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Barium Titanate Nanocomposite Capacitor FY09 Year End Report

Todd C. Monson, Tyler E. Stevens, Ben D. Fellows, Chris B. Diantonio, Mike R. Winter,
Tom P. Chavez, Pin Yang, Alex. W. Roesler

Prepared by
Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550

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Todd C. Monson, Tyler E. Stevens, Ben D. Fellows
Nanomaterials Sciences

Chris B. Diantonio, Mike R. Winter, Tom P. Chavez, Pin Yang, Alex. W. Roesler
Ceramics and Glass

Sandia National Laboratories
P.O. Box 5800
Albuquerque, New Mexico 87185-MS1415

Abstract

This late start RTBF project started the development of barium titanate (BTO)/glass nanocomposite capacitors for future and emerging energy storage applications. The long term goal of this work is to decrease the size, weight, and cost of ceramic capacitors while increasing their reliability. Ceramic-based nanocomposites have the potential to yield materials with enhanced permittivity, breakdown strength (BDS), and reduced strain, which can increase the energy density of capacitors and increase their shot life. Composites of BTO in glass will limit grain growth during device fabrication (preserving nanoparticle grain size and enhanced properties), resulting in devices with improved density, permittivity, BDS, and shot life. BTO will eliminate the issues associated with Pb toxicity and volatility as well as the variation in energy storage vs. temperature of PZT based devices. During the last six months of FY09 this work focused on developing syntheses for BTO nanoparticles and firing profiles for sintering BTO/glass composite capacitors.

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NOMENCLATURE

Ba	Barium	
Ba(OH) ₂ ·8H ₂ O	Barium Hydroxide Hydrate	
BaTiO ₃	Barium Titanate	
BTO	Barium Titanate	
BDS	Breakdown Strength	
DOE	Department of Energy	
EDA	Ethylenediamine	
ELNG	Electric Neutron Generators	
HEV	Hybrid Electric Vehicle	
MEA	Ethanolamine	
MLCC	Multilayer Ceramic Capacitor	
PLZT	Lanthanum-Doped Lead Zirconium Titanate	
PVA	Polyvinyl Alcohol	
PZT	Lead Zirconium Titanate	
RTBF	Readiness in Technical Base and Facilities	
SEM	Scanning Electron Microscope	
SNL	Sandia National Laboratories	
TEM	Transmission Electron Microscope	
Ti(OBu) ₄	Titanium Tert-Butoxide	
Ti(OPr) ₄	Titanium Isopropoxide	
XRD	X-ray Diffraction	

1. INTRODUCTION

To meet the needs of future and emerging applications, there is a requirement to decrease the size, weight, and cost of ceramic capacitors while increasing their reliability. Ceramic-based nanocomposites have demonstrated enhanced permittivity, breakdown strength (BDS), and reduced strain, which can increase the energy density of capacitors (reducing their size and weight) and increase their shot life. However, to date, ceramic nanoparticles have only been incorporated at loadings of 30% or less in polymer-based composite capacitors where they have demonstrated high energy densities but not extended performance in a robust device.

This Readiness in Technical Base and Facilities (RTBF) project has a long term goal to develop barium titanate (BTO)-glass nanocomposite capacitors with a high ceramic loading and energy densities $> 10 \text{ J/cc}$. Composites of BTO in glass will limit grain growth during device fabrication (preserving nanoparticle grain size and enhanced properties), resulting in devices with improved density, permittivity, BDS, and shot life. BTO will eliminate the issues associated with Pb toxicity and volatility as well as the variation in energy storage vs. temperature of Lead Zirconium Titanate (PZT) based devices. As the project progresses, the effort will shift from precursor synthesis to materials processing, and finally to fabrication and characterization of devices. The size of current pulsed power capacitors will be reduced by a factor of two due to increased materials properties and performance, while increasing their pulse life, environmental performance, temperature stability, and decreasing their cost.

2. BTO NANOPARTICLE SYNTHESIS AND CHARACTERIZATION

In order to fabricate BTO/glass nanocomposite capacitors a reliable source of BTO nanoparticles is required. Since the long term goal of this project is to develop a process for fabricating large quantities of capacitors which can eventually be transferred to industry and DOE applications, a relatively simple and scalable synthesis will need to be developed. Expensive reagents should be avoided and the synthesis should be performed close to room temperature and ambient pressure, yet still yield crystalline BTO particles. Additionally, nothing about the synthesis should preclude it from being scaled to large volumes. During the early stages of research and development, a large degree of control over the particle size and crystalline phase will be desired. This will enable the research team to explore a variety of nanoparticle precursor parameters and determine which particles produce the best performing capacitors. Finally, an ideal synthesis would produce particles that could be coated with a glass shell while still in solution, since this is one avenue for producing BTO/glass composites that will be explored.

