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SINGLE STEP COMBUSTION SYNTHESIS TO PREPARE BISMUTH TITANATE CERAMICS FOR POTENTIAL APPLICATIONS

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ABSTRACT

Bismuth titanate, $Bi_4Ti_3O_{12}$ or BIT was systematically prepared by a single step combustion synthesis, and subsequently, sintered at different temperatures. In this work, the formation of corresponding phase with better degree of crystallinity was strongly affected by NH_4OH and hydrolysis temperature. It was supported by XRD and Raman. Based on TG-DTA, the reaction was completely decomposed at around 500°C with larger exothermic peaks in the range of 250 – 300°C. The HRTEM micrograph showed clearly the agglomeration particles with irregular shape. Determination on sintering temperature was clearly showed by dilatometry curve whereby it was in the range of 800°C and above. The maximum relative density (86%) was achieved at 1000°C. The microstructure of BIT was randomly oriented, thus leading to a decrease in relative density. The conductivity of BIT was relatively high at elevated temperature, which can be explained in terms of larger grain size. Based on electrical measurement, the dielectric constant, dielectric loss, conductivity, resistivity, remanent polarization and coercive field of optimized BIT were recorded to be around 130 - 150, 0.00552 - 0.05212, $3.78 \times 10^{-8} - 3.97 \times 10^{-5}$ S/m, $2.52 \times 10^{4} - 2.64 \times 10^{7}$ Ω/m, 7 μC/cm^2 and 30 kV/cm, respectively. In addition, the Curie temperature was approximately at 675°C.

Keywords: $Bi_4Ti_3O_{12}$, combustion, NH_4OH, electrical properties

INTRODUCTION

Recently, ferroelectric materials have been intensively studied as candidates for use in a wide range of potential applications [1]. Bismuth Layer-Structured Ferroelectrics (BLSFs) consist of a layer structure of Bi_2O_2 and a pseudo-perovskite $Bi_2Ti_3O_{10}$ along the *c*-axis; which is different from a simple perovskite structure. $Bi_4Ti_3O_{12}$ is a BLSFs and is known to have a high Curie temperature (675°C), a large remanent polarization (P_r = 5 – 6.5 μC/cm^2), a low coercive field (E_c = ~50 kV/cm), and a low processing temperature (750 – 800°C); giving this material a high potential in real applications [2]. Furthermore, BIT is very familiar as a lead-free ceramic material. For the above reason, BIT was investigated by several other researchers. BIT is usually prepared in many ways. Among them, soft combustion has been chosen and employed in this study, due to its simple, more economical and rapid preparation process. Hence, an attempt has been made to investigate the possibility of firing pure BIT at low temperature assisted by other possible parameter studies. Besides that, the characterization on other properties i.e. thermal behaviour, particle morphology, density and electrical properties were also included.

EXPERIMENTAL PROCEDURE

Aqueous solution of Bi^{3+}, Ti^{4+} and citric acid were constantly stirred for 24 hour at 60°C. The prepared solution was also added with NH_4OH (29% solution) to obtain pH 7. The temperature was raised to 80°C for a dehydration process, prior to the combustion process. The combustion reaction lasted for

less than 5 minutes and produced a dry yellowish powder. The as-burnt powders were unixially pressed and sintered at different temperatures. The characterization on the as-burnt powders and sintered samples were carried out, i.e. XRD, Raman, TG-DTA, HRTEM, SEM, and several electrical measurements.

RESULTS AND DISCUSSION

In order to determine the important factor in combustion route, the XRD patterns of as-burnt powder prepared with and without NH_4OH were analyzed. The result was obviously different whereby better degree of crystallinity was observed from the sample with NH_4OH addition. This implies that the combustion reaction is more beneficial in formation of pure phase BIT at low temperature assisted by extra fuel agent. Thus, NH_4OH has plays double functions i.e. fuel agent and chelating agent. Based on TG-DTA curve, the decomposition temperature was approximately around 500°C, which might be considered as combustion temperature. The as-burnt powder was calcined at 500°C as to get more insight in phase formation. As seen, there is no other phase formed in XRD pattern. This result is in agreement with TG-DTA analysis. Agglomeration of the as-burnt BIT particles can be seen in HRTEM micrograph, as shown in Fig. 1. The powder was found to be crystalline from the selected area diffraction (SAD) pattern inserted in the HRTEM micrograph. This confirms the presence crystalline particles. In order to discuss the characteristic of ferroelectric properties, the compacted powders were sintered at different temperature whereby the optimized temperature was achieved at 1000°C. As can be seen in Fig. 2, the hysteresis loop of BIT was relatively good with electrical field. The remanent polarization (P_r) and coercive field (E_c) were approximately around 7μC/cm^2 and 30kV/cm, respectively.

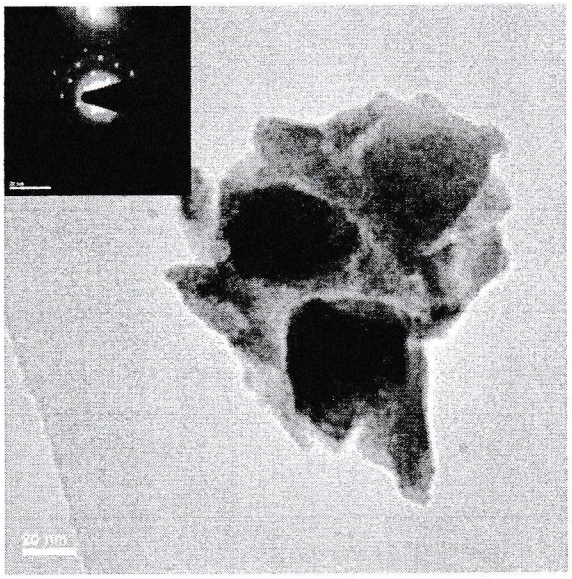

Figure 1: HRTEM image of BIT powder along with SAD pattern.

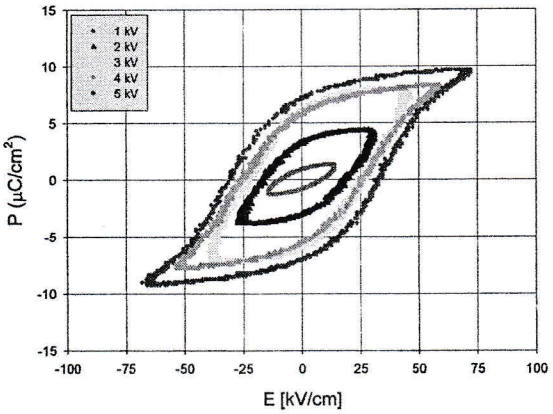

Figure 2: P-E hysteresis loops of BIT as a function of electrical field.

CONCLUSIONS

Single step combustion synthesis was successfully used to produce BIT powders with better degree of crystallinity. Several electrical measurements were also performed.

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