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Tailoring the microstructure by a proper electric current control in flash sintering: The case of barium titanate

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ABSTRACT

Flash sintering is arousing growing interest because high-density ceramics can be obtained at lower temperatures and shorter dwell times than conventional sintering. However, not only temperature and dwell times should be controlled during flash sintering but also parameters such as the electric field and electric current should be considered. Controlling all the parameters during the processing allows comprehensive control of the microstructure and, consequently, functional properties can be improved. In this work, it is evidenced that an exhaustive control of the flash electric current is a crucial factor for tailoring the microstructure of $BaTiO_3$ ceramics. The results reveal that the most suitable way to control the sintering process is by using non-linear current profiles because better densification and improved grain growth is achieved. Although the results focus on $BaTiO_3$, this work offers a new pathway to tailor the microstructure of flash sintered ceramics, which may be extended to other materials.

1. Introduction

The massive production of ceramics entails high energy consumption associated with its manufacturing because long-time, high-temperature heat treatments are required. Accordingly, non-conventional faster kinetics sintering routes have emerged in order to mitigate the environmental impact associated to conventional ceramic sintering. In this perspective, the field-assisted flash sintering has started to be used since the application of a proper electric field has proven to significantly reduce sintering time and temperature [1]. Particularly, at a specific combination of electric field and temperature, an abrupt drop of the sample resistivity takes place and the flash event occurs, thereby allowing for rapid densification (on the order of seconds to minutes). A proper control of the flash sintering technique parameters such as atmosphere, electric field, electric current, and current profile, among others, has shown to crucially determine the sample properties [2,3]. Although several phenomenological descriptions have been proposed to describe the flash sintering process, the mechanisms leading to the densification are still controversial [4,5].

Even though flash sintering has been used to obtain a wide array of structural ceramics [6,7], its use for sintering functional materials is still moderated, even scarce for ferroelectrics. In fact, works related to flash sintering of ferroelectric materials have been focused on obtaining dense ceramics with appropriate microstructure [8–12], but comprehensive studies related to microstructure-properties relationship are difficult to be found. In this work, commercial micropowder of $BaTiO_3$ (BTO) is subject to flash sintering experiments in order to evaluate the influence of some operating conditions on the flash-sintered sample microstructure. A thorough control of the sintering parameters such as dwell time, electric field, current density and current profile, is shown to results in exhaustive tailoring of the sample microstructure and, consequently, of the functional properties.

BTO is a well-known polar oxide that has been extensively studied because it is considered a prototype of ferroelectric perovskites [13]. Therefore, BTO is used as model system for a wide variety of studies such as grain and scaling effects, doping effect, among other. Furthermore, it is also used to form binary and ternary ferroelectric systems, chemically designed to show enhanced properties for specific applications [14,15]. Undoped and doped BTO was flash-sintered for the first time recently [8, 9], showing that dense BTO ceramics are possible to be obtained under different electric field and current conditions. Here, a step further is done and a control of the grain size is achieved by a suitable electric current profile management. Results suggest that flash-sintered ceramics with customized microstructure can be achievable by a proper electric

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current control.

2. Experimental procedure

2.1. Sintering experiments

Commercial $BaTiO_3$ powder (Sigma Aldrich, 338842–500G, Lot# MKCJ0127), with ~2 µm medium grain size, is pressed into dog bone pellets (supplementary Fig. S1a) using a 270 MPa uniaxial load to form the initial green sample (GS). A typical flash sintering setup (supplementary Fig. S1b) is used, where GS is hanged in the center of the furnace using two platinum electrodes attached to its handles. A programmable DC power supply (Ametek XG 600–2.6) is remotely controlled by homemade software. The power supply changes the operation mode automatically when the current limit is reached. Electrical contact between the sample and the electrodes is assured using platinum paint (SPI Supplies, 04990-AB, Lot# 1240813) mixed with platinum paint thinner (SPI Supplies, 04989-AB, Lot# 1241022). Contacts are adequately painted so that a uniform electric field flows along the sample. The shrinkage of the sample is monitored by a CCD camera.

