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Chemical Microanalysis of SnO_2–MnO_2 Nanofibers in an Electron Probe Microanalyzer

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Tin dioxide (SnO_2) is an N-type semiconductor for a wide range of applications [1]. Hierarchical SnO_2 nanocrystals, in the forms of nanoparticles, nanotubes or nanosheets, have been used for gas sensors [1, 2], supercapacitors [3], lithium-ion batteries [1], and solar cells [1]. With the addition of MnO_2, it was found that SnO_2–MnO_2 nanocomposite powders exhibited remarkable improvement in electrochemical performance of in terms of discharge capacity and low capacity fading, compared with pristine MnO_2 [4]. SnO_2–MnO_2–SnO_2 sandwich-structured structure was also demonstrated to be a high-performance anode in a lithium-ion battery [5]. In this work, we synthesized SnO_2, MnO_2 and mixed SnO_2–MnO_2 nanofibers and conducted microanalysis using an electron probe microanalyzer (EPMA).

Polymer solutions were prepared by dissolving 2.0 g polyvinylpyrrolidone (PVP) (molecular weight ~1,300,000 g/mol) in a 20 mL (50/50) mixture of N,N-dimethylformamide (DMF) with ethanol. Tin chloride ($SnCl_2$), or manganese chloride ($MnCl_2$) or a mixture of both, was added to the polymer solution. The electrospinning was conducted at room temperature with applied voltage of 18 kV. The prepared nanofibers were subsequently calcined at 500 °C in air for 5 h to obtain oxide nanofibers. Samples were coated with carbon and analyzed in a JEOL field-emission JXA-8530F EPMA, which was equipped with a SDD X-ray energy-dispersive spectrometer (EDS) and five wavelength-dispersive spectrometers (WDSs), and xCLent IV Hyperspectral Cathodoluminescence System, worked at 10 kV.

Fig. 1(a) shows an SEM image of SnO_2 nanofibers after calcination. The nanofibers become porous, with a diameter of ~150 nm. After calcination, the fibers are in fact composed of small nanoparticles (~50 nm). The EDS analysis, as shown in Fig. 1(b), confirms that its composition is almost stoichiometry of SnO_2. The MnO_2 nanofibers, on the other hand, are composed of large nanoparticles (~150 nm) in the form of chains. Since the melting point of SnO_2 is 1,630 °C while the melting point of MnO_2 is only 535 °C, the MnO_2 nanofibers were melted during the calcination. The EDS spectrum of MnO_2 is shown in Fig. 1(d), with a stoichiometric composition of MnO_2.

The image of mixed SnO_2–MnO_2 nanofibers is shown in Fig. 2(a), with larger diameter (~300 nm) in a porous shape. As shown in Fig. 2(b) from the EDS analysis, the Mn content is very low although the starting molar ratio of Sn:Mn is 1:1, indicating the melting and volatilization of MnO_2 from the mixture during the calcination [6].

