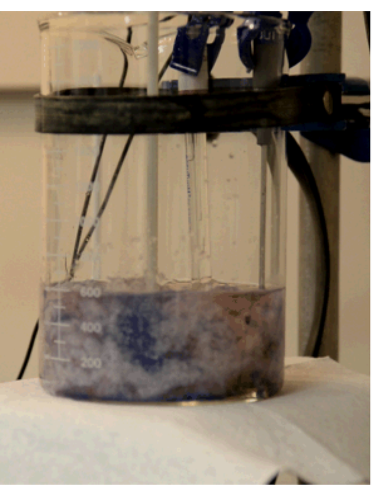
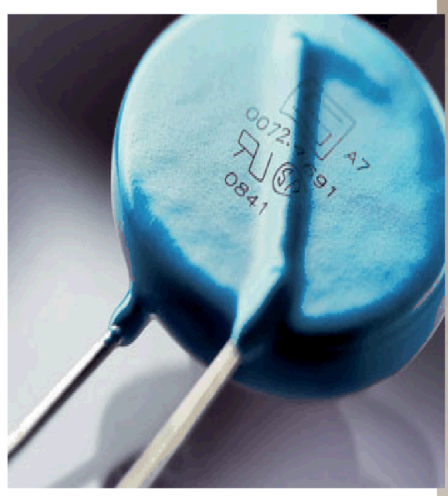
SAND2017-7611C

The Effect of Calcination and Milling on ZnO Varistor Powder Characteristics





Joshua Nordlander (2568)

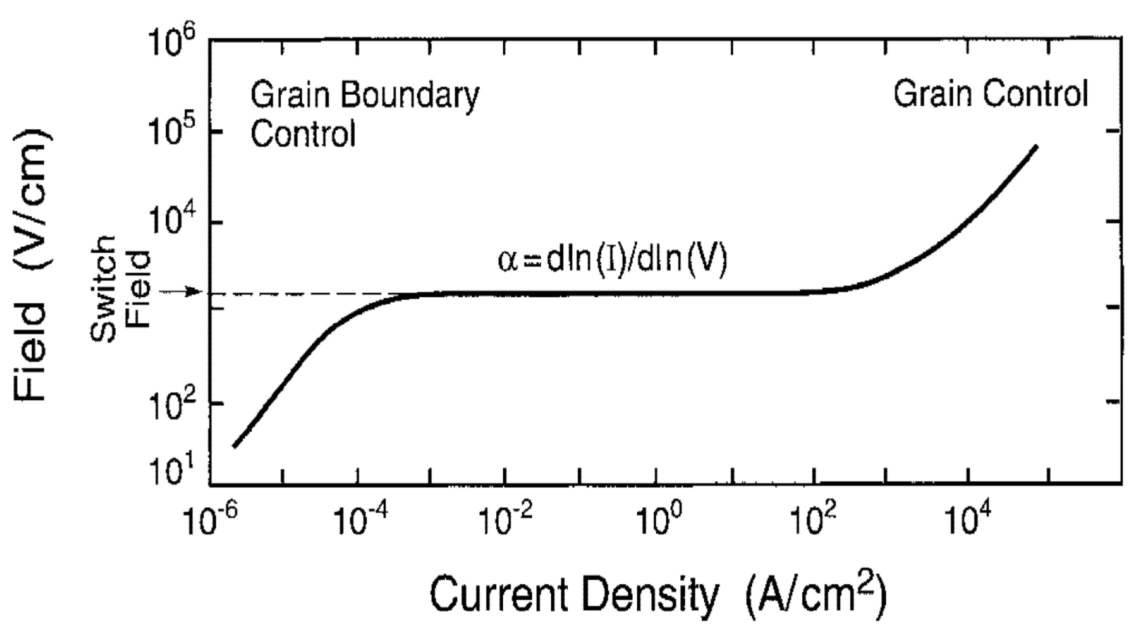
Colorado School of Mines; B.S. Metallurgical & Materials Engineering
Expected Graduation: Dec. 2017