Two flash sintering methods are put into operation: conventional flash sintering (CF) and controlled current flash sintering (CCF). On the one hand, for CF experiments, the sample is heated at a rate of 10 °C/min from room temperature while being held in electric field control until the preset current limit is achieved. After dwelling, both field and furnace are turned off and the sample cools down naturally. On the other hand, for CCF experiments, the sample is heated at a rate of 10 °C/min from room temperature until reaching 950 °C, 1000 °C or 1085 °C and then an electric field value of 300 V/cm, 200 V/cm or 150 V/cm, respectively, is applied. The sample is held in current control exclusively until the preset current limit is achieved. Then, current control is kept for 10 min. Different current profiles are used (i.e., conventional, ramp, step, quadratic, square root) so that the current limit is always reached at the same time (supplementary Fig. S2). The evolution of the samples' resistivity is measured to evaluate the influence of the electrical contacts. Similar resistivity values are obtained for all tested samples during the dwell time (supplementary Fig. S3) when the same electrical conditions are reached. Therefore, electrical contacts do not introduce significant uncertainty in the flash sintering experiments.

After sintering, dog bone shaped specimens are cut into 10 mm × 3 mm × 1 mm samples to avoid density inhomogeneities, which could be a critical issue in the vicinity of the electric contacts. Although the formation of density gradients is an intrinsic issue related to the flash sintering technique, this phenomenon can be minimized by proper control of the flash event. Scanning electron microscopy (SEM) images show that no noticeable density gradient has arisen, as shown as a representative example in supplementary Fig. S4.

Conventional sintering at 1350 °C for 4 h of dwell time is also carried out to obtain a control sample. Both heating and cooling are reached at a temperature ramp of 10 °C/min. The sintered sample shows 97% of density and 2.5 µm of medium grain size. Microstructure, dielectric response, and ferroelectric hysteresis loop show the expected behavior for a 2.5 µm grain-sized $BaTiO_3$ sample (supplementary Fig. S5).

2.2. Microstructure and functional properties

Flash-sintered samples are polished and then chemically etched for 1 min. Archimedes method is used to measure the samples density. The microstructure is assessed by using a field-emission scanning electron microscope (JEOL, JSM-7001 F). Medium grain size is determined from SEM images by taking a grain size population of at least 300 grains. After that, samples are gold sputter coated on its parallel faces to ensure electrical contact for electrical characterizations. An LCR meter (Agilent E4980A) is used to measure permittivity data of unpoled samples from room temperature to around 250 °C at various frequencies, ranging from 100 Hz to 1 MHz. Electric field induced polarization (*P-E*) hysteresis loops are measured in a typical Sawyer-Tower configuration by applying a triangular electric field of amplitude of 2 kV/mm at a frequency of 1 Hz, at room temperature. Samples are then poled in a silicone bath at 80 °C for an hour under a DC electric field of three times their coercive field and subsequently aged for a day before further measurements to avoid influence of the aging process. A d_{33}-meter (KCF Technologies, PM3500) is used to determine the static longitudinal direct piezoelectric constant.

3. Results and discussion

3.1. Conventional flash sintering

Since powders are known to strongly influence the microstructure of flash-sintered specimens [16], a throughout study of flash parameters is required in order to fully characterize the sintering conditions. Fig. 1 shows the obtained correlation between the applied electric field and the onset temperature, which can be understood as an imbalance between Joule generated heat and dissipated heat, i.e., the Dong-Chen model [17]. For these experiments, electric current density and dwell time are set at 15 mA/mm^2 and 10 min, respectively. The conductivity evolution of the samples is tracked to understand better the flash onset and the sintering thermal runway (supplementary Fig. S6).

