# MgO nanofibres *via* an electrospinning technique

CHANGLU SHAO*, HONGYU GUAN, YICHUN LIU
*Center for Advanced Optoelectronic Functional Materials Research, Northeast Normal University, People's Republic of China*
*E-mail: changlushao@yahoo.com.cn*

RIXIANG MU
*Nanoscale Materials and Sensors, Department of Physics, Fisk University, Nashville, TN, USA*

**Published online:** 10 April 2006

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


## 1. Introduction

One-dimensional (1D) nanostructures of metal oxides have been recently become the subject of extensive research due to their potential application of functional components for the fabrication of nanoscale electronic, optical, optoelectronic, electromechanical, and sensing devices [1, 2]. In order to obtain these materials, various preparation methods have been developed including arc discharge [3], laser ablation [4], template [5, 6], precursor thermal decomposition [7], and other methods [8–10]. Recently, we found a new method to produce inorganic nanofibres by using electronspun fibres of polymer/inorganic composite as the precursor [11, 12]. This processing involved the following steps: (1) Preparation of a sol with suitable inorganic precursor and polymer content, and achieving the right rheology for electronspinning. (2) Spinning of the solution to obtain fibres of polymer/inorganic composite. (3) Calcinations of the composite fibres to obtain final oxide fibres. It is important, however, to control all of the above three stages in order to obtain high quality fibres with the desired final properties. On the other hand, magnesium oxide (MgO) has been developed as an intensifier used in superconductive and spaceflight composite materials, since these materials show some superior properties such as high melting point, low density and high modulus of rupture [13–15]. Several studies have been previously reported on the preparation of nanostructures and MgO, such as MgO nanowires [16], MgO nanorods [17], MgO fishbone fractal nanostructures [18], MgO nanobelts [19]. In this paper, we report a novel and simple approach to large-scale fabrication of MgO nanofibres by calcination of the electrospun fibres of PVA/magnesium acetate composite.

## 2. Experimental

### 2.1. Materials

PVA (Mn 80000) was supplied by Shanxi Chemical Co., Ltd. Analytical-grade magnesium acetate ($Mg(CH_3COO)_2{\cdot}4H_2O$) was obtained from Aldrich. Distilled water was used as a solvent.

### 2.2. Preparation of PVA/magnesium acetate composite gels

Aqueous PVA solution (about 10 wt%) was first prepared by dissolving PVA powder in distilled water and heating at 80°C with stirring for 2 h, then cooling to room temperature and stirring for 12 h. 20.0 g of prepared aqueous PVA solution (10 wt%) was added slowly into the solution of magnesium acetate (1.5 g $Mg(CH_3COO)_2{\cdot}4H_2O$ 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/magnesium acetate composite was obtained.

### 2.3. Preparation of nanofibers

The viscous solution of PVA/magnesium acetate composites was taken in a plastic capillary. A copper pin connected to a high-voltage generator was placed in the

*Author to whom all correspondence should be addressed.
0022-2461 
DOI: 10.1007/s10853-005-5623-3

