Malaysian Journal of Science **21A**: 109-111 (2002)

Formation of barium strontium titanate powder by solid state reaction using different calcination temperatures

Teoh Wah Tzu, Ahmad Fauzi Mohd Noor and **Zainal Arifin Ahmad**

School of Materials & Mineral Resources Engineering, Universiti Sains Malaysia, 14300 Nibong Tebal, Pulau pinang, Malaysia.

ABSTRACT The unique electrical properties of large permittivity in Barium Strontium Titanate have been widely used to make capacitors; it can be produced by solid state reaction. In this study, the mixture of Barium Carbonate, Strontium Carbonate and Titanium Dioxide was calcined at 500°C, 1000°C, 1100°C, 1150°C, 1200°C, 1250°C and 1300°C. The results of the phases change in each stage were investigated via X-Ray Diffraction. The results show that the formation of Barium Strontium Titanate started at 1100°C with the presence of other phases. The mixture is fully reacted to form Barium Strontium Titanate at 1150°C. Only Barium Strontium Titanate was formed as the calcination temperature was set higher.

(Electrical properties, Barium, Strontium, XRD)

INTRODUCTION

One of the common methods to produce Barium Strontium Titanate (BST) is by solid state reaction of Barium Carbonate ($BaCO_3$), Strontium Carbonate ($SrCO_3$) and Titanium Dioxide (TiO_2) mixture. Calcination temperature and time are the key parameters that would determine the characteristic of BST produced. The optimum calcination temperature and time must be obtained to ensure the mixture completely formed BST through solid state reaction. If the calcination temperature and time is set too low, the mixture will only be partially reacted [1-3]. This paper report the phases detected in the mixture as calcination temperature changed.

MATERIALS & METHOD

The raw chemicals used to prepare BST are $BaCO_3$ (Fluka), $SrCO_3$ (Fluka) and TiO_2 (BDH). The mixture prepared is to form $Ba_{1-x}Sr_xTiO_3$ with x = 0.3, this compound is referred as $Ba_{0.7}Sr_{0.3}TiO_3$ hereafter. The raw chemicals were weighed out in stoichiometric proportion and mixed by grinding lightly in a mortar and pestle [4]. Eight batches of mixture were prepared and calcined at 500°C, 1000°C, 1100°C, 1150°C, 1200°C and 1300°C for soaking time of 3 hours, with one remain uncalcined for comparison purposes. All the calcination processes were carried out using the same furnace to minimize variation. After calcination, all the samples were ground to fine powder and send for X-Ray diffraction (XRD), using Philips Model PW1710, with Copper (Cu) anode. The diffractograms obtained were matched against the Powder Diffraction File (PDF) from International Centre for Diffraction Data.

RESULTS AND DISCUSSION

The diffractograms obtained were partially superimposed with one to another as shown in Fig. 1. Major peaks in the diffractograms were labelled with the phase detected. However, the diffractograms that have similar patterns will only be labelled once.

In general, Fig. 1 shows that a change in diffractogram is detected as the calcination temperature increased. This change is due to the presence of other phases in the samples before $Ba_{0.7}Sr_{0.3}TiO_3$ is completely formed.

