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Prevention of tapering in the tube-shaped sputtering target via initial heat treatment under external pressure

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Abstract

The tube-type transparent conducting oxide target is preferred to the planar type owing to a prolonged life. The uniaxial pressing of 2 wt% Al–ZnO powder into a tube shape and subsequent sintering resulted in a tapered product owing to the nonuniform green density. To suppress the taper, an initial heat treatment (IHT) under an external pressure was applied prior to the main final sintering. The degree of the taper was estimated from the difference in the percentage between the longer and shorter diameters of the tube. The application of pressure (1 MPa) during the IHT at 800 °C decreased the taper from 4.3% to 0.9% after pressureless final sintering at 1250 °C. The application of pressure during the IHT activated shrinkage at the bottom part of the tube and consequently reduced the tapering. With the IHT under pressure, micron-sized voids fragmented into the submicron pores, and the sintering potential was improved.
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Keywords: Al–ZnO; External pressure; Nonuniformity; Taper; Tube type target

1. Introduction

Sputtering is a widely accepted route for the preparation of thin film in various electronic devices. By the collision of accelerated ions onto the target, film with the same composition as that of the target is obtained. For the sputtering, a dense target is preferred to prevent the formation of nodules and extend the target life [1,2]. The nodules, produced by the arcing during the sputtering, can result in defective devices [1]. The arcing is related to the chemical and electrical nonuniformity in the target. In the case of indium tin oxide, a more uniform SnO_2 distribution reduced both the arcing and nodule formation during the sputtering [3]. The uneven distribution of the electric charges caused by pores also needs to be controlled to prevent the formation of nodules.

The extension of the target life can be achieved by preparing a tube-shaped target (also known as rotary type [4]) with increase in density. In the planar target, intensive erosion occurs selectively above the magnet. In contrast, the tube-type target is uniformly eroded as a result of the rotation during the sputtering. The tube-shaped target can be sustained until it loses 70% of its initial mass, whereas the planar target needs to be replaced after consuming ~30% of the initial weight [5]. Thus, the tube-shaped target is required for various materials. In the case of ceramics, as the target is prepared by sintering of the powder compact, the sintering step should support sufficient and uniform densification. However, the preparation of the uniformly compacted green body is often troublesome, particularly for a complex shaped product such as a tube.

A dense green body is obtained by fine filling with particles. Fluidic powder is favorable for the dense filling; however, the migration of powder is usually impeded by the agglomeration caused by the van der Waals attraction. In the preparation of the tube-shaped green body, the bottom of the mold is the hardest part to fill, as it is narrow and distant from the entrance of the powder supply. Besides, during the uniaxial compression, the pressure transmitted to the bottom is reduced [6]

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owing to the friction with the side wall. In the tube-shaped green body, the reduction in the pressure is even greater at the bottom, because of the presence of the inner wall. Thus, the bottom part is prone to have reduced green density, because of the reduced pressure [6] as well as the unfavorable particle filling. The difference in green density between the top and bottom parts of the tube can affect the shrinkage during the sintering, resulting in the tapered product.

The nonuniformity may be practically controlled by the introduction of the granulated fluidic powder. The use of a lubricant is also an effective approach to reduce nonuniformity by decreasing friction with the side wall. However, few reports are available that document a sintering approach. The two-step sintering process has been introduced for the sintering of various materials to increase the sinterability of the aggregated materials [7], volatile materials [8] or reducing the sintering temperature [9]. The two-step sintering process involves an initial heat treatment (IHT) under pressure and a final pressureless sintering. The role of the IHT was reported to be the recovery of the sintering potential by the fragmentation of the microsized pore into submicron pores [10].

Since the tapering is the result of the difference in the shrinkage, the two-step sintering route can be applied to prevent tapering by the promotion of uniform densification throughout the green body. In this study, ZnO doped with 2 wt% Al was chosen as the transparent conducting oxide (TCO) and the two-step sintering route was tested as a route for the preparation of a tube-shaped TCO target with a controlled taper.

