Electrospun Rare-Earth Orthovanadate Scintillator Nanofibers Analyzed by EPMA

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Luminescent rare-earth (R) orthovanadates (RVO₄) fall into the mineral group wakefieldite, crystallizing in zircon type (I₄/amd) structure which consists of VO₄ tetrahedra sharing corners and edges with RO₈ dodecahedra [1]. The rare-earth orthovanadates are recognized widely as optical maser materials. Moreover, they are identified as potential candidates for optical sensing [2], photocatalytic degradation of textile effluents [3], hosts for luminescent ions [4], battery electrodes [5], etc. The plausible applications of several orthovanadates are not addressed very well in the literature. The hightemperature stability and the relatively high concentration of rare-earth ions in the rare-earth orthovanadates make them ideal candidates for scintillator materials [6-8]. Large RVO₄ crystals are commercially used as a promising candidate for magneto-optical applications in visible and near-infrared regions [9]; however, the nanosized counterparts of RVO₄ are multifunctional with magnetic, photocatalytic and antibacterial properties [10]. In this work, we fabricated nanosized orthovanadate fibers using electrospinning method and confirmed the morphology and composition using an Electron Probe Microanalyzer (EPMA).

0.07 M ammonium metavanadate and 0.07 M terbium (III) nitrate were dissolved in a 20 mL 20/40/40 mixture of H₂O/Ethanol/N,N-Dimethylformamide to prepare the electrospinnable precursor solution. Then 3.0 g of polyvinylpyrrolidone (PVP) (molecular weight ~1,200,000 g/mol) was added to the above solution and stirred vigorously for 12 h to ensure uniform mixing. The electrospinning was conducted at atmospheric conditions with an applied voltage of 18 kV, a flow rate of 300 μL/h, and a spinneret to collector distance of 17 cm. The xerogel fibers obtained through electrospinning process were subsequently calcined at 700 °C in air for 5 h at a ramp of 1 °C/min to obtain the orthovanadate nanofibers. Samples were coated with carbon and analyzed in a JEOL field-emission JXA-8530F EPMA, which was equipped with an SDD X-ray energy-dispersive spectrometer (EDS), and five wavelength-dispersive spectrometers (WDSs) worked at 15 kV.

The SEM images of RVO₄ nanofibers after calcination are shown in Fig. 1 (a, b). The observed size of the RVO₄ fibers are <150 nm. The diameter of the fibers is significantly reduced upon calcination as compared to xerogel precursor fibers. The EDS analysis, as shown in Fig. 1(c), confirms that the composition of the obtained fibers has the significant peaks arising from Tb, V and O. The composition is also validated with WDS analysis, which clearly reveals the stoichiometric composition to that of anticipated RVO₄ [11].

References:

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Figure 1. (a) Low and (b) high magnification SEM images of electrospun RVO$_4$ scintillator nanofibers, and (c) the corresponding EDS spectrum.

Figure 2. WDS spectra of RVO$_4$ nanofibers obtained using different crystals.