

FLASH SINTERING OF SrTiO₃

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Key Words: defects chemistry, flash sintering, perovskites, modelling

Since the first reports of flash sintering in 2010 the community is continuously growing. Several parameters have been identified to be important gaining access to flash sintering, e.g. the temperature change due to joule heating, changes in the microstructure, polarization of defects or other effects on the defect chemistry. Analyzing the process is raised to another level of complexity compared to conventional sintering. Sintering and grain growth experiments under an electrical field are conducted with SrTiO₃. The results are compared to our existing database, implying simulations of the defect-chemistry, to discriminate the most important parameters in the flash sintering process. Experiments were carried out in attempt to isolate some of the listed parameters.

By controlling the maximum current of the power source, the joule heating of the sample can be controlled. Therefore experiments with different power densities in the samples up to 100 mW/mm³ were carried out to obtain different effective sintering temperatures. At a power density of 28 mW/mm³ experiments with different holding times are conducted to obtain information about the microstructural evolution. A gradual transition from flash sintering to regular sintering can be found. We elucidate the relationship between conventional sintering and flash sintering by application of the Coble model. The microstructural evolution during flash sintering seems to be similar to conventional sintering at higher temperatures.

A strong correlation of the onset of flash sintering with the defect chemistry was found in iron doped SrTiO₃ by manipulating the oxygen vacancy concentration. Increasing defect concentrations by doping SrTiO₃ leads to a decrease of the trigger temperature for flash sintering of about 500 degrees. The results of our experiments emphasize the importance of joule heating for flash sintering and grant access to parameters to control the process.

FLASH SINTERING OF TCP BIOCERAMICS: EFFECT OF PARTICLE SIZE AND INFLUENCE ON $\beta \rightarrow \alpha$ TRANSITION

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Key Words: TCP nanopowders, flash sintering, $\beta \rightarrow \alpha$ phase transition

In this work, sintering behavior of tricalcium phosphate ceramics under the effect of an external electrical field (in Flash sintering configuration) was analyzed to obtain dense bio-resorbable components. The aim was to understand whether the application of the external E-field, which allows to reduce drastically sintering time and temperature, limits also the undesired $\beta \rightarrow \alpha$ -TCP phase transition.

TCP powders were synthesized by solid state reaction and by wet synthesis through precipitation from aqueous solutions, this allowing to obtain amorphous calcium phosphate nano-particles, which were crystallized into nano β -TCP by thermal treatment. Then, cold-pressed green pellets were prepared and their sintering behavior was studied by dilatometry under different E-field at constant rate heating. The presence of α -TCP and the microstructure were investigated by XRD and SEM techniques.

It is shown that a Flash phenomenon takes place for both TCP powders morphology below 1000°C it being more evident at higher E-fields and when smaller particles were used. Moreover, although $\beta \rightarrow \alpha$ transition occurs at ~1150°C for pure TCP, the detection of both polymorphs indicates a local higher temperature, which could be correlated to Joule effect induced by the current flow along the material.

FAST SINTERING OF ALUMINA, SPINEL AND YTTRIA-STABILIZED ZIRCONIA THREE-PHASE COMPOSITES

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Field assisted sintering experiments in air were carried out on multiphase ceramics composed of Al_2O_3 , MgAl_2O_4 and cubic 8 mol% Y_2O_3 stabilized ZrO_2 (8YSZ) to investigate the grain boundary interactions as well as the sintering outcomes of an applied electric field. The ultimate goal is to develop a method to produce a dense nanocrystalline multiphase oxide ceramic for energy applications in extreme environments, without exposing the material to carbon contamination or a reducing atmosphere as is common in "spark plasma sintering" (SPS). Experiments were carried out in a modified compression furnace in air with an applied DC voltage of 50V/cm or less applied to the sample. Preliminary results with single-phase 8YSZ and three-phase alumina/spinel/8YSZ show that the experimental set up is successful for producing FAST sintered samples, with densification occurring at lower temperatures and shorter times compared to non-FAST sintered samples. Data on densification parameters, minimum sintering temperatures, and high temperature compression behavior under an applied electric field will be presented. Grain size and grain boundary energy measurements using SEM and AFM will be compared for FAST three-phase samples and control three-phase samples sintered without an applied electric field.

IN-SITU SYNTHESIS AND COATING OF NiAl-Al₂O₃ NANOCOMPOSITE BY SPARK PLASMA SINTERING

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Key Words: Coating, Composite, Spark Plasma Sintering, Nickel aluminide, Alumina.

