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ScienceDirect

Physics Procedia 72 (2015) 374 – 377

Physics Procedia

Conference of Physics of Nonequilibrium Atomic Systems and Composites, PNASC 2015, 18-20 February 2015 and the Conference of Heterostructures for microwave, power and optoelectronics: physics, technology and devices (Heterostructures), 19 February 2015

Influence of Pressure in Flash Sintering Technique

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Abstract

An innovative pressure-assisted flash-sintering technique has been developed to investigate the effect of pressure applied on microstructure and the sintering behavior of titanium dioxide. There are numerous applications for titanium dioxide in ceramics including microeletronics, glass ceramics, refractive materials, structural ceramics and titanium-containing ceramic materials and chemical intermediates. The traditional sintering of titanium dioxide usually requires several hours at over 1200°C. The conducted research indicates that titanium dioxide can be sintered to full density in only a few seconds at 800-1000°C, when subjected to a DC electrical field at a certain temperature moment.

Keywords: flash sintering, titanium dioxide, pressure applied

1. Introduction

There are traditional methods (convectional sintering, hot pressing, etc.), but now developed new method flash sintering [1]. The point this method is application electrical potential to sample at a certain temperature point, wherein the sample becomes conductive. In result resist of material flowing electrical current occurs instantaneous rise temperature into the sample and his sintering. Until that time scientists application electrical potential to sample without pressure assist, but we applicated pressure together with application electrical potential.

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doi:10.1016/j.phpro.2015.09.056

2. Material

In this work we used powder of titanium dioxide. This material have melting point around 1800 C and semiconductor properties. The powder used in this work have particle size around 30-50 mkm and lower fraction 4-6 mkm.

3. Experiments and results

In this work besides method flash sintering for comparison we used classical methods such as convectional sintering in dilatometr and hot pressing.

For sintering in dilatometr we used green compact with diameter equal 10 mm which was pressed at 130 MPa. For hot pressing we used molybdenum punches and graphite die, sintering was carried out in hot press. For method flash sintering was used same hot press with plug electrical block which have force equal 16 A and voltage 500 V. By the electrical block current flowing through the sample. For method flash sintering we used molybdenum punches like in hot pressing, but the material of die was mulit which has insulation properties. This isolation die was surrounded steel clamp for safe his integrity. Isolation die is needed for flowing current only through the sample.

First stage, we made hot pressing and sintering in dilatometr powder titanium dioxide. Modes of heating and value of density sample shown in table 1. Obviously, in case of hot pressing even with minimal applied pressure (13 MPa) the shrinkage more intensive, then in case of convectional sintering in dilatometr.

Table 1. Modes of heating and value of density sample.

Name	T(max), C^0	Ramp rate, C^0/min	Dwell time, min	Pressure, MPa	Density, %TD
HP1000	1000	25	5	13	77,00
HP1200	1200	25	120	64	98,48
Dil1	1000	25	5	0	59,63
Dil2	1200	10	0	0	88,15
Dil3	1600	10	120	0	81,26

Second stage – flash sintering experiment. On this stage is observed two outcomes of events. The first exit then at large density of current and small values of temperature we observed poor quality structure within the sample as on fig 1a. All samples have the same structure, all they have five zones:

1) Spherical cavity within the sample. Cavity have surface as shown on fig 1b.
2) Monolithic, nonporous material around the spherical cavity (fig 1c).
3) Transition zone between nonporous material and material sintered until average density. (Fig 1d).
4) Average density material (fig 1e).
5) Unsintered powder of titanium dioxide (fig 1f).

The second exit – at smaller density of current and larger values of temperature we observed quality structure like at on fig 1 (e).

Fabio Trombin and Rishi Raj describe the same outcomes of events in [2].