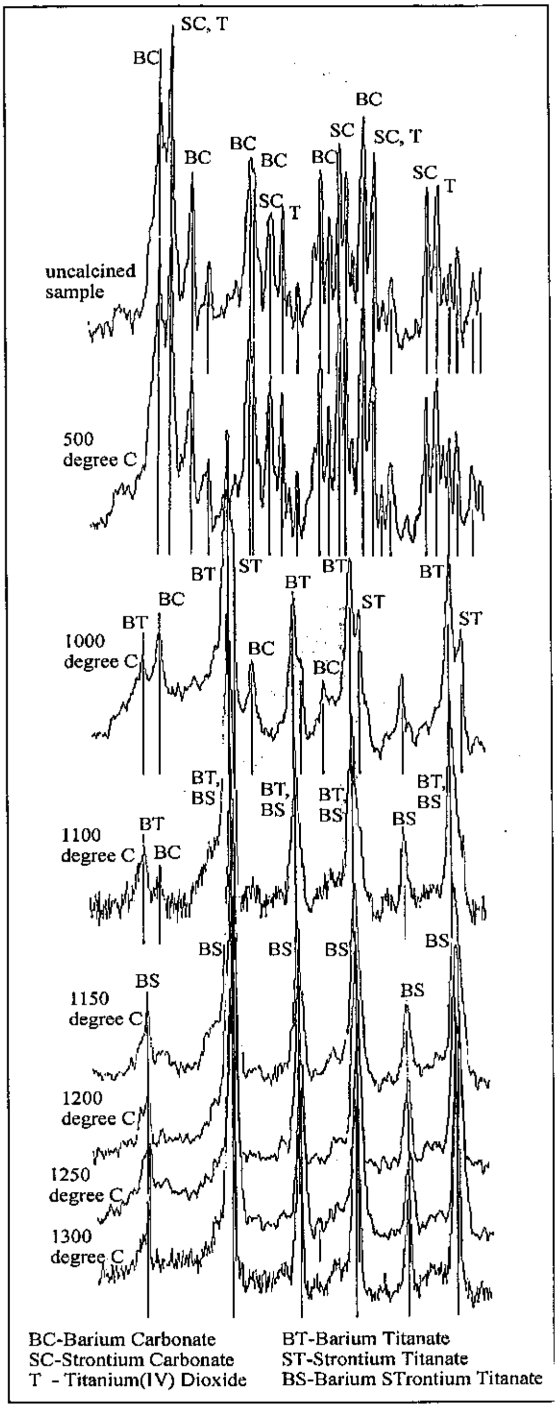

Figure 1. Diffractograms at Different Calcination Temperature

XRD was performed on the uncalcined mixture of $BaCO_3$, $SrCO_3$ and TiO_2, and the diffractograms obtained confirmed the presence of the raw chemicals used. The diffractograms for the 500°C calcined sample has similar pattern to the uncalcined sample. None of new phases detected as the calcination temperature is not high enough for solid state reaction to happen [5].

In the 1000°C calcined sample, a change in diffractograms was observed compared to the 500°C calcined sample and the presence of Barium Titanate ($BaTiO_3$), Strontium Titanate ($SrTiO_3$) and $BaCO_3$ was detected. On the other hand, $SrCO_3$ and TiO_2 were no longer detected in the 1000°C calcined sample. At this temperature, $BaCO_3$ & $SrCO_3$ start to decompose and reacted with TiO_2 to form $BaTiO_3$ and $SrTiO_3$ [6].

The sample calcined at 1100°C indicated the presence of $BaTiO_3$, $Ba_{0.7}Sr_{0.3}TiO_3$ and $BaCO_3$. The formation of $Ba_{0.7}Sr_{0.3}TiO_3$ took place at this temperature. However existence of $BaTiO_3$ and $BaCO_3$ in the sample indicated that the reaction to form $Ba_{0.7}Sr_{0.3}TiO_3$ is not completed.

At 1150°C, the diffractogram shows only the presence of $Ba_{0.7}Sr_{0.3}TiO_3$ (PDF No. 44-93), indicating the completion of the solid state reaction in the mixture [6]. When the calcination temperatures were increased further to 1200°C, 1250°C and 1300°C, the diffractograms remained similar to the 1150°C. No further reaction took place after 1150°C.

The summary of each phases detected at different calcination temperature is tabulated in Table 1.

Table 1: Phases Detected at Different Calcination Temperature

	Phases
uncalcined	$BaCO_3$, $SrCO_3$ & TiO_2
500°C	$BaCO_3$, $SrCO_3$ & TiO_2
1000°C	$BaTiO_3$, $SrTiO_3$ & $BaCO_3$
1100°C	$BaTiO_3$, $Ba_{0.7}Sr_{0.3}TiO_3$ & $BaCO_3$
1150°C	$Ba_{0.7}Sr_{0.3}TiO_3$
1200°C	$Ba_{0.7}Sr_{0.3}TiO_3$
1250°C	$Ba_{0.7}Sr_{0.3}TiO_3$
1300°C	$Ba_{0.7}Sr_{0.3}TiO_3$

CONCLUSION

A systematic experiment had been carried out to study the impact of calcination temperature on $Ba_{1-x}Sr_xTiO_3$ formed through solid state reaction of $BaCO_3$, $SrCO_3$ and TiO_2 mixture. By performing XRD on the samples, the results show that complete formation of $Ba_{0.7}Sr_{0.3}TiO_3$ happened at 1150°C and above, and several different phases are detected if the calcination is below 1150°C.

Acknowledgement The authors wish to thank Mr. Shahrul, Mr. Razak and Madam Fong Lee Lee for their technical support and useful discussions.

REFERENCES

1. Hilton, A.W. and Ricketts, B.W. 1996, *J.Phys. D: Appl. Phys.*, **29**: 1321.
2. Shaw, T.M., Suo, Z., Huang, M., Liniger, E., Laibowitz, R.B. and Baniecki, J.D. 1999, *Appl. Phys. Lett.*, **75**: 2129.
3. Seung-Eek Park, Kelley Markowski, Shoko Yoshikawa, and L. Eric Cross, 1997, *J. Am. Ceram. Soc.*, **80**: 407.
4. Long Wu, Yih-Chien Chen, Cheng-Liang Huang, Yi-Ping Chou, and Yuan-Tu Tsai, 2000, *J. Am. Ceram. Soc.*, **83**: 1713.
5. Moulson, A.J. and Herbert, J.M. 1990, *Electroceramics*, London: Chapman & Hall
6. James S. Reed, 1987, *Introduction to the Principles of Ceramic Processing*, Singapore: John Wiley & Sons.