Rapid way to obtained translucent ZnO ceramics by Spark Plasma Sintering

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Highlights

- Pure nanometric ZnO powder is synthesized by coprecipitation.
- Fully dense ZnO ceramics obtained in short dwell time (5'), low T° (700°C) under 400 MPa.
- Optimized cycle allow to obtain translucent ZnO pellet (RIT ~ 40% at λ = 640 nm).

1. Introduction

Obtaining transparent polycrystalline ceramic oxides is a challenge today. It is possible to achieved optical transparency if the ceramic is fully dense by limiting as much as possible the presence and the size of defects such as porosity or grain boundaries which are diffusing elements [1]. Here we present our work on the optimization of the spark plasma sintering cycle of a synthesized powder to produce fully dense pellet (>99%). Under these conditions, a translucency of about 40% for a wavelength of 640 nm was obtained.

2. Methods

First, zinc oxalate powder was synthesized by coprecipitation [2] in order to obtain pure homogeneous nanocrystalline powder. Zinc oxide powder is then obtained after calcination at 500°C of zinc oxalate. The synthetized powder is composed of grains of 700 ± 65 nm with crystallites size of 42 ± 8 nm.

The samples were sintered using a Dr Sinter 2080 Spark Plasma Sintering apparatus (Sumitomo Coal Mining Co. Ltd.,Japan) available at the "Plateforme Nationale CNRS de Frittage Flash" (PNF2-CNRS) located at Université Toulouse III Paul Sabatier (France). The powder was introduced in an 8 inner diameter tungsten carbide die lined with 0.2 mm thick graphite foil then pre-compacted and placed in the SPS chamber under vacuum (5 Pa). A pulse sequence of 12:2 was applied to heat the sample. The temperature was monitored using a thermocouple in a hole 1.8 mm in diameter and 3 mm deep located at the external surface of the die.

The real in-line transmission (RIT) measurements in the range of λ 200 – 1200 nm were conducted on a PVE300 photovoltaic EQE device equipped with an integrating sphere taking into account the diffused part of the beam.

3. Results and discussion

In order to fully densified the ZnO powder, the sintering was carried out by Spark Plasma Sintering [3]. The test parameters from previous works [2] have been optimized. Indeed the pressure was applied on a 2 minutes stage at 520°C at the time when the sintering speed is the highest. Then a pressure of 400 MPa (250 MPa in previous work) is applied and maintained all along the cycle with a sintering temperature of 700°C and a dwell time of only 5 minutes.

In this case the grain size is about 4 µm with a relative density of 99,9%. But due to very low oxygen partial pressure in the SPS chamber there is the formation of a large number of oxygen vacancies. They may result from the reduction of Zn^{2+} cations to Zn metal by charge compensation [2].

Then, the ceramics are heat treated for 10 h at 600°C in an air furnace to reoxydize ZnO. The pellets change from grey to un-colored. The transmittance determined before and after annealing on a ZnO pellet is reported on figure 1. One can observe that the transmittance at low wavelengths (< 500 nm), but also at wavelengths in the IR (> 800 nm) is improved after annealing. A high value of RIT is obtained (40%) is obtained at a wavelength of 640 nm.

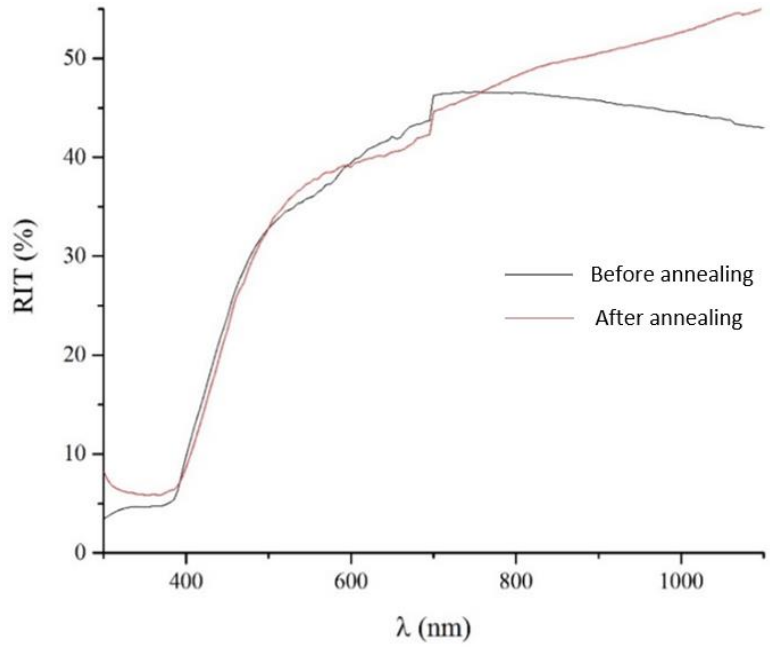

Figure 1. Transmittance before and after annealing on a ZnO pellet (700°C - 400 MPa, dwell time 5 min) thickness 0,5 mm.

4. Conclusions

Nanopowder of ZnO were succefully synthesized by soft chemistry route and densified by Spark Plasma Sintering at low temperature i.e. 700°C for 5 min under a high pressure of 400 MPa. These parameters permitted to obtain translucent ZnO pellet, which after annealing, demonstrated for an optimal value of RIT: 40% at λ =640 nm.

References

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Keywords
ZnO, Spark Plasma Sintering, Translucent