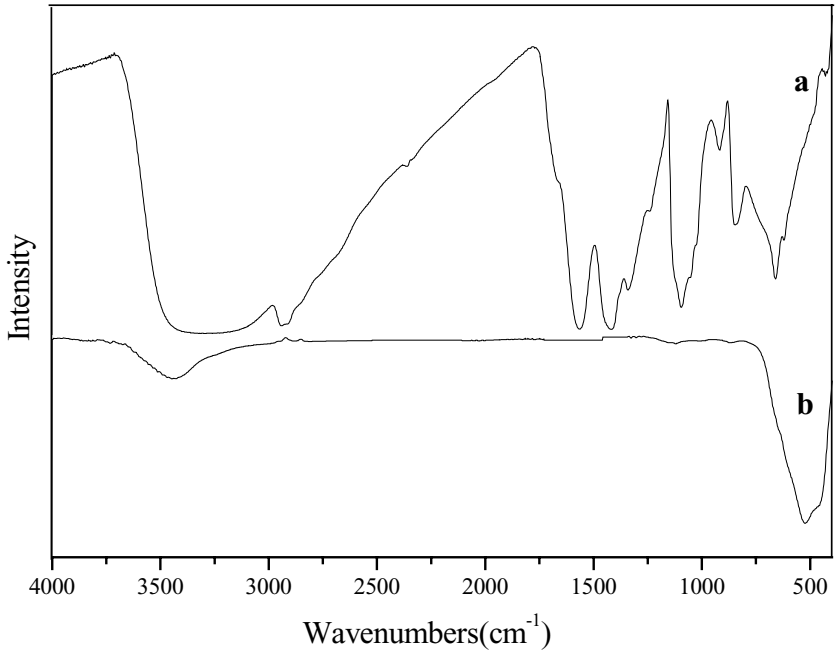


*Figure 1* IR spectra of (a) PVA/magnesium acetate composite fibres; (b) fibres after calcinations at 800°C.

solution, and the capillary was placed at an angle of around 20° to horizontal in order to get uniform flow of the solution. A grounded iron drum, covered with an aluminium foil, served as the counter electrode. A voltage of 18 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 129 h at 70°C under vacuum, and then calcined at 400–800°C at a rate of 240°C $h^{-1}$ and remained 10 h at the required temperature.

### 2.4. Characterization

For SEM investigation, a JSM-6700 F was used. XRD patterns of the samples were recorded by a Siemens D5005 Diffractometer, scans were made from 4° to 70° ($2\theta$) at the speed of 2° $min^{-1}$, Ni-filtered CuK$\alpha$ was used. IR spectra were obtained on Magna 560 FT-IR spectrometer with a resolution of 1 $cm^{-1}$.

## 3. Results and discussion

### 3.1. IR spectra

Fig. 1 gave the IR results of as-synthesized composite as well as calcined (at 800°C) fibres. As one could see that all the organic molecules could be removed completely from PVA/magnesium acetate composite fibres after calcination at 800°C, and a new peak around 525 $cm^{-1}$ assigned to $\nu_{Mg-O}$ of MgO [20] appeared after calcination at 800°C,

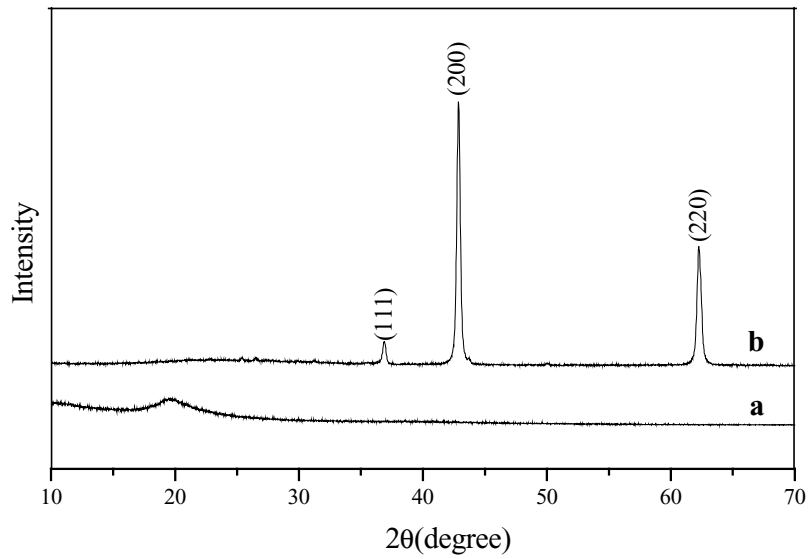


*Figure 2* XRD patterns of (a) PVA/magnesium acetate composite fibres; (b) fibres after calcinations at 800°C.

