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THE EFFECT OF SINTERING TEMPERATURE AND MIXING TREATMENT ON CRYSTAL STRUCTURE AND MAGNETIC PROPERTIES OF Y-358 SUPERCONDUCTORS

WG Suharta[1]*, IK Giri Nata[2], IG Antha Kasmawan[3], S Poniman[4] and GN Sutapa[5]

[1,2,3,4,5] Faculty of Mathematics and Natural Sciences, Udayana University, Indonesia

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ABSTRACT: This study aims to determine the effect of adding sintering temperature to changes in crystal structure and magnetic properties of the Y-358 superconductor. The raw material consisting of Y_2O_3 (99.9%), $BaCO_3$ (99.9%) and CuO (99.9%) were mixed in 3 days using a magnetic stirrer. Then heated at 100°C until crust. The calcination process was carried out at 600 for 3 hours and sintered at 850, 875, 900 and 925ºC for 10 hours each. From the X-Ray diffraction (XRD) result, the peak intensity of Y-358 superconductor increases with the addition of sintering temperature. The lowest peak intensity is 58 cps at 2θ of 86.4925 and the highest peak is 2041 cps at 2θ of 32.78. The addition of sintering temperature at 850, 875, 900 and 925ºC resulted in the addition of volume fraction, respectively 42.3%, 49.7%, 57.7% and 67.9%. The addition of sintering temperature also resulted in larger particle size, respectively 35.31, 47.72, 49.07 and 57.85 nm. The 3-day mixing treatment in the synthesis process resulted in a sample of nanometer size (nm) as seen in the TEM characterization results. The magnetic properties of the sample are known from the vibrating sample magnetometer (VSM) characterization results, showing the ferromagnetic properties with a magnetization saturation value of 0.08 emu/gr, the magnetization value of remanent of 0.02 emu/gr and the coercive force value of 684.12 Oe. The Fourier transform infrared spectroscopy (FTIR) characterization results show the absorption of OH groups in wave numbers 2814.14 and 3608.81 for samples calcined at 600°C and their intensity decreases with the addition of sintering temperature.

Keywords: wet-mixing, superconductor, XRD, TEM, VSM, FTIR

1. INTRODUCTION

Superconductors are materials that have a resistivity and a magnetic field equal to zero. Now, the development of superconductor technology is increasing rapidly, given the many applications that can be applied using such materials, such as Magnetic Levitation (MagLev), power transmission cables and superconducting magnetic energy storage system [1-3]. One of the ingredients that have great opportunities in applications is the $YBa_2Cu_3O_{7-\partial}$ (Y-123) superconductors found by Wu et.al [4]. The Y-123 superconductors have two CuO_2 layers in a single unit cell with a critical temperature of 90 K. To be applicable, the material has a high critical temperature (Tc), high critical current density (Jc) and high critical magnetic field (Hc). In that many different ways have been done, including with the substitution of rare earth elements such as Nd, Eu, and Gd which is a magnetic material [5]. The substitution of rare earth element is not only used in YBCO superconductors but also use for substitution the Ca element on the BSCCO superconductors [6].

Besides, the researchers made changes in the molar composition of the constituent compounds from Y-123 $YBa_2Cu_3O_{7-\partial}$ to Y358 ($Y_3Ba_5Cu_8O_{18}$). The superconductor of Y-358 has five layers of CuO_2 with a critical temperature of 100 K [7,8].

Generally, the synthesis of the YBCO superconductors has been done with solid state reaction and melting methods, to produce a sample with big particle size. However, in this research, Y-358 superconductors synthesis has been done by wet mixing method, using HNO_3 as a digesting agent, to produce samples with nanometer scale. For that, in this research is a long mixing process, with sintering time variation.

2. EXPERIMENT

Initial materials to form Y-358 superconductor is using Y_2O_3 (99.9%), $BaCO_3$ (99.9%) and CuO (99.9%). Each of the starting materials that had been weighed according to the molar compound was added HNO3, then stirred using a magnetic stirrer for 3 days at room temperature. Then the temperature is raised slowly (maximum 100ºC) until the precipitate is obtained. A compound in the form of precipitate calcined at temperature 600ºC for 3 hours, so obtained from powder samples with black color. Samples of the powder

are then sintered with temperature variations of 850, 875, 900 and 925ºC, respectively for 10 hours.

To see the success of Y-358 sample synthesis process, X-Ray Diffraction (XRD), Transmission Electron Microscopy (TEM) Vibrating Sample Magnetometer (VSM) and Fourier Transform Infrared Spectroscopy (FTIR) are used. Flow diagram The complete of Y-358 superconductors synthesis process is shown in Figure 1.

