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Sintering of Barium Strontium Titanate for Use in Dielectric Components

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INTRODUCTION

Dielectric materials are used for a wide-range of applications, but current research is focused on utilizing dielectrics to increase capacitor's energy storage capabilities. This study aims to prove that high dielectric constants can be achieved with the absence of a dopant, through strategic selection of material concentrations and processing.

RESEARCH HYPOTHESIS

A dielectric constant of approximately 1,000 can be achieved without the addition of a dopant, by carefully selecting material concentrations and monitoring processing techniques, based on literature previously discussed.

EXPERIMENTAL CHANGES

STOICHIOMETRY

Our initial concentrations were changed to meet literature values; our initial proposed ratios were chemically impossible to produce.

FINAL CONCENTRATION:

- Concentration
 - $\text{BaCO}_3 + \text{SrCO}_3 + \text{TiO}_3$
 - BaSrTiO_3



ERROR AND DATA ANALYSIS

■ SOURCES OF ERROR

- Contamination
 - Could affect purity of samples
- Incomplete sintering
 - Inhomogeneous phase distribution
- Furnace Temperature Inconsistencies
- Grinding
- Density, porosity, and grain size discrepancies

■ ERROR MITIGATION

- Prepare samples in clean space
- Monitor all processing standards

■ DATA COLLECTION/ANALYSIS

- Measure Dielectric
- SEM
- EDS
- XRD

PRESSING

PRESSING PROCEDURE

Pellets did not meet expectations with our original pressing standards. After experimental trial and error successful mass and pressure values were determined.

A 6mm pressing apparatus was used to press powders.

UNSUCCESSFUL STANDARDS:

Pellet Mass: ~ 0.230 grams

Pressure: 1.5 Metric Tons

Pellet Mass: ~ 0.270 grams

Pressure: 1.5 Metric Tons

SUCCESSFUL STANDARDS:

Pellet Mass: ~ 0.300 grams

Pressure: 2 Metric Tons

Pellet	Diameter (mm)	Thickness (mm)	Area (mm ²)	Capacitance (pF)	K
1	5.43	2.12	23.15738631	48.8	1,292.38
	5.43	2.12	23.15738631	43.3	1,146.72
	5.43	2.12	23.15738631	46.5	1,231.47
2	5.36	1.7	22.56417508	51.52	1,382.23
	5.36	1.7	22.56417508	51.48	1,381.16
	5.36	1.7	22.56417508	51.49	1,381.43
3	5.4	1.64	22.90221045	67.3	1,792.22
	5.4	1.64	22.90221045	66.8	1,778.91
	5.4	1.64	22.90221045	67	1,784.23
4	5.56	2.04	24.27948467	72	1,862.21
	5.56	2.04	24.27948467	71.3	1,844.10
	5.56	2.04	24.27948467	71	1,836.34

CALCINING

PROCESSING

- CALCINATION

- 5 samples were calcined at 1200°C for 5 Hours

SEM imaging enabled us to conclude that our parameters were unsatisfactory. The pellets were composed of heterogeneous regions, with different concentrations of Ba and Sr. This lack of homogeneity was most likely due to poor grinding/mixing prior to calcining. Furthermore, some regions of the sample contained high carbon concentrations, which means the gas was not properly evolved. It's possible that the requirements to calcine our ceramic was different than the requirements found in literature, which dealt with oxides instead of carbonates.

SINTERING

PROCESSING

- Sintering
 - Sinter both at 1280°C for 2 hours
 - Increase 200°C/hour

- Sintering parameters were successful.
- The pellets kept their structure, but showed signs of internal cracks along the diameter.
- These defects could have been caused by the previous issues associated to calcining, or due to the fact that the pellets bonded to the ceramic plate during their furnace time.

MEASUREMENT OF DENSITY

DENSITY	SCALE MASS (g)	SUB MASS (g)	TEMP	CALC. DENSITY
1	0.2192	0.192	22.5	6.344
2	0.2249	0.196	22.5	6.126
3	0.2352	0.203	22.5	6.79
			AVG	6.42
			STD	0.338461224

