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Fabrication of superconducting YBCO agglomerated particles (ANPs) by electrospinning

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Superconducting $YBa_2Cu_3O_{7-\delta}$ (YBCO) agglomerated nanoparticle (ANPs) was fabricated by electrospinning technique and sol-gel of a homogeneous precursor of Y-Ba-Cu acetate and Poly (vinyl pyrrolidone) (PVP). The sol-gel solution of YBCO is conducted by adding 4.0 g of (Y-Ba-Cu) metal acetate according to a stoichiometric ratio of 1:2:3 molar mass and 5.0 g of PVP powder in 25.0 ml solution contains propionic acid 10 ml, acetic acid 5 ml and methanol 10 ml. By optimizing electrospinning process, sol-gel parameters and heat treatment, YBCO ANPs were obtained with a transition temperature of T_c ~90 K using the AC susceptibility. YBCO ANPs was found with high surface area 6.8310 m^2/g, and not affected by high calcination temperature at 950 ^oC in comparison with bulk YBCO sample. XRD Characterization of YBCO was demonstrated YBCO has orthorhombic phase, FESEM images of the electrospun YBCO sample showed ANPs of size in the 200 – 400 nm range. A closer examination revealed that agglomerates contain finer particles of size ~50 nm. Electrospinning is an effective technique can produce various morphologies of YBCO superconductor at the nanoscale with unique properties for practical applications.

Keywords: Agglomerated nanoparticle; Electrospinning; Superconductor; YBCO.

Exp. Theo. NANOTECHNOLOGY 3 (2019) 181-188

1. INTRODUCTION

The high-temperature superconductor (HTS) $YBa_2Cu_3O_{7-\delta}$ (YBCO) is characterized by several unique properties in comparison with the conventional conductors [1-3]. HTS YBCO became popular among other ceramic superconductors due to their high transition temperature (~92 K), current density (~10^6 A/cm^2, 77 K), magnetic field (~16 T), and chemical stability [4, 5]. In addition, YBCO is easy to produce into a single phase and crystalline using relatively low cost starting material such as acetates, nitrates, and so on [6, 7]. Several structures of the superconducting $YBa_2Cu_3O_{7-\delta}$ (YBCO) at nanoscale were synthesized successfully by employing various techniques with structure, pure, uniform and, high performance [8]. Structures of YBCO superconductor like fibres, wires, tube, tape and particles have been prepared [9, 10]. Many reports have been published introduced the preparation of YBCO at the nanoscale by several techniques such as pulsed laser deposition (PLD), chemical vapor deposition (CVD), electron-beam lithography (E-beam) and anodic aluminum oxide (AAO) template, most of these techniques are complex and expensive [11-15].

Recently, electrospinning among various methods is relatively simple, effective, cheap, controllable and versatile technique used to prepare YBCO with various structures at nanoscale, such as nanowires (NWs), nanotube (NTs), and nanofiber (NFs), intensive research works focusing on their properties and practical applications have been widely undertaken [7, 13]. Electrospinning process is one of the renowned techniques used to prepare the material in nanopattern structures, which was observed by formulas in 1934 [16, 17]. Electrospinning process consists of four setups; the high voltage power supplier, spinneret, syringe pump and collector. The electrospinning can be defined as an electric field is applied between a tip nozzle and the collector. Normally the high voltage is used to generate a fragile charged jet solution from the droplet at the tip of the needle; the drop began to stretch and form a monofilament. The monofilament leaves the drop when the electric field strength increase and the electrostatic repulsive force overcomes the surface tension [18, 19]. Electrospinning is a powerful technique, generally used in combination with sol-gel technique followed by calcination at high temperature [14, 20]. The typical sol-gel solution used with electrospinning technique consists of the precursor of the desired material plus polymer and a relatively volatile solvent. Solvent utilized to control the solution product viscosity, conductivity, and morphology. The proper solvent was used typically like water, methanol, isopropanol, and ethanol [4, 7, 13, 14, 18]. In this work, we show that by optimizing the electrospinning, sol-gel, and heat treatment process of YBCO ANPs could be obtained with serval unique properties. Detailed results are described in this paper.