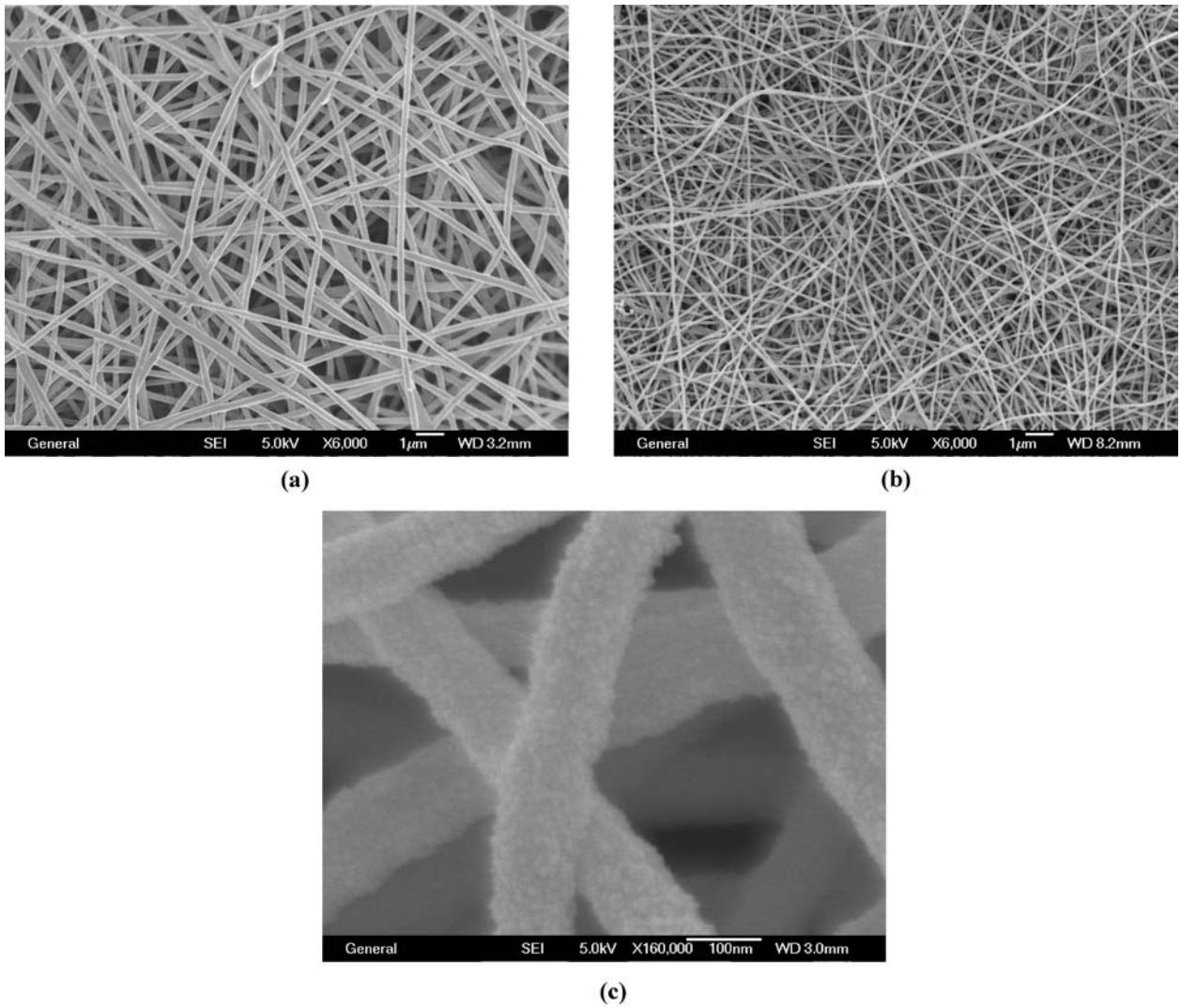


*Figure 3* Scanning electron microscopy images of (a) PVA/magnesium acetate composite fibres; (b) and (c) fibres after calcinations at 800°C.

indicating that the fibres obtained at this temperature were pure inorganic MgO species.

### 3.2. X-ray diffraction

Fig. 2 gave the XRD curves as-synthesized composite as well as calcined (at 800°C) fibres. In Fig. 2a, it was observed that a broad peak around $2\theta = 20^\circ$ corresponding to the (101) plane of semi-crystalline PVA [21] in PVA/magnesium acetate composite fibres appeared. This result indicated an amorphous property of the PVA/magnesium acetate composite fibres, suggesting that there might be some interaction between magnesium acetate and PVA in the composite fibres. Notably, after the PVA/magnesium acetate composite fibres were calcined at 800°C (Fig. 2b, crystalline peak of PVA disappeared, and three reflection peaks corresponding to the pure MgO crystalline [19, 22] appeared at $2\theta = 36.8^\circ$ (111), 42.8° (200), 62.2° (220), respectively. The results clearly showed that the calcined fibres were pure MgO nanofibres.