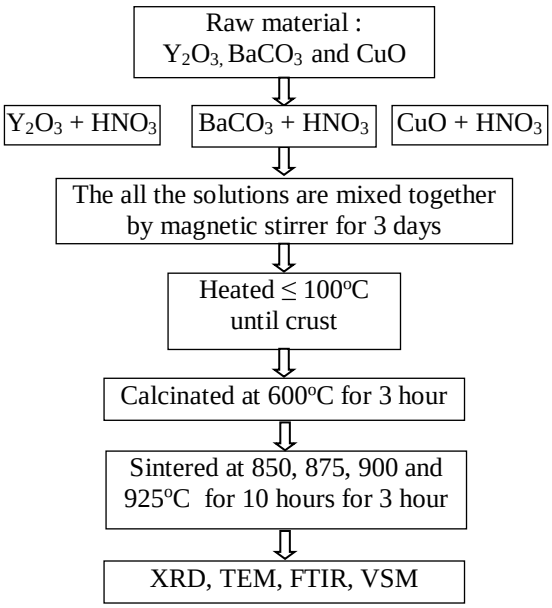

Fig.1 Flow diagram of synthesis process of Y-358 superconductors

3. RESULTS AND DISCUSSIONS

The result of the XRD characterization of the superconductor sintered at 850, 875, 900 and 925ºC, respectively for 10 hours is shown in Fig. 2. In general, the diffractogram pattern generated by each sample exhibits a similar pattern. The crystallization has occurred well, seen from the sharp peaks detected in the sample. The peak intensity of the Y-358 superconductor sample is greater with the addition of sintering temperature. This indicates that the sintering temperature of 925ºC is the optimal temperature of Y-358 phase formation, compared to the other three temperatures (850, 875 and 900ºC) given in this study. The value of the increase and decrease of peak intensity from superconductor samples, can be seen in Table 1.

Phase identification is done using Match program! 3.4.2 Build 96 [9]. The result of phase identification was obtained that the sample was dominated by Y-358 phase with high intensity, and the impurity phase was compound and with little intensity. Match results using Match program! shown in Figure 3. The results showed that the addition of sintering temperature resulted in a higher phase intensity of superconductor Y-358, followed by decreasing the intensity of the impurity phases of the compound. The increasing of the percentage of Y-358 superconductors and the decrease in phase intensity of the compound's impurities and can be determined by calculating the volume fraction of each phase. The result of the volume fraction calculation is shown in Table 2. Besides the addition of sintering temperature resulted in new peak growth with different Miller Index planes, as shown in Figure 3 and 4.

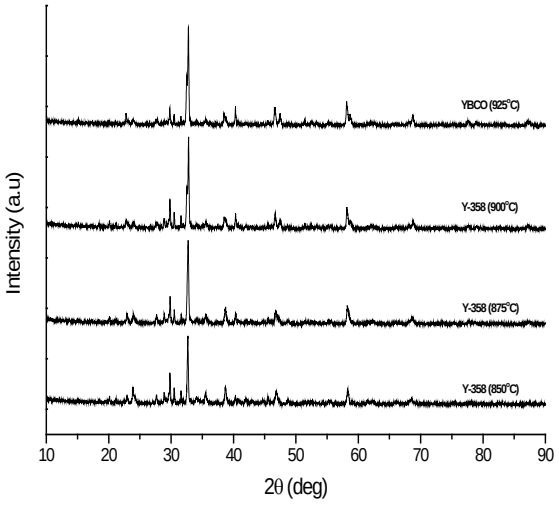

Fig.2 XRD pattern of Y-358 superconductor with sintering temperature at 850, 875, 900 and 925ºC

Table 1. The peak intensity value generated from the superconducting sample

Sample	2θ (º)	Intensity (cps)			
		850ºC	875ºC	900ºC	925ºC
Y-358	15.14	163	170	188	207
	22.81	170	205	289	318
	32.77	943	1598	1665	2026
	32.78	806	1512	1794	2041
	32.80	666	1321	1828	1981
	38.48	175	178	320	329
	40.32	282	341	399	474
	46.67	227	285	400	451
	58.12	172	228	420	520
	68.78	130	182	278	293
	77.67	129	139	155	192

The lattice parameter of the Y-358 sample by

the wet-mixing method in this study is similar to the lattice parameter of the Y-358 sample that added 0.5 mol Y-211 by solid-state reaction method. The lattice parameter of Y-358 for sintering temperature of 850ºC for 10 hours by a wet-mixing method in this study is a=3.7985 Å, b=3.8501 Å, and c=30.5732 Å. While the result of the lattice parameter of Y-358 sample added 0.5 mol Y-211 with solid state reaction method with a sintering temperature of 900ºC for 10 hours by Kruaheong T, yields a=3.7772 Å, b=3.8460 Å and c=30.3921 Å [10].

