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Preparation of Co_3O_4 Nanofibers *via* an Electrospinning Technique

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Abstract: Thin PVA/cobalt acetate composite fibers were prepared by using sol-gel processing and electrospinning technique. After calcination of the above precursor fibers, Co_3O_4 nanofibers with a diameter of 50-150 nm could be successfully obtained. The fibers were characterized by SEM, FT-IR, WAXD, respectively.

Keywords: PVA/cobalt acetate composite, Co_3O_4 nanofibers.

Co_3O_4-based materials are suitable candidates for the construction of solid-state sensors[1, 2], heterogeneous catalysts[3, 4], electrochromic devices[5], and solar energy absorbers[6, 7]. A few methods on the preparation of Co_3O_4 nanocrystalline powder, films and nanotube were reported[8~10]. However, to our knowledge, there have been no reports on the preparation of Co_3O_4 nanofibers. In this paper, we obtained the electrospun fibers of PVA/cobalt acetate composite by using sol-gel processing and electrospinning technique. And, the precursor fibers were calcined to get the Co_3O_4 nanofibers. The procedure was as follows. 20.0 g aqueous PVA solution of 10 wt% was dropped slowly into the solution of cobalt acetate (1.5 g $Co(CH_3COO)_2{\cdot}4H_2O$ and 2.0g H_2O), and the reaction proceeded in a water bath at 50 °C for 5 h. Thus, a viscous gel of PVA/cobalt acetate composite was obtained. Then it was contained in a plastic capillary. A copper pin connected to a high-voltage generator was placed in the solution, and the solution was kept in the capillary by adjusting the angle between capillary and the fixed bar. A grounded iron drum, sprayed with an aluminium foil, served as counter electrode. A voltage of 20 kV was applied to the solution and a dense web of fibers was collected on the aluminium foil. The fibers thus formed were dried initially at 70 °C for 12 h under vacuum, and then calcined at 800 °C at a rising rate of 240 °C h^{-1} and remained 10 h at the required temperature.

IR results showed that all the organic groups could be removed completely from PVA/cobalt acetate composite fibers after calcination at 800 °C, two peaks around 667 cm^{-1} and 577 cm^{-1} assigned to $\nu_{Co\text{-}O}$ of Co_3O_4[11, 12] appeared, indicating that the fibers

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Figure 1 WAXD results for (a) PVA/cobalt acetate composite fibers; (b) calcination at 800 °C.

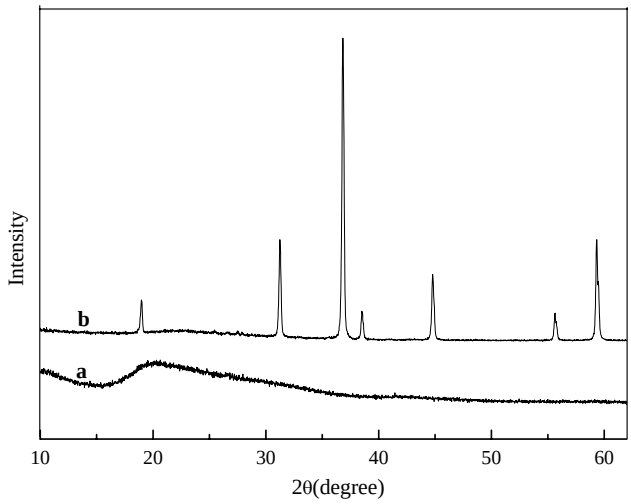
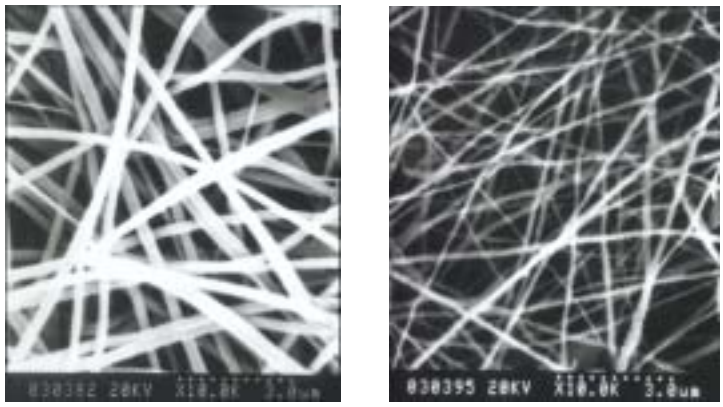


Figure 2 SEM of various fibers



(a) PVA/cobalt acetate fibers (b) calcination at 800 °C

obtained at this temperature were pure inorganic Co_3O_4 species. The WAXD results in the next section also supported this suggestion.

Figure 1 gave the WAXD curve for various fibers samples. As showed in **Figure 1(a)**, there existed a broad peak around $2\theta = 20°$, corresponding to the (101) plane of PVA semi-crystalline[13] in PVA/cobalt acetate composite fibers. This result indicated that the crystallinity of PVA was largely influenced by the presence of cobalt acetate in the PVA/cobalt acetate composite fibers, saying that there might be some interaction

between PVA and cobalt acetate molecules. Notably, after the PVA/cobalt acetate composites fibers were calcined at 800 °C (**Figure 1(b)**), crystalline peak of PVA disappeared, and eight reflection peaks appeared at 2θ = 19.0°, 31.3°, 36.8°, 38.6°, 44.8°, 55.8°, 59.3°, 65.2°, respectively, corresponding to the pure Co_3O_4 crystalline with cubic phase[14]. Comparing with the IR results, the products obtained at 800°C were pure Co_3O_4 fibers.

The SEM photographs of PVA/cobalt acetate composite fibers and the fibers calcined at 800 °C were showed in **Figure 2**. It could be seen that nanofibers of Co_3O_4, with alveolate surface and diameters of 50-150 nm, were obtained after calcining the PVA/cobalt acetate composite fibers at 800 °C. Meanwhile, due to the removal of PVA and the CH_3COO group of cobalt acetate molecule, the diameters of the fibers calcined at 800 °C (**Figure 2 (b)**) become smaller than which were not calcined (**Figure 2 (a)**).

For the first time, nanofibers of Co_3O_4 phase, with diameters of 50-150 nm, were prepared by using the electrospun thin fibers of PVA/cobalt acetate composites as precursor and through calcinations treatment. A new method for making nanofibers of inorganic materials might be provided. By modifying the parameters of sol-gel or electrospinning processing, one could also expect to be able to make nanofibers of inorganic materials with smaller diameter.

Acknowledgments

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