



Investigation on Some Physical and Microstructural Behavior of Composite Ceramic Capacitors Prepared from Nano Scale Powder

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Abstract

High purity of (99.999%) Nano grain size from both SrTiO₃ and BaTiO₃ was used to prepare the composites ceramic capacitors. The fabricated discs were sintered at 1300 °C, for 3 hours under static air. X-ray diffraction, thermo gravimetric and differential thermal analysis, sintering behavior, SEM and AFM were conducted to study its physical and microstructural behavior. The best results indicated at the combination (BaTiO₃ 72 -SrTiO₃ 28) Wt.%, after sintering as composites ceramic fabricated from nano powder.

Key Words: Nano Grains, Composite Ceramic, Sintering, Thermal Analysis, SEM, XRD.

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Introduction

The unique properties of Nano powders like large surface area of quantum effect and formability, make them a good choice for countless application. In other words, materials will be constructed from the bottom up instead of conventional methods, top – down method [1]. The properties and structure of materials has made Nano powders a quickly developing field that has been gaining interests among the public due in part to the possibilities that the technology provides. The basis is the ability to from Nano – sized particles to build materials can be adapted for certain purposes, for example, the electronics filed, like fast and high energy strong capacitors that are smaller and faster. Improving the performance of ceramic capacitors are also being developed for sintering. It's very important for economic aspects. Their potential applications in Nano-electronics, op to

electronic chemical sensors, catalysts, biological medicines[2]. As atypical representative, barium strontium titanate is a kind of useful electronic ceramic material with fine performance and high dielectric constant, especially in the application of sensitive components and high voltage capacitors. moreover, the substitution of barium by strontium in barium titanate can improve the properties such as lowering the temperature of ferroelectric transformation, increasing di electric constant, lowering di electric dissipation and eluting pyro electric coefficient [3], there have been methods developed for preparing barium strontium titanate including convention at mixing and calcination method[4], co-precipitating method synthesis and sol-gel process.

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Many researchers focus on its electrical properties and specially on the combination BST of BaTiO₃ 72 Wt.% as ceramic type [5]. In present investigation wide ranges from barium titanate were used to study its effect on microstructural and thermal behavior.

Experimental

Pure of 99 nm particle size powdered barium titanate and strontium titanate were dry mixed for 18hours,different weight percentage of BaTiO₃ ranging from (68,70,72,74,76)Wt.% were used and pressed in to disc of 2cm diameter and 3cm thickness. Sintering were done for all the combinations in discs form at 1300 °C, under static air for soaking time 3hours. X- ray diffraction was carried out by SHERMADZU XRD – 6000 (Japan), for all the sintered combinations. TG and DTA thermal analysis were examined too at 1400 upon heating at scanning of 10C⁰ /min, under static air by using LINSEIS STA (Germany). Densities were measured by the geometrical method using high accurate electronic balance and high accurate digital micrometer and according to the ASTM C29/C29M-17a, before and after sintering for all prepared

discs, followed by scanning electron microscopy test to study the microstructure using VEGA, TESCAN (Geska Republic). AFM analysis were used to study the sintered discs tomography and for all the BST samples.

Results and Discussion

The XRD pattern of Ba TiO₃ -SrTiO₃ sintered at 1300 C⁰ for 3 hours under static air was presented in figure 1, its evident that the x-ray diffraction pattern is exactly like the barium strontium titanate pattern of cubic crystal structure according to the PCPDFWIN – [PDF NO. 35 – 0734]. When the barium substituted by strontium which will affect the diffraction peaks. Therefore the barium peak of 111 (hkl) at 2 theta = 40 having a tetragonal structure was appeared, while the other peaks 002(hkl) and 200 (hkl), belongs to strontium titanate can't be shown in the pattern. Thus due to the size difference in the atomic weight between barium (larger) and strontium (smaller), which caused the overlapping of 002 and 200 peaks and only the 200 peak can be shown at 2 theta = 46.48 in the pattern[4].