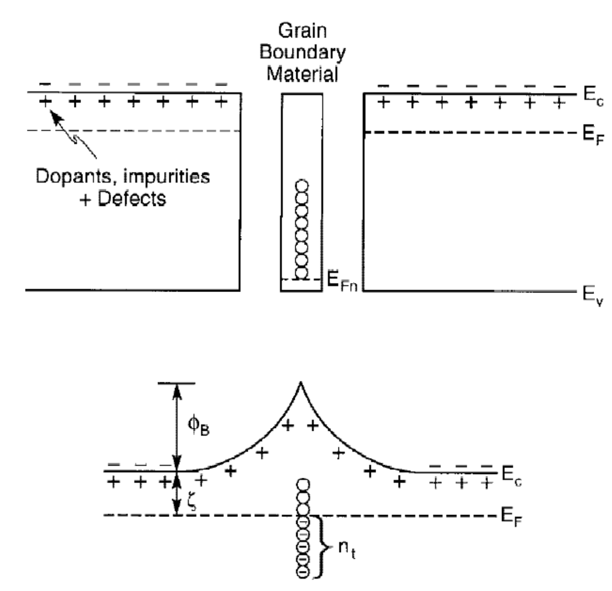
Mentors: Pin Yang (1816),Erik Spoerke (1816)