As reported in Fig. 1a, sample density decreases as electric field rises in accordance with the literature [8]. However, while an inverse relation between electric field and grain size was reported [8], results of this work show no discernible correlation as standard deviation overshadows average grain size (it is remarkable to note that measured grain size is barely above the micropowder size). Dielectric and ferroelectric responses show the expected BTO behavior for all flash-sintered samples, although better properties are depicted when sintering is done at 200 V/cm (supplementary Fig. S7). Taking into account a reasonable compromise between a low onset temperature, a high density and good functional properties, a region between 150 V/cm and 200 V/cm is defined as optimal electric field for the flash sintering.

A second set of experiments is performed in order to evaluate the influence of the electric current over sintering temperature and densification. An electric field of 150 V/cm and a dwell time of 10 min are used. Results are collected in Fig. 2a, where a current-induced grain growth is evidenced. However, densification has a non-linear relation with current, achieving a maximum value around 20 mA/mm^2. Dielectric and ferroelectric responses also show the expected BTO behavior for all flash-sintered samples, but ferroelectric properties and dielectric losses degrade as current flow increases (supplementary Fig. S6), which is in accordance with the formation of microstructural defects (Fig. 2b-e). A current density value of 15 mA/mm^2 offers the best combination of properties.

The effect of dwell time is also analyzed, as it is often neglected in the literature. Even though dwell time effect is well-known in conventional sintering, studies concerning dwell time in flash sintering are conducted in a non-rigorous manner since dwell time is usually changed between experiments, so that several variables are modified at the same time. Fig. 3a shows an increment in both density and grain size with dwell time, but an increase of one order of magnitude is necessary to achieve a significant change. Dielectric and ferroelectric responses follow the expected BTO behavior without a significant change for all flash-sintered samples (supplementary Fig. S7) whereas the microstructural characterization evidences the aforementioned improvement of density as dwell time increases (supplementary Fig. S8). As a result, a dwell time value of 10 min may be then considered as short enough time to obtain well-density BTO ceramics.

Summarizing, a comprehensive study of flash sintering conditions of BTO micropowder has been carried out. As discussed above, the electric field, the current density as well as the dwell time play a non-trivial role on the sintered sample microstructure and, therefore, notably influence its functional properties. Here, it is shown that electric field induces an increase in dielectric losses due to the formation of microstructure noise,

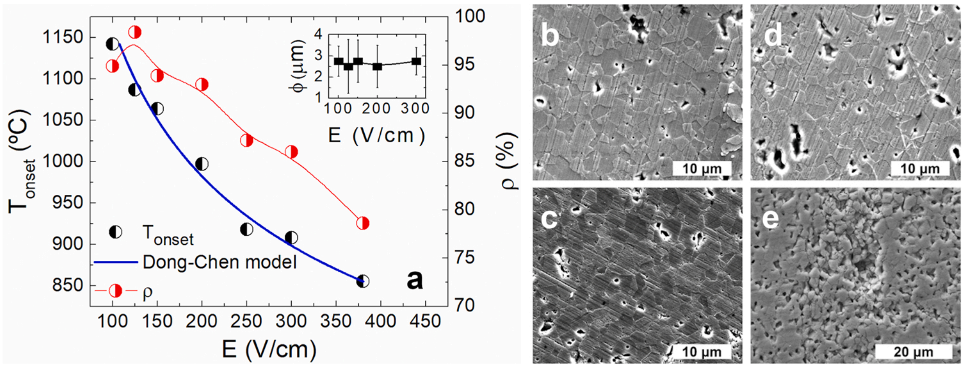

Fig. 1. (a) Sintering temperature (T_{onset}), relative density (ρ) and grain size (ϕ) of samples obtained in conventional flash experiments carried out using different electric field values (E). Current density and dwell time have been maintained at 15 mA/mm^2 and 10 min, respectively. The Dong-Chen model is used to validate the dependence of T_{onset} with E showing a reasonable fit to the experimental data. (b-e) Representative micrographs of samples obtained in conventional flash sintering experiments carried out using different electric field values: (b) 125 V/cm, (c) 150 V/cm, (d) 200 V/cm, and (e) 300 V/cm. Current density and dwell time have been maintained at 15 mA/mm^2 and 10 min, respectively.

