DFG FOR 1509 Ferroische Funktionsmaterialien
Mehrskalige Modellierung und experimentelle Charakterisierung

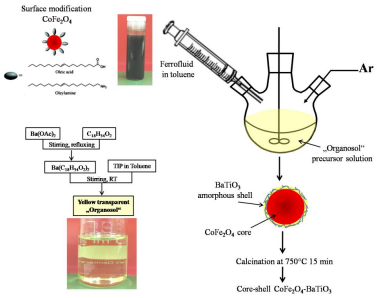
TP2 "Synthesis of magneto-electric composites"

Introduction

- Multiferroic materials have drawn much attention because they display the coexistence of ferroelectric and magnetic properties. Spinel/perovskite multiferroic composites $CoFe_2O_4/BaTiO_3$ were prepared by both organosol and hydrothermal route.
- Combining the organosol and co-precipitation methods we have successfully synthesized $CoFe_2O_4/BaTiO_3$ (CFO-BTO) core-shell nanoparticles, where cobalt iron oxide nanoparticles are cores and barium titanate forms a shell.
- By hydrothermal technique in the first step CFO was prepared by co-precipitation of salts of cobalt and iron with different concentrations of the capping agent polyvinylpyrolidone (PVP). Polyvinylpyrolidone (PVP) was used to stabilize the particles and prevent them from agglomeration. The BTO shell was synthesized by the hydrothermal method. The electron microscopy of CFO-BTO showed two-phase composite nanostructures of cobalt ferrite cores coated with a $BaTiO_3$ shell.
- The converse magnetoelectric coefficient measured for sintered ceramics reaches the value 4.4 x 10^{-12} s/m.

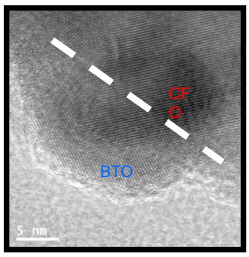
Organosol route

"Organosol"- precipitation: Intimate mixing of components in solution, precipitation, filtration, washing, drying, and calcination.

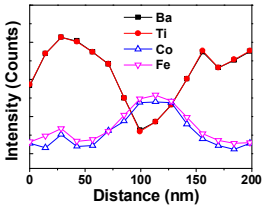


Advantages

- Control of stoichiometry by initial reagent ratio.
- Complex compositions, mixture of metal ions.
- Good homogenity, mixing at the molecular level.
- Low firing temperatures.

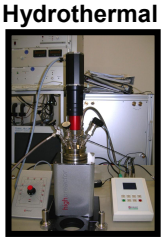


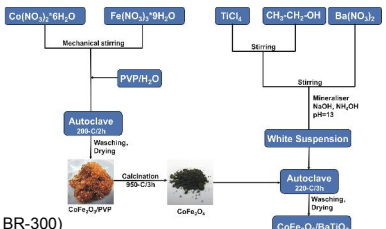
HRTEM of a single CFO-BTO particle indicating its core-shell structure.



Distribution of Ba, Ti, Co, Fe elements across a single CFO-BTO particle measured using **EDS**.

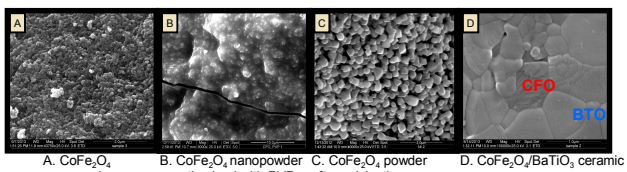
Hydrothermal route





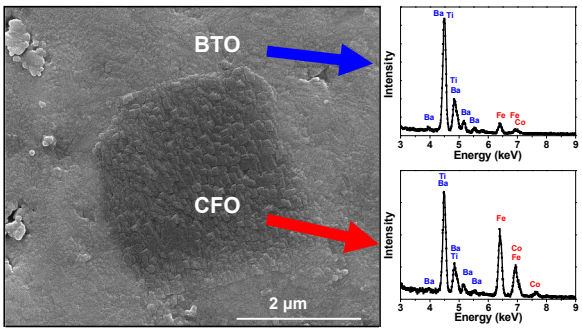
Laboratory autoclave (Berghoff BR-300)
- Max. operating pressure: 200 bar
- Max. temperature: 230°C PTFE seal

SEM Analysis $CoFe_2O_4$- $BaTiO_3$ (50-50)



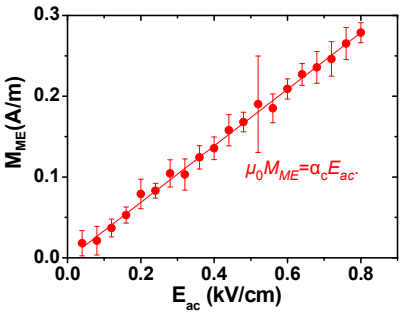
A. $CoFe_2O_4$ nanopowder synthesized without PVP | B. $CoFe_2O_4$ nanopowder synthesized with PVP | C. $CoFe_2O_4$ powder after calcination | D. $CoFe_2O_4/BaTiO_3$ ceramic

Ceramic $CoFe_2O_4$- $BaTiO_3$ (20-80)



SEM micrograph of the polished surface of a $CoFe_2O_4$ - $BaTiO_3$ ceramic sintered at 1200°C show a $CoFe_2O_4$ grain (size ~ 2 microns) in a $BaTiO_3$ matrix. The dark and bright areas correspond to $CoFe_2O_4$ and $BaTiO_3$ phases.

ME Measurements SQUID



Electric field dependence of the electrically induced magnetization, M_{ME}, for the $CoFe_2O_4$-$BaTiO_3$ (20-80) ceramic composite measured at $\mu_0 H_{dc}$ = 0.15 T and T = 285 K follows well a linear law. The best fit yields the value of the ME coefficient α_c = 4.4 × 10^{-12} s/m.

M. Etier et al. Ferroelectrics 2013

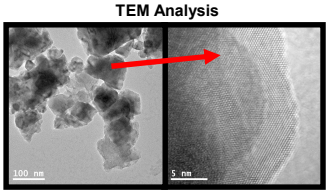
Outlook

Terfenol – D / $BaTiO_3$ composite

Experimental

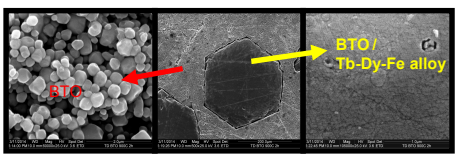
- Fe acetate, Dy acetate, and Tb acetate
- (Fe, Dy, Tb) oleate precursor
- $BaTiO_3$ „organosol"
- Mixture and precipitation (Methanol)
- Calcination, Sintering

TEM Analysis



HRTEM analysis confirms crystalline phases of Terfenol-D - $BaTiO_3$.

SEM Analysis



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