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Electron Probe Microanalysis of Electrospun Nd_2O_3 Nanofibers Doped with Ce/Zn

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Nanoscale one-dimensional (1D) oxides have exhibited enhanced physical and chemical properties for a wide range of applications, such as lithium-ion-battery electrodes, chemical gas sensors, filters, surface coatings, and biomedical applications. The compound Nd_2O_3 has been used to improve electrical properties of materials and for photocatalytic applications. In order to study the Nd_2O_3 properties at the nanoscale, Nd_2O_3 nanoparticles have been synthesized by a number of different routes [1]. Recently, nanorods have been synthesized by a hydrothermal method [2]. In this work, we report long Nd_2O_3 nanofibers by an electrospinning method, and conduct chemical analysis using an electron probe microanalyzer (EPMA).

Polymer solutions were prepared by dissolving 6.0 g polyvinylpyrrolidone (PVP) (molecular weight ~40,000 g/mol) in a mixture of N,N-dimethylformamide (DMF) with ethanol. Neodymium (III) nitrate hexahydrate, cerium (III) nitrate hexahydrate and zinc acetate dihydrate were 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 700 °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), worked at 10 kV.

Fig. 1(a) shows an image of the Nd_2O_3 nanofibers, with an average diameter less than 100 nm. The EDS spectrum confirms the Nd_2O_3 compositions, as shown in Fig. 1(b). The C K_α line is from the sample coating. No other impurities are observed. In the samples doped with Ce or Zn, the EDS spectra display addition of Ce or Zn, as shown in Fig. 1(c) and (d), respectively. Note that in the EDS spectrum in Fig. 1(c), the Nd L_α line (5.229 keV) and Ce L_β line (5.261 keV) are overlapped; and in Fig. 1(d), the Zn L_α (1.012 keV) and Zn L_β (1.034 keV) lines are overlapped, as indicated in a single arrows.

These elements are further analyzed by WDS. The WDS from Nd_2O_3 shows Nd only in the spectrum by LIFH crystal, as shown in Fig. 2(a). O and C are observed in the spectrum by LDE crystal, which is not shown here. However, in the Nd_2O_3/Ce sample, isolated Ce peaks appear, as shown in Fig. 2(b). Noticeably, the Nd L_α and Ce L_β lines, which are overlapped in EDS in Fig. 1(c), are well separated in the WDS spectrum, as indicated by double arrows in Fig. 2(b). In the Nd_2O_3/Zn sample, Zn L_α and Zn L_β lines, which are overlapped in EDS in Fig. 1(d), are also well separated, as indicated by double arrows in Fig. 2(c). Both EDS and WDS are complementary methods in the chemical analysis [3].