### 3.3. Scanning electron microscopy (SEM)

The SEM photographs of PVA/magnesium acetate composite fibres and the fibres calcined at 800°C were showed in Fig. 3. It could be seen that nanofibres of MgO, with rough surface and diameters of 50–150 nm, were obtained after calcining the PVA/magnesium acetate composite fibres at 800°C. Meanwhile, due to the removal of PVA molecule and acetate group, the diameters of the fibres calcined at 800°C Figs. 3b and c become smaller than that were not calcined (Fig. 3a).

## 4. Conclusion

For the first time, nanofibres of MgO, with diameters of 50–200 nm, were prepared by using the electrospun thin fibres of PVA/magnesium acetate composite as precursor and through calcination treatment. This simple approach should promise us some actual applications of one-dimensional MgO.

## Acknowledgments

The present work is supported financially by the Key Project of Chinese Ministry of Education (No. 104071) and the Project Sponsored by the Scientific Research Foundation for the Returned Overseas Chinese Scholars, State Education Ministry (No. 200314).

## References

1. M. H. HUANG, S. MAO, H. FEICK, H. YAN, Y. WU, H. KIND, E. WEBER, R. RUSSO and P. YANG, *Science* **292** (2001) 1897.
2. X. DUAN, Y. HUANG, Y. CUI, J. WANG and C. M. LIEBER, *Nature* **409** (2001) 66.
3. S. IIJIMA, *Nature* **354** (1991) 56.
4. A. M. MORALES and C. M. LIEBER, *Science* **279** (1998) 208.
5. C. R. MARTIN, *ibid.* **266** (1994) 1961.
6. W. HAN, S. FAN, Q. LI and Y. HU, *ibid.* **277** (1997) 1287.
7. Y. D. WANG, C. L. MA, X. D. SUN and H. D. LI, *Inorg. Chem. Commu.* **5** (2002) 751.
8. W. S. SHI, Y. F. ZHENG, N. WANG, C. S. LEE and S. T. LEE, *Adv. Mater.* **13** (2001) 591.
9. Z. W. PAN, Z. R. DAI and E. L. WANG, *Science* **291** (2001) 1947.
10. S. W. LIU, J. YUE and A. GEDANKE, *Adv. Mater.* **13** (2001) 656.
11. C. L. SHAO, H. Y. KIM, J. GONG and D. R. LEE, *Nanotechnology* **13** (2002) 635.
12. H. Q. DAI, J. GONG, H. Y. KIM and D. R. LEE, *ibid.* **13** (2002) 674.
13. D. F. HASSON, *J. Mater. Sci.* **20** (1985) 4147.
14. P. KONDILIS, G. MOUSDIS and G. KORDAS, *Physica C* **235–240** (1994) 467.
15. Y. S. YUAN, M. S. WONG and S. S. WONG, *ibid.* **250** (1995) 247.
16. Y. D. YIN, G. T. ZHANG and Y. N. XIA, *Adv. Funct. Mater.* **12**(4) (2002) 293.
17. Z. CUI, G. W. MENG, W. D. HUANG, G. Z. WANG and L. D. ZHANG, *Mater. Res. Bull.* **35** (2000) 1653.
18. Y. Q. ZHU, W. K. HSU, W. Z. ZHOU, M. TERRONES, H. W. KROTO and D. R. M. WALTON, *Chem. Phys. Lett.* **347** (2001) 337.
19. J. ZHANG and L. D. ZHANG, *Chem. Phys. Lett.* **363** (2002) 293.
20. *SADTLER RESEARCH LABORATORIES, INC. U.S.A., Inorganics Grating Spectra Y* 89K (1965).
21. Y. NISHIO and R. S. MANLEY, *Macromolecules* **21** (1988) 1270.
22. Z. Q. WEI, H. QI, P. H. MA and J. Q. BAO, *Inorg. Chem. Commu.* **5** (2002) 147.

*Received 2 October*
*and accepted 22 July 2005*