2. Materials and methods

The Al-doped ZnO powder was prepared following the literature procedure [9,10]. $Al(NO_3)_3 9H_2O$ (Showa Chemicals, 98%) was weighed for the weight percentage of Al_2O_3 to be two after oxidation; therefore, $Al_2O_3/(Al_2O_3+ZnO)$ was 2 wt%. The powder was labeled as 2Al–ZnO. After the heat treatment at 400 °C for 30 min, the powder was ground in an agate mortar and then passed through a 360 mesh (40 μm) sieve to obtain secondary particles of regulated size. The resulting granules were ranged from 20 to 35 μm with an average primary particle size of 0.3 μm. The prepared 2Al–ZnO powder was placed in a cylindrical mold with a central pillar to obtain a tube-shaped green body. The mold was designed for the green body to have outer diameter and inner diameters of 10 and 5 mm, respectively. 0.9 g of 2Al–ZnO was weighed and pressed in a single action mold by pressing at 10 MPa for 3 min. The green tube was then cold isostatically pressed at 100 MPa for 10 min.

The prepared green tube was placed between two SiC rams in a SiC mold. Coarse (5 μm on average) alumina powder was placed around the specimen as the medium for transferring the external pressure from the rams, while minimizing the chemical reaction. The mold unit was placed in a furnace attached to a material testing machine (DYMH-100, Daeyeong Co., Korea). The specimen was heated to 800 or 900 °C at the rate of 5 °C/min under an external pressure of 0 or 1 MPa for 10 min. As soon as the specimen was taken out of the mold after the heat treatment, the coarse alumina powder was removed from the specimen.

After the IHT, some specimens were horizontally bisected to separate the green body into top and bottom parts. For each part, the specific surface area and pore-size distribution were measured using a BET surface area analyzer (ASAP-2010, Micrometrics, USA) and mercury intrusion porosimeter (Autopore IV 9500, Micrometrics, USA), respectively.

The specimens were sintered without pressure in the range of 1100–1350 °C for 2 h in air using a separate furnace. Before and after sintering, the diameters at the top and bottom of the green body were measured using a vernier caliper. The sintered specimens were then vertically bisected using a diamond saw for the microstructural analysis. The exposed surface was sequentially polished with SiC abrasive paper and diamond pastes (6, 3, and 1 μm). The polished specimens were then thermally etched in the range of 1100–1200 °C for 1 h to develop the microstructures. Scanning electron microscopy (JSM-6300, JEOL, Japan) was used at 20 kV for the surface morphological observation. The density and porosity of the bulk specimen were also estimated using an image analyzer from the microstructure.

3. Results

Fig. 1 shows the top and side views of the tube shaped green body after cold isostatic pressing (CIP). As shown in the side view of the tube in Fig. 1, the diameter at the bottom (d_b) differed from that at the top (d_t). The degree of taper was determined from Eq. (1) in this study. According to Eq. (1), the degree of taper increases with increasing difference between d_b and d_t. The degree of taper is 0 for the tube with the same d_b and d_t

$$\text{taper}(\%) = \left(\frac{d_b - d_t}{d_b}\right) \times 100 \quad (1)$$

The uniaxially pressed green tube had the height and diameter of 6.32 ± 0.18 and 9.98 ± 0.02 mm, respectively, at both the top and bottom. After the CIP, d_t and d_b decreased to 8.73 ± 0.11 and 8.91 ± 0.08 mm, respectively, and the height of the tube decreased to 5.61 ± 0.06 mm. Therefore the shrinkage at the top of the tube was greater than at the bottom as a result of the CIP. The inner diameter was found to be 4.52 ± 0.05 mm at both the top and bottom. From the result of the CIP, the top of the tube shrank by 12%, whereas the bottom of the tube shrank by 11%. The degree of taper was thus 1.93% after the CIP. The density of the green tube after the CIP was 2.46 g/ml.

According to a previous report [10], the bonding across the secondary particles needs to be achieved during the IHT with an external pressure. In the case of 2Al–ZnO, the pressure of 1 or 2 MPa was sufficient during the IHT at 700–900 °C to induce bonding among the secondary particles. Thus, similar experimental conditions (IHT temperature of 800–900 °C, external pressure of 1 MPa, and final sintering temperature of 1250–1350 °C) were selected.

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