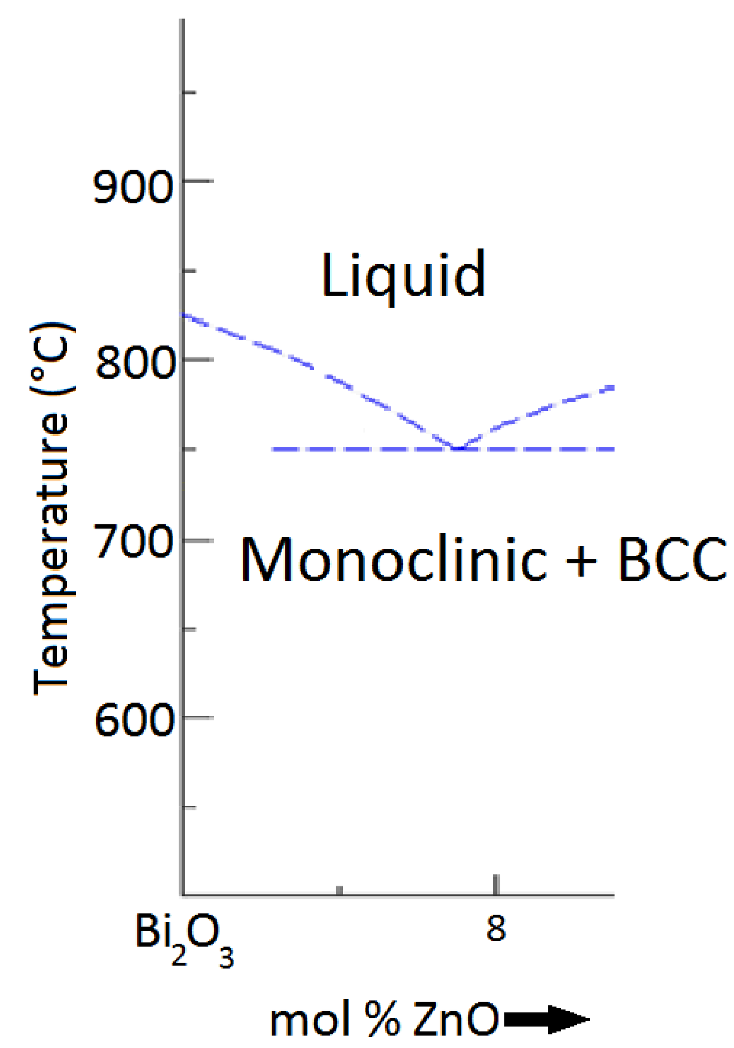
Manager: Michael Winter

Collaborators: Daniel Stefan, Dara Gough

Background

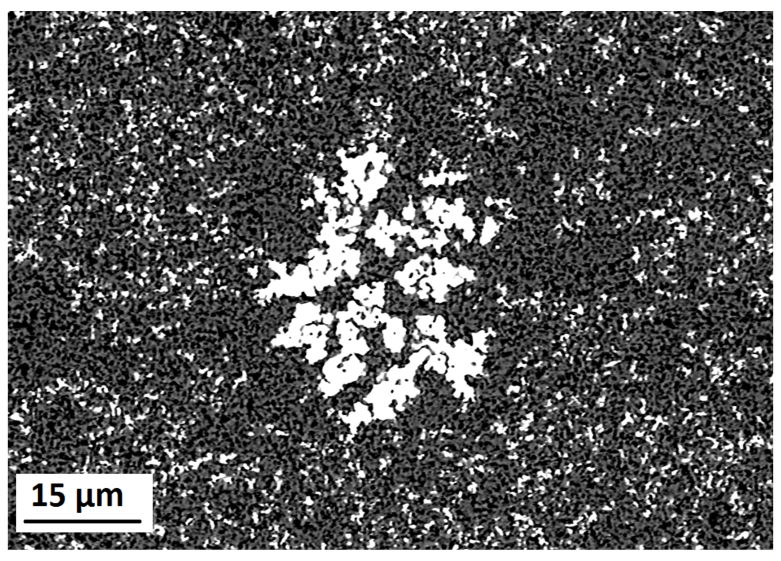






- Non-linear Current-Voltage properties
- Electrical properties modeled as double Schottky barrier (~3.5 V/grain boundary)
- Microstructure consists of n-doped ZnO grains surrounded by thin grain boundary phase.
- Grain boundary phase is sintering aid and establishes electrostatic barrier.

Clarke, D. R. *Journal of the American Ceramic Society*, (1999) **82** (3), 485-500.

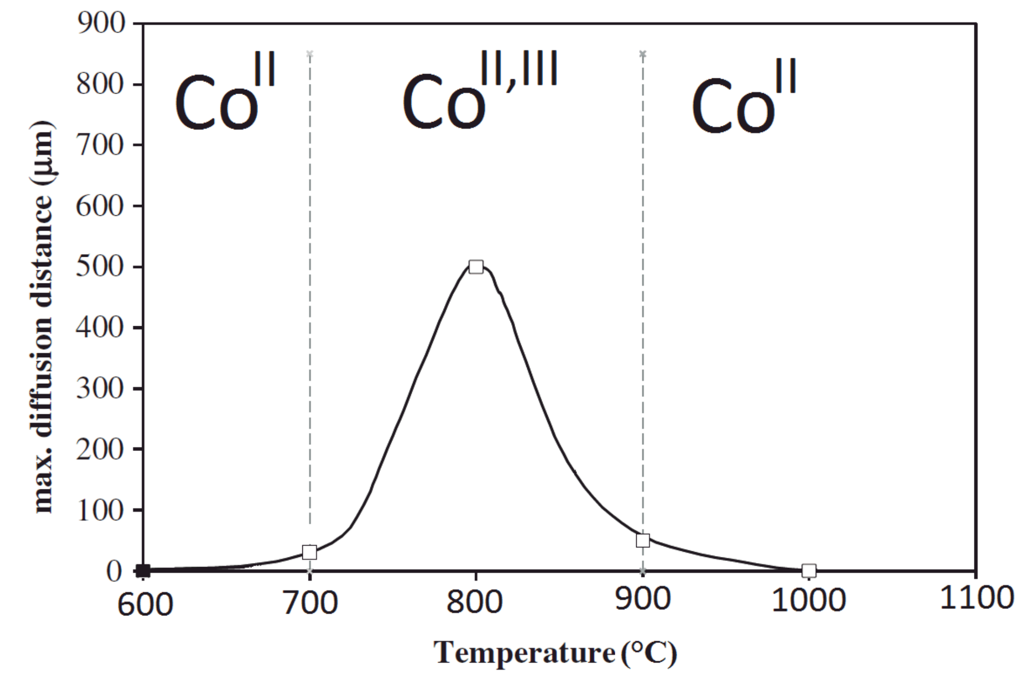


Motivation from Literature

- M. Peiteado et al. have shown via a diffusion couple experiment that the first calcination temperature (600°C) is too low to observe Mn^{3+} diffusion in ZnO. The oxidation state of Mn is temperature dependent.
- M. Peiteado et al. have shown in similar experiments that no significant diffusion of Co in ZnO occurs at the first calcination temperature. The oxidation state of Co is also temperature dependent.