2. EXPERIMENTAL

1) Materials

YBCO ANPs sample was prepared through electrospinning the starting materials were; Poly (vinyl pyrrolidone), (PVP) (Mw = 1,300,000 g/mole), were purchased from Sigma-Aldrich,

Exp. Theo. NANOTECHNOLOGY 3 (2019) 181-188
Yttrium (III) acetate tetrahydrate (99.9%), Y ($OOCCH_3)_3.4H_2O$), Barium acetate $Ba(OOCCH_3)_3$) (99.0%), and Copper (II) acetate monohydrate ($C_4H_6CuO_4.H_2O$) were purchased from Alfa Aesar. The solvents Propionic acid ($C_3H_6O_2$), Acetic acid ($C_2H_4O_2$) and Methanol (CH_4O) used (purity, 99%) supplied commercially. All the material was used without any purification.

2) Sample Fabrication

The sample sol-gel solution was conducted by adding 4.0 g of Y-Ba-Cu acetate according to a stoichiometric ratio of 1:2:3 molar mass and 5.0 g of PVP powder in 25.0 ml solution contain the propionic acid 10 ml, acetic acid 5 ml and methanol 10 ml. Commonly propionic acid utilized to dissolve the metallic acetate [13, 14], while acetic acid was added to avoid any hydrolysis of the polymers [7, 13, 14]. However, the acid medium is necessary to create a stable solution and to avoid the hydrolysis in the sol-gel precursor. Finally, the methanol addition was made to dissolve the polymer and to tune the viscosity of the precursor [13]. The addition of the polymer leads to increase the viscosity and achieve a spun-able solution [2, 3, 14]. The solutions then stirred at 70 ^oC for 6 h in a closed beaker. The viscosity of the solutions was controlled by adding the polymer and solvent. Both the viscosity and the conductivity of the solution were measured and the results was consistent with other work [21, 22].

The set-up of the electrospinning processes includes a syringe (2 cm diameter, 20 ml volume), needle (18-gauge, stainless steel), rotated collector (8 cm diameter, 25cm length), high voltages supplier and the pump injection [17, 18, 23]. The sol-gel solution was loaded to the syringe and electrospun at 22 kV with the tip-collector distance of 15 cm, flow rate 0.3 ml/h and collector speeds with 1200 rpm. The electrospinning process was carried out at room temperature and closed environment with humidity ~50% and the electrospun sample was placed in a closed desiccator for 48 h for drying purpose.

3) Heat treatment

The dried sample of YBCO was prepared with two heat treatment process, the first step is to burn out the polymer using the box furnace (Nabertherm, 30-3000 ^oC) up to 500 ^oC for 3 h at a heating rate of 50 ^oC/h. The second is for sintering process to produce the single structure of YBCO ANPs using tube furnace (Nabertherm, 30-3000 ^oC), from 500 ^oC up to 900 ^oC for 4 h with a rate of 250 ^oC/h, which is followed by the heating rate of 25 ^oC/h up to 950 ^oC for 3 h in oxygen environment. The sample then grained to a fine powder and transferred to the characterization and measurements.

4) Characterizations

The viscosity and conductivity of the solution were measured at room temperature by the high-resolution laboratory Viscometer Brook Field (model: LVDV -11+P) and the conductivity meter respectively. The thermal analysis of the electrospun sample was examined by a thermogravimetric analyzer (Mettler Toledo, TGA DSC). The sample morphology was observed by the Field Emission Scanning Electron Microscope (JEOL, model: JSM–7600) operated with 5.0 kV. The structure of the sample was analyzed using X-ray diffractometer (Rigaku, model: Miniflex II) from the angle 2θ 5 to 85^{o}, count speed of 1 degree per minute, operating at 30 kV, 15 mA. The critical temperatures (T_c) of YBCO sample was measured using the AC Susceptometer of closed cycle liquid helium refrigeration systems (Cryo Industry model REF-1808-ACS. The BET surface area measurements for electrospun YBCO nanoparticle sample was examined using the BET surface area and porosity analyzer (Micrometrics, model: ASAP 2020), and the degas temperature of YBCO sample is found at 400 ^{o}C for 7 h.

3. RESULTS AND DISCUSSION

In The viscosity of the YBCO sample was found in the range from 188 to 193 cP, while the conductivity is from 149 to 155 mS/cm range at room temperature. This work the solution indicated with high conductivity in comparison with the work of Edgar et al (2014), the conductivity is around 12.7 µS/cm [13]. Electrospun solution with high conductivity and low viscosity are desired for an effortless process for the charged jet transformation from the nozzle tip to the collector [24]. Cui *et al* [14] have studied the relation between the nanofiber diameter produced by electrospinning and the viscosity of Y-Ba-Cu acetate solution, the fiber diameter was increased slightly by increasing their solution viscosity from 200 to 400 cP and then increased steadily by increasing the viscosity from 500 to 700 cP [14].

