DTA-TG Analysis of $Gd_{0.95}La_{0.05}Ba_{1.95}Sr_{0.05}Cu_3O_y$ Compounds

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Abstrak – *The sintering temperature is played a vital role in the evolution of phase structure, microstructure, and the properties of the superconductor. In this study, the $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ phase compound has been synthesized by the wet method using HNO3 as a solvent. The samples were divided into two groups. The first sample was calcined at 400 °C for 2 hours + 500 °C for 2 hours + 600 °C for 6 hours. The second sample treated by the same process and then continued by heating at 900 °C for 15 minutes. The effect of the calcination temperature for the synthesis of $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ bulks was investigated using the DTA-TG method. The results showed that the optimum reaction temperature for the formation of $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ phase was 938 °C. The additional heating temperature e.g. 900 °C for 15 minutes on the calcination process can reduce the optimum formation temperature of $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ compounds by 20 °C. The peritectic melting reaction temperatures of the sample without the addition of heating and with the addition of heating at temperature 900 °C for 15 minutes are 1032°C and 1035°C, respectively. The melting temperatures of both samples are 1164 °C and 1200 °C.*

Keywords: *DTA-TG method, calcination temperature, sintering temperature, $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ compounds, wet method*

1. Introduction

Thermal stability of cuprate superconductor ceramic compounds, $RE_1Ba_2Cu_3O_{7-\delta}$ (REBCO) (RE means "rare earth"), is limited by sintering temperature Ts and incongruent melting at Tm [1]. Knowledge of Ts and Tm is crucial for growing bulk superconductors from the melt. Melting temperature Tm constitutes an essential input to construct the ternary REO-BaO-$CuO_{7-\delta}$ phase diagram and so to control nucleation and growth phenomena for epitaxial thin film and coated conductor preparation.

Among REBCO high-temperature superconductors, $GdBa_2Cu_3O_{7-\delta}$ (GBCO-123) is the extensively studied because of it's the high critical temperature of 94 K [2] and because, when prepared in the form of thin film or coated conductor, it can carry high electric currents at high magnetic fields seems a good candidate to compare with YBCO. However, it still needs to be improved by its performance at high temperatures and high magnetic fields. For this reason, in this study substitution was made in one of its constituent cations, which substituted a small portion of Gd with La and Ba with Sr simultaneously on $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ compound. Because of the change of cation, it is possible that the change in reaction of the $GdBa_2Cu_3O_{7-\delta}$ phase formation. Therefore, it is necessary to examine the sintering temperature for the formation of these compounds and their melting temperatures.

2. Experiment Method

In this study, the synthesis process of $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ was carried out by the wet mixing method [3]. High-purity powder of metal-oxide Gd_2O_3, La_2O_3, BaO, CuO was used as starting materials. The stoichiometric amounts of each metal-oxide with ratio Gd:La:Ba:Cu=0.9:0.1:1.95:0.05:3.00 in 6 gram were mixed in HNO_3 solution and stirred on a magnetic stirrer with spin of 400 rpm, the suspension blue-white was obtained. The blue-white suspension was stirred for 24 hours. Then, while stirring the suspension was heated at temperature 200 °C for 2.5 hours. A vigorous reaction with release NO_2 gas occurred, followed by the appearance of a blue-green viscous gel was formed. Without stirred the viscous gel was heated at 250 °C until a black crust was obtained. The black crust was crushed in a mortar, then was calcinated i.e. heated at temperature 400 °C for 2 hours, next at temperature 500°C for 2 hours and finally at temperature 600 °C for 6 hours in a furnace. The resulting was crushed and one of the samples

was given heating at 900 °C for 15 minutes. This final powder as a precursor is subjected to high-temperature heat treatments (sintering) for production of $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ compound.

To studies the thermal properties, the sample has been characterized by using of DTA-TG of STA 1600C. The precursor sample was heated at a maximum temperature of 1203 °C with an air atmosphere with flow velocity 0.5/minute and at a rate of 600 °C/hours.

3. Results and Discussion

Figure 1 is the result DTA-TGA measurement for $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ precursor without heating at 900 °C. From Figure 1, the TGA curve at temperature between 168-1200 °C shows a decrease in the total mass of 26%. At temperatures between 168-668 °C show a mass decrease of 13%. From the DTA curve there was a complex reaction in which there was evaporation of nitrates and decomposition of metal-nitrate salts. At temperatures between 668-954 °C a mass decrease of 10% occurred. From the DTA curve, the peak at 938 °C is observed, according some researchers have previously shown that the $Gd_1Ba_2Cu_3O_{7-\delta}$ phase can form at temperatures between 850-950 °C [4-6], therefore we conclude that $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ phase can be formed at temperature 938 °C. On the TG curve, a 3% mass decrease is observed at temperatures between 954-1200 °C, whereas on the DTA curve the peak at 1032 °C was observed.

