UDC 66

STUDY ON THE PROPERTIES OF IN2O3·SNO2 TARGETS ASSISTED BY ULTRASOUND

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Abstract.

In this paper, ITO (In2O3: SnO2= 9:1, wt%) with diameter of 20~70nm and purity ≥99.99% was prepared into fluffy particles with size of 15 ~ 20um. Then, ITO (In2O3: Sno2 = 9:1, wt%) was filled in a mold that could be introduced into strong ultrasound and placed under cold isostatic pressure to obtain high quality blank. Under the dual action of the collector pressing and powerful ultrasound assistance (CP+ PUA) with the power P=3Kw and the cold isostatic pressure Pw=250MPa, the pressure is maintained for 1min. ITO blank with diameter of φ=14mm, density up to 68% and uniform microstructure was prepared. Finally, ITO targets with density ≥99.5%, uniform microstructure and fine grain were obtained by sintering at 1550℃ for 2h in a vacuum tungsten furnace.

1 Introduction

Indium tin oxide composite ceramics (In2O3· SnO2 referred to as ITO) has the characteristics of transparency, strong reflectivity and refractive index. Transparent conductive films made by magnetron sputtering using its transparent conductive properties are widely used in touch sensors of mobile phones and tablets, solar cells and gas sensors, accounting for more than 95% of the market share. However, due to the defects of density and uniformity, the domestic ITO target can only be used in the low-end market. How to obtain ITO targets with high density (≥99.5%), high purity (≥99.99%), uniform microstructure and fine grain is one of the hot topics in materials research. The preparation of ITO target is mainly divided into three parts: preliminary powder treatment, blank forming process and sintering process. Earlier researchers, such as H. Wang et al., have conducted detailed and systematic research on sintering process. How to obtain high quality blank is the key to sintering high quality target material, and is also the focus of current research.

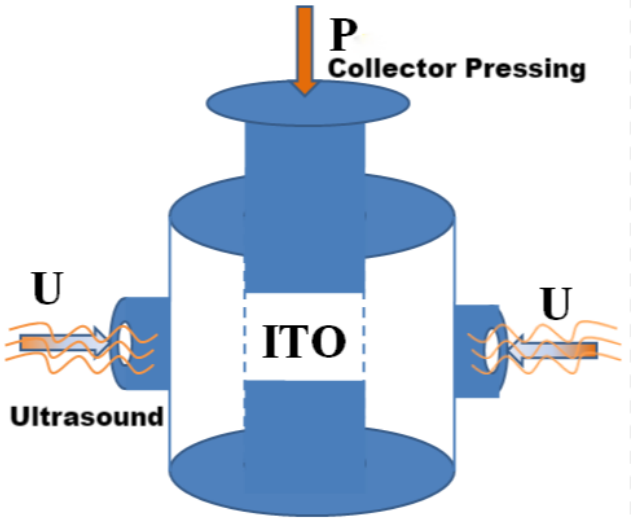


Figure 1. Schematic diagram of powerful ultrasound assistance mold

High quality blank is obtained by ultrasonic shock in the process of forming the blank. Using polyvinyl alcohol (PVA) as binder, ITO nanoparticles were prepared into ITO particles with the size of 15~20um. Then, as shown in Figure 1, ITO particles were put into the mold, high-power ultrasonic wave was introduced, and cold isostatic pressing was performed to produce ITO blank with uniform microstructure and density. ITO blank was pre-sintered in Muffle furnace to remove binder and other impurities, and then it was sintered in vacuum tungsten wire furnace to obtain ITO target material with high density and uniform microstructure.

2 Experimental

2.1 Material and reagents

ITO (In2O3: SnO2= 9:1, wt%) with particle size of 20~70nm and purity ≥99.99%, adding 1.5% polyvinyl alcohol (PVA) as binder, through the Suzhou Guolang GLZ2-25 dry granulator, the size of 15-20um fluffy particles.

2.2 ITO blank was prepared

The particles are filled in the mold and placed under cool pressure. Adjust the pressure of the hydraulic press, and set the internal pressure of the die to 400MPa, 600MPa, 800MPa, 1000MPa, 1200MPa and 1500 MPa respectively. The samples obtained by direct extrusion molding under isostatic pressure (the method referred to as CP) were placed in Muffle furnace and sintered at 550℃ for 1 hour. The obtained ITO blank samples were denoted as an (a1, a2,..., a6). Under the same pressure, same frequency ultrasonic wave with power P=3Kw is introduced (Figure 1). The samples formed under the double action of isostatic pressure and strong ultrasonic assistance (CP+PUA for short) were placed in Muffle furnace and sintered at 550℃ for 1 hour. The obtained ITO blank samples were denoted as bn (b1, b2,..., b6).

