FABRICATION COMPOSITE CERAMIC CAPACITOR FROM NANO- SCALE POWDER

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Abstract . The aim of present work is the development of fast and high energy storage capacitor , capable of with standing electrical stresses at room temperature X-ray diffraction, thermo gravimetric and differential thermal analysis ,sintering behavior and SEM were conducted . The brake down voltage and capacitance value were measured for all the $BaTio_3$ –$SrTio_3$ sintered discs .

Keywords : Composite ceramic , Sintering , Thermal analysis , SEM , Capacitance values

1. 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 button 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. Its very important for economical 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 .

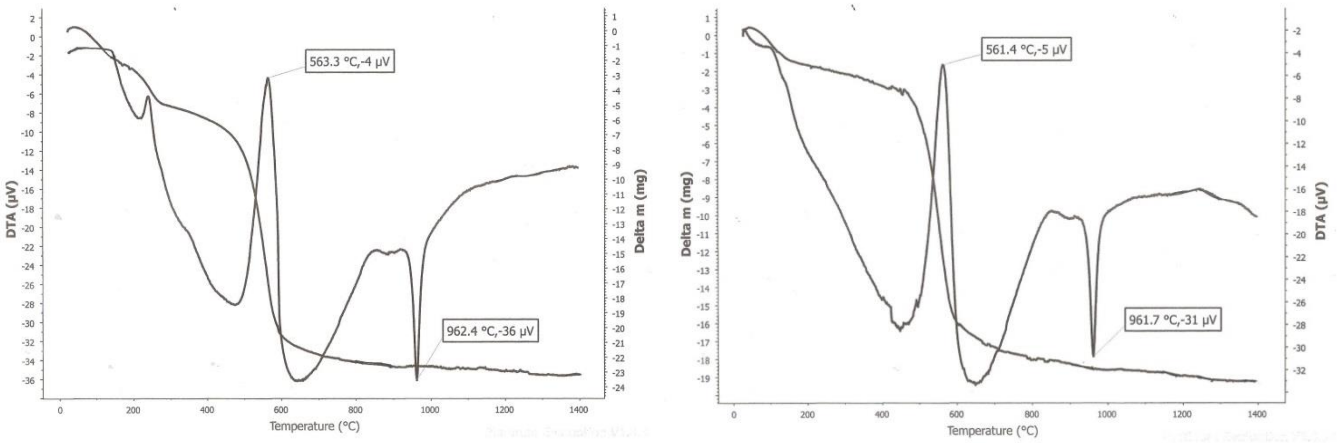
2. EXPEREMENATAL

Pure of 99 nm particle size powdered barium titanate and strontium titanate were dry mixed for 18hours , combination from $BaTiO_3$ 72% - $SrTiO_3$ 28wt% and $BaTiO_3$ 70wt% –$SrTiO_3$ 30 wt.% were pressed in to disc of 2cm diameter and 3cm thickness . Sintering were done for the discs at $1100C^0$ and 1300C0,in static air for socking time 3hours . X- ray diffraction was carried out by SHEMADZU XRD – 6000 (Japan) , for the combination sintered at 1300 C^0 . TG and DTA thermal analysis were examined for both combinations at 1400 C^0 upon heating at scanning of $10C^0$ /min ,under static air by using LINSEIS STA (Germany) . Densities were measured before and after sintering for all discs by using the geometric method, followed by scanning electron microscopy test to study the microstructure for the sintered discs using VEGA ,TESCAN (Geska Republic) . Finally the capacitance value and brake down voltage were conducted for both combinations after sintering process. .

3. RESULTS AND DISCUSSION

The typical TG - .DTA, curves of Ba Tio3- SrTio3 ,sintered at 1300 C^0 for 3 hours under static air shown in figure 1(a,b). 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^0 . This is regarding to the volatilization of water molecules entrapped within the composition, figure 1(a) ,for the Ba $TiO_3$70 wt. % - $SrTiO_3$ 30 wt.% .The second thermal weight loss of about (-22mg) at temperature between (300 – 600) C^0, while the weight loss of about (-17 mg) was conducted in figure 1(b) ,for the Ba $TiO_3$72 wt. % - $SrTiO_3$ 28 wt.% , that difference in the amount of weight loss between the both combinations are because of the difference in weight percept used . Those are represent both endotherm and exotherm proses respectively , caused by further decomposition and solid –solid reaction between different components shows a broad exothermic peak ranging between (563.3 – 561.4) C^0 (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^0 ,its lower than the published [3] at 700 C^0 . The last thermal decomposition conducted when the temperature is further elevated to about 1000 C^0 , shows a sharp endothermic peak ranging between (962.4 – 961.7). 2 C^0 (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 1(a,b) : TG – DTA curves for a) Ba $TiO_3$70 wt. % - $SrTiO_3$ 30 wt.% , b) Ba $TiO_3$72 wt. % - $SrTiO_3$ 28 wt.% ,sintered at 1300 C^0 for 3 hours under static air