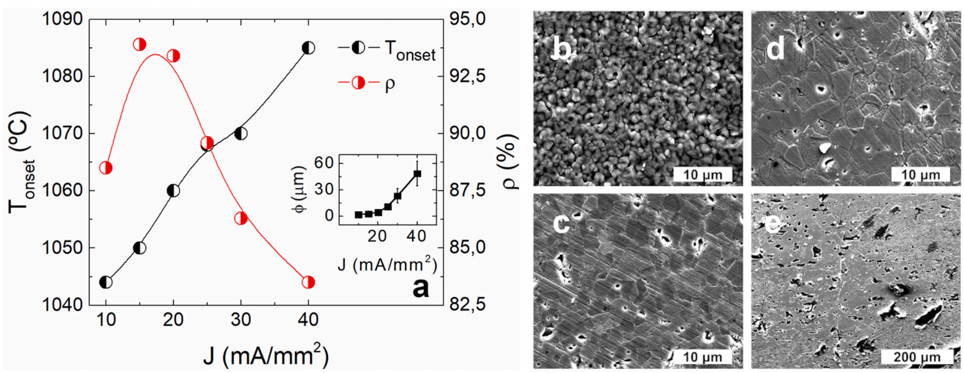

Fig. 2. (a) Sintering temperature (T_{onset}), relative density (ρ) and grain size (ϕ) of samples obtained in conventional flash experiments carried out using different electric current density values (J). Electric field and dwell time have been maintained at 150 V/cm and 10 min, respectively. (b-e) Representative micrographs of samples obtained in conventional flash sintering experiments carried out using different electric current density values: (b) 10 mA/mm^2, (c) 15 mA/mm^2, (d) 20 mA/mm^2, and (e) 40 mA/mm^2. Electric field and dwell time have been maintained at 150 V/cm and 10 min, respectively.

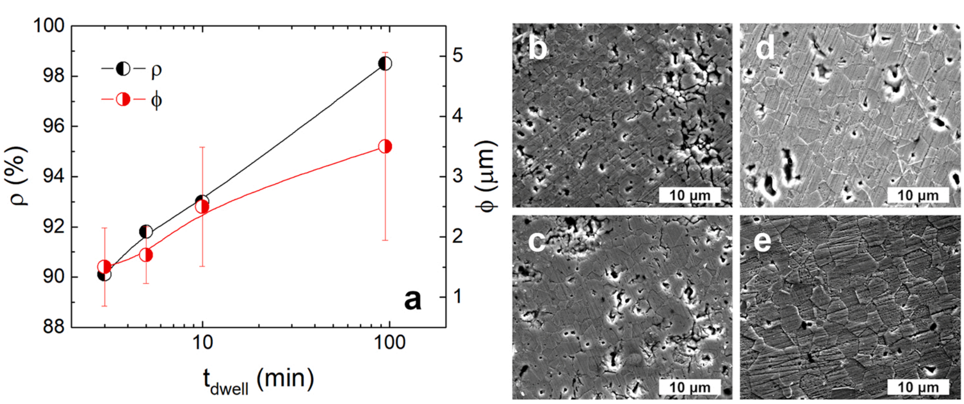

Fig. 3. (a) Relative density (ρ) and grain size (ϕ) of samples obtained in conventional flash experiments carried out using different dwell times (t_{dwell}). Electric field and current density have been maintained at 200 V/cm and 15 mA/mm^2, respectively. Electric field value of 200 V/cm was selected instead 150 V/cm because it showed a lower density for a dwell time of 10 min, therefore yielding better room for improvement at higher dwell times. (b-e) Representative micrographs of samples obtained in conventional flash sintering experiments carried out using different dwell times: (b) 3 min, (c) 5 min, (d) 10 min, (e) 95 min. Electric field and current density have been maintained at 200 V/cm and 15 mA/mm^2, respectively.