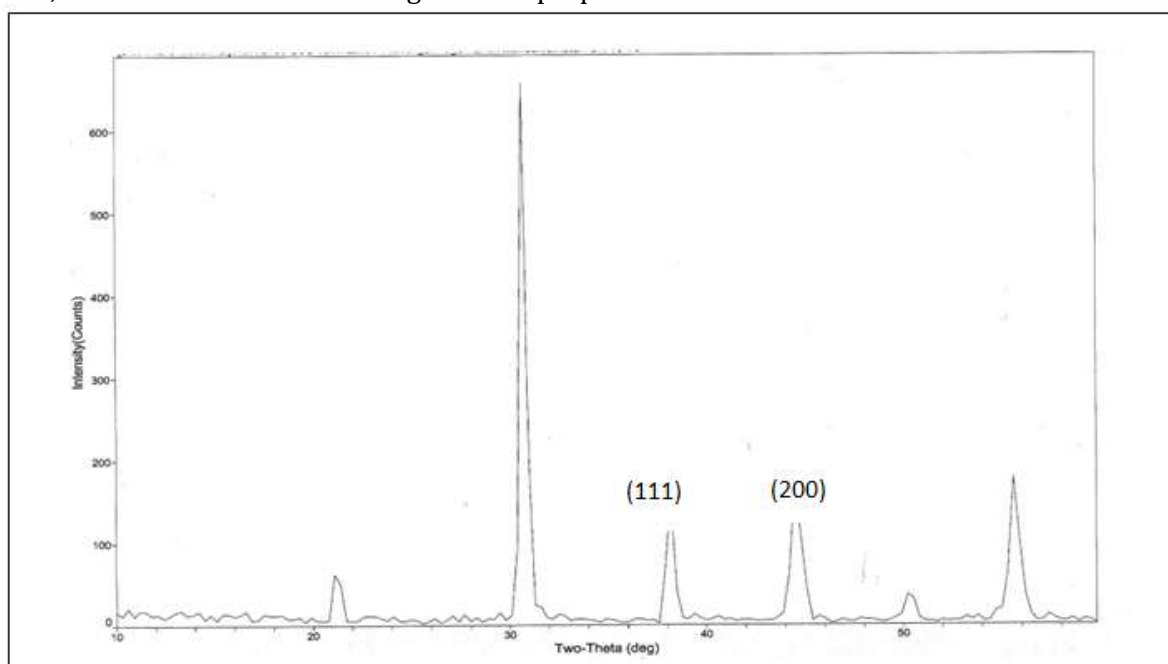


Figure 1. X –ray diffraction pattern for (Ba TiO₃ - SrTiO₃) Wt. % Composites ceramics, sintered at 1300 °C, for 3 hours under static air

The TG -.DTA, profile curves of (BaTiO₃- SrTiO₃) Wt.%,sintered at 1300 °C for 3 hours under static air shown in figure 2. The curves reflected the weight loss of water content and phase transition. From the TG –DT curves, the sintered process could be investigated as the volatilization of water, new

phase formation of barium strontium titanate and finally the decomposition of titanate to titania. The first endotherm weight loss of about (-4mg), appears in the temperature range below 200 °C. This is regarding to the volatilization of water molecules entrapped within the composition. The

second thermal weight loss of about (-22mg) at temperature between (300–600) °C, those are represent both endotherm proses respectively, caused by further decomposition. And solid –solid reaction between different components shows a broad exothermic peak ranging between 563.3 °C (onset point) and, which indicates the formation of barium strontium titanate phase. The crystallization temperature of BTS is found to be at

549.1 °C, its lower than the published [3] at 700 °C. The last thermal decomposition conducted when the temperature is further elevated to about 1000, °C shows a sharp endothermic peak at 962.4 °C (onset point), which is belongs to the decomposition of titanate to titania[4] without any weight loss can be measuring indicating the thermal stability of BST.