Figure 1 shows the TGA – DSC data of the as-spun sample after drying for 48 hours. The DSC showed an endothermic event at ~100^{o}C; correspondingly ~8% weight loss was observed in the TGA which is assigned to evaporation of water and other low-volatile impurities such as adhered solvent molecules. The DSC showed a major endothermic event centered at ~230^{o}C with a ~15% weight loss due to melting and evaporation of polymer. Two more endothermic events are observed in the DSC, centered at 300, 400, and 450^{o}C with a total weight loss of ~30% showing complete removal of polymers. An exothermic event and correspondingly a slight change in weight of the sample were observed at ~750°C, which is assigned to the crystallization of YBCO and oxygenation of the samples. These assignments are consistent with previous reports [4, 7, 13]. Therefore, the DSC and TGA results suggest that crystalline nanostructured samples of YBCO could be obtained from electrospinning by combining two heat treatments processes. The first heat treatment is to fully evaporate the polymers and the second one is to get the required YBCO in crystallized phase.

Exp. Theo. NANOTECHNOLOGY 3 (2019) 181-188

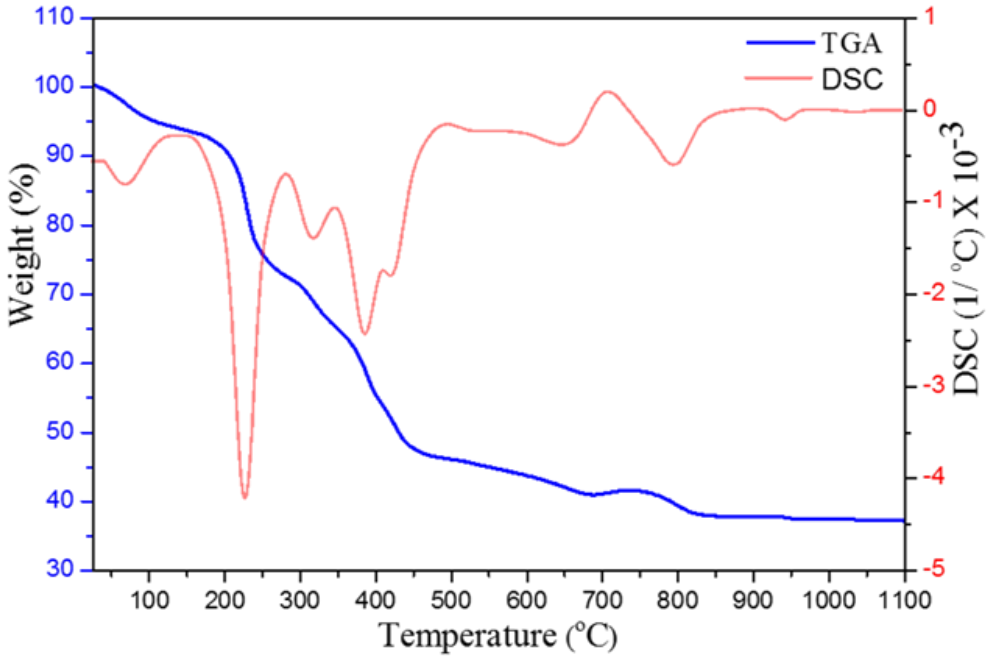

Figure 1 Simultaneous thermogravimetric analysis (TGA / DSC) curves vs. temperature for YBCO nanostructure mats.

Figure 2 summarizes the morphology, crystal structure, and superconducting properties of the ANPs sample. The top FESEM images showing the morphology of the electrospun YBCO agglomerated particles sample sized in the 200 – 400 nm range, the agglomeration most likely results from the elevated heat treatment involved. A high magnification revealed that agglomerates contain a fine particle size <50 nm. The bottom left panel of Figureure 2, displays the XRD pattern of the prepared sample; the peaks can be indexed to the orthorhombic structure with lattice parameter found are; a = 3.897 Å, b = 3.8889 Å, c = 11.707 Å, V = 177.4064 Å^3 and corresponding to the superconducting phase [7, 13]. The bottom right panel of Figure 2, displays the AC susceptibility result as function of temperature, the complex susceptibility (χ) consists of the real and imaginary parts ($\chi = \chi' + i\chi''$); clearly the real part of susceptibility suddenly decreased as the temperature decreased after the transition temperature, the T_c of the YBCO sample was estimated from the curve ~ 90 K.

