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# Preparation and characterization of NiO nanofibres via an electrospinning technique

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**Abstract**

Thin PVA/nickel acetate composite fibres were prepared by using sol–gel processing and electrospinning technique. After calcinations of the above precursor fibres, NiO nanofibres with a diameter of 50–150 nm could be successfully obtained. The fibres were characterized by SEM, FT-IR, WAXD, respectively. The results showed that the crystalline phase and morphology of NiO fibres were largely influenced by the calcination temperature.


*Keywords:* NiO nanofibres; PVA/nickel acetate composite; Electrospinning; PVA

In recent years, nanostructured materials have been actively studied due to both scientific interests and potential applications [1,2]. Among them, much attention has been focused on the research field of one-dimensional nanostructured materials, such as nanorods, nanowires, or nanofibres, because of their potential applications in nanodevices [3–5]. Nickel oxide (NiO) is a very important material extensively used in catalysis [6], battery cathodes [7,8], gas sensors [9], electrochromic films [10,11], and magnetic materials [12,13]. A few methods on the preparation of NiO nanocrystalline powder and films were reported [14–16]. However, to our knowledge, there have been no reports on the preparation of NiO nanofibres.

In this paper, we obtained the electrospun fibres of PVA/nickel acetate composite by using sol–gel processing and electrospinning technique. And, then got the NiO nanofibres by calcinations of the precursor fibres. The procedure was as follows. Fifteen grams aqueous PVA (Mn 80,000) solution of 10 wt% was dropped slowly into the solution of nickel acetate (1 g $Ni(CH_3COO)_2 \cdot 4H_2O$ and 4.0 g $H_2O$), and the reaction proceeded in a water bath at 50 °C for 5 h. A viscous gel of PVA/nickel 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 fixing bar. A grounded iron drum, covered with an aluminium foil, served as counter electrode. A voltage of 20 kV was applied to the solution and a dense web of fibres was

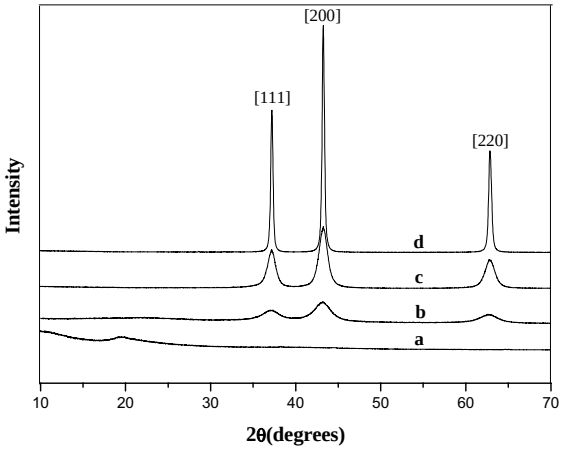


Fig. 1. WAXD results for various fibre samples: (a) PVA/nickel acetate composite fibres; (b) calcinations at 400 °C; (c) calcinations at 550 °C; (d) calcinations at 700 °C.

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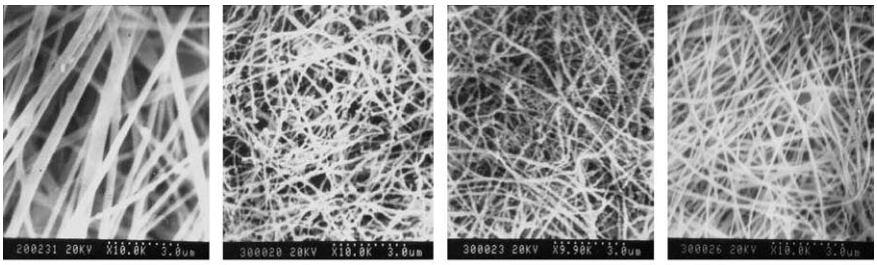


Fig. 2. Scanning electron microscopy photographs of various fibre samples: (a) PVA/nickel acetate composite fibres; (b) calcinations at 400 °C; (c) calcinations at 550 °C; (d) calcinations at 700 °C.

collected on the aluminium foil. The fibres thus formed were dried initially 12 h at 70 °C under vacuum, and then calcined at 400–700 °C at a rate of 240 °C $h^{-1}$ and remained 10 h at the required temperature.

IR results showed that all the organic molecules could be removed completely from PVA/nickel acetate composite fibres after calcination at 700 °C, and a new peak around 442 $cm^{-1}$ assigned to $\nu_{Ni-O}$ of nickel oxide (NiO) appeared [17,18], indicating that the fibres obtained at this temperature were pure inorganic NiO species. Fig. 1 gave the WAXD curve for various fibres samples. As showed in Fig. 1(a), there existed a broad peak around $2\theta = 20°$, corresponding to the (1 0 1) plane of semi-crystalline PVA [19] in PVA/nickel acetate composite fibres.

However, after the PVA/nickel acetate composite fibres were calcined at 400 °C (Fig. 1(b)), crystalline peak of PVA disappeared, and three broad reflection peaks corresponding to pure cubic NiO [16] appeared at $2\theta = 37.2°$ (1 1 1), 43.2° (2 0 0), 62.8° (2 2 0). Notably, when increasing calcination temperature to 550 and 700 °C (Fig. 1(c) and (d)), all the peaks belong to NiO cubic phase were markedly sharpening with high intensity, which suggested that the crystallinity of NiO phase was higher at high calcination temperature than which obtained at low calcination temperature. As compared with the IR results, the products obtained at 700 °C were pure NiO fibres. The SEM photographs of PVA/nickel acetate composite fibres and the fibres calcined at 400–700 °C were showed in Fig. 2. It can been seen that the surface of PVA/nickel acetate composite fibres (Fig. 2(a)) was smooth due to the amorphous nature of PVA and nickel acetate composites. On calcinations at 400 and 550 °C (Fig. 2(b) and (c)), due to the decomposition of PVA and the development of NiO crystalline, the diameters of fibres became smaller, and the surface became rougher than samples that were not calcined. After increasing the calcination temperature to 700 °C (Fig. 2(d)), nanofibres of NiO, with smooth surface and diameters of 50–150 nm, were prepared.

For the first time, NiO nanofibres, with diameters of 50–150 nm, were prepared by using the electrospun thin fibres of PVA/nickel acetate composites as precursor and through calcinations treatment. This route might open a new door to making nanofibres of inorganic materials. By modifying the parameters of sol–gel or electrospinning processing, one could also expect to be able to make nanofibres of inorganic materials with smaller diameter.

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