To determine the value of lattice parameters, volume, and density of unit cell, then performed Rietveld analysis with refinement using Rietica software [11]. The refinement results are shown in Table 3 and 4. It appears that the addition of sintering temperature results in the value of the lattice parameter toward the a-axis and c-axis increases, whereas the lattice parameter value toward the b-axis is reduced. The change in lattice parameter values causes the value of the unit cell volume to increase, while density decreases with the addition of sintering temperature.

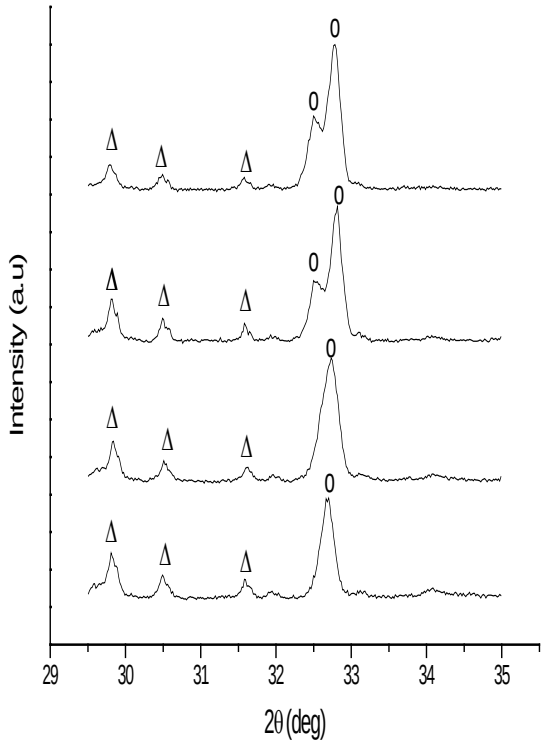

Fig.3 The results of search match of Y-358 samples at 2θ from 29º to 35º. Symbol o ($Ba_4Cu_6O_{13}Y_2$), Δ ($BaCuO_5Y_2$)

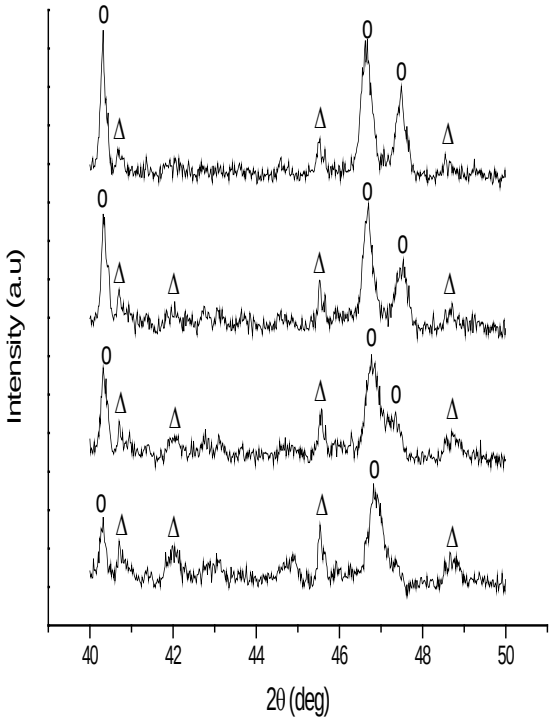

Fig.4 The results of search match of Y-358 samples at 2θ from 40º to 50º. Symbol o ($Ba_4Cu_6O_{13}Y_2$), Δ ($BaCuO_5Y_2$)

Table 2. The result of the volume fraction calculation of Y-358 superconductors and impurity compound

Sintering temperature (ºC)	Volume fraction (%) Y-358
850	42.3
875	49.7
900	57.7
925	67.9

To know the sample morphology and particle size, the characterization was done with TEM. The characterization result using Transmission Electron Microscopy (TEM) is a sample surface image. The results of TEM characterization can be seen in Figure 5. The results are then analyzed using the Image-J and the Origin software. The results of particle size calculations of the sample shown in Fig. 6. It can be seen that the particle size of the sample is equal to 2.5 ± 1.30708 nm.