Exp. Theo. NANOTECHNOLOGY 3 (2019) 181-188

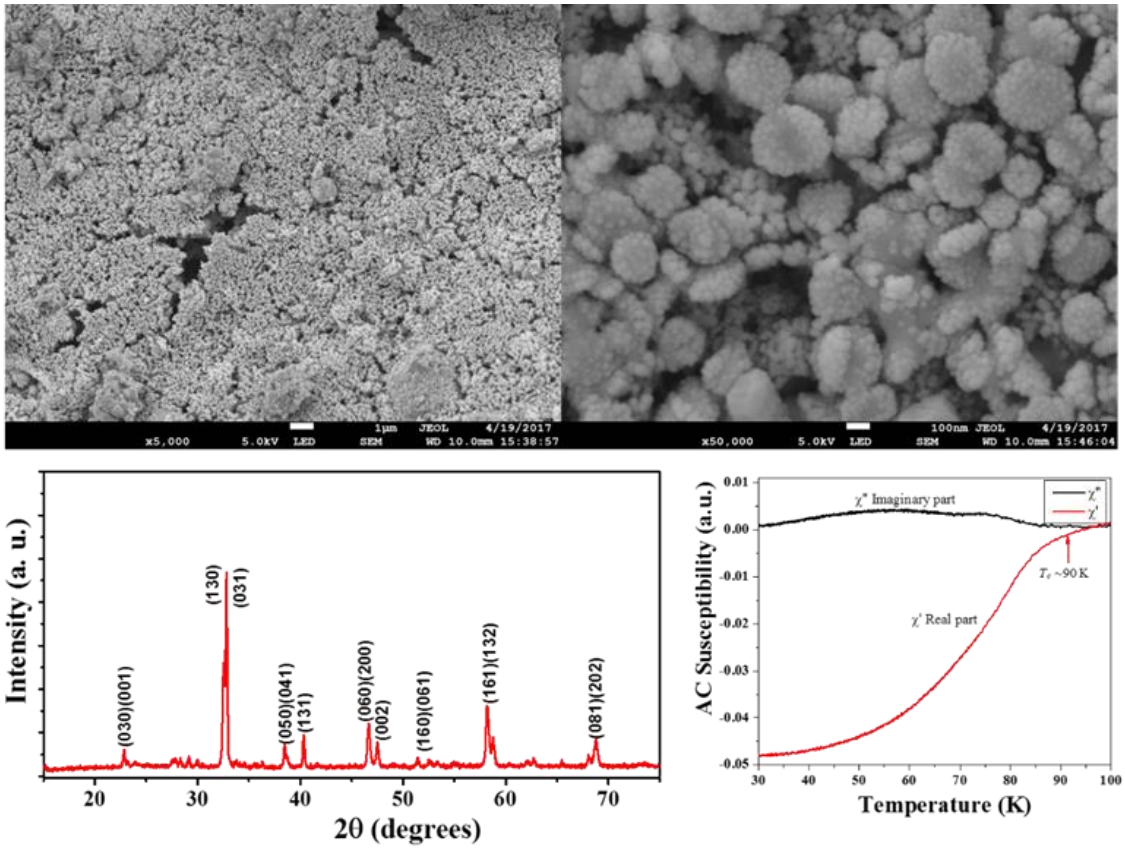

Figure 2 FESEM images, X-ray diffraction pattern, T_c measurement of the electrospun YBCO ANPs.

BET surface area measurements for the electrospun YBCO ANPs sample was 6.8310 m^2/g. The sample was heated and annealed up to 950 °C in oxygen atmosphere. G. E. Shter *et al*.[25], have studied the interrelation between the surface area and the calcination temperature, the author found the surface area of the YBCO sample was decreased from 2.36 to 0.46 m^2/g by increasing the calcination temperature from 750 °C to 910 °C. However, G. E. Shter and G. S. Grader [26], measured the surface area of YBCO sample with 1.7 m^2/g corresponding to the calcined at 780°C for 12 h, while calcining YBCO at 920 °C lead to decrease the surface area to 0.3 m^2/g which is attributed to the reduce in particle size to submicron**.** However, this work presented a new result proved that, the surface area of YBCO nanoparticle is not affected by the high calcination temperature. This result due to the nanoscale size of YBCO particle, which is definitely, lead to increase the surface area [27].