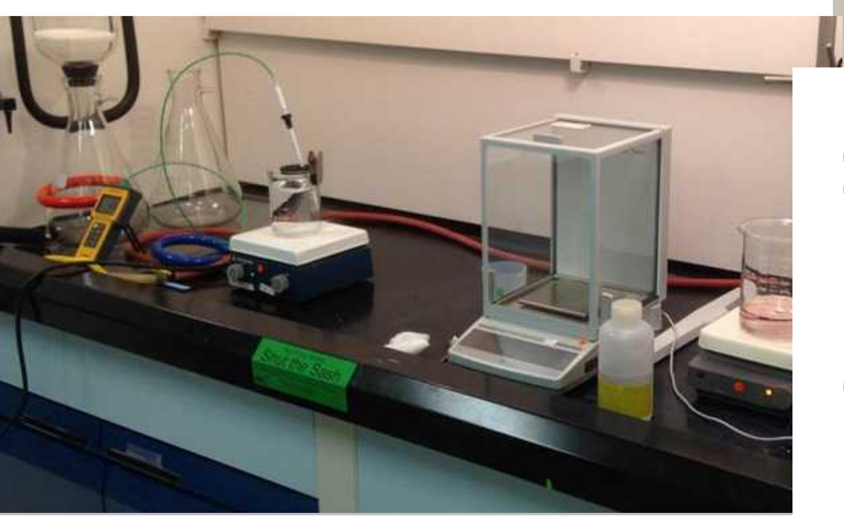
M. Peiteado et al., (2007), *Diffusion and reactivity of ZnO-MnO_x system.* J. Solid State Chemistry (180), 2459-2464

M. Peiteado et al., (2008), *Influence of crystal structure on the Co^{II} diffusion behavior in the $Zn_{1-x}Co_xO$ system.* J. Solid State Chemistry (181), 2456-2461



