

Novel Production and Characterization of Nano Fibers of Nickel Alumina Borosilicate Ceramic Composite

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Abstract- Composite nanofibres can be formed in many different deposition techniques. A sol-gel recipe that allows the formation of a homogeneous four component alkoxide solution and provides a control over solution viscosity for electrospinning process was developed. In addition to, electrospinning is the most reliable and commonly used techniques to obtain desired fiber properties. Aluminum borate fibres have also attracted more attention due to their stability at high temperatures. The nickel alumina borosilicate ($\text{NiO}/\text{Al}_2\text{O}_3/\text{B}_2\text{O}_3/\text{SiO}_2$) nanofibres was produced by calcinations at the certain temperature 750°C of the nanofibres obtained with this method.

Keywords- Composites, Ceramics, Nanostructures, Sol-gel chemistry, electrospinning process

I. INTRODUCTION

In recent years, nanostructural materials such as nanorods, nanowires and the nanofibres have been actively studied due to both their scientific use and potential applications in nano devices. In particular, the dispersion of metal Nano particles in an inorganic matrix has aroused great interest. Nickel oxides are well-studied materials due to their high activity, low cost, and abundant element, nickel-based catalysts have been extensively employed and investigated for catalysis, chemical and energy applications. The electrospun nanofibres are able to form a highly porous mesh and their large surface-to-volume ratio improves performance for numerous applications [1-3].

The sol-gel method has been compounded with the electrospinning to produce very fine polymer/ceramic nanofibres. The chemical and physical features of the materials obtained by these methods (e.g., surface areas, particle sizes and mechanical properties) can be changed with the temperature, operating conditions, and the used precursor, which means that control, is provided over the microstructural properties of these materials thanks to this technique. The essential feature of this method is to give a desirable particle size and distribution at high yield and low preparation cost [4]. This study reports an easily applicable and inexpensive approach of the fabrication of the nickel alumina borosilicate composite nanofibres using the sol-gel processing and the electrospinning technique. The PVA /nickel nitrate/ aluminum izopropoxide / boric acid / TEOS (Tetraethyl orthosilicate) composite was used as precursor through calcination

treatments. The product obtained in this way was characterized by FT-IR (Fourier transform-infrared spectroscopy), DTA-TGA (Thermogravimetric/differential thermal analysis), SEM (Scanning electron microscopy), TEM (Transmission electron microscopy), and XRD (X-ray diffractometry).

II. MATERIALS AND METHODS

The sol-gel solutions used in the electrospinning were composed of nickel nitrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, Sigma-Aldrich, >98%), tetraethyl orthosilicate (TEOS, $\text{Si}(\text{C}_2\text{H}_5\text{O})_4$, Sigma-Aldrich, >98%), aluminum isopropoxide ($\text{Al}(\text{OC}_3\text{H}_7)_3$, Sigma-Aldrich, >97%), boric acid (H_3BO_3 , Etibank A.Ş.), polyvinyl alcohol (PVA $M_w = 80.000$ g/mol, Sigma-Aldrich), hydrochloric acid, absolute ethanol (EtOH), isopropyl alcohol (i-PrOH) and deionized water.

PVA aqueous solution (10wt%) was first prepared by dissolving 10 g PVA powder in 100 ml of distilled water while stirring for 2 hours at 80°C , then the solution was cooled to room temperature and stirring was maintained for 12 hours. A silica gel solution was prepared by mixing 10 ml aluminum isopropoxide ($\text{Al}(\text{OC}_3\text{H}_7)_3$) 10 ml of tetraethyl ortosilicate (TEOS), 10 ml of ethanol, 10 ml of water, 10 ml isopropyl alcohol and 2 ml of concentrated HCl together. The mixed solution was maintained in a 40°C water bath with vigorous magnetic stirring for 2 hours. In another container, 10 ml of boric acid and 20 ml of PVA was mixed with 10 ml of nickel nitrate. Next, the above solutions were separately added into the prepared 30 ml PVA aqueous solution under vigorous stirring at a slow speed. The pH of medium is below 2 to protect of peptization during the forming of the sol-gel. The resulting solution was placed in a 10 ml plastic capillary-tipped syringe. Fibers on a metal collector which is the aluminum foil were collected in the electrospinning apparatus under these conditions that are 10 kV, capillary tip to metal collector distance of 10 cm, 10% PVA solutions and 3 ml/h flow rate of solution. Thus, the formed fibres were dried initially at 110°C for 2 hours and then, calcined at a heating rate of $2^\circ\text{C}/\text{minute}$ in air at 400 , 750°C , and 1000°C temperatures, respectively. It remained at the required temperature for 2 hours to obtain the $\text{NiO}/\text{Al}_2\text{O}_3/\text{B}_2\text{O}_3/\text{SiO}_2$ nanofibres.