which is related to a lower densification as shown in the SEM images (Fig. 1b-e). Poor dielectric response is obtained for sample flash-sintered at 100 V/cm (supplementary Fig. S5), which can be attributed to the fact that 100 V/cm might not be a high enough electric field value to allow flash event to take place, as will be further discussed. Current density experiments show that densification is improved up to a certain value of current density, from which microstructure damage is induced (Fig. 2e) []. Finally, large dwell times slightly improve the density but no significant improved properties are obtained (supplementary Fig. S7).

3.2. Controlled current flash sintering

Electric power appears to be an important parameter for microstructure evolution during flash sintering [18–20]. When comparing CF experiments, it is found that samples flash-sintered in shorter times (i.e., higher instant power values) exhibit not only bigger grains for the same total applied electric power (Fig. 4a) but also higher microstructural damage (Fig. 4b-c). This result hints the influence of the electric power profile seems to be non-trivial. Therefore, the control of the electric current profile should be introduced as a new sintering parameter, thereby yielding refined microstructure. That is, for some given sintering conditions, modifying the electric profile allows to carefully adjust the total power supplied to the sample and, therefore, a fine tuning of the microstructure could be achieved. In fact, recent works report an improvement of both density, grain size and sample homogeneity when applying a current ramp during flash sintering [21–23]. Hence, an exhaustive study of different current profiles and their influence over the microstructure is hereafter presented.

Fig. 5 shows the experimental electric profiles for CCF experiments, where electric fields of 150 and 200 V/cm, current density of 15 mA/mm^2, and dwell time of 10 min were used as starting sintering conditions. First results showed a remarkable samples density enhancement

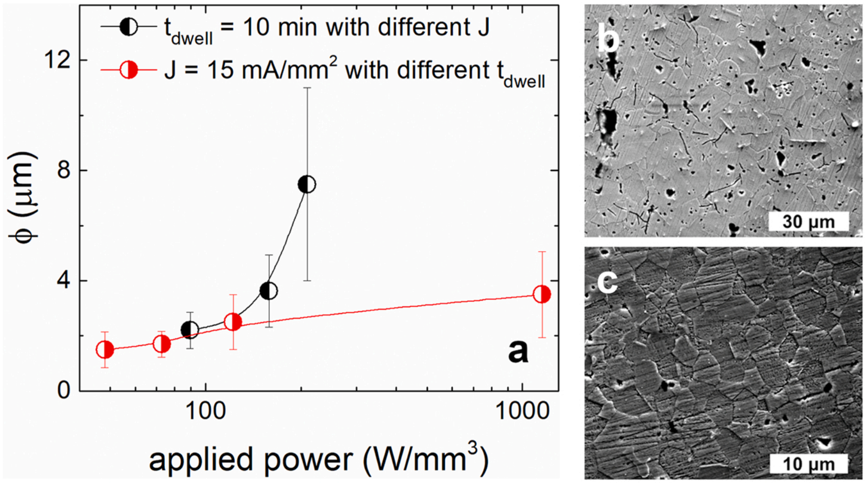

Fig. 4. (a) Comparison of grain size (ϕ) respect to total applied electric power for samples obtained in conventional flash experiments carried out using different dwell times and current densities. Electric field has been maintained at 200 V/cm for both cases. Normalized total applied power is used as means to compare samples sintered under different times and electric conditions. (b-c) Representative micrographs of samples obtained in conventional flash sintering experiments carried out using different dwell times and current densities: (b) 25 mA/mm^2 and 10 min, (c) 15 mA/mm^2 and 95 min. Electric field has been maintained at 200 V/cm.

