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INVESTIGATION OF ZINC STANNATE SYNTHESIS USING PHOTOACOUSTIC SPECTROSCOPY

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Abstract

Mixtures of ZnO and SnO_2 powders, with the molar ratio of 2:1, were mechanically activated for 40, 80 and 160 minutes in a planetary ball mill. The resulting powders were compacted into pellets and non-isothermally sintered up to 1200°C with a heating rate of 5°C/min. X-ray diffraction analysis of obtained powders and sintered samples was performed in order to investigate changes of the phase composition. The microstructure of sintered samples was examined by scanning electron microscopy. The photoacoustic phase and amplitude spectra of sintered samples were measured as a function of the laser beam modulating frequency using a transmission detection configuration. Fitting of experimental data enabled determination of photoacoustic properties including thermal diffusivity.

Introduction

Zinc stannate belongs to A_2BO_4 compounds (A = group II, e.g. Zn, B = group IV, e.g. Sn, Ge). They are called spinels and have semiconducting properties. Presumably their sensor properties are mostly derived from the fact that their electrical conductivity is sensitive to oxygen stoichiometry and environmental atmosphere. Zinc stannate spinel, Zn_2SnO_4, investigated in this work is potentially good gas and humidity sensor. In this paper, we present the results of a photoacoustic investigation of thermal and transport properties of bulk zinc stannate synthesized by reaction sintering process. Photoacoustic (PA) spectroscopy has been used lately, besides for the characterization of electronic, optical and defects stuctures, for defining the electronic states and structural disorders of ceramic materials.

Experimental

Mill type		Planetary ball mill (Fritsch Pulverisette 5)	
Starting powders		ZnO SnO_2	
ZnO:SnO_2 molar ratio		2:1	
m_{powder}/m_{ball} ratio (D_{ball} = 10 mm)		1:40	
Grinding time, min.	40	80	160
Sample	ZSO-40	ZSO-80	ZSO-160

-X-ray diffractometer (Norelco-Philips PW-1050) with CuKα radiation and a step scan mode of 0.02/0.4s

-Scanning electron microscopy (JSM 5300 JEOL)

-Sensitive dilatometer (Bähr Gerätebau GmbH Type 702s)

-Photoacoustic set-up with an infrared laser (25 mW) as the optical source (Fig. 7)

Results

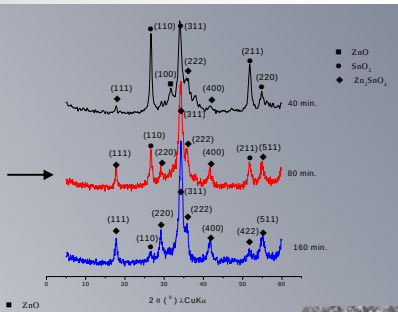

Fig. 1 XRD patterns of ZTO powder mixtures as a function of the time of activation

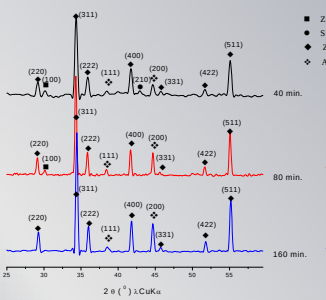

Fig. 2 XDR patterns of ZSO samples non-isothermally sintered up to 1200°C with a heating rate of 5ºC/min.

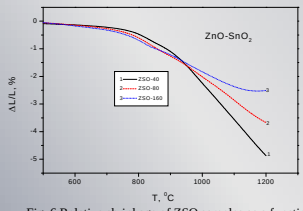

Fig. 6 Relative shrinkage of ZSO samples as a function of the heating temperature and time of activation during non-isothermal sintering up to 1200ºC with a heating rate of 5ºC/min.

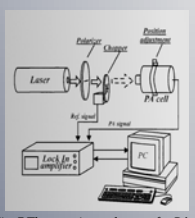

Fig. 7 The experimental set-up for PA measurements.

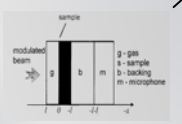

Fig. 8 The gas-sample-backing-microphone detection configuration.

$$S(-l_s,\omega)=\frac{\gamma\cdot P_0}{T_0\cdot k_b\cdot l_b}\Phi_s(-l_s,\omega)$$

- l_s - sample thickness
- ω - frequency of the laser beam
- γ - adiabatic constant of the gas
- P_0, T_0 - ambiant pressure and temperature
- $k_b=\frac{(1+j)}{\mu_b}, \mu_b$ - thermal diffusion length of the backing gas
- l_b - distance between the sample and the microphone membrane
- $\Phi_s(-l,\omega)$ - temperature variation of the sample surface that is in contact with the microphone

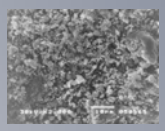

Fig. 3 SEM fractured surface of the ZSO-40 sample sintered at 1200°C.

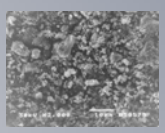

Fig. 4 SEM fractured surface of the ZSO-80 sample sintered at 1200°C.

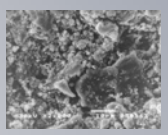

Fig. 5 SEM fractured surface of the ZSO-160 sample sintered at 1200°C.

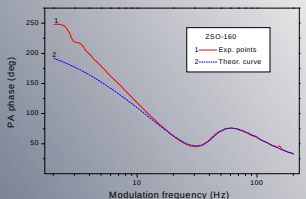

Fig. 9 Phase photoacoustic spectra for ZSO-160 sample.

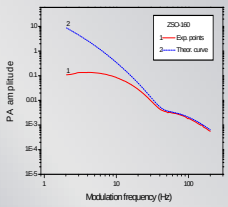

Fig. 10 Amplitude photoacoustic spectra for ZSO-160 sample.

Conclusion

-monophased zinc stannate was synthesized when the mixture milled for 160 min was sintered at 1200°C

-grinding leads to the formation of a structure with reduced grain size that accelerates spinel formation (SEM and XRD analysis) but agglomerates also present

-grain growth of spinel with increasing activation time could inhibits densification and cause the formation of a porous microstructure (dilatometry and SEM)

-the value of the thermal diffusivity obtained for ZSO-160 (pure zinc stannate phase) is almost identical to thermal diffusivity value we calculated for thin film zinc stannate (D_T – thermal diffusivity = $0{,}1006 \cdot 10^{-5}$ m^2s^{-1})

-to our best knowledge no other thermal diffusivity values for Zn_2SnO_4, synthesized in this way, are available in the literature

Acknowledgement

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