III. RESULTS AND DISCUSSION

A. FT-IR Spectra

According to Fig. 1a, the peaks are shown, especially Si-O-Si at around 1091 cm^{-1} . When calcinations were at 750°C and 1000°C (Fig. 1b-c), all of the peaks fitting to the organic groups of PVA and other components faded away and a new peak at 467 cm^{-1} is appointed to the Ni-O bond [5]. Formation of the anticipated B-O-Si linkage is confirmed by (FT-IR). The FT-IR spectrum of nanofibres indicates the presence of the B-O bonds at around 1430 cm^{-1} . In particular, this distinct band assigns the asymmetric stretching of the BO_3 unit [6]. In addition, in the all of the FT-IR spectra, the peaks at around 800 cm^{-1} are assigned Al-O-Si (bending). In addition, when the composite is calcinated, a peak at 1331 cm^{-1} assigned to Si-OEt or ethanol normally disappears [7].

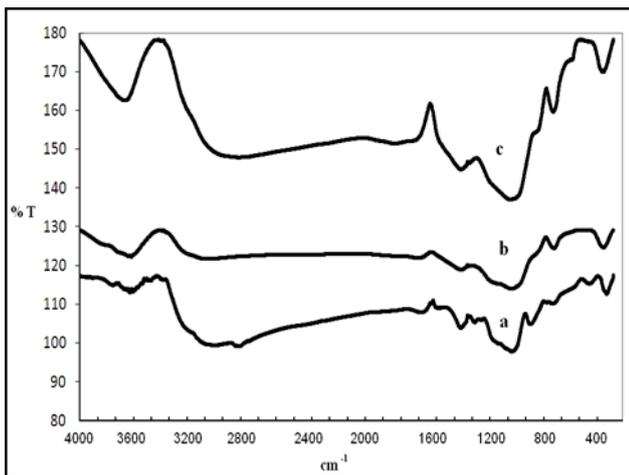


Figure 1. FT-IR spectra for (a) before applied the thermal treatment PVA / nickel nitrate / tetraethyl orthosilicate / aluminium izopropoxide / boric acid composite fibres; (b) calcined at 750°C ; (c) calcined at 1000°C .

B. TGA-DTA

The DTA curve is description as an endothermic peak at 115°C , which could be referring to the loss of moisture and removed solvent (water, ethanol from TEOS). The endothermic peak around 370°C could be referring the degradation of PVA by dehydration on the polymer side chain, which was manifested by a weight loss in the TGA curve at the about temperature range. The other degradation between 400°C and 500°C corresponds to the decomposition of the PVA main chain, which was shown in the TGA curve. Finally, as shown in the endothermic at around 680°C in the DTA curve, the inorganic component-PVA complex decomposes to oxides at 680°C [8]. No further weight loss above 680°C indicated the formation of clear inorganic oxide in a crystalline form (Fig. 2).

C. X-Ray Diffraction (XRD)

Fig. 3 shows the XRD patterns of nanofibres calcinated at 750°C . As shown in the figure, all the diffraction peaks were extremely similar to those of the nickel alumina borosilicate phase. In unison with Guan et al., the crystalline region of all of the Al_2O_3 , B_2O_3 , SiO_2 and also NiO was observed [5].

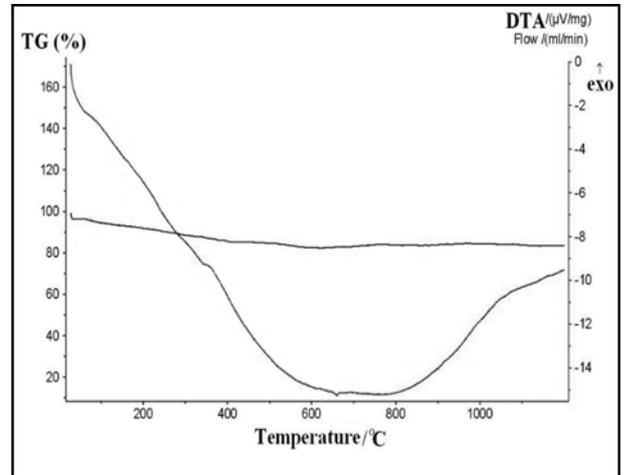


Figure 2. TG-DTA thermograms for the PVA /nickel nitrate/ tetraethyl orthosilicate/aluminium izopropoxide/boric acid composite.