U.S. DEPARTMENT OF ENERGY
NNSA National Nuclear Security Administration

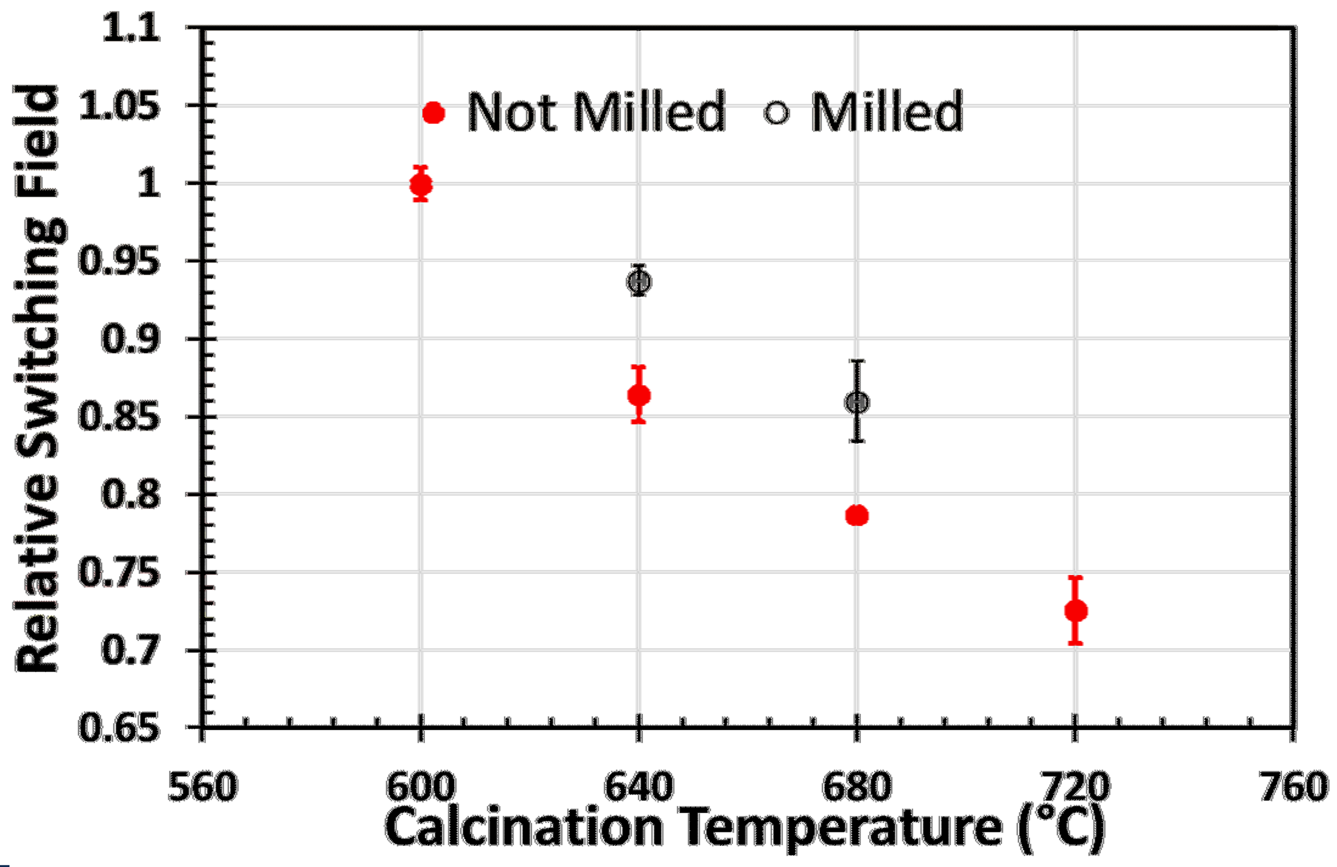
Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc. for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.
SAND No. 2011-XXXXP