Table 3. The value of lattice parameter of Y-358 superconductors

Sample	Lattice parameter		
	a (Å)	b (Å)	c (Å)
Y-358 (850°C)	3.7985	3.8301	30.5732
Y-358 (875°C)	3.8246	3.7962	30.6701
Y-358 (900°C)	3.8317	3.77456	30.7167
Y-358 (925°C)	3.8338	3.7662	30.7337

Table 4. The value of the volume of the unit cell and density of Y-358 superconductor

Sample	Volume $Å^3$	Density (g) $Å^{-2}$
Y-35 (850°C)	444.791	6.532
Y-358 (875°C)	445.306	6.522
Y-358 (900°C)	444.255	6.538
Y-358 (925°C)	444.941	6.528

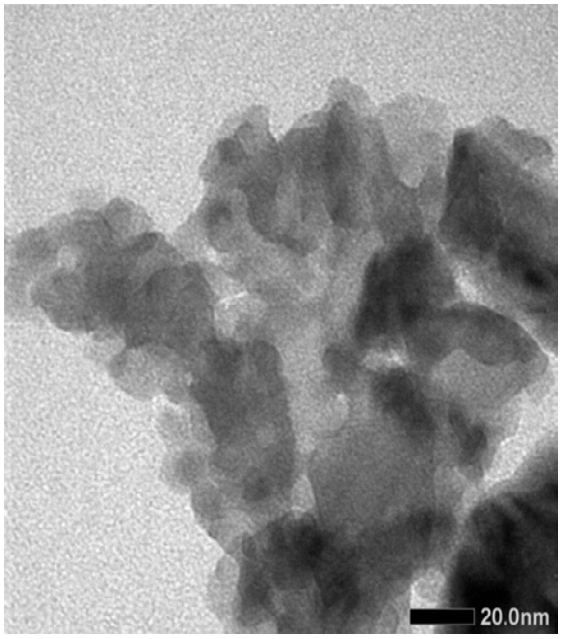

Fig.5 TEM image of Y-358 superconductors

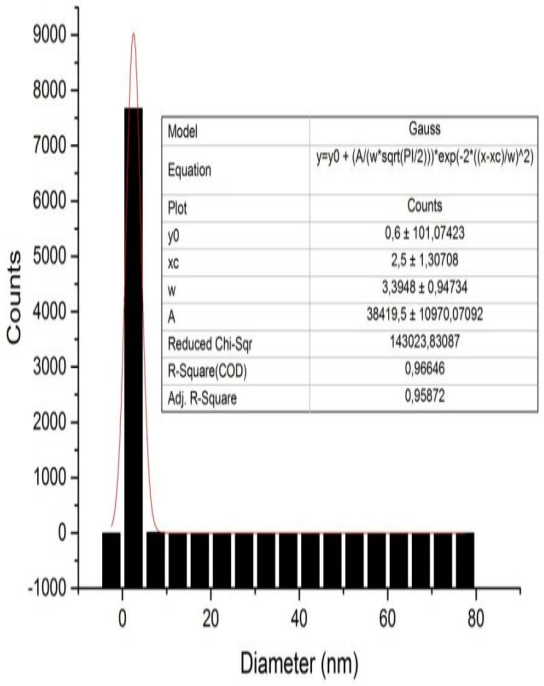

Fig.6 The results of TEM analysis using imageJ and Origin software of Y-358 superconductors

The TEM characterization of Figure 5 shows a less homogeneous crystal size, possibly due to long sintering time. The long sintering time resulted in the agglomeration process happening quickly. However, overall, the average particle size is below 50 nano meters.

The magnetic properties of the material are seen from the relationship between the external magnetic field and the magnetization, therefore characterization is performed using Vibrating Sample Magnetometer (VSM) at room temperature. The result of VSM characterization is a hysteresis curve as shown in Figure 7, which indicates that the sample is ferromagnetic. The hysteresis curve shows the magnetization value of saturation (Ms) 0.08 emu/gr, a remanent magnetization (Mr) 0.02 emu/gr and coercivity field (Hc) 684.12 Oe. The magnetization value of saturation, remanent magnetization, and coercivity field are shown in Table 5.

The results are in accordance with the results of a study reported by Shipra et.al, that the Y-123 sample with nano-meter particle size exhibits ferromagnetic properties at room temperature [12]. The nanoparticles of Y-123 with a critical temperature of 91 K shows a linear magnetization curve at room temperature.

While A. Sundaresan et.al reported that any ingredients such as cerium oxide, aluminum oxide, zinc oxide, indium oxide, a tin oxide with the particle size of 7-30 nano meter shows ferromagnetic properties at room temperature [13].