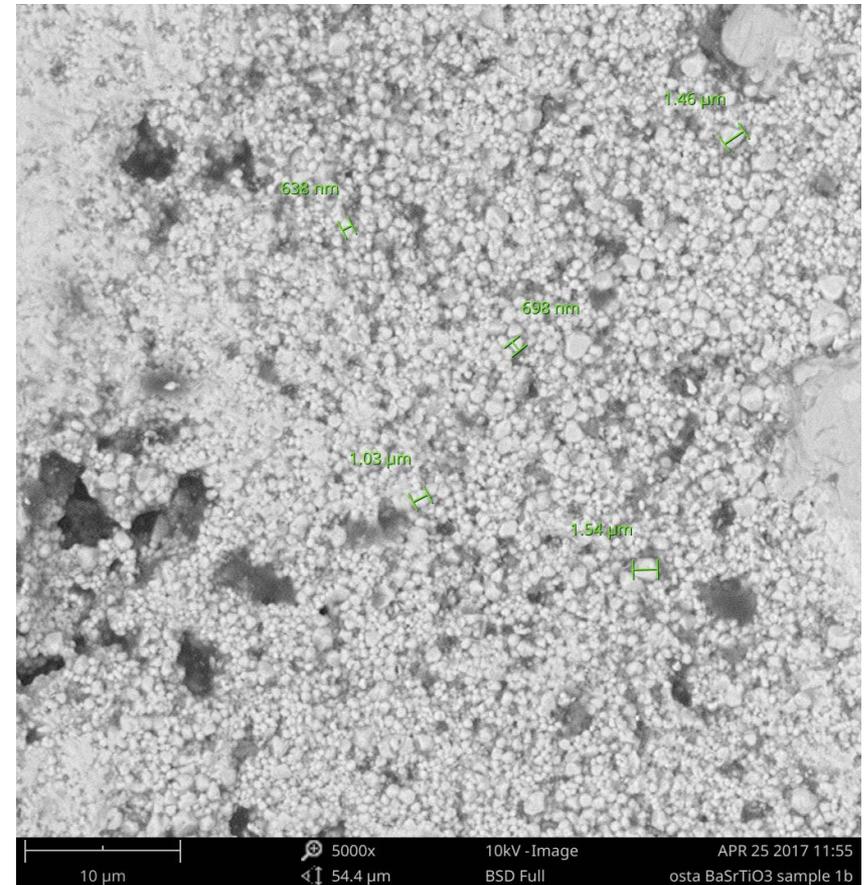
ARCHIMEDES PRINCIPLE DENSITY CALCULATION

$$\rho_{sample} = \frac{A}{A-L} \rho_{Liquid}$$

- 3 samples were randomly selected for density testing
- Samples were polished on 1200 grit for 100 rotations
- Archimedes' Method was used for measurements
- Average density was 6.42 g/cm³
- Standard deviation was 0.34

GRAIN SIZE ANALYSIS

- 5 different regions (randomly selected) on 2 different pellets were observed under SEM at 5,000x
- Each pellet was polished with 1200 grit for 100 rotations before analyzing
- From each region, 5 randomly selected grains were measured using software on SEM computer
- Average grain size was 830 nm
- Standard deviation was 16 nm