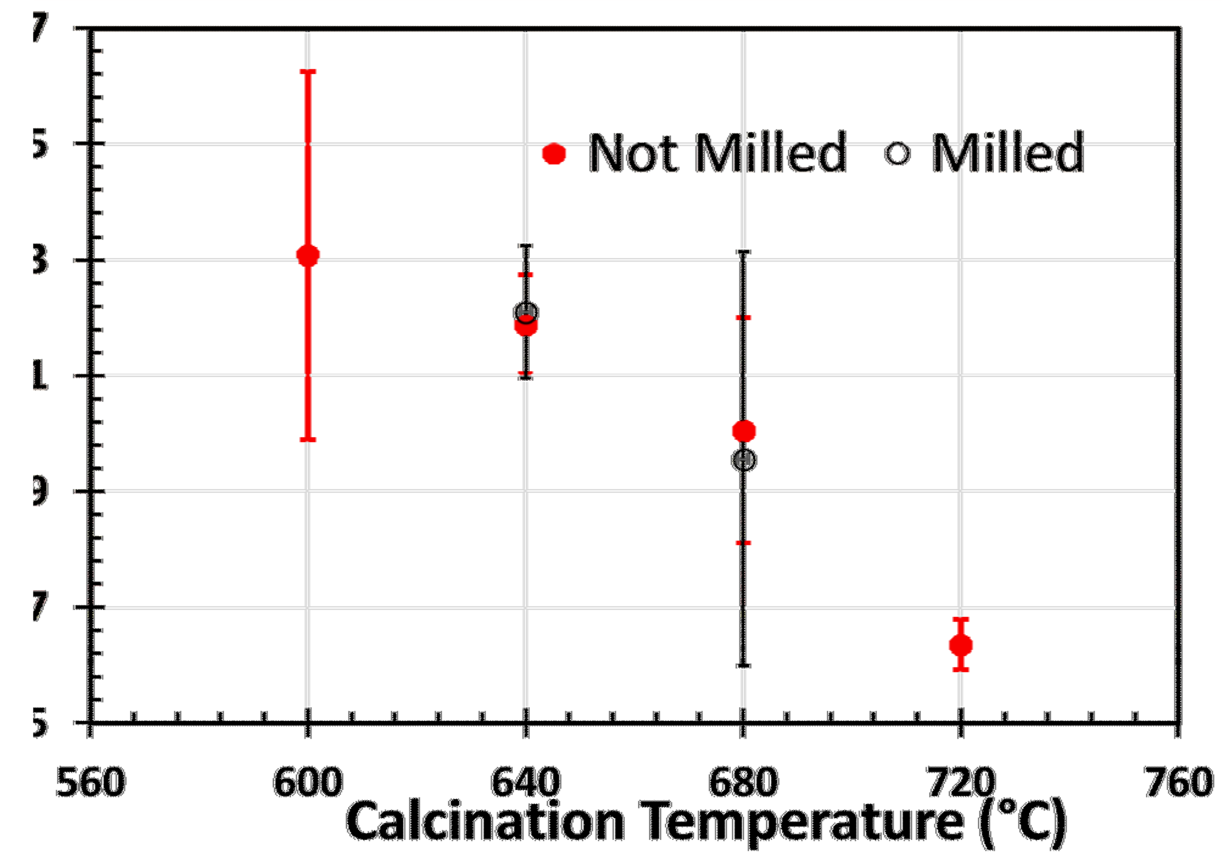


Experimental Procedure

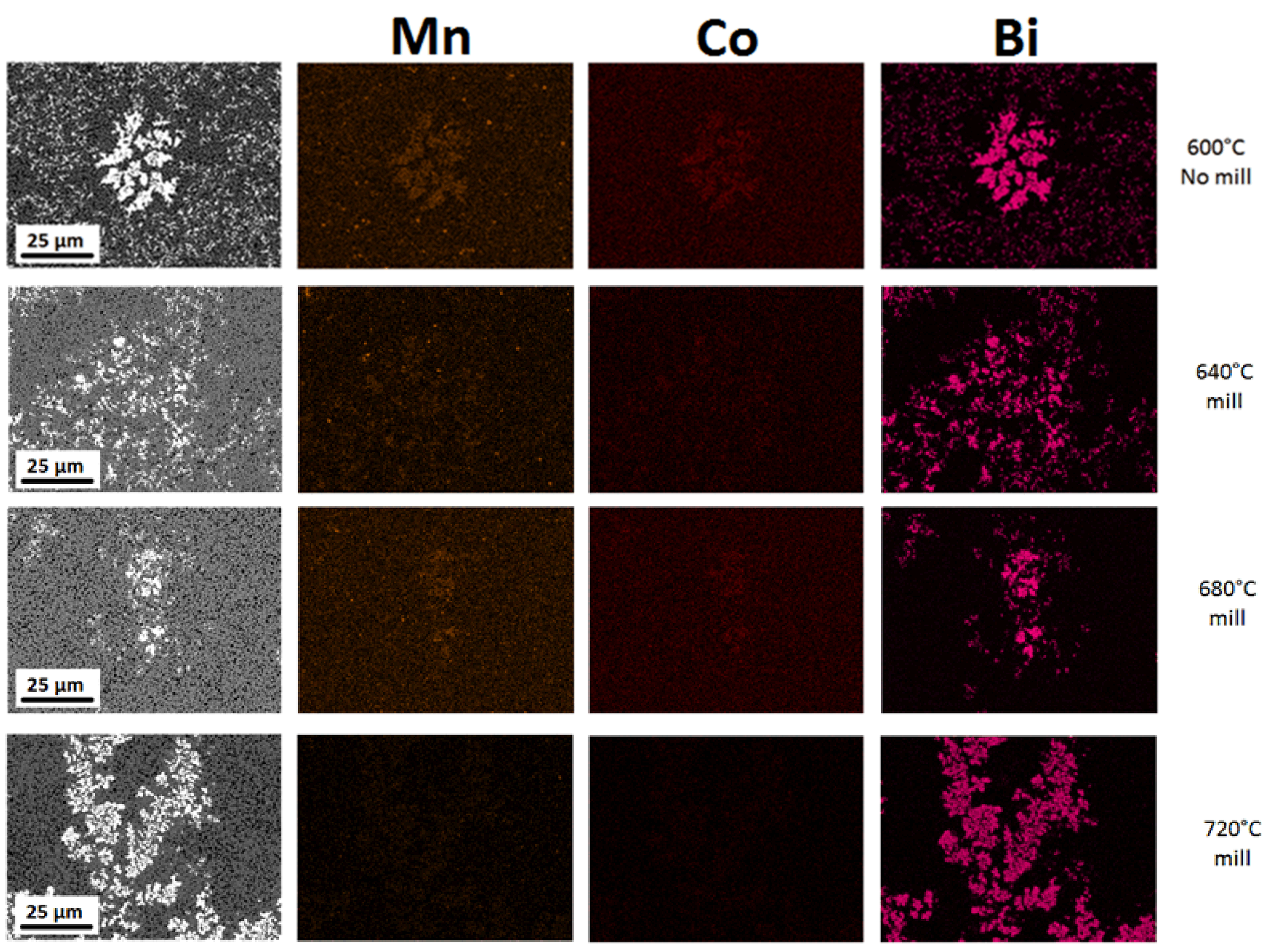
- Vary the first calcination temperature of co-precipitation process to see the effect on α values
- Mill half of the powder calcined at a given temperature to determine the effect of particle size on switching electric field
- Compare mixtures of highly doped ZnO/Mn_3O_4 with XRD after firing at 600°C or 720°C to understand how the oxidation state changes.