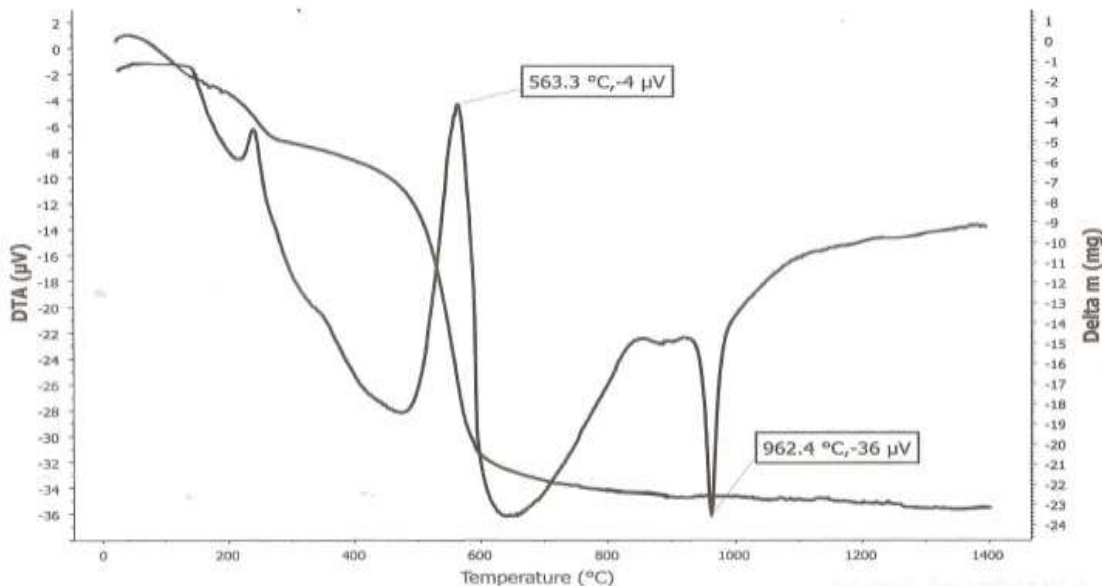


Figure 2. TG – DTA curves (BaTiO₃ -SrTiO₃) Wt.%, composites ceramics sintered at 1300 °C, for 3 hours under static air

Sintered density were calculated for all compacted combinations, which were sintered at 1300 °C under static air. The increasing in sintering was observed at the added weight percentage (68,70 and 72) from BaTiO₃, reached its maximum value of 6.040 gm /cm³, at the combination(BaTiO₃ 72 - SrTiO₃28) wt. %.And then decreased at the (74 and76) Wt.% BaTiO₃, with minimum value of 3.010

gm /cm³. The rapid increasing in sintered density in about 50% was achieved for the combination BaTiO₃ 72 wt. % -SrTiO₃28 Wt.%, sintered at 1300 °C, as shown in figure 3. This is because of the Nano particles create a very high surface to volume ratio can be comprised three to five molecules together [5, 6], which lead to the rapid grain growth with closed porosity.

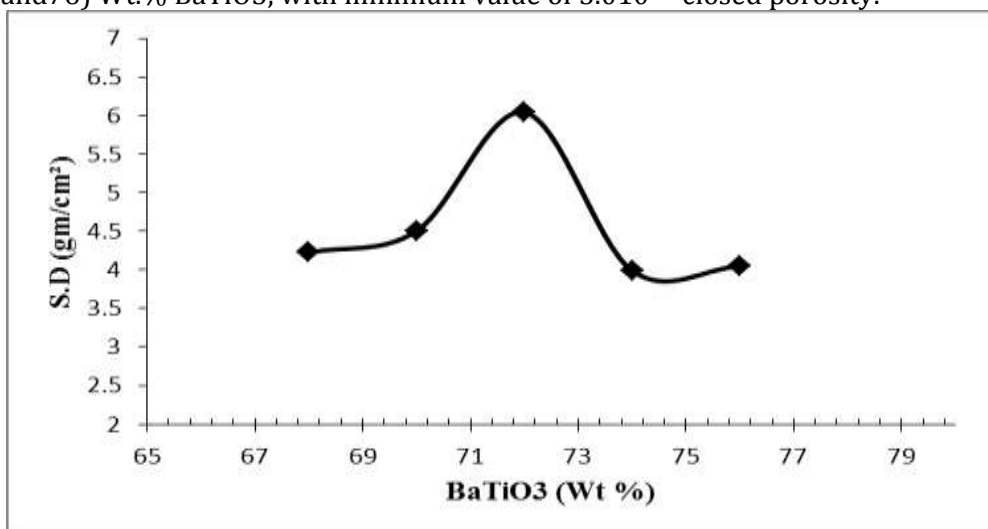


Figure 3. The sintered density of (BaTiO₃-SiTiO₃) Wt.%, composites ceramic sintered at 1300 °C, for 3 hours under static air



The SEM analysis were shown in figure 4(a,b,c) the Combinations (BaTiO₃ - SrTiO₃) wt.% sintered at 1300 °C, for 3hours under static air. The grains contacting with grain size of 10µm and open porosity represent the first stage of sintering [7], figure 3(a) the combination (BaTiO₃ 68 - SrTiO₃ 28) Wt.%, while figure 3(b), the combination BaTiO₃ 72 wt.% - SrTiO₃28 wt.%, we can notes a closed porosity with a wide homogenization,

remarkable microstructure and grain size of 2 µm represent the final stage of sintering [7]. These analysis reflect the consistency results with those obtained from the combinations sintered at 1300. In figure3 (c) the combination (BaTiO₃ 76- SrTiO₃24) wt.%, showing the microstructure with a clear open porosity which reflect the decreasing in the sintered density.