NiAl and NiAl composite coatings with marvelous high temperature properties like good oxidation resistance can enhance surface properties and prolong application range of metallic substrates. In this paper, bulk pellets and coatings of NiAl/Al₂O₃ composites on gray cast-iron substrates are fabricated by spark plasma sintering (SPS) at 700°C and 1050°C using a highly reactive powder-mixture of "8Ni+3NiO+13Al". Mechanical activation of powder mixture is achieved by ball milling for 1 hour at 280 rpm and with ball to powder ratio of 10. X-ray diffractometry (XRD), differential thermal analysis (DTA), scanning electron microscopy (SEM) equipped with energy-dispersive X-ray spectroscopy (EDS), Vickers hardness and scratch hardness tests are performed to characterize the samples. The reactions are complete in all cases means NiAl and Al₂O₃ are produced, except for the coating produced at the lower temperature which consist of NiAl, Ni₃Al, Ni₂Al₃ besides Al₂O₃. At both temperatures, the pellets experienced internal explosions, due to the intense reactivity of the powder, producing inhomogeneous microstructures. At 1050°C, the heat absorption from the substrates resulted in damped reactions producing homogenous, dense, fully reacted NiAl/Al₂O₃ composite coatings. In the coating sample sintered at 1050°C, crystallite sizes for NiAl and Al₂O₃ determined by the Scherrer method based on XRD peak broadening were 73nm and 65 nm respectively. In addition, a bond layer with a diffusion zone of Fe, Ni and Al forms at the interface between coating and substrate which indicates extensive diffusion of atoms from coating towards the substrate and vice versa. For a polished substrate, the bond layer is thicker and more uniform compared to the bond layer on a machined surface. In all cases the adherence of coatings to substrates is good with no signs of pores or cracks. Application of NiAl/Al₂O₃ (nano)-composite coating on cast iron substrate can remarkably improve its scratch resistance. The coating sample produced at 700°C showed lower scratch resistance than the one fabricated at 1050°C. The coating sample produced at lower temperature has softer phases as Ni₃Al and Ni besides NiAl and Al₂O₃ while coating sample fabricated at higher temperature has just NiAl and Al₂O₃ in a more homogenous microstructure. Scratched surface of coatings did not reveal any peeling off at coating-substrate interface indicating their good adherence.

FAST ONE-STEP SYNTHESIS AND SINTERING OF MATERIALS PROMOTED BY ELECTRIC FIELDS

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Key Words: $\text{CaCu}_3\text{Ti}_4\text{O}_{12}$; Crystallization; Synthesis; Sintering; Field effect.

Functional dielectric materials have a recognized wide range of applications in electro-electronic devices such as capacitors, oxygen sensors and varistors [1], frequently in the form of ceramics. Processing of such materials traditionally involves several steps including calcination at a relatively low temperature for synthesis of the powder phase of concern, followed by milling, cold-pressing and then sintering at a high temperature for densification. Here we show that this processing protocol can be merged into a one-step experiment by the application of electric fields. This is illustrated with experiments carried out on perovskite-structured $\text{CaCu}_3\text{Ti}_4\text{O}_{12}$ (CCTO). The starting point was a chemically-prepared amorphous CCTO precursor powder [2], which is shown on heating to experience crystallization and synthesis of the end phase, plus densification depending on field adjustment. The processing times and temperatures are considerably reduced, enabling production of dense ceramics with microstructures showing grains noticeably finer than in conventional approach. This entire scenario, implying low-cost materials processing, demonstrates that enhanced synthesis and sintering rates apply under field input, the underlying mechanism(s) of which will be discussed. In addition, this work shows that using fields is as well a cost-effective way to detecting the temperatures at which crystallization and/or chemical reactions occur during processing. Fig. 1 is a representative example of such results. The X-ray diffraction data in Fig. 1a show that the precursor CCTO powder is still amorphous after heat treatment at 400 °C for 2h, and converts into crystalline CCTO when heat treated, for instance, at 800 °C for 2h. Under field processing, Fig. 1b,

