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Original

Availability: This version is available at: 11583/2665959 since: 2017-02-24T12:26:37Z

Publisher: Piezo Institute

Published DOI:

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Reduction of PbO loss in PZT-cobalt ferrite composites through quite-fast sintering and its quantification by means of XRD analysis

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ABSTRACT
In the near future (within ten years) magnetoelectric multiferroics could be implemented into the emerging technologies such as wireless power, internet of things, machine-to-machine communication services, mesh network, etc. A great research effort is in progress to improve the fabrication of PZT–CoFe2O4 (PZT–CF) composites due to the excellent piezoelectric properties showed by the PZT material class and the large magnetostriective coefficient of the CF. Unfortunately, during the sintering process of the particulate PZT-CF composites, side reactions do occur that are detrimental to the properties of the so-obtained material.

In this study:
1. we have avoided such reactions and PbO loss by setting a quite-fast sintering process.
2. The extent of PbO loss was determined by means of XRD analysis of the densified samples taking into account the amount of ZrO2 and the variations of the perovskite’s tetragonality.

EXPERIMENTAL

CONVENTIONAL SINTERING
- Heating rate < 300 °C/h
- Sintering temperature = 70% melting point
- Soaking time > 0.5 h
- Natural cooling

QUITE-FAST SINTERING
- Heating rate > 300 °C/h
- Sintering temperature < 70% melting point
- Soaking time < 0.5 h
- Cooling rate > 30 °C/min

RESULTS

CONCLUSIONS
For the first time fully dense particulate PZT-CF composites were produced by quite fast sintering. Full densification and suppression of chemical reactions are required to improve the magnetoelectric coupling.

You can find further results, about the microstructure evolution, chemical reaction, and functional properties, in the papers [1, 2].

Appendix: how to calculate the PbO loss (δ) through XRD analysis

Supposed reaction due to the PbO loss:

\[ \text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3 \rightarrow f \text{PbO} + (1 - f) \text{Pb}(\text{Zr}_{0.52}y\text{Ti}_{0.48-z})\text{O}_3 + 0.52(1 - y + fy) \text{ZrO}_2 + 0.48(1 - z + fz) \text{TiO}_2 \]

Once ZrO2 molar fraction (b) and the change of the amount of Zr in the produced perovskite (y) has been quantified by quantitative XRD analysis, PbO loss can be calculated through the following equation [1]:

\[ \delta = \frac{m_{\text{Pb}}(b/m_{\text{Zr}} - 1 + y)/y}{y} \]

where \( m_{\text{Pb}} \) and \( m_{\text{Zr}} \) are the stoichiometric coefficients of lead and zirconium in the starting perovskite phase, respectively. In our case, \( m_{\text{Pb}} = 1 \) and \( m_{\text{Zr}} = 0.52 \).

References