During the last half of FY09 three different syntheses for BTO nanoparticles were explored. The first synthesis was a modified version of a synthesis of Lanthanum-Doped Lead Zirconium Titanate (PLZT) developed at Sandia.[1] In the original reaction, metal salts are dissolved in a

solution of nitric acid and water. After stirring overnight, titanium isopropoxide ($\text{Ti}(\text{OPr})_4$) and a solution of polyvinyl alcohol (PVA) in water are added. Finally, the acidic aqueous solution is boiled off, decreasing the solubility of the dissolved metals and leading to particle formation. The PVA forms a semi-dilute solution which limits the final particle size. Since we were attempting to synthesize BTO, barium nitrate was the only metal salt required. The synthesis was attempted both with equal moles of barium and titanium and a 20% excess of barium to aid final BTO formation. At room temperature, the product of all reactions was amorphous. After a post-anneal of 700 °C a mixed phase product was formed. Post-annealing at 1000 °C was required to fully convert the particles synthesized via this method to crystalline BTO. XRD data of particles formed from this reaction, including those treated by post-annealing is shown in Figure 1.

Figure 1. XRD of particles synthesized using barium nitrate reagents. Phase pure and crystalline barium titanate was only achieved after post-annealing at 1000 °C

The second synthesis attempted was based on a prep. found in literature which used barium acetate and titanium tert-butoxide as precursors.[2,3] A mixture of ethylenediamine (EDA) and ethanolamine (MEA) were used as the solvent. These solvents have a basic pH and can catalyze room temperature formation of crystalline BTO. The amine functionality of these solvents also allows them to bind to the BTO surface and act as ligands. Size of the particles can be controlled by varying the EDA:MEA ratio. In addition to repeating the EDA:MEA ratio of 1:1 as in the literature, the reaction time of the 1:1 ratio sample was increased to 96 hrs. (the longest reaction time reported in the literature was 48 hrs.), and larger particles were produced as desired. Also, ratios with much higher concentrations of MEA than reported in literature were synthesized. We were hopeful that greater amounts of MEA would produce larger particles and potentially

particles that were tetragonal in phase (rather than cubic). These included EDA:MEA ratios of 1:5, 1:10, 1:15, 1:20, 1:50, and 1:100. Increasing the amount of MEA yielded larger cubic particle sizes up to a ratio of 1:15. X-ray diffraction (XRD) data of BTO particles with EDA:MEA ratios of particles from 1:1 through 1:15 are shown in Figure 2. The intensity of the XRD peaks can allow for a relative comparison of peaks. Peak intensity increases as the amount of MEA goes up. These same nanoparticles were also characterized using Transmission Electron Microscope (TEM), and TEM images for the particles synthesized over the course of 96 hrs. are displayed in Figure 3. As determined from the TEM images, the approximate diameters of BTO nanoparticles synthesized with EDA:MEA ratios of 1:1, 1:5, 1:10, and 1:15 are, respectively: 30 nm, 30 nm, 45 nm, and 65 nm. Beyond a 1:15 ratio only amorphous particles were formed and could not be converted to crystalline BTO even with a post-annealing temperature of 1000 °C. XRD data of particles synthesized with EDA:MEA ratios in excess of 1:15 are shown in Fig. 4.

Figure 2. XRD of particles synthesized using barium acetate reagents with varying EDA:MEA ratios. Increased reaction time and MEA yields larger particles, as indicated by the intensity of the XRD peaks.

Figure 3. TEM images of BTO nanoparticles synthesized with EDA:MEA ratios of: a) 1:1, b) 1:5, c) 1:10, d) 1:15

Figure 4. XRD of particles synthesized using barium acetate reagents with EDA:MEA ratios having greater concentration of MEA than 1:15. All reactions, even when post-annealed at 1000 °C failed to produce crystalline BTO.

Other reaction parameters were explored with the barium acetate based synthesis and the 1:15 EDA:MEA ratio, all while holding the reaction time at 96 hrs. Yet another modification was to add additional water to the reaction. When additional water was added to a synthesis with a EDA:MEA ratio of 1:15 amorphous particles were still produced. However, when 50 mL of extra water were added to a synthesis using only EDA, larger cubic BTO particles were produced. A plot comparing the XRD data of both the 1:15 synthesis and the synthesis with no MEA and extra water is displayed in Fig. 5. This sample was also analyzed in a TEM. A TEM image of particles synthesized with 50 mL of extra water and no MEA is shown in Figure 6. In an attempt to grow larger and potentially tetragonal particles, the 1:15 synthesis was performed at an elevated temperature of 100 °C but resulted in amorphous particles. The reaction was also run for an extended period in order to produce larger particles. However, when the reaction was run for 211 hrs. amorphous particles were again the result. The XRD data from all the modifications to the 1:15 reaction which resulted in amorphous particles are shown in Figure 6.

