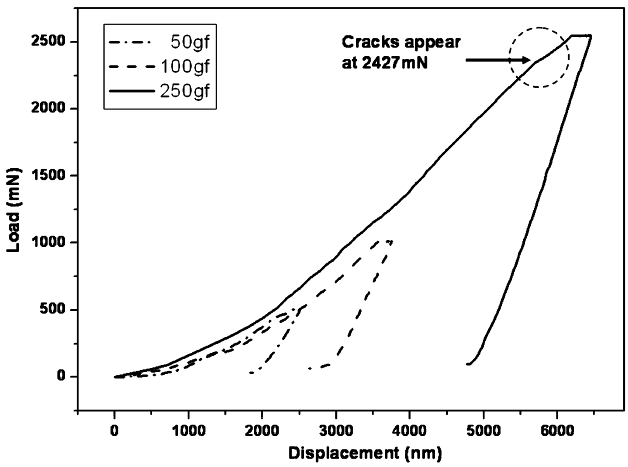

FIGURE 5. Load and displacement of P1 barrier rib.

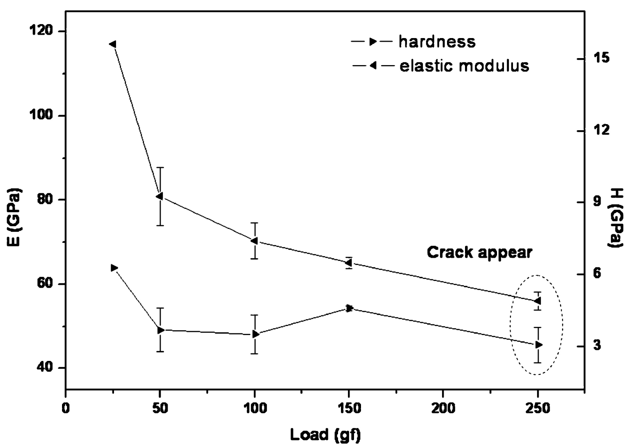

FIGURE 6. Mechanical properties (elastic modulus and hardness) of P1 barrier rib.

Compared with the mechanical properties of D1 (Figs. 3-4), P1 is significantly similar to the mechanical properties of D1 except for its hardness, which shows a nearly constant value in a certain range (less than 250gf) (Figs. 5-6). The elastic modulus decreased as the load increased. However, the hardness of P1 was less than D1 for a load greater than 100gf and yet cracks appeared for a load greater than 150gf in P1 barrier rib. In addition, the mechanical properties of P1 have larger error bars than D1 owing to two factors: the small size indenter (tip edge radius: $<0.1\mu$m) and the complex microstructure, which are composed of alumina powders (3-4§) as a filler and whose pores (>5um) are distributed in the matrix. The large error occurs for a lower load, 50-100gf load. For instance, at 50gf, the elastic modulus and hardness are about 80.9±7GPa and 3.7±1GPa, respectively. In Fig. 5, in the load and displacement of P1 barrier rib, the occurrence of cracks was confirmed for the load of 2427mN.

When D1 and P1 barrier ribs are compared in terms of the occurrence of cracks, the application of elastic and plastic deformation is significantly different. In the case of P1 barrier rib, the plastic deformation is more dominant than elastic deformation so that the displacement is longer than D1 while the recovering displacement is low. This supposed to be the reason for low hardness of P1 for the load of 50-100gf compared with the hardness of D1.

The microstructure of the barrier rib, which is a composite material, is similar to that discussed in the work of Kim *et al.* [13]. They showed that the porosity dependence of the hardness and elastic constant of ceramic tiles decreases rapidly initially, then more slowly by an increasing porosity. They suggest that the mechanical properties of ceramic tiles should be governed with the relationship between hardness (elastic modulus and strength) and porosity. It has also been reported that the constants (K_i and b) for elastic modulus and hardness are 69.1, 6.64 and 4.38, 6.27, respectively, in the empirical equation (H, S, E=$K_i \exp(-bP)$ where H, S and E is hardness, strength and elastic modulus and P is volume fraction of porosity) [13]. Using this formula, the elastic modulus of the D1 and P1 is predicted to be 64.7 and 35.6GPa, respectively. However, the elastic modulus of D1 and P1 was measured in the range of $72.5 - 87.8$ GPa and $65.1 - 117.0$ in our experiment. Based on the empirical relation of Kim *et al.*, *i.e.* E/H=18, H/MOR=100 for dense ceramic, E/H=12, H/MOR=85 for ceramic tiles (porosity <20%) [8,14], the strength of D1 and P1 is determined to be around 36.25 and 24.84MPa, respectively.

There is a slight difference in the elastic modulus between the prediction and our experiment. The difference can be explained in two ways based on our result and results of Kim *et al.* [13]. In one way, the components of the composites are significantly different. In the other way, in view of the microstructure, the effects of the components size, grain boundary, and shape of pores should be accounted for. The hardness of the D1 and P1 barrier ribs was similar to the elastic modulus. Table I shows in detail the hardness of the D1 and P1 barrier ribs for the empirical formula with constants reported by Kim *et al.* [13] and our results. However, the mechanical properties revealed by our experiment were insufficient for evaluation data according to porosity. We suggest that porosity is correlated to the mechanical properties, and that the mechanical properties of a composite depending on porosity call for further research. The minute parts of electronic devices for materials are useful for evaluating physical and mechanical properties using a nanoindenter, although the result might have a low reliability since they have to deal with a nano scale.

4. Conclusion

Nano indentation gave reliable values for mechanical properties of barrier ribs in PDP and in tiny bulk porous samples. The dense and porous types of barrier ribs showed characteristics of an abrupt curve in displacement and load obtained by nano indentation, which suggests an indication of crack initiation in the matrix. The hardness and elastic constant of samples are totally dependent on applied loads. An empirical equation to give strength, hardness, and elastic constant showed a slight difference value from the previous work because of the complex microstructure. However, porosity dependence of elastic constant and hardness is a key factor in predicting the values. Therefore, we suggest that the mechanical properties of nano scale materials should be evaluated for reliability of the minute electronic devices with a nanoindenter.

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