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Cathodoluminescence and EPMA Analysis of Alkaline Earth Fluoride Nanowires

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Alkaline earth fluorides are widely recognized as the hosts for the fabrication of inorganic scintillators and phosphors, due to favorable properties such as chemical inertness, low lattice phonon energy, etc. [1-3]. Mn^{4+}-doped barium hexafluorometallates, $BaXF_6$ (X = Si, Ge, Ti, or Sn) can be potentially used in commercial LEDs in order to convert the UV-blue emissions from the conventional white light emitting LEDs to red emissions to reduce the health-related impacts that may arise after prolonged exposure UV-blue light [4]. These materials have excellent photoluminescence properties and can be useful for several applications such as color multiplexing [5], optical sensing [6], and temperature sensing [7]. Furthermore, when doped with rare-earth ions, these materials exhibit fast decay time. In this work, high aspect-ratio single-crystal $BaXF_6$ nanowires were synthesized by a solvothermal method and are analyzed using an Electron Probe Microanalyzer (EPMA).

The nanowires were synthesized using a solvothermal method in a solvent system comprising of cetyltrimethylammoniumbromide (CTAB)/water/cyclohexane/1-pentanol. Two identical solutions containing 25 mL cyclohexane, 2 g of CTAB, and 1 mL 1-pentanol were prepared first. 1 mL of 1 M of the respective tetravalent element in 20% HF was added dropwise at a rate of 2 mL h^{-1} to one of the above solutions and 1 mL of 1 M barium chloride ($BaCl_2$) solution was added to the other solution, under vigorous stirring at a speed of 700 rpm. 1 M solution of the tetrvalent metal in 20% HF was prepared by dissolving 0.9573 g the respective metal in 10 mL 20% hydrofluoric acid (HF) and 30% hydrogen peroxide was added dropwise to the solution under stirring until the solution becomes transparent. After 30 min of continuous stirring, the above two solutions were mixed and stirred for an additional 20 min at a rate of 1,000 rpm. The solution was then sealed in an autoclave reactor and heated at 120 °C for 12 h and the reactor was allowed to cool down naturally to room temperature. The nanowires separated from the solution obtained after hydrothermal treatment were subsequently washed using ethanol and water and then dried at 80 °C overnight. The nanowires were coated with approximately 10 nm of carbon before their analysis in a JEOL field-emission JXA-8530F EPMA, which was equipped with an SDD X-ray energy-dispersive spectrometer (EDS), five wavelength-dispersive spectrometers (WDSs), and xClent IV Hyperspectral cathodoluminescent (CL) system, worked at 15 kV.

Fig. 1a shows an SEM image of $BaXF_6$ nanowires obtained by the solvothermal method. These long nanowire diameter is measured 50-100 nm. The nanowires emit blue-green light under the electron beam irradiation as observed in the CL spectrum as in Fig. 1b. The compositional studies using EDS analysis (Fig. 1c) and WDS analysis (Fig. 2), confirms the stoichiometric composition of intended $BaXF_6$ nanowires, with significant emission, originated from the elements Ba, Ti and F [8].

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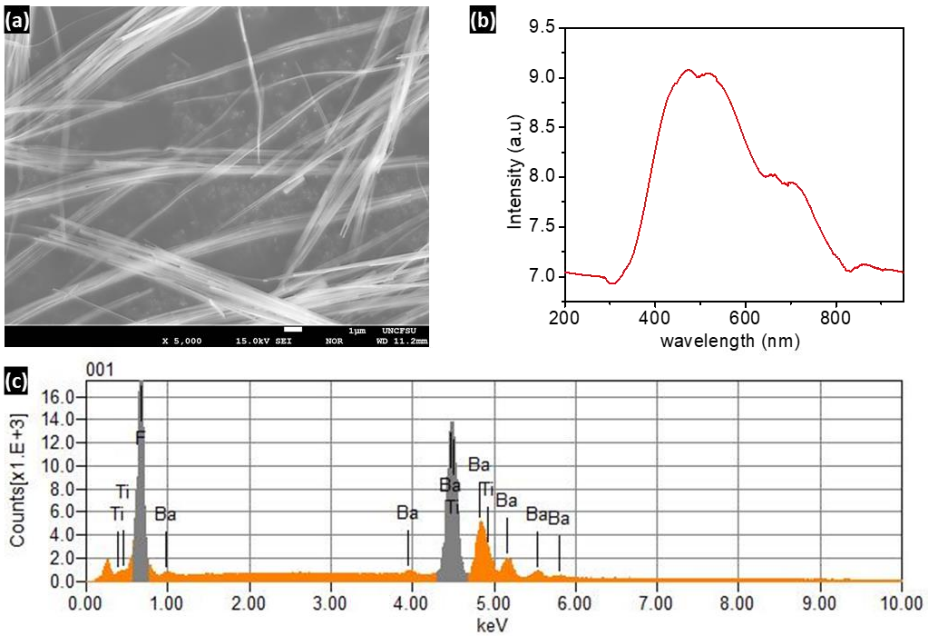

Figure 1. (a) SEM image, (b) CL spectrum, and (c) EDS spectrum of $BaXF_6$ scintillator nanowires.

