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A Novel Method for Making 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 calcination 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.

Keywords: PVA/nickel acetate composite, NiO nanofibres, PVA.

In recent years, nanostructural 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 nanostructural materials, such as nanorods, nanowires, or nanofibres, because of their potential applications in nanodevices[3, 4, 5]. Nickel oxide (NiO) is a very prosperous material extensively used in catalysis[6], battery cathodes[7], gas sensors[8], electrochromic films[9], and magnetic materials[10]. A few methods on the preparation of NiO nanocrystalline powder and films were reported[11~13]. 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, the NiO nanofibres was obtained by calcination of the precursor fibres. The procedure was as follows. 15.0 g aqueous PVA (Mn 80,000) solution of 10 wt% was dropped slowly into the solution of nickel acetate (1.0 g $Ni(CH_3COO)_2 \cdot 4H_2O$ and 2.0 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, sprayed 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 collected on the aluminium foil. The fibres thus formed were dried initially 12h at 70°C

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nder 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 ν_{Ni-O} of nickel oxide (NiO) appeared[14, 15], indicating that the fibres obtained at this temperature were pure inorganic NiO species. The WAXD results in the next section also supported this suggestion.

Figure 1 gave the WAXD curve for various fibres samples. As showed in **Figure 1(a)**, a broad peak around 2θ=20° corresponded to the (101) plane of PVA semi-crystalline[16] in PVA/nickel acetate composite fibres. This result indicated that the crystallinity of PVA was largely influenced by the presence of nickel acetate in the PVA/nickel acetate composite fibres, saying that there might be some interaction between PVA and nickel acetate molecules. Notably, after the PVA/nickel acetate composite fibres were calcined at 700°C (**Figure 1(b)**), crystalline peak of PVA disappeared, and three reflection peaks appeared at 2θ = 37.2° (111), 2θ = 43.2° (200), 2θ = 62.8° (220), corresponding to the pure NiO crystalline with cubic phase[13]. 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 700°C were showed in **Figure 2**. It could be seen that nanofibres of NiO, with smooth surface and diameters of 50-150 nm, were obtained after calcining the PVA/nickel acetate composite fibres at 700°C. Meanwhile, due to the removal of PVA and the CH_3COO group of nickel acetate molecule, the diameters of the fibres calcined at 700°C (**Figure 2 (b)**) become smaller than which were not calcined (**Figure 2 (a)**).

For the first time, nanofibres of NiO phase, 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 make 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.

Figure 1 WAXD results for (a) PVA/nickel acetate composite fibres; (b) calcination at 700°C.

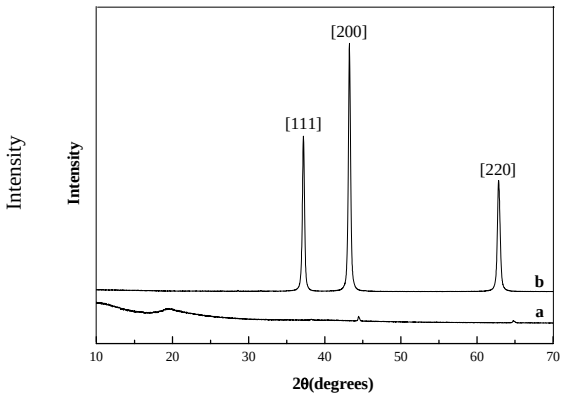
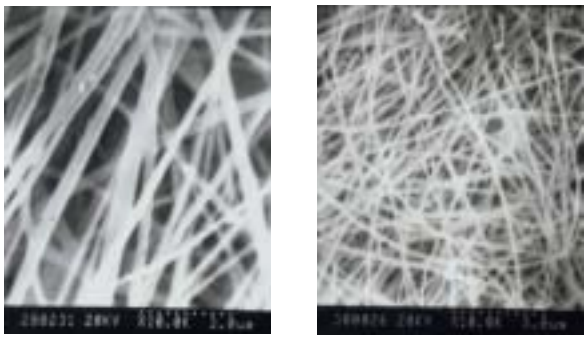


Figure 2 SEM of various fibre samples



(a) PVA/nickel acetate fibres (b) calcination at 700°C

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References

1. R. E. Cavicchi, R. H. Silsbe, *Phys. Rev. Lett.*, **1984**, *52* (16), 1435.
2. P. Ball, G. Li, *Nature*, **1992**, *355*, 761.
3. J. Hu, M.Y. Ouyang, C. M. Lieber, *Nature*, **1999**, *399*, 48.
4. D. S. Xu, G. L. Guo, L. L Gui., Y. Q. Tang, E. J. Shi, E. X. Jin, *App. Phys. Lett.*, **1999**, *75*, 481.
5. J. R. Heath, P. J. Kuekes, Snyderg, R. S. Williams, *Science*, **1998**, *280*, 717.
6. D. Levin, J. Y. Ying, *Stud. Surf. Sci. Catal.*, **1997**, *110*, 367.
7. H. X. Yang, D. F. Dong, X. H. Hu, *J. Power Sources*, **1999**, *79* (2), 256.
8. T. Hidehiro, *Patent J P 09263444 7*, **1997**, *10*, 5.
9. Y. Wu, G. Wu, X. Ni, *J. Vac. Sci. Technol.*, **1999**, *19* (3), 228.
10. Y. Wang , J. Ke, *High Technol. Lett.*, **1997**, *3* (1), 92.
11. J. Estelle, P. Salagre, Y. Cesteros, et al., *Solid State Ionics*, **2003**, *156*, 233.
12. S. Y. Wang, W. Wang, W. Z. Wang, Y. W. Du, *Mater. Sci. and Eng.*, **2002**, *1390***,** 133.
13. L. Xiang, X. Y. Deng, Y. Jin, *Scripta Materialia*, **2002**, *47***,** 219.
14. P. S. Patil, L. D. Kadam, *Appl. Surf. Sci.*, **2002**, *199*, 211.
15. R. C. Korosec, P. Bukovec, *Journal of Thermal Analysis and Calorimetry*, **1999**, *56*, 587.
16. Y. Nishio, R. S. Manley, *Macromolecules*, **1998**, *21*, 1270.

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