2.3 Sintered ITO target

According to previous scientific research results, ITO will lose evaporation when it exceeds 1600℃, and the sintering temperature of ITO target is generally around 1600℃. Therefore, high quality ITO blanks with high density and uniform microstructure were selected to prepare ITO targets by vacuum sintering at 1550℃. The high-quality ITO blank obtained only by isostatic pressing was put into the vacuum tungsten wire furnace manufactured by Haichenhua and sintered at 1550℃

for 60min, 120min, 150min, 180min, 240min and 300min respectively. The samples obtained were CPn (CP1, CP2,..., CP6). The high-quality ITO blank formed under the double action of isostatic pressure and strong ultrasonic assistance was put into the vacuum tungsten wire furnace made by Haichenhua and sintered at 1550℃ for 60min, 80min, 100min, 120min and 140min respectively. The samples obtained were CAn (CA1, CA2,..., CA5).

3 Experimental results testing and analysis

3.1 Effect of strong ultrasonic shock on ITO blank forming under pressure

Testing the density of ITO blank samples (a_n and b_n), and Figure 2 was obtained according to the results.

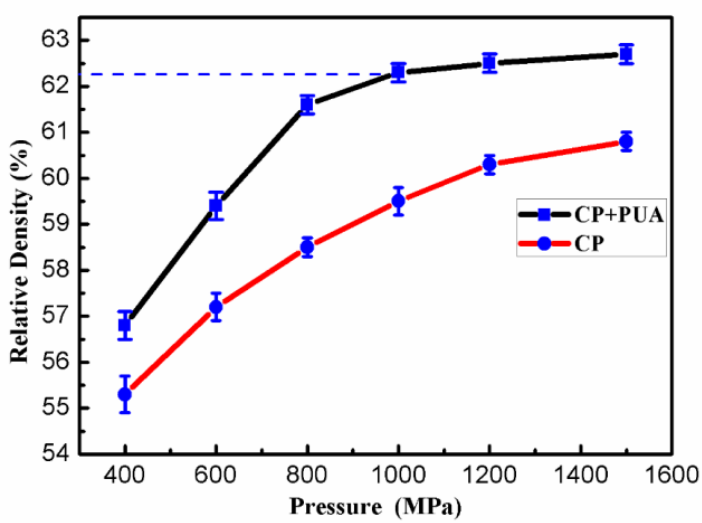


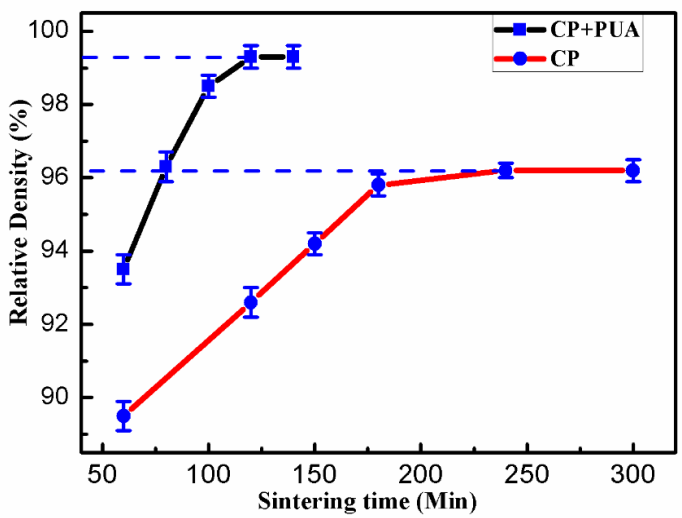
Figure 2. The effect curve of ultrasonic oscillation of Pw=3Kw on ITO blank forming under different cold isostatic pressure

As shown in Figure 2, with the increase of pressure, the density of blank also gradually increased, but the increasing trend gradually weakened. When the pressure reached 1000MPa, the density of the blank was 62.2±0.2%. As the pressure continued to rise, the density of the blank was very slow, so the pressure of 1000MPa was the optimal pressure condition of this method.

3.2 Influence of sintering time on density of ITO target

Testing the density of ITO target samples (CPn and CAn), and Figure 2 was obtained according to the results.

Figure 3. Density curve of ITO target sintered at different times



As shown in Figure 3, the density of ITO target made by high temperature sintering of ultrasonic assisted forming blank is significantly higher than that of ITO target made by direct extrusion forming blank made by high temperature sintering. The highest density was (99.3±0.3)% after sintering for 120min at 1550℃. The density of the blank was (96.2±0.2)% after sintering at 1550℃ for 4h.

3.3 Microstructure analysis of ITO target

The author used FEI's Quanta-250 scanning electron microscope (SEM) to test the morphology of ITO target obtained after the raw blank with ultrasound assisted molding was sintered at 1550℃ for 60min, 80min, 120min and 140min, as shown in Figure 4.