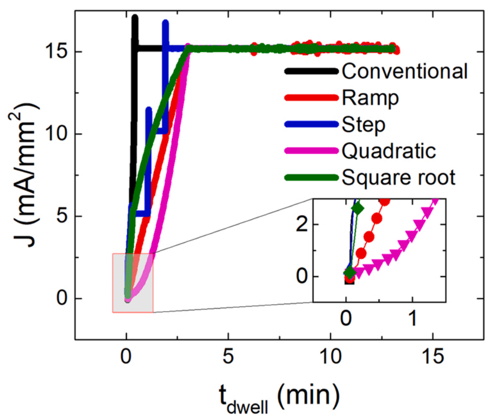

Fig. 5. Experimentally obtained current density profiles. Profile slopes are selected so that maximum current is reached in 3 min for all cases. Samples are left to dwell for 10 min under maximum current. The inset shows a zoom of the first minute of the experiment (shaded region).

when some current profiles were used (in particular, the quadratic profile), which suggested harsher electrical conditions could be adopted for CCF experiments. Fig. 6(a) summarizes the experiments results, evidencing that density depends on both the electric power and the applied current profile. Results show that high densities can be achieved even under high electric power for current profiles with a low initial slope (i.e., ramp and quadratic profiles). A proper selection of the electric current profile allows to use high electric power without a remarkable drop of densification. This is a relevant result because grain size can be tuned by varying the electric power, as shown in Fig. 6(b). Therefore, an adequate combination of current profile and electric power gives rise to a tailored microstructure. Table S1 summarizes obtained densities for three representative flash sintering conditions for more information.

The functional properties of BTO ceramics are known to be strongly dependent on their grain size [13], as happens for many other ferroelectric systems. Supplementary Fig. S10 shows permittivity versus temperature curves and *P-E* hysteresis loops at different electric power values. As may be observed, all samples exhibit the expected behavior for a BTO ceramics. However, the properties improve depending on current profile and electric power; that is, depending on density and grain size, respectively. Fig. 7 shows an expected behavior for the room temperature dielectric constant and the direct piezoelectric constant of BTO ceramics [13,24], but a significant ε' and d_{33} improvement is revealed for certain current profiles as compared with CF and

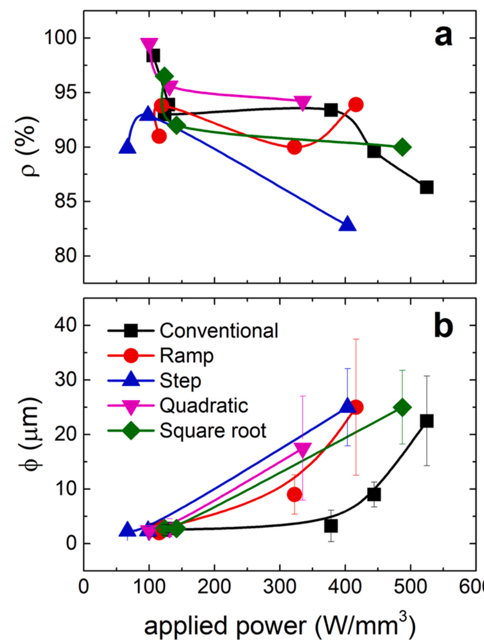

Fig. 6. (a) Relative density (ρ) and (b) grain size (ϕ) of samples obtained in controlled current flash experiments carried out using different electric field and current density values and profiles. Normalized total applied power is used as means to compare samples sintered under different electric conditions.

conventional sintering samples, which may be understood to be purely related to the tailored microstructure of CFF samples. Results demonstrate enhanced functional properties can be achieved by a proper control of the electric current profile.

3.3. The role of a proper electric current control

The effect of the electric current control during flash sintering via the application of a steady current ramp has been studied for ZnO and 3YSZ [3,22,23]. It has been reported that higher densification and higher grain size is achieved by using slow ramps, which is thought to be a consequence of lower lattice reduction and, therefore, improved mass transport. Ramping current entails ramping electric field thus less oxygen vacancies are produced and recombination with atmospheric oxygen is possible [25,26]. Densification is therefore not prevented, but enhanced. In summary, at slower profiles the sample is kept in this “densification region” longer and, therefore, better sintering is achieved.

