Fabrication Composite Ceramic Capacitor from Nano - Scale Powder

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Abstract. Barium strontium titanate compact were prepared from nano particles .Sintering were conducted at 1100 C⁰ and 1300 C⁰ respectively under static air. X-ray diffraction, pattern analysis shows the BST phase present ,differential thermal analysis indicating the BST formation with the decomposition of titanate to titania. SEM were done to study the sintering behavior of resulting combination BST.

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 . Improving the performance of ceramic capacitors are also being developed for sintering. Their potential applications in Nano-electronics, chemical sensors ,catalysts ,biological medicines (2). As a typical 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).

EXPEREMENATAL

Pure of 99 nm particle size powdered barium titanate and strontium titanate were dry mixed for 18hours , combination from BaTiO₃ 72% - SrTiO₃ 28wt% and BaTiO₃ 70wt% –SrTiO₃ 30 wt.% were pressed in to disc of 2cm diameter and 3cm thickness . Sintering were done for the discs at 1100C⁰ and 1300C⁰,in static air at 10 C⁰ /min for socking time 3hours . X- ray diffraction was carried out by SHEMAZDU XRD – 6000 (Japan). DTA thermal analysis were examined for both combinations at 1400 C⁰ upon heating at scanning of 10C⁰ /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) ..

RESULTS AND DISCUSSION

The typical DTA, curve of Ba TiO₃- SrTiO₃ ,sintered at 1300 C⁰ for 3 hours under static air shown in figure 1(a,b) . The curves reflected the phase transition . From the 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 endothermic peak ,appears in the
temperature range below 200 C°. This is regarding to the volatilization of water molecules entrapped within the composition, figure 1(a), for the Ba TiO₃ 70 wt. % - SrTiO₃ 30 wt.%. At temperature between (300 – 600) C°, another endothermic peak was conducted in figure 1(b). Those are represent both endothermic and exothermic peaks respectively, caused by further decomposition and solid–solid reaction between different components shows a broad exothermic peak ranging between (563.3 – 561.4) 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 ranging between (962.4 – 961.7) C° (onset point), which is belongs to the decomposition of titanate to titania (4).

Figure 1(a,b) : DTA curves for a) Ba TiO₃ 70 wt. % - SrTiO₃ 30 wt.% , b) Ba TiO₃ 72 wt. % - SrTiO₃ 28 wt.% , sintered at 1300 C° for 3 hours under static air .

The XRD pattern of Ba TiO₃ -SrTiO₃ sintered at 1300 C° 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).
Figure 2: X-ray diffraction pattern for BaTiO$_3$ 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$^0$.

<table>
<thead>
<tr>
<th>Sample</th>
<th>S.D gm./cm$^3$ 1100 C$^0$</th>
<th>S.D gm./cm$^3$ 1300C$^0$</th>
</tr>
</thead>
<tbody>
<tr>
<td>BaTiO$_3$ 70 wt% - SrTiO$_3$30</td>
<td>3.502</td>
<td>4.504</td>
</tr>
<tr>
<td>BaTiO$_3$ 72 wt% - SrTiO$_3$28</td>
<td>3.905</td>
<td>6.040</td>
</tr>
</tbody>
</table>

The SEM analysis were shown in figure 3 and 4 respectively. From figure 3, the combination BaTiO$_3$ 70 wt.% - SrTiO$_3$30 wt.% sintered at 1100 C$^0$ for 3 hours under static air reflecting the grain contacting with grain size of 10µm and open porosity represent the first stage of sintering (5), figure 3(a) while the combination BaTiO$_3$ 72 wt.% - SrTiO$_3$28 wt.% figure 3(b) shows little open porosity and with grain size of 5µm. Figure 4, the combinations sintered at 1300 C$^0$ for 3 hours under static air. In figure 4(a) the combination BaTiO$_3$ 70 wt.% - SrTiO$_3$30 wt.%, showing the microstructure homogeneity with a clear grain growth of 5µm, and closed porosity represent the final stage of sintering (5). From the combination BaTiO$_3$ 72 wt.% - SrTiO$_3$28 wt.%, figure 4(b) we can notes a closed porosity with a wide homogenization, remarkable microstructure and grain size of 2µm. 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$28 sintered at 1100 C$^0$ for 3 hours under static air.
CONCLUSION

The fabricated sintered discs of the combination BaTiO$_3$ 72 wt.% - SrTiO$_3$28 wt.% sintered at 1300°C for 3 hours under static air, indicating a unique DTA curve, XRD pattern for the barium strontium titanate phase, having a maximum sintered density. SEM results show a closed porosity with a wide homogenization and remarkable microstructure.

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