SEM image with grain size measurements

COMPOSITIONAL ANALYSIS

- Multiple regions of 2 samples were analyzed for compositional makeup using EDS

Bulk

Atomic percentage



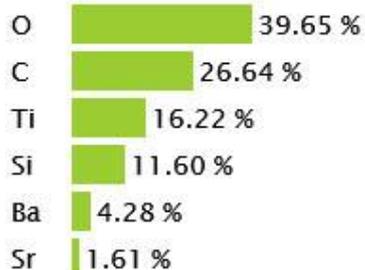
Dark Grains in Bulk

Atomic percentage



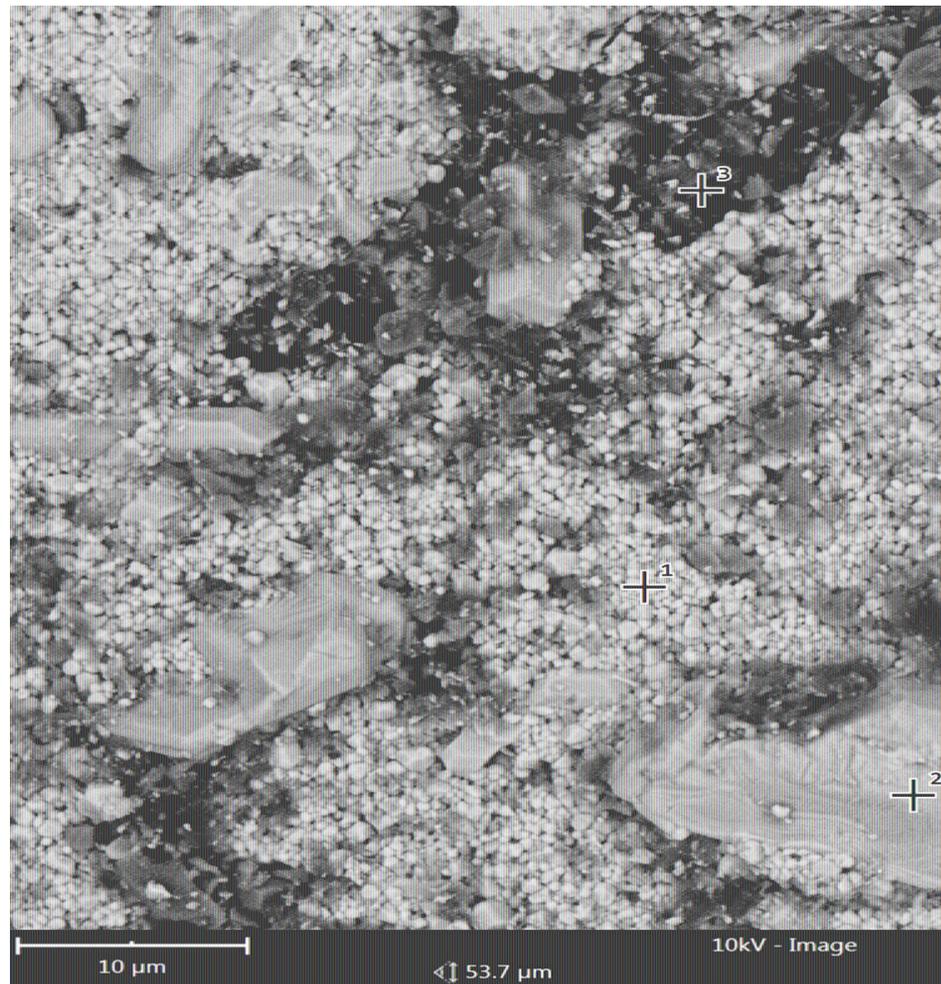
Large Particulates

Atomic percentage



Crack

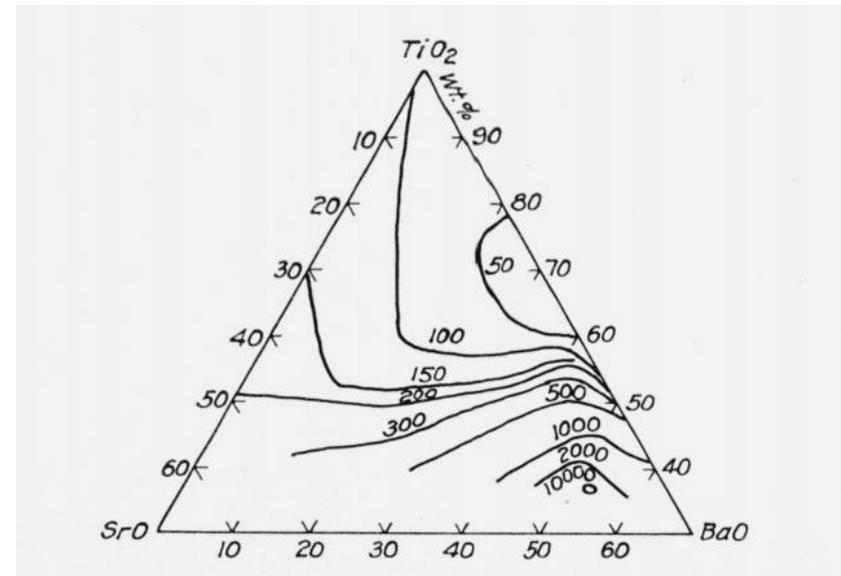
Atomic percentage



Example of one region analyzed for composition

MEASUREMENT OF DIELECTRIC CONSTANT

- 4 pellets were polished with 1200 grit for 100 rotations
- Each was coated in silver paint
- 3 measurements were taken per sample
- Expected Dielectric Constant from background research was about 2,000
- Average Dielectric Constant was 1,560
- Standard Deviation 275



Ternary phase diagram of TiO₂, SrO, BaO showing expected value of 2,000
<http://nvlpubs.nist.gov/>

INITIAL BUDGET

	Amount Needed	Per Unit	Price (USD)	Percent of Total Cost
SEM/EDS	2	hr	80.00	4.75%
XRD	2	hr	140.00	8.32%
Furnace (non-sequential use)	53.53	hr	1338.13	79.48%
Hydraulic Press	1	hr	25.00	1.48%
Processing Equipment	1	week	100.00	5.94%
BaCO ₃	3.39	g	0.29	0.02%
SrCO ₃	0.75	g	0.03	0.00%
TiO ₂	2.11	g	0.19	0.01%
	Total Budget		1683.63	100.00%

FINAL BUDGET

	Amount Needed	Per Unit	Price (USD)	Percent of Total Cost
SEM/EDS	1.5	hr	60.00	5.33%
XRD	2	hr	140.00	12.44%
Furnace (non-sequential use)	31	hr	775.00	68.86%
Hydraulic Press	2	hr	50.00	4.44%
Processing Equipment	1	week	100.00	8.88%
BaCO ₃	3.39	g	0.29	0.03%
SrCO ₃	0.75	g	0.03	0.00%
TiO ₂	2.11	g	0.19	0.02%
	Total Budget		1125.51	100.00%

With furnace use being 80% of initial costs, we were able cut heating costs by 42% by combining pellets in a single runs to be treated at a common temperature. An extra 30 minutes of SEM were required but did not significantly impact the reduction in spending.

CONCLUSION AND JUSTIFICATION

- Dielectric constant expected for BaSrTiO₃ with concentrations used was 5,000
- Measured dielectric constant was 1,560.
- Reasoning
 - SEM showed large particulates distributed throughout samples
 - Likely cause for breaking of pellets before testing due to acting as stress concentrator
 - Possible a result of not calcining long enough or not grinding fine enough
 - EDS revealed multiple phases present in sample including large amounts carbon that did not burn off from precursors
 - Multiple phases likely due to not calcining for long enough to burn of carbon from carbonates
 - Likely a factor in why dielectric constant was not as high as expected
 - Concavity of pellet surfaces due to fracture
 - Likely a factor in why dielectric constant was not as high as expected value