Figure 5. Comparison of XRD from particles synthesized using barium acetate reagents with a EDA:MEA ratio of 1:15 and using EDA only and 50 mL of extra water.

A negative of this reaction is the high cost of titanium tert-butoxide as a reagent. Titanium tert-butoxide is used to produce titanium hydroxide, which then reacts with the barium acetate to form BTO. It was shown that less expensive titanium isopropoxide could be used to prepare titanium hydroxide. A comparison of the XRD data from titanium hydroxide formed from both titanium isopropoxide and titanium tert-butoxide is shown in Figure 8. The data clearly shows that the titanium hydroxide produced in both cases were identical.

Figure 6 TEM image of BTO nanoparticles synthesized with EDA only (no MEA) and an additional 50 mL of water.

Figure 7. XRD data from barium acetate syntheses using a EDA:MEA ratio of 1:15 and modified in reaction temperature, water content, or reaction time. Each of these syntheses resulted in amorphous material.

The third synthesis attempted was one which used barium hydroxide hydrate ($\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$) as a reagent along with titanium isopropoxide. The entire reaction was performed in a solvent of

isopropanol at a temperature of 80 °C. Although this is higher than room temperature it is still a low enough reaction temperature such that mass production of cheap particles would not be precluded. Following the procedure described in the literature, the particles were synthesized without the addition of any water.[4,5] However, this resulted in predominantly amorphous particles. Yoon and coauthors reported that the water molecules available from the $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ provided enough water for the reaction between the barium ions and titanium isopropoxide to move forward. We believe that due our location's dry climate and our thorough glassware drying procedure that less water was available for the reaction. The reaction was repeated using 6 mol of water and this time the reaction produced cubic BTO nanoparticles. The XRD data of the particles synthesized both with and without the addition of excess water is displayed in Figure 9 and a TEM image of the particles synthesized with 6 mol of water is shown in Figure 10. These particles are roughly 10 nm in diameter.

Figure 8. XRD of titanium hydroxide synthesized with both titanium isopropoxide and titanium tert-butoxide ($\text{Ti}(\text{OBu})_4$).

Figure 9. XRD data of BTO particles synthesized using Ba hydroxide as a reagent.

Figure 10. TEM image of BTO nanoparticles synthesized with Ba hydroxide reagents and 6 mol of water.

Further exploration of these syntheses is ongoing, particularly for the synthesis which utilizes barium hydroxide reagents. Continued work will develop more control over the size of the particles produced as well as the phase (i.e. the ability to produce tetragonal nanoparticles).

3. MATERIALS CHARACTERIZATION AND OPTIMIZATION OF NANO-COMPOSITE

In order to fully evaluate the potential of a truly engineered nano ceramic particle-glass composite system for use as a high energy density capacitor a nano barium titanate – barium borosilicate glass composite has been formulated for characterization. The initial composite has been formulated from a nano engineered barium titanate (BaTiO_3) powder purchased from TPL, Inc., NanOxide HPB-1000, and a glass composition obtained from Missouri-Rolla having the composition as listed in Table I. The as-received nano-particle barium titanate powder has a measured BET surface area of $16.26 \pm 0.0669 \text{ m}^2/\text{g}$ and Scanning Electron Microscope (SEM) images of the primary particle size and morphology are shown below (Figures 11 and 12). The nano-particle barium titanate powder was attrited to produce a powder having a BET surface of $18.65 \pm 0.0459 \text{ m}^2/\text{g}$.

Figure 11. SEM image of a primary particle of BTO from TPL, Inc. (NanOxide HPB-1000)

Figure 12. SEM image showing the morphology of BTO particles from TPL, Inc.

Table 1. Composition of the Missouri-Rolla barium borosilicate glass.

Components	Mole %	Weight %
BaO	50	68.73
SiO ₂	20.97	11.29
Al ₂ O ₃	102	0.93
H ₃ BO ₃	24	14.97
ZrO ₂	2.01	2.22
SrO	2	1.86

Simultaneous thermal analysis (STA) was performed to characterize the conversion of the cubic form of the nano barium titanate from TPL, Inc. to the tetragonal crystal structure. This phase transition is apparent from the following calorimetric results where the cubic to tetragonal phase transition is highlighted following a high temperature, approximately 1300°C, thermal treatment. The STA data is displayed in Figure 13.