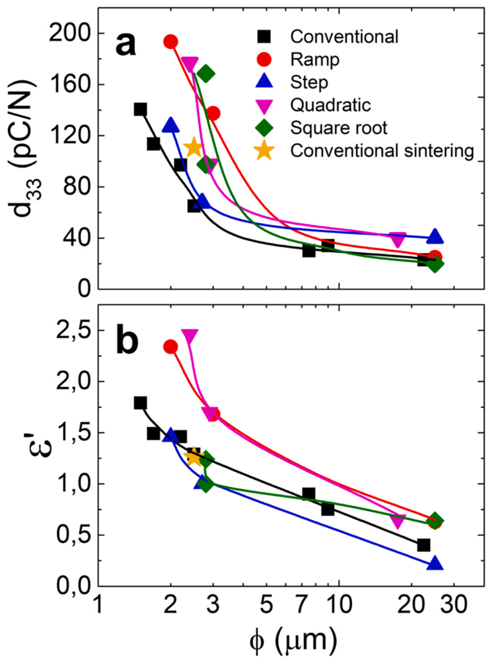

Fig. 7. Comparison of the (a) static longitudinal direct piezoelectric constant (d_{33}) and (b) room temperature dielectric constant of samples obtained in conventional flash and controlled current flash experiments carried out using different current profiles. Values corresponding to the conventional sintered sample are also shown.

Generally, electric field and electric current are closely related during a flash experiment. Controlling the current flow imposes a constrain over electric field values. When restricting the current flow, applied electric field slowly rises to account for resistivity evolution. Fig. 8 shows a comparison between current and electric field evolution when different current profiles are used. On the one hand, the evolution for steep profiles is similar to conventional flash. The sharp increase of electric field for these profiles yields densification retardation in the early stages of sintering due to high lattice reduction, leading to defect accumulation in the grain boundaries and dislocations [26]. Initially, electric current increases as electric field rises. In fact, steeper profiles have higher initial current rates, which should (theoretically) lead to enhancement of densification and grain growth due to the fast Joule heating [21]. As it tuns out, heating phenomena are not allowed to fully densify the sample because they compete with electric fields densification retardation. Defect accumulation leads to the formation of field-induced percolation paths and the flash event then takes place [18], triggering the electric field to abruptly drop. This phenomenon is thought to be a consequence of the sudden conductivity enhancement due to the release of accumulated oxygen vacancies. Densification is then partially allowed via mass transport and Joule heating.

Smoother profiles, on the other hand, follow a steady increase of electric field and electric current. Initially, electric field is very low, so oxygen vacancy recombination is possible. Mass transport is therefore the leading mechanism for densification. Electric field increases as current rises, thereby starting densification retardation phenomenon and defect accumulation. At a high enough electric current, a maximum value of electric field is observed (Fig. 8) and the flash event takes place. Electric field slowly declines and the characteristic jump of the vertical profiles is not present, leading to hypothesize that defect accumulation is less prominent on smooth profiles. In fact, this could explain why electric field maximum is broader the lower the slope of the applied current profile. If defect accumulation is below a saturation point, mass transport and densification will enhance [26], which happens at low electric field values. As electric field increases, defect saturation is reached and grains grow only via thermal gradients and rapid heating.