References

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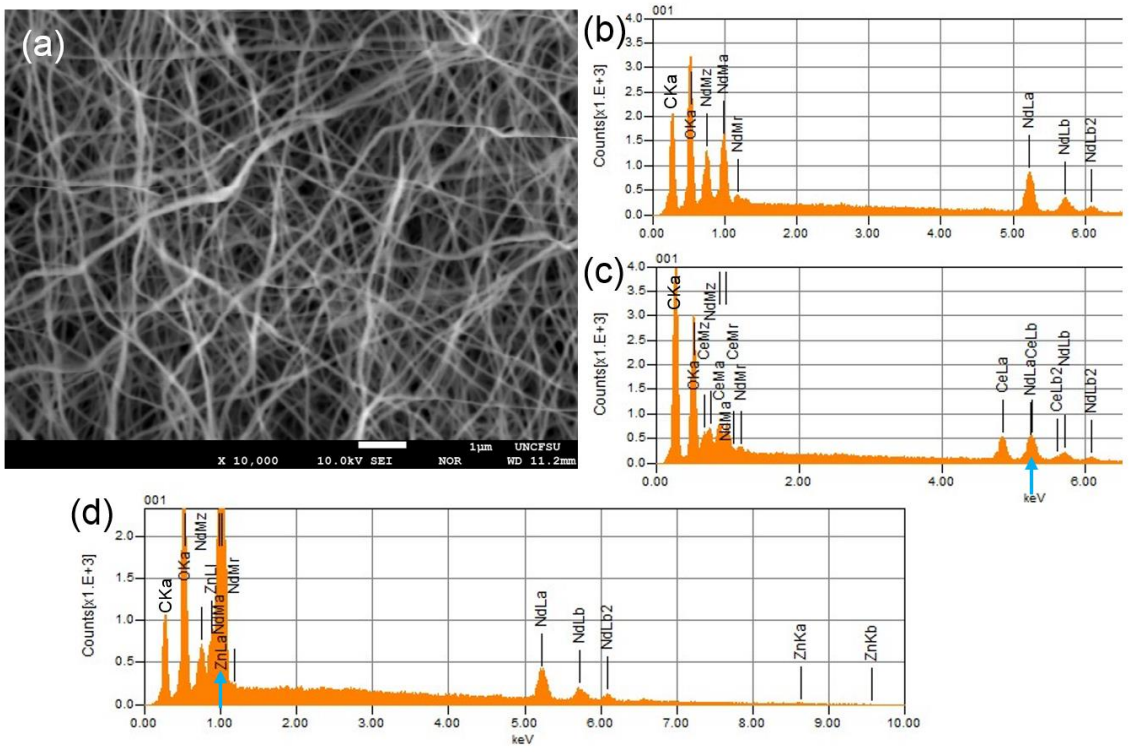

Figure 1. (a) SEM image of Nd_2O_3 nanofibers; (b–d) EDS spectra from Nd_2O_3, Nd_2O_3 doped with Ce, and Nd_2O_3 doped with Zn, respectively. Arrows indicate overlapped lines.

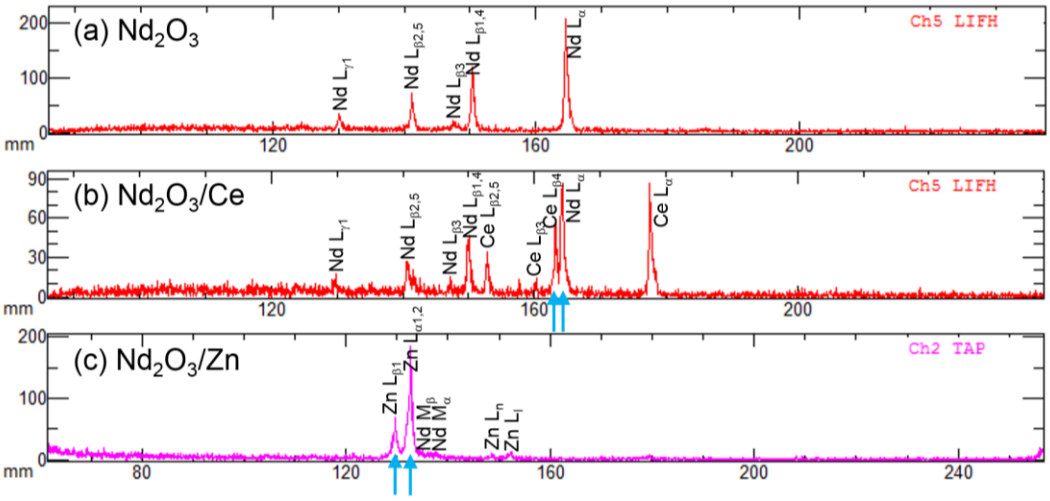

Figure 2. WDS spectra from Nd_2O_3 (a), Nd_2O_3 doped with Ce (b), and Nd_2O_3 doped with Zn (c). Double arrows in (b) and (c) indicate separated lines that are overlapped in the EDS spectra in Fig. 1 (c) and (d), respectively.

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