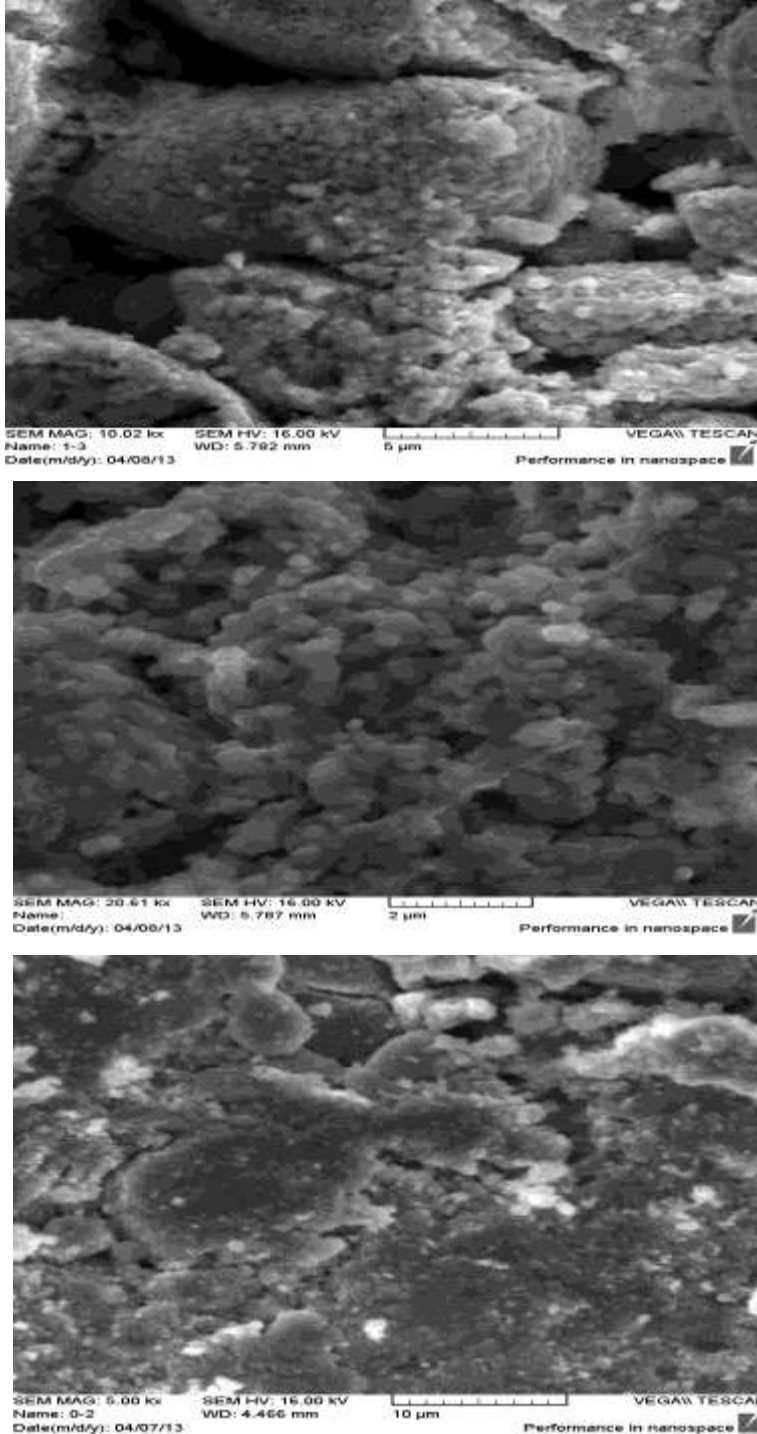


Figure 4. The SEM micrographs of the BST sintered at 1300 °C, for 3 hours under static air, a),(BaTiO₃ 68 -SrTiO₃ 72)Wt.%, b) (BaTiO₃ 72 - SrTiO₃28)Wt.%, c) (BaTiO₃ 76 -SrTiO₃ 24)Wt.%



Conclusion

The fabricated sintered discs of the combinations (BaTiO₃-SrTiO₃) Wt.% sintered at 1300 °C, for 3hours under static air, indicating a unique TG – DTA curves, and XRD pattern for the barium strontium titanate phase, having a maxima sintered density, and uniform microstructure with mass transportation behaviour and closed porosity and highly surface tomography indicated in the combination (BaTiO₃ 72 -SrTiO₃ 28) Wt.%.

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