(a) (b)

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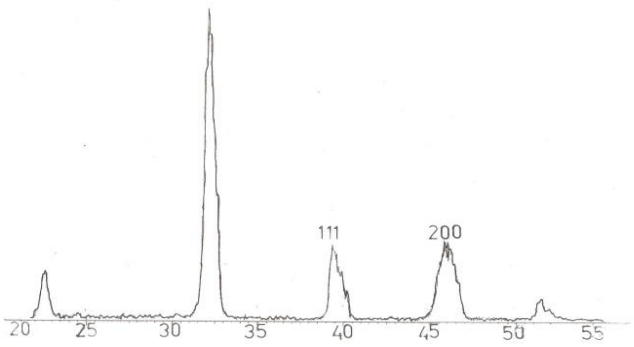
The XRD pattern of Ba TiO_3. -$SrTiO_3$ sintered at 1300 C^0 for 3 hours under static air was presented in figure 2 , its evident that the x-ray diffraction pattern is exactly like the barium strontium titanate pattern of cubic crystal structure according to the PCPDFWIN – [PDF NO. 35 – 0734] . When 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].

Figure2: X –ray diffraction pattern for Ba $TiO_3$72 wt. % - $SrTiO_3$ 28 wt.% , ,sintered at 1300 C^0 for 3 hours under static air



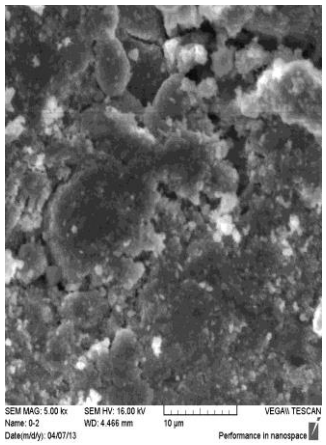
Sintered density were calculated for both compacted combinations ,which were sintered at 1100 C^0 and 1300 C^0 .The resulting data showing a lower value for both combinations sintered at 1100C^0 ,with a slight increasing indicated with the combination $BaTiO_3$ 70 wt.% -$SrTiO_3$30wt % . while a rapid increasing in sintered density in about 90% was achieved for the combination $BaTiO_3$ 72 wt.% -$SrTiO_3$28 wt% ,sintered at 1300C^0 as shown in table 1. 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.

TABLE 1. sintered density for $BaTiO_3$ - $SrTiO_3$ sintered at 1100C^0 and 1300C^0

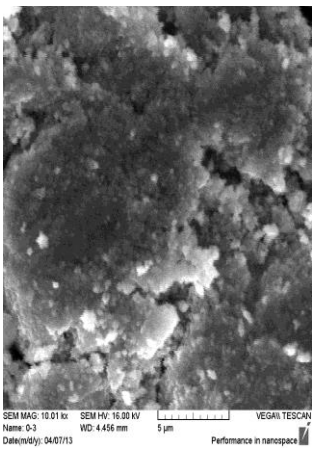
Sample	S.D gm./cm^3 1100 C^0	S.D gm./cm^3 1300C^0
$BaTiO_3$ 70 wt. % - $SrTiO_3$30 wt. %	3.502	4.504
$BaTiO_3$ 72 wt. % - $SrTiO_3$28 wt. %	3.905	6.040

The SEM analysis were shown in figure 3and 4 respectively . From figure 3 , the Combination BaTio3 70 wt.% - SrTio330 wt.% sintered at 1100 C^0 for 3hours under static air reflecting the grain contacting with grain size of 10µm and open porosity represent the first stage of sintering [7], figure 3(a) while the combination BaTio3 72 wt.% - SrTio328 wt.% ,figure 3(b) shows little open porosity and with grin size of 5µm . Figure 4 , the combinations sintered at 1300 C^0 for 3 hours under static air. In figure4(a) the combination $BaTiO_3$ 70 wt.% - $SrTiO_3$30 wt.%, showing the microstructure homogeneity with a clear grain growth of 5µm, and closed porosity represent the final stage of sintering [7] .From the combination $BaTiO_3$ 72 wt.% - $SrTiO_3$28 wt.% ,figure4 (b) we can notes a closed porosity with a wide homogenization, remarkable microstructure and grain size of 2 µm .These analysis reflect the consistency results with those obtained from sintered densities .

