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Availability:
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Publisher:

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Heterostructured ceramic materials based on PZTN-CFO compounds

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Abstract

Multiferroic composites are currently one of the hot research topics [1]. A great research effort is in progress to improve the fabrication of PZT-CoFe$_2$O$_4$ (PZT-CFO) composites due to the excellent piezoelectric properties showed by the PZT material class and the large magnetostrictive coefficient of the CFO. Unfortunately unwanted reactions occur during densification of PZT-CFO materials at 1100-1200 °C. They are promoted by initial PbO loss that is calculated through XRD analysis, considering the amount of ZrO$_2$ and variation of perovskite’s tetragonality. The resulting titania reacts with CFO to form cobalt titanate [2].

The microstructure of the composites at 26-81 mol% CFO content was thoroughly investigated; the CFO grain size distribution can be mono- or bi-modal and overgrowth [3] occurs. By setting a quite-fast sintering full densification and prevention of unwanted reactions was achieved for the PZT:CFO 74:26 composites.

Experimental

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

**QUITE-FAST SINTERING [2]**
- Heating rate > 300 °C/h
- Sintering temperature < 70% $T_m$
- Soaking time < 0.5 h
- Cooling rate > 30 °C/min

Results

![Reaction Products](image1)

- Relative density = 82%
- PbO loss: 11%
- Bi-modal CoFe$_2$O$_4$ grain size distribution
- CoFe$_2$O$_4$ overgrowth by multiple parallel twinning [3]
- Coercivity: 239 Oe
- Reduced remnant magnetisation $M_r/M_s = 0.17$

![Euhedral CoFe$_2$O$_4$ Grains](image2)

- Relative density = 99%
- PbO loss < 0.2%
- Mono-modal CoFe$_2$O$_4$ grain size dist.
- Euhedral CoFe$_2$O$_4$ grains = 250 nm
- Coercivity: 789 Oe
- Reduced remnant magnetisation $M_r/M_s = 0.38$

Discussion

Reactions due to the PbO loss at the PZT/CFO interfaces [2]

$1^o$) $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})_3\text{O}_9 \rightarrow f\text{PbO} + (1 - f)\text{Pb}(\text{Zr}_{0.52}\cdot\text{Ti}_{0.48})_3\text{O}_9 + 0.52(1 - y + fy)\text{ZrO}_2 + 0.48(1 - z + fz)\text{TiO}_2$

$2^o$) Displacement reaction [2, 4]:

$\text{TiO}_2 + \text{CoFe}_2\text{O}_4 \rightarrow \text{CoTiO}_3 + \text{Fe}_2\text{O}_3$

It has been demonstrated that twin boundaries on CFO (111) planes act as the effective pinning centers for the hindrance of domain wall movement [2, 3].

![Globus model extension](image3)

In the Globus model the linear correlation between the initial susceptibility ($\chi_i$) and the mean grain diameter ($D_m$) is expressed as:

$$\chi_i = 2\pi M_s^2 D_m / K$$

where $M_s$ is the saturation magnetization, and $K$ is the global anisotropy. But, where the domain walls are pinned at twinning boundaries, $D_m$ should be interpreted as the distance between the twinning boundaries, i.e. the span of the domain wall [3].

Acknowledgements

RITMARE flagship project is gratefully acknowledged

References


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