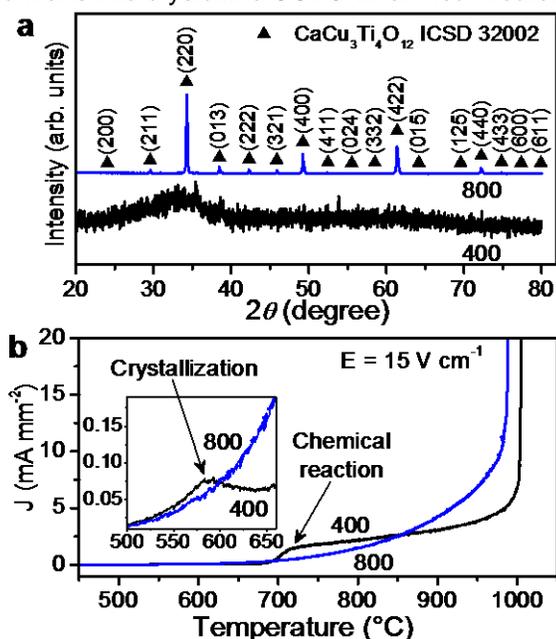


Figure 1 – a. X-ray diffraction patterns of a precursor CCTO powder heat treated at 400 °C for 2h (amorphous aspect) and 800 °C for 2h (single crystalline phase). b. Temperature dependence registered for the current density (J) under a field (E) input of 15 V/cm.

two anomalies are observed in the thermal behavior of current density (J), which were identified to be an expression of crystallization through intermediate phases toward low temperatures, and chemical reaction ending with CCTO synthesis at the intermediate temperatures. Notice that these anomalies are observed only for the amorphous powder; this is the detection power mentioned above in relation to field input. The last-processing stage in Fig. 1b, involving a remarkable non-linear increase in current density toward the highest temperatures, refers in both cases to development of flash sintering [3,4], which promoted rapid material densification. We believe that simplicity coupled with novelty of these approaches merit great attention for in-depth explorations on synthesis and sintering of materials.

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DENSIFICATION AND MICROSTRUCTURAL DEVELOPMENT OF A NEW VARISTOR CERAMIC

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Key Words: Varistor ceramic, sintering, densification, dilatometric analysis, microstructural analysis.

Varistors are nonlinear voltage dependent ceramic resistors used to suppress and limit transient voltage surges. The work reported in this paper involves the relationship between densification behavior during sintering and microstructural development which affects the varistor performance of ZnO-based ceramics doped with rare-earth oxides. Appropriate molar reagent grades were used to prepare a varistor formulation ZM (mol %) 99.00.ZnO + 0.50.CoO + 0.50.MexOy, where MexOy = 25.00.Pr₆O₁₁ + 15.00.La₂O₃ + 15.00.Nd₂O₃ + 15.00.Sm₂O₃ + 15.00.Eu₂O₃ + 15.00.Dy₂O₃. The investigations on the densification and sintering processes that occur in this varistor ceramic were made based on the phenomenological approach developed by Su and Johnson [1] and Hansen [2], also used by Choron *et al.* [3] in a recent work on a similar ceramic system, which makes it possible to group the set of kinetic (time) and thermal (energy) parameters in the global thermal-time function (GTT), which describes the densification behavior during the sintering, allowing identify their stages.

From the curve of Figure 1(a), the inclination of the region (intermediate) between two approximately horizontal segments, where there is a continuous increase in the degree of densification to the ceramic system studied, it is broad in terms of the densification degree, denoting the sintering in the presence of liquid phase. In the graph of the Figure 1(b) it is noted that the first stage is rather short and others are long, but there is no evidence of transitional characteristic areas of solid solution formation. In fact, as can be seen in the SEM photomicrograph, Figure 1(c), characteristic of the studied system, there is a clear formation of intergranular layers that are remnants of the percolation liquid phase during sintering, so that the microstructure of ZM based varistor ceramics consists of two phases: ZnO grains (doped with Co) and rare-earth elements rich phase (also doped with Co) segregated at grain boundaries.