Figure 3: SEM micrographs for the a) $BaTiO_3$ 70 wt.% - $SrTiO_3$30 and b) $BaTiO_3$ 72 wt.% - $SrTiO_3$28 ,sintered at1100 C^0 for 3hours under static air .

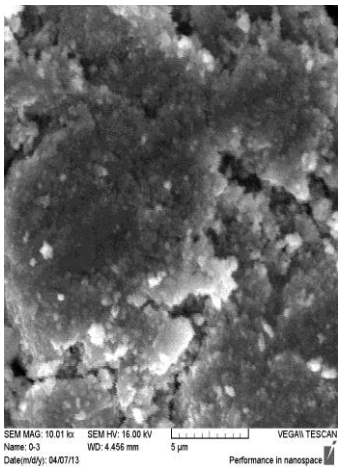


(a)

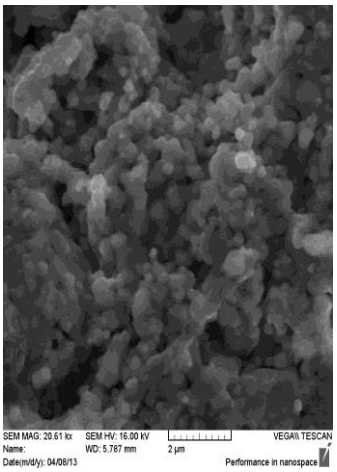


(b)

Figure 4: SEM micrographs for the a) $BaTiO_3$ 70 wt.% - $SrTiO_3$30 and b) $BaTiO_3$ 72 wt.% - $SrTiO_3$28 ,sintered at1300 C^0 for 3hours under static air .



(a)



(b)

Brake down voltage , capacitance values and the dielectric constant measurements ,also enhance the densification behaviour as shown respectively and indicated a maximum brake down voltage 3.081 Kv/mm , maximum capacitance value of 437 P F and maximum dielectric constant value of 4030 for the combination $BaTiO_3$ 72wt.% -$SrTiO_3$28wt% sintered at 1300C^0 . This is because of the rapid grain growth during the sintering stages [8,9], which were strongly drive both the strength ,energy storage and dielectric to be increased rapidly , and the solid solution formed of crystal grains constituting the dielectric ceramic. Herein, when crystal grains mainly composed of barium titanate has a cubic crystal structure. Ferro electricity caused by a tetragonal system is suppressed and the crystal grains mainly exhibit Para electricity . As a result, a dielectric ceramic can be provided that has a high relative dielectric constant, and stable temperature [10].

TABLE 2. brake down voltage for $BrTiO_3$ - $SrTiO_3$ sintered at $1100C^0$ and $1300C^0$ for 3hours under static air

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Sample	Sintering temperature $1100C^0$ Brake down voltage kv/mm	Sintering temperature$1300C^0$ Brake down voltage kv/mm
$BaTiO_3$ 70 wt.% - $SrTiO_3$30 wt. %	1.840	2.020
$BaTiO_3$ 72 wt.% - $SrTiO_3$28 wt. %	2.440	3.081

TABLE 3. capacitance values for $BaTiO_3$ - $SrTiO_3$ sintered at $1100C^0$ and $1300C^0$ for 3hours under static air.

Sample	Sintering temperature 1100 C^0 Capacitance PF	Sintering temperature $1300C^0$ Capacitance PF
$BaTiO_3$ 70 wt.% - $SrTiO_3$30 wt. %	26	398
$BaTiO_3$ 72 wt.% - $SrTiO_3$28 wt. %	400	437

TABLE4. Di Electric constant values for $BaTiO_3$ - $SrTiO_3$ sintered at $1100C^0$ and $1300C^0$ for 3hours under static air.

Sample	Sintering temperature 1100 C0 Di Electric constant	Sintering temperature 1300C0 Di Electric constant
BaTiO3 70 wt.% - SrTiO330 wt. %	2398	3670
BaTiO3 72 wt.% - SrTiO328 wt. %	3689	4030

4. CONCLUSION

The fabricated sintered discs of the combination $BaTiO_3$ 28 wt.% -$SrTiO_3$72wt% sintered at $1300C^0$ 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 ,brake down voltage, remarkable capacitance value and dielectric constant .

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