Electrical Characterization and EDXS





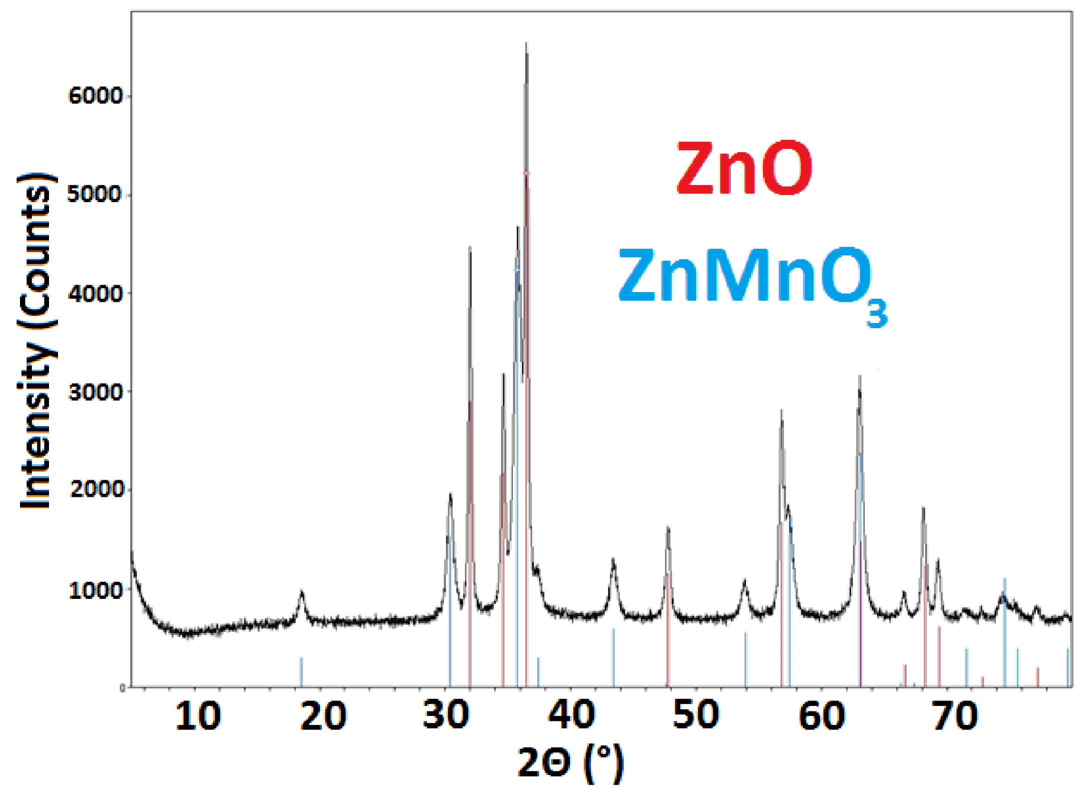
Higher calcination temperatures increase particle size (lower switching field), but lead to a lower α