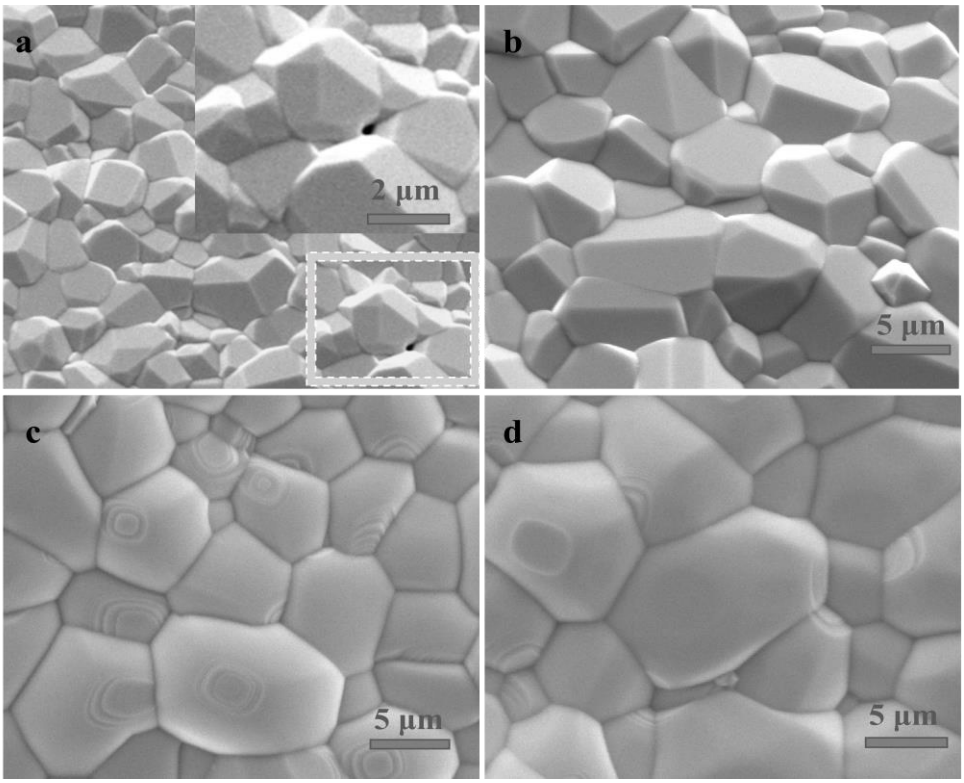


Figure 4. Microstructure of ITO targets prepared at different sintering times

As can be seen from Figure 4, different sintering times have a great influence on the densification of the product. In Figure 4a, most of the grains are between 2 and 5um, and obvious pores can be seen. With the extension of sintering time, the grains grow up gradually and the pores disappear. As shown in Figure 4c, most of the grains are between 5 and 7um, without pores and other defects, and each grain is closely arranged with obvious grain boundaries. With the extension of sintering time, the grains continue to grow to about 8um, but there is no obvious change in the structure of the grains.

3.4 The resistivity of ITO target

The resistivity of ITO target samples (CPn and CAn) was tested by Van der Burgh method, and Figure 5 was obtained according to the results.

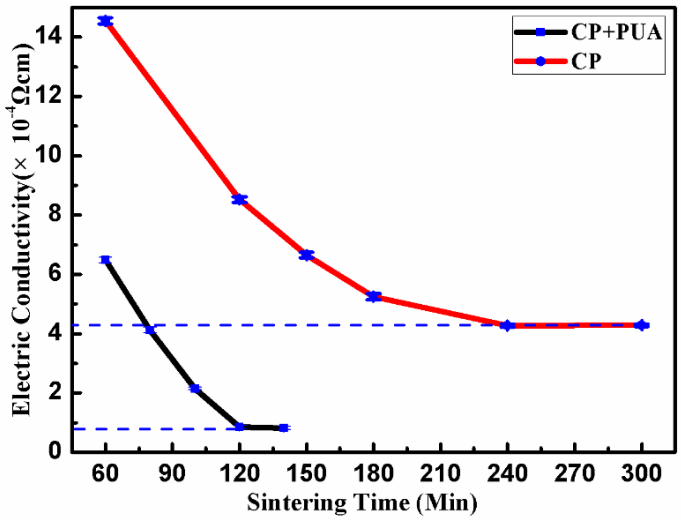


Figure 5. Resistivity curves of ITO targets at different sintering times

According to the Figure 5, the resistivity of the blank formed by ultrasonic assisted pressing after sintering at high temperature is significantly higher than that of the sample sintered after direct pressing. After sintering for 120min, the conductivity of the sample is (0.85±0.05)×10-4Ωcm. The lowest conductivity of the directly extruded sample is (4.28±0.05)×10-4Ωcm, which is basically consistent with the density variation of the target material shown in Figure 3.

Conclusions

When strong ultrasonic shock is introduced into the forming of ITO particle blank, the ITO blank prepared at 1000MPa reaches 62.2%, which is about 3% higher than the direct extrusion forming. After sintering at 1550℃ for 120min, the ITO target material has a density of (99.3±0.3)% and an electrical conductivity of (0.85±0.05)×10-4Ω cm. The density of ITO blank formed by extrusion is only about 92.5% and the conductivity is only (4.28±0.05)×10-4Ω cm under the same sintering condition.

Acknowledgments

This work was the Project Supported by Scientific and Technological Research Program of Chongqing Municipal Education Commission (Grant No. KJQN202101335)

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