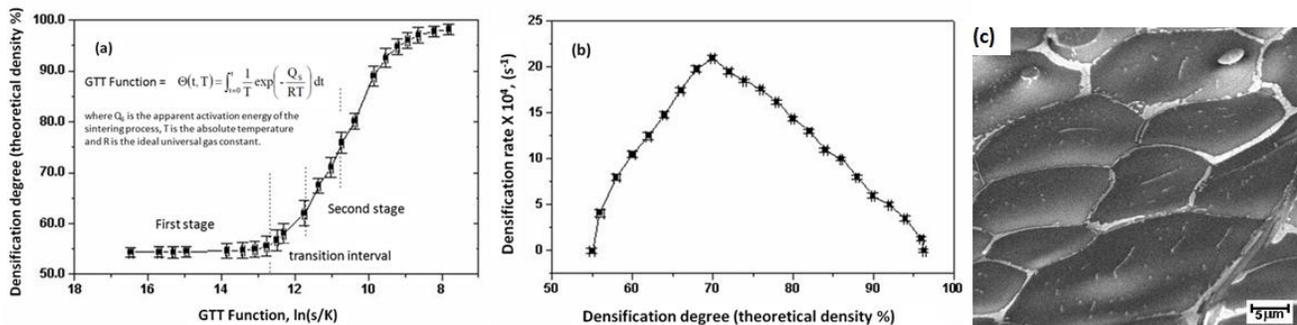


Figure 1 – (a) Microstructural sintering evolution and (b) Densification rate as a function of the densification degree (relative density) for the varistor ceramic studied. (c) SEM micrograph of densified varistor ceramic studied specimen.

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EFFECT OF NANO-SIZED Si_3N_4 ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF SPARK PLASMA SINTERED GRAPHITE-ALUMINUM COMPOSITES

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The demand for lightweight materials in the aerospace and automobile industries has led to researches on graphite and graphite aluminum (Gr-Al) composites. The composites have gained application in a lot of fields but not without their own drawbacks. These drawbacks have generated many research interest with the aim of improving the properties and expanding the applicability of Gr-Al composites. This work is aimed at studying the effect of nano Si_3N_4 on the microstructure and coefficient of thermal expansion (CTE) property of Gr-Al composite. In this study, micro Si_3N_4 powder was milled for 30 hrs using planetary mill (Restch PM100). The particle sizes and shapes of the micron and nano-sized particles were studied using SEM and image J software respectively. Sintering of Gr-Al composite reinforced with various percentages (4, 8, 12 and 16%) of nano Si_3N_4 particles was done at a temperature of 560°C and pressure of 50 MPa. Sintering was carried out at a heating rate of $50^\circ\text{C}/\text{min}$ with a dwelling time of 10 min. In addition, a study of the microstructure, CTE and the phases present in the resulting composites were done after full sintering was achieved. The results also showed that nano Si_3N_4 reduced the relative density of Gr-Al composite down to 89.47%. This reduction was more obvious particularly at 16% composition and heating rate of $50^\circ\text{C}/\text{min}$. However, the CTE continues to fluctuate with different compositions, with the best CTE occurring in the composite reinforced with 8 wt% Si_3N_4 . This work which is part of an on-going research has shown that we can take advantage of some of the properties of Si_3N_4 to improve the performance of graphite-aluminum composite.

OBSERVATIONS ON FLASH SINTERING OF URANIUM DIOXIDE

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Key Words: Urania, Uranium Dioxide, Flash Sintering, Nuclear Fuel

The recent prioritization on development of nuclear fuels with enhanced accident tolerance has led the community to consider fuel systems that offer improvements in certain performance characteristics such as fission gas retention, oxidation resistance and thermal conduction. Advanced fuels being studied for enhanced accident tolerance often have unique characteristics that make conventional sintering routes undesirable for or incapable of yielding the required fuel pellet characteristics. For illustration, UO_2 -composite fuels with second phases for increased thermal conductivity, such as SiC, can have deleterious reactions with the UO_2 fuel above operating temperatures but below temperatures required to sinter a high quality fuel pellet. In such cases, field assisted sintering techniques can enable these compositions to be considered for application as nuclear fuel in addition to offering more efficient and economical fuel pellet fabrication.

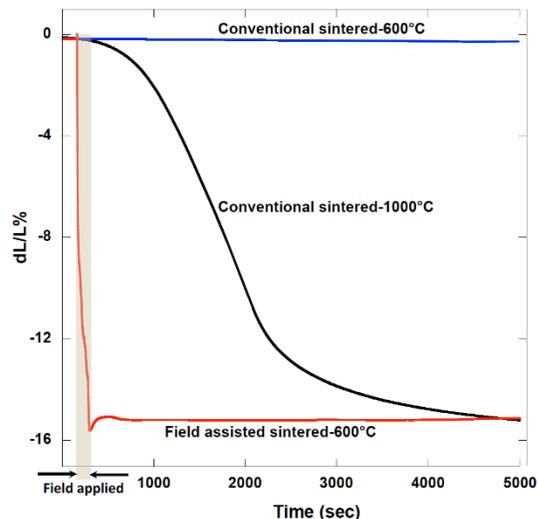


Figure 1 – Sintering strain ($dL/L_0\%$) obtained from $\text{UO}_{2.16}$ comparing flash sintering at 600°C to conventional sintering at 600°C and 1000°C . Samples were sintered under gettered argon.