The aforementioned ideas could explain why some profiles allow for better densification and improved grain growth. Fig. 8 shows that a lower initial current slope allows to maintain smaller electric field over longer time, which translate to better densification via mass transport. Electric field keeps rising and, at a certain value, densification retardation is triggered. After electric field reaches its maximum value, it starts to drop, thereby weakening densification retardation and promoting grain growth via fast heating. It is noteworthy that the quadratic profile transits from exhibiting the broadest low electric field region to show a high current rate, which explains why better density and improved grain

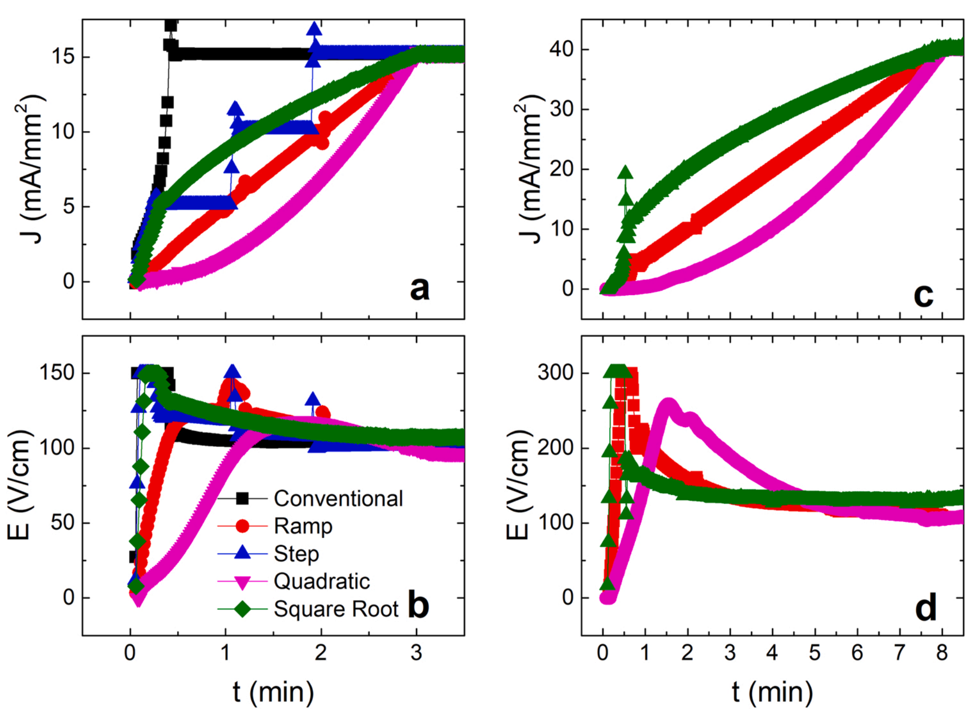

Fig. 8. Current density and electric field profile comparison of samples obtained in controlled current flash experiments carried out using different electric current profiles and conditions: (a, b) low electric power and fast current rates (current density, electric field and dwell time have been maintained at 15 mA/mm^2, 150 V/cm and 10 min, respectively); (c, d) high electric power and slow current rates (current density, electric field and dwell time have been maintained at 40 mA/mm^2, 300 V/cm and 10 min, respectively).

growth can be achieved by using this profile, as reported in Fig. 6. In other words, two-step sintering is desirable: (i) the lower the starting slope is, the better is the densification and (ii) the greater the current slope is after electric field maximum is reached, the greater is the grain growth.

4. Conclusion

In this work a comprehensive study of flash sintering parameters for BTO has been carried out. Results have shown that an adequate selection of the electric current profile is crucial to refine the sintering processes. Profiles exhibiting lower electric current slopes lead to better results, which can be further improved by using slower electric current rates. A refined microstructure can be accomplished by controlling the conventional sintering parameters but it can be fine-tuned by a suitable current profile selection. It is demonstrated that high density (> 90%) BTO ceramics for a broad array of grain sizes (ranging from 2 μm to 25 μm), short dwell times (10 min) and relatively low furnace temperatures (around 1000 °C) can be obtained by flash sintering taking into account a proper electric current control. Although results are focused on BTO, the here proposed exhaustive control of the electric current may be extended to other materials.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jeurceramsoc.2022.05.053.

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