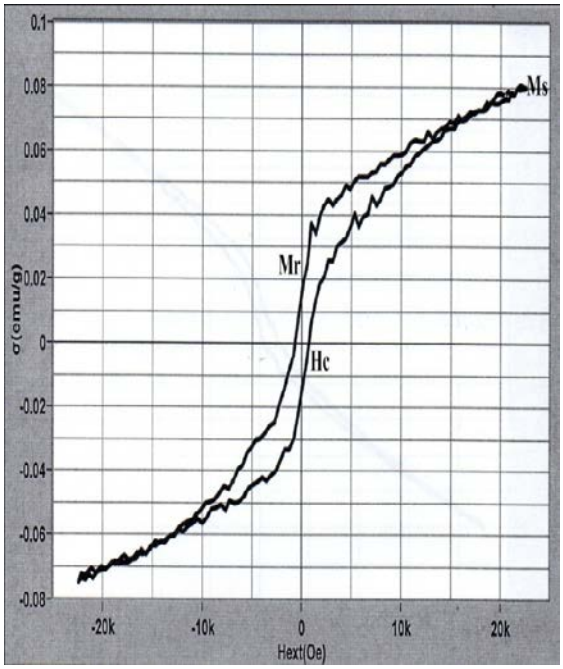

Fig.7 Hysteresis curve of Y-358 superconductor at room temperature

Table 4. The value of saturation magnetization (Ms), a remanent magnetization (Mr) and coercivity field (Hc).

Sample	Ms (emu/gr)	Mr (emu/gr)	Hc (Oe)
Y-358	0.08	0.02	684.12

To know the absorption that occurs in the sample, then it is done the characterization using Fourier Transform Infrared Spectroscopy (FTIR). The results of the FTIR characterization of the samples that sintered at 850, 875, 900 and 925ºC for 10 hours can be seen in Fig. 7.

FTIR spectrophotometer analysis was used to determine the groups formed from the resulting sample. This analysis is based on an analysis of the characteristic peak wavelengths of the sample. The wavelengths of the peaks indicate the presence of a particular functional group present in the sample since each functional group has a specific characteristic peak for a particular functional group. The functional group data obtained at the wave numbers possessed by the superconductor samples as shown in Table 5. Where at wavenumber 2814,14 and 3608,81 nm the presence of OH group on samples in the calcination process at 600ºC, but in the sintering process at 850, 875, 900 and 925ºC there was no return of the OH functional group to the superconductor samples.

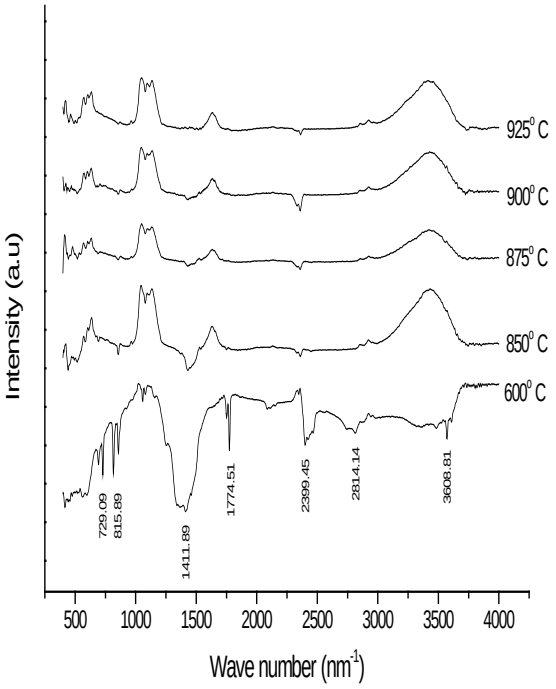

Fig.8 The results of FTIR characterization of Y-358 superconductors

4. CONCLUSION

From the result of research, hence can be concluded:

The increasing of sintering temperature from 850 to 925ºC have been resulted in the increasing the value of volume fraction (from 42.3% to 67.9%), while it has been resulted decreasing of density (from 6.532 $Å^{-2}$ to 6.528 $Å^{-2}$).

The increasing of sintering temperature from 850 to 925ºC have been resulted in the increasing of lattice parameter toward a-axis from 3.79848 Å to 3.83379 Å and c-axis from 30.57318 Å to 30.73371 Å. While the increasing of sintering temperature from 850 to 925ºC have resulted in the decreasing of lattice parameter toward b-axis from 3,83006 Å to 3,76624 Å.

The particle size of the Y-358 superconductor obtained in this study is nanometer-scale and shows ferromagnetic properties at room temperature

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