The GBCO-123 family superconductor has a peritectic melting temperature at temperatures between 980-1090 °C [7, 8, 9]. Prado *et al* also reported that the endothermic DTA peaks under pure oxygen corresponding to the peritectic melting reaction which takes place at 1073 °C [10] and the actual peritectic temperature of GdBCO bulk superconductors, i.e. 1030 °C [11]. Therefore, we suspect a temperature at 1032 °C is a peritectic melting reaction temperature of $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ phase. Furthermore, the DTA curve drops sharply at temperature 1164 °C, we suspect that at this temperature a complete melting of the $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ phase has occurred.

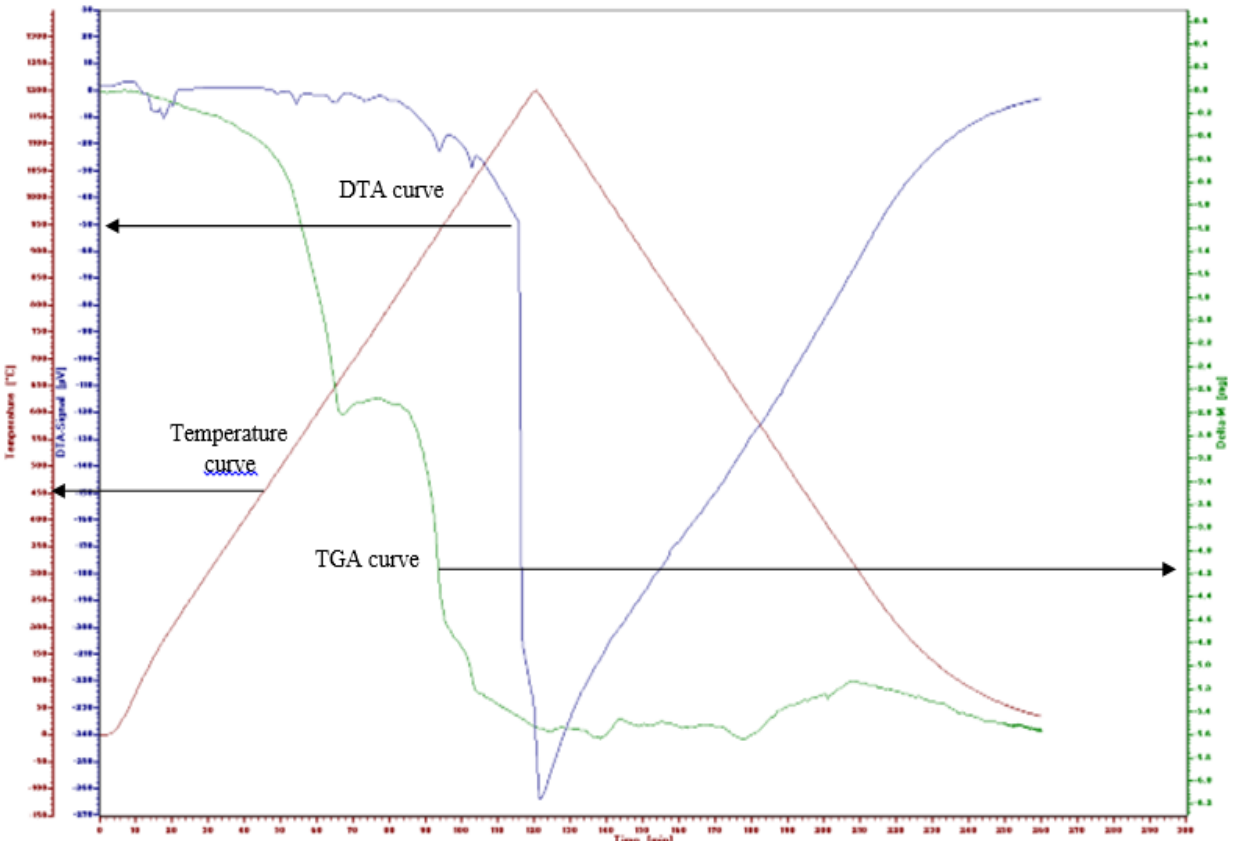

Figure 1. DTA-TG measurement result of $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ precursor without heating at 900 °C**.** Initial of sample is 21.3 mg.

The result DTA-TGA measurement of the $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ precursor with heating at 900 °C for 0.25 hours as of Figure 2. From Figure 2, the TGA curve between 168-1200 °C shows a decrease

in the total mass of 23%. At temperature between 2 °C to 777 °C there was a mass reduction of 12%, the DTA curve showed a widen peak with a peak at 285 °C and a small peak at 750 °C, it is we suspect a complex reaction in which nitrate evaporation and decomposition of metal-nitrate salts occurred. At temperatures between 777 °C to 962 °C there was a mass decrease of 9%. From the DTA curve, the peak at 918 °C was observed. According some researchers have previously shown that the $Gd_1Ba_2Cu_3O_{7-\delta}$ phase can form at temperatures between 900-950 °C [4-6], therefore we suspect that the $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ phase can be formed at optimum temperature 918 °C. On the TG curve, 2% mass decrease is observed too at temperatures between 962-1200 °C, whereas on the DTA curve the peak at 1035 °C was observed. We suspect it is the peritectic melting reaction temperature of the $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$. Furthermore, the DTA curve drops sharply, we suspect that the melt complete of the $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ phase occurring at temperatures above 1200 °C.