4. CONCLUSIONS

Electrospinning is a very competitive technique by which researchers could have built several constructions in nanostructured morphologies with high porosity, surface area, small volume

and unique properties. This work presents synthesis the superconducting YBCO ANPs using a new procedure by electrospinning process. In this work used a homogeneous polymeric solution and the precursor of metal acetate were electrospun and followed by the heat treatment process. The measurements of the transition temperature of YBCO ANPs by the AC susceptibility was found T_c ~90 K. The BET measurements of YBCO were found with highly surface area 6.8310 m^2/g, and there is not influenced by the high calcination temperature compared with the last works. The XRD and FESEM structural Characterization of YBCO was demonstrated that, the YBCO sample has the phase of orthorhombic structure, the images of the electrospun YBCO sample showed ANPs of size in the 200 – 400 nm range. A closer examination revealed that agglomerates contain finer particles of size ~50 nm. Electrospinning is an effective technique to produce various morphologies of YBCO superconductor at the nanoscale with unique properties for practical applications.

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References

[1] D. van Delft, Physica C: Superconductivity 479 (2012) 30
[2] S. Bose and P. Ayyub, Reports on Progress in Physics 77 (2014) 116503.
[3] R. P. Huebener, Conductors, Semiconductors, Superconductors, ed: Springer (2015) 144.
[4] I. Uslu, M. Kemal Ozturk, M. Levent Aksu, and F. Gokmese, Current Nanoscience 6 (2010) 408
[5] S. Nawaz, R. Arpaia, T. Bauch, and F. Lombardi, Physica C: Superconductivity 495 (2013) 33
[6] Z. Shen, Y. Wang, W. Chen, L. Fei, K. Li, H. L. W. Chan, Journal of Materials Science 48 (2013) 3985
[7] Aruna P. Wanninayake, Benjamin C. Church, Nidal Abu-Zahra, Exp. Theo. NANOTECHNOLOGY 2 (2018) 31
[8] Q. Wali, A. Fakharuddin, I. Ahmed, M. H. Ab Rahim, J. Ismail, and R. Jose, Journal of Materials Chemistry A 2 (2014) 17427
[9] R. ARPAIA, University of Technology G¨oteborg, Sweden (2016)
[10] Z. Shen, Y. Hu, L. Fei, K. Li, W. Chen, H. Gu, Journal of Nanomaterials 1 (2015) 1
[11] E. A. Duarte, N. G. Rudawski, P. A. Quintero, M. W. Meisel, and J. C. Nino, Superconductor Science and Technology 28 (2014) 015006
[12] X. M. Cui, W. S. Lyoo, W. K. Son, D. H. Park, J. H. Choy, T. S. Lee, Superconductor Science and Technology 19 (2006) 1264
[13] G. Zhang, X. Lu, T. Zhang, J. Qu, W. Wang, X. Li, Nanotechnology 17 (2006) 4252
[14] D. Manimekalai, Pradipkumar Dixit, Exp. Theo. NANOTECHNOLOGY 2 (2018) 43
[15] A. Valizadeh and S. M. Farkhani, Nanobiotechnology, IET 8 (2014) 83
[16] D. Li and Y. Xia, Advanced materials 16 (2004) 1151
[17] N. Bhardwaj and S. C. Kundu, Biotechnology advances 28 (2010) 325

Exp. Theo. NANOTECHNOLOGY 3 (2019) 181-188
[18] J. Doshi and D. H. Reneker, Journal of electrostatics 35 (1995) 151
[19] Y. Greenberg, Y. Lumelsky, M. Silverstein, and E. Zussman, Journal of Materials Science 43 (2008) 1664
[20] B. Sun, Y. Long, H. Zhang, M. Li, J. Duvail, X. Jiang, Progress in Polymer Science 39 (2014) 862
[21] A. Baji, Y.-W. Mai, S.-C. Wong, M. Abtahi, and P. Chen, Composites science and technology 70 (2010) 703
[22] R. Nirmala, R. Navamathavan, S.-J. Park, and H. Y. Kim, Nano-Micro Letters 6 (2014) 89
[23] K. Garg and G. L. Bowlin, Biomicrofluidics 5 (2010) 013403
[24] G. Shter, A. Schwartzman, and G. Grader, Applied superconductivity 3 (1995) 543
[25] G. E. Shter and G. S. Grader, Journal of the American Ceramic Society 77 (1994) 1436
[26] A. Knizhnik, *NEW RESEARCH ON YBCO SUPERCONDUCTORS*, Nova Science, New York (2008)