As indicated above, urania-based composite fuels are being considered for these enhanced fuels, and field assisted sintering, particularly spark plasma sintering, has been used recently to both process UO_2 and $\text{UO}_2\text{-SiC}$ ¹ fuel pellets and as a method to fabricate UO_2 with volatile fission products². As a potential approach to employ the benefits of field assisted sintering for “enhanced UO_2 ” while mitigating changes from the existing nuclear fuel fabrication infrastructure, we have examined flash sintering in UO_2 and $\text{UO}_2\text{-UB}_x$ composites with second-phase contents up to ~10 vol%.

Early studies on uranium dioxide showed that flash sintering of cylindrical compacts resulting in relative densities as high as 94% could be achieved in seconds as opposed to several hours at temperatures hundreds of degrees lower than necessary under conventional sintering (Fig. 1). The critical parameters governing flash sintering of urania were examined, such as, oxygen-to-metal ratio, applied field, temperature, current and hold times. The flash event was observed in hyperstoichiometric urania at room temperature with applied fields as low as 25 V/cm with incubation times of several minutes.

Results of the parametric studies will be presented along with early data from in-situ x-ray diffraction studies. The role of electric field on defect behavior in this semiconducting compound will be discussed.

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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF SPARK PLASMA SINTERED TUNGSTEN-COPPER – ZINC COMPOSITES

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Tungsten/Copper (W/Cu) composites, generally used for applications whereby the combination of high heat resistance, high electrical and thermal conductivity and low thermal expansion is required, are suitable for hard working conditions such as intensive electrical sparks, gouging spark erosion, surface melting, welding, material transfer etc. However, the large disparity between the melting point of tungsten (3410 °C) and that of copper (1083 °C) make their production through traditional processes very difficult. Furthermore the difficulty of approaching near theoretical densities even by using liquid phase sintering, due to the negligible mutual solubility of the constituents and high wetting angle of liquid copper on tungsten is quite challenging. In the present work, the densification mechanisms during spark plasma sintering of tungsten/copper and the effect of the addition of zinc on both microstructural and mechanical properties were investigated. Commercially pure tungsten powders were dry mixed with varied amounts of copper (25, 30 and 50 wt.%) with the addition of different weight fractions of zinc (Zn) (5, 10, 15 and 20 wt.%) using a T2F Turbular mixer for 8 h and at a speed of 49 rpm. The blended composite powders were then sintered using spark plasma sintering system (model HHPD-25 from FCT Germany) with varying dwell times, sintering temperatures, pressures and a constant heating rate of 100°C min⁻¹. The sintering of W/Cu was used as a base study prior to the addition of Zn for a better understanding of the interplaying mechanisms. Densification was monitored through the analysis of the recorded punch displacement and the measured density of the sintered samples using Archimedes method. Microstructural evolutions and phase changes were investigated using scanning electron microscope and X-ray diffraction techniques. The effect of Zn addition on the mechanical properties was investigated through hardness, three point bending flexural test and fracture toughness measurements as well as the analysis of fracture surfaces.

SELF-ORGANIZED STRUCTURE IN CURRENT-ACTIVATED PRESSURE-ASSISTED DENSIFICATION (CAPAD)

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Using nanostructured thermoelectrics led to a significant improvement of the figure of merit. An easily upscalable and cheap way of producing nanostructured bulk material is current-activated pressure-assisted densification (CAPAD). This process is quite complex and it has been shown that the powder morphology gives a feedback to the process itself [1,2]. We investigated the influence of particle properties of binary mixtures on the evolving structure. For this purpose we use a network model [3] based on the Onsager-de Groot-Callen theory. In the simulations we find that depending on the difference between the Seebeck coefficients of both materials either a well mixed or a segregated sintered structure is obtained.

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BURST MODE/FLASH SINTERING OF HYDROXYAPATITE NANOCRYSTALS WITH THE ADDITION OF TiO₂ FOR BIOMEDICAL APPLICATIONS

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Flash sintering is a novel method of sintering which reduces the sintering time and temperature by applying an electric field while heating the sample. Hydroxyapatite, the major constituent of bone grafts, is flash sintered in this experiment in order to improve the mechanical properties for its application. Titania is added to the material system in order to optimize the specimen with respect to electrical and mechanical properties while maintaining biocompatibility. With 10 wt.% addition of titania, a hydroxyapatite composite specimen was flash sintered at 1000 °C. Additionally, Energy Dispersive X-ray Diffraction patterns were collected every two seconds as the sample flash sintered, from which d-spacing and lattice parameters were calculated as a function of scan number and temperature.