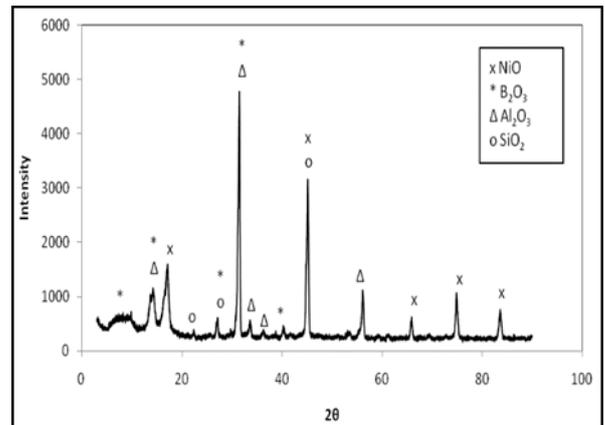


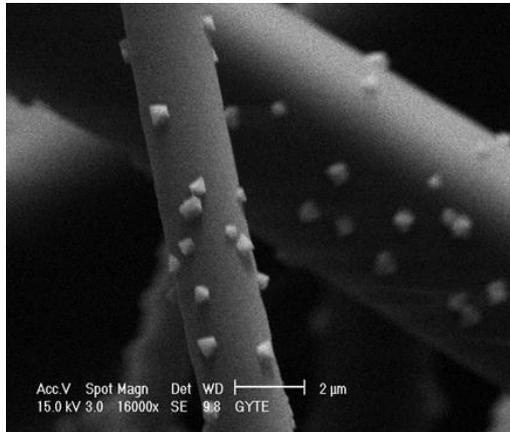
Figure 3. XRD patterns of the NiO / Al_2O_3 / B_2O_3 / SiO_2 nanofibres calcined at 750°C .

D. Scanning Electron Microscopy (SEM)

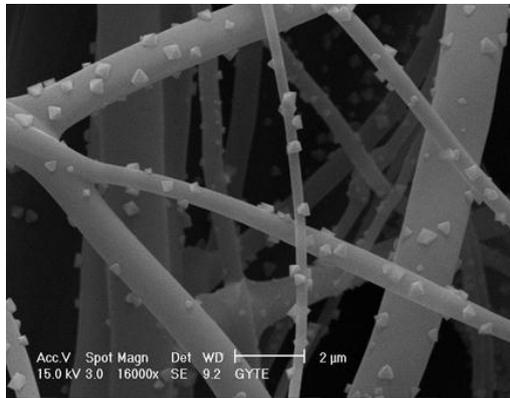
The SEM photographs of the PVA/ /nickel nitrate/ aluminum izopropoxide / boric acid / TEOS composite fibres was shown in Fig. 4 a-b. As shown in Fig. 4a, before the applied thermal treatment, the PVA /nickel nitrate/ aluminum izopropoxide / boric acid / TEOS composite nanofibres demonstrated a uniform morphology. After calcination at 750°C (Fig. 4b), the diameters of the fibres became smaller. This situation was thought to be due to the complete removal of organic molecules and the development of the NiO/ Al_2O_3 / B_2O_3 / SiO_2 composite fibres [2- 9].

E. Transmission electron microscopy (TEM)

The TEM photographs of the nickel alumina borosilicate composite fibres were shown in Fig. 5. As shown in Fig. 5, after the applied thermal treatment, the nickel alumina borosilicate composite nanofibres demonstrated morphology. The average diameter of the nanosized crystals growth on the composite nanofibers is about smaller than 100 nm [9-10-11].



(a)



(b)

Figure 4. (a) SEM images of the NiO/Al₂O₃/B₂O₃/SiO₂ nanofibres (before the applied thermal treatment). (b) SEM images of the NiO/Al₂O₃/B₂O₃/SiO₂ nanofibres calcined at 750 °C.

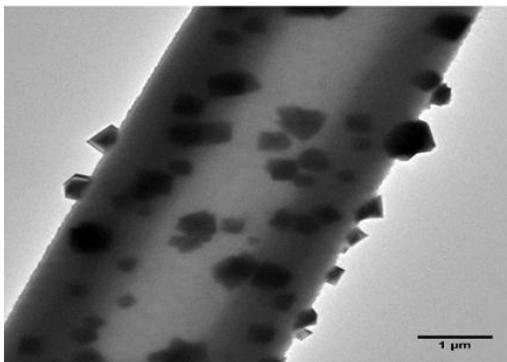


Figure 5. TEM images of the NiO/Al₂O₃/B₂O₃/SiO₂ nanofibres (before the applied thermal treatment)

IV. CONCLUSIONS

In conclusion, nanofibres of the nickel alumina borosilicate composite have been successfully prepared using the sol-gel processing and the electrospinning techniques. A sol-gel recipe that allows the formation of a homogeneous four component alkoxide solution and provides a control over solution viscosity for electrospinning process was developed. This situation was thought to be due to the complete removal of organic molecules and the development of the nickel alumina borosilicate composite fibers. The effect of the spinning distances was more pronounced at higher applied voltages. Increasing the applied voltage increases the surface charge of the jet and helps to reduce the frequency of occurrence of beads.

Borates are structurally analogous to silicates except for the modes of coordination. Silicates are coordinated to oxygen in a tetrahedral geometry while borates can coordinate to oxygen both in triangular and tetrahedral geometry. Nevertheless, it is still not easy to foretell which coordination of boron will dominate in a structure at environmental conditions. The side of the best of our works there is no presented on nickel alumina borosilicate composite although various morphologies of metal borates have been investigated.

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