Figure 13. STA of BTO nanoparticles from TPL, Inc.

For the first order study on the nano-composite systems, the following compositions were prepared and characterized;

1. Nano-barium titanate – 0 volume % barium borosilicate glass
2. Nano-barium titanate – 1 volume % barium borosilicate glass
3. Nano-barium titanate – 20 volume % barium borosilicate glass

Sintering and electrical characterization test samples were prepared for all three of these systems using dry pressed pellets. Sintering studies are being performed in a dilatometer to fully characterize the densification of the various composite systems. The figure below summarizes the dilatometric sintering results showing the temperature-displacement trajectory for each of the nano-composite formulations. It is apparent from these results that as the amount of glass in the formulation is increased we see a decrease in the onset of sintering temperature and the maximum shrinkage of the body occurs at a lower temperature. The dilatometric sintering results are shown in Figure 14.

Figure 14. Dilatometric sintering results of 0, 1, and 20 vol% barium borosilicate glass and BTO nanoparticle composite.

4. ELECTRICAL CHARACTERIZATION

Electrical characterization has been evaluated using both weak-field impedance analysis (weak-field permittivity and dielectric loss as a function of temperature and frequency) and high-field hysteresis behavior characterization (high AC electric field as a function of temperature). Figures 15, 16, and 17 show the results from the weak-field analysis for the 0, 1, and 20 vol% barium borosilicate glass and BTO composites, respectively. Figure 18 shows the high field hysteresis behavior of TPL nano-barium titanate and 0 volume % barium borosilicate glass and Figure 19 shows the high field hysteresis behavior of TPL nano-barium titanate and 1 volume % barium borosilicate glass. For comparison purposes the high field hysteresis behavior of a chem.-prep PLZT with 0 volume % Viox glass addition and chem.-prep PLZT with 1 volume % Viox glass addition is shown in Figures 20 and 21, respectively. The energy density of these nonlinear dielectrics was calculated from the integral of the applied electric field and measured polarization relationship. The results are tabulated at maximum and normalized applied electric fields in Table 2. A direct comparison of these values should be considered with caution as the results are not normalized to density and optimized processing parameters.

Figure 15. Weak-field impedance analysis of 0 vol% barium borosilicate glass and BTO composite

Figure 16. Weak-field impedance analysis of 1 vol% barium borosilicate glass and BTO composite

Figure 17. Weak-field impedance analysis of 20 vol% barium borosilicate glass and BTO composite

Figure 18. High field hysteresis behavior of TPL nano-barium titanate and 0 volume % barium borosilicate glass.

Figure 19. High field hysteresis behavior of TPL nano-barium titanate and 1 volume % barium borosilicate glass.

Figure 20. High field hysteresis behavior of chem.-prep PLZT and 0 volume % Viox glass.

Figure 21. High field hysteresis behavior of chem.-prep PLZT and 1 volume % Viox glass.

Table 2. Comparison of the energy density for nonlinear dielectrics as calculated from the integral of the applied electric field and measured polarization. (*Note: results are not normalized for density and optimized processing parameters.).

Material	Energy Density (J/cm ³) @ Applied Electric Field (kV/cm)
TPL Nano-Barium Titante + 0 volume % Barium Borosilicate Glass	0.038 J/cm ³ @ 14 kV/cm
TPL Nano-Barium Titante + 1 volume % Barium Borosilicate Glass	0.033 J/cm ³ @ 14 kV/cm
Chem-Prep PLZT + 0 volume % Viox Glass	0.360 J/cm ³ @ 46 kV/cm
Chem-Prep PLZT + 1 volume % Viox Glass	0.331 J/cm ³ @ 46 kV/cm
Chem-Prep PLZT + 0 volume % Viox Glass	0.040 J/cm ³ @ 14 kV/cm
Chem-Prep PLZT + 1 volume % Viox Glass	0.038 J/cm ³ @ 14 kV/cm

5. DISCUSSION AND CONCLUSIONS

In half a year, the room temperature synthesis of crystalline BTO nanoparticles in large batches was demonstrated. The particles' size can be tuned between diameters of 30 – 100 nm. Other syntheses were explored in order to demonstrate additional control over BTO nanoparticles' crystalline phase and will be continued into FY10. In parallel to the synthesis effort BTO-glass and PLZT-glass nanocomposites were fabricated and tested. The initial results are encouraging and laid the groundwork for further optimization of ceramic-glass nanocomposite capacitors which should lead to significant improvement in device performance.

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