From the above explanation it can be concluded that there is a difference reaction temperature for formation of the $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ phase. For samples calcined without heating and by heating at 900 °C i.e. at 938 °C and 918 °C respectively.

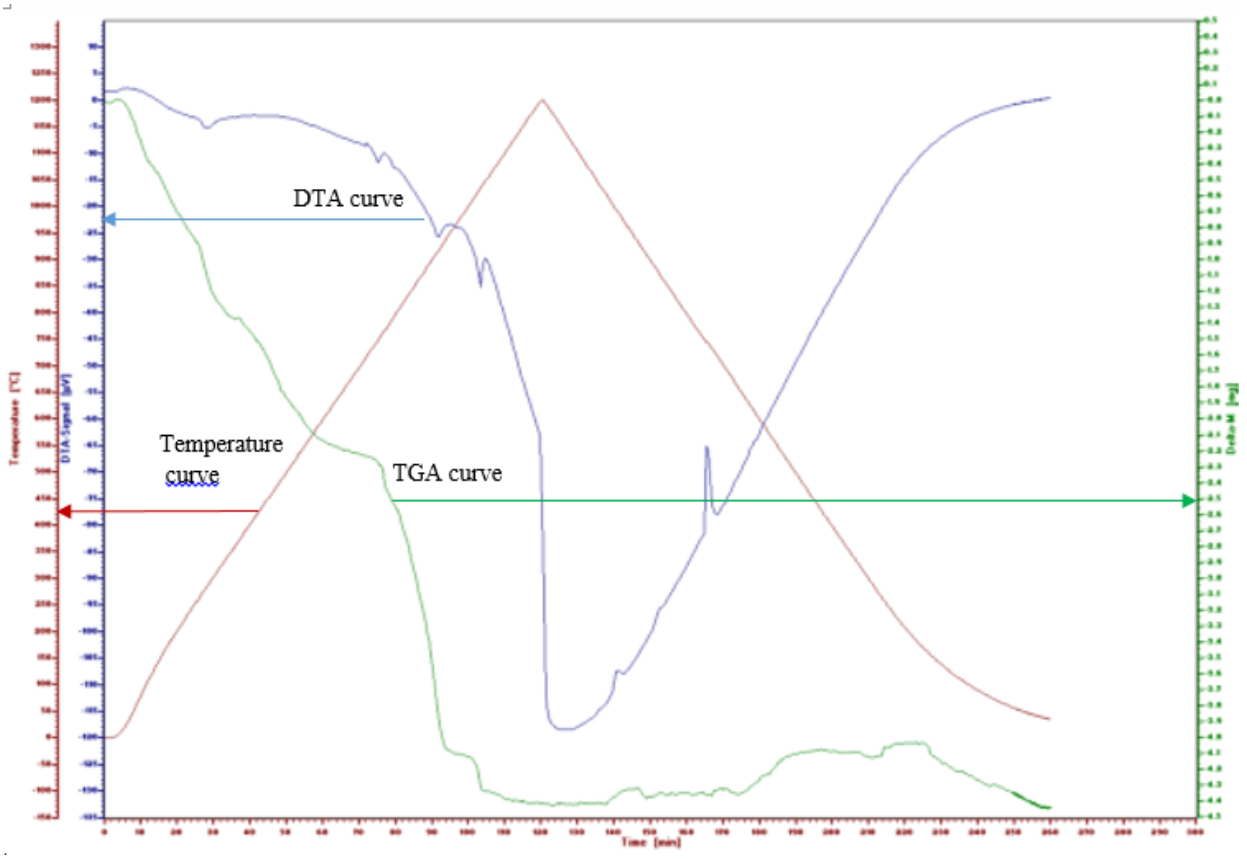

Figure 2. DTA-TG measurement result of $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ precursor with heating at 900 °C for 0.25 hours. Initial of sample is 20.7 mg.

4. Conclusion

Calcination affects the reaction temperature of the formation of $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ compounds. The reaction temperature optimum for the $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ phase formation is 938 °C and peritectic melting reaction temperature at 1032 °C for calcined samples without heating at 900 °C. The reaction temperature optimum for the $Gd_{0.9}La_{0.1}Ba_{1.95}Sr_{0.05}Cu_3O_{7-\delta}$ phase formation is 918 °C and peritectic melting reaction temperature at 1035 °C for calcined samples accompanied by heating at 900 °C.

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