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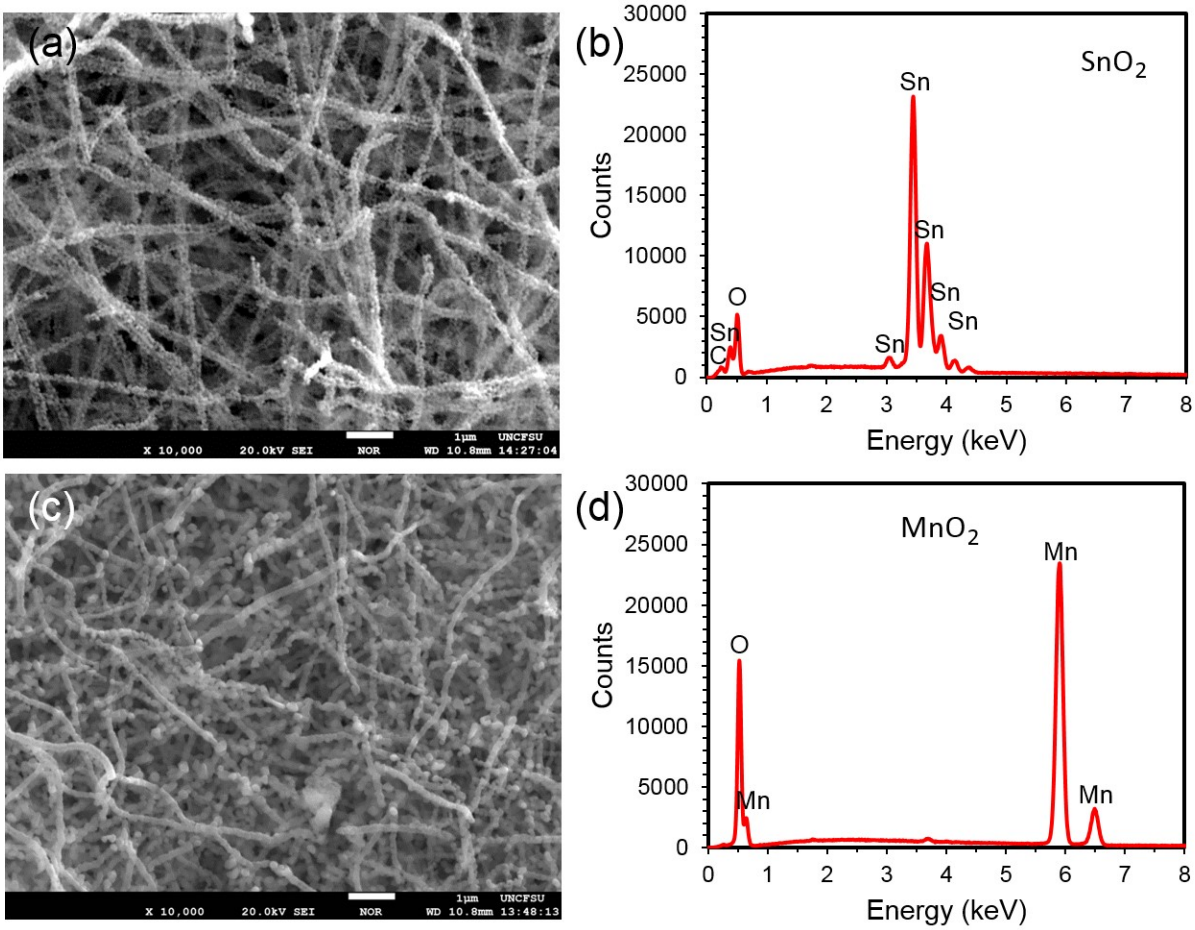

Figure 1. (a, b) SEM image of SnO_2 nanofibers and EDS spectrum; (c, d) SEM image of MnO_2 nanofibers and EDS spectrum.

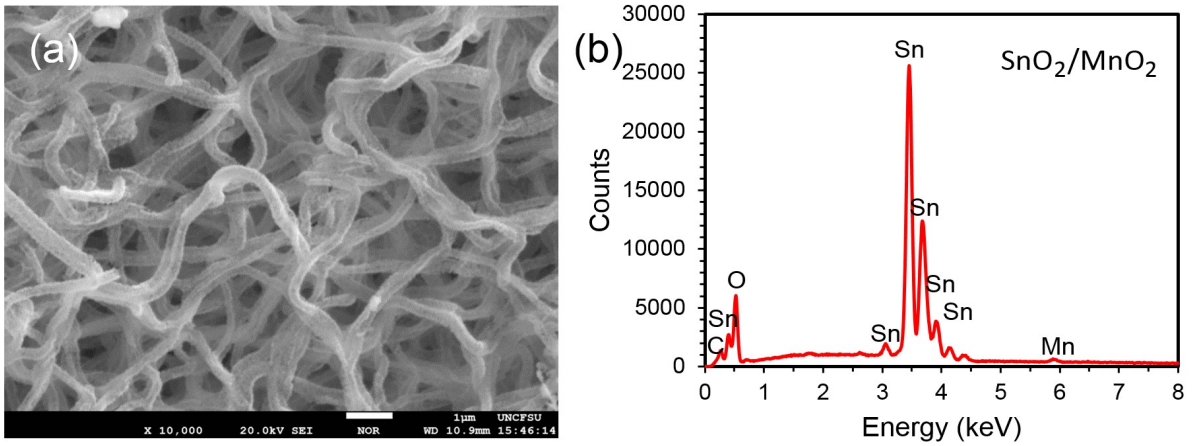

Figure 2. (a) SEM image of SnO_2–MnO_2 nanofibers; (b) EDS spectrum.

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