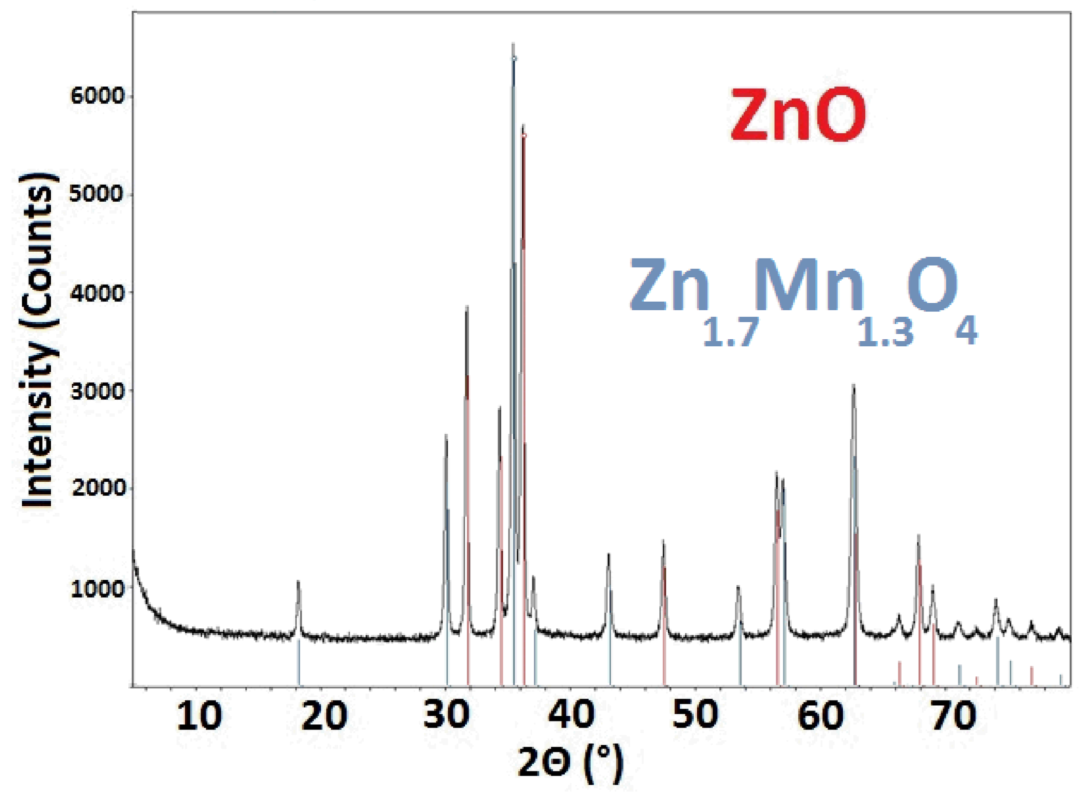
Higher calcination temperatures result in a more homogeneous distribution of Mn, Co rather than being predominantly localized with the Bi_2O_3 phase

X-Ray Diffraction of Highly Doped ZnO/Mn_3O_4

600°C Calcination



720°C Calcination



The Mn^{4+} reduces in this temperature range.

Summary

- An apparent homogeneous distribution of Mn,Co dopants appears to reduce alpha values
- Milling increases E_b, no statistical effect on α
- Mn changes oxidation state between 600-720°C, but it is difficult to characterize on XRD

Future Work

- Use ESR to understand oxidation state of Mn
- Develop other characterization (e.g. impedence analysis) to understand trap state effect

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc. for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.
SAND No. 2011-XXXXP