FIELD – ASSISTED DENSIFICATION OF TRICALCIUM PHOSPHATE – ZIRCONIA COMPOSITES

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A high degree of densification (~90% theoretical density) was observed in samples of pure β – tricalcium phosphate (TCP), as well as of β – tricalcium phosphate and 3% yttria stabilized zirconia (YSZ) mixtures. Densification was done with the assistance of an applied electric field, a method also known as flash sintering or burst mode densification. Hardness testing of the mixed samples yielded an average Vickers Hardness of around 1.05 GPa, a value that is much lower than the Vickers Hardness of human bone.

In conjunction with field – assisted densification experiments, in situ EDXRD scans showed a change in the structure of the YSZ phase upon heating around 400 °C. This was attributed to the phase transformation from tetragonal to monoclinic zirconia.

FLASH SINTERING OF BARIUM TITANATE

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Flash sintering (also called burst-mode densification) is a relatively new method for sintering high melting temperature ceramic materials, such as barium titanate. Barium titanate is a ferroelectric ceramic of the perovskite family with piezoelectric properties and a photorefractive effect. Barium titanate is used across a wide number of applications including capacitors, transducers, thermistors, and nonlinear optics. Flash sintering allows for sintering of barium titanate at very low temperatures in ultra-short timeframes. By applying a voltage gradient across a sample, coupled with the application of a weak electric field, barium titanate was sintered at times under ten seconds at temperatures as low as 420 °C. Energy Dispersive X-Ray Diffraction was performed during *in-situ* flash sintering experiments to demonstrate the lattice expansion of the of barium titanate during burst-mode densification.

A STUDY OF THE RECRYSTALLIZATION AND DENSIFICATION OF AMORPHOUS BN USING APPLIED THERMAL & ELECTRIC FIELDS BY SYNCHROTRON PROBE

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This research study deals with the recrystallization and densification behavior of amorphous BN under concurrent application of thermal and electric fields.

Recrystallization is facilitated by heat alone above 900C. Recrystallization was present in samples heated above 750C with an applied field of 1000V/cm.

Results suggest that a different phase is preferred during recrystallization when an electric field is present in addition to a thermal field. Further testing is needed to determine the nature of the phase formed in this unique processing method. Results of flash sintering of BN under higher applied electric field will be also discussed.

**GROWTH BEHAVIOR OF FACETED $\text{Na}_{1/2}\text{Bi}_{1/2}\text{TiO}_3\text{-BaTiO}_3$ GRAINS
IN SINGLE AND TWO-STEP SINTERING IN SUPPORT FOR
THE MICROSTRUCTURAL EVOLUTION PRINCIPLE**

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We investigated the grain growth behavior in $89\text{Na}_{1/2}\text{Bi}_{1/2}\text{TiO}_3\text{-}11\text{BaTiO}_3$ (mol %, NBT-11BT) at 1100 and 1200 °C, and also under two-step sintering at 1200 (first) and 1100 °C (second). When the powder compacts were sintered at 1100 and 1200 °C, the initial growth behavior was quite normal; however, the subsequent growth behavior was distinctively abnormal and moderately abnormal, respectively. This difference in growth behavior is attributed to a change in the critical driving force for appreciable migration of the boundary with temperature and can be explained by the coupling effect of the maximum driving force for the growth of the largest grain and the critical driving force for appreciable migration of the grain boundary, viz. the principle of microstructural evolution. In contrast, two-step sintered samples exhibited stagnant grain growth behavior at the second sintering step up to 10 h at 1100 °C after appreciable initial growth of grains during the first sintering step at 1200 °C. Measurement of the grain size distribution after the first sintering step at 1200 °C indicated that the maximum driving force for the growth of the largest grain was reduced to a lower value than that in the sample conventionally sintered at 1100 °C without the first sintering step. This suggests that the beneficial effect of two-step sintering for suppressing grain growth is due to the reduction of the maximum driving force after the first sintering step compared with the maximum driving force in the conventionally sintered sample with the same average grain size. The experimental results confirm the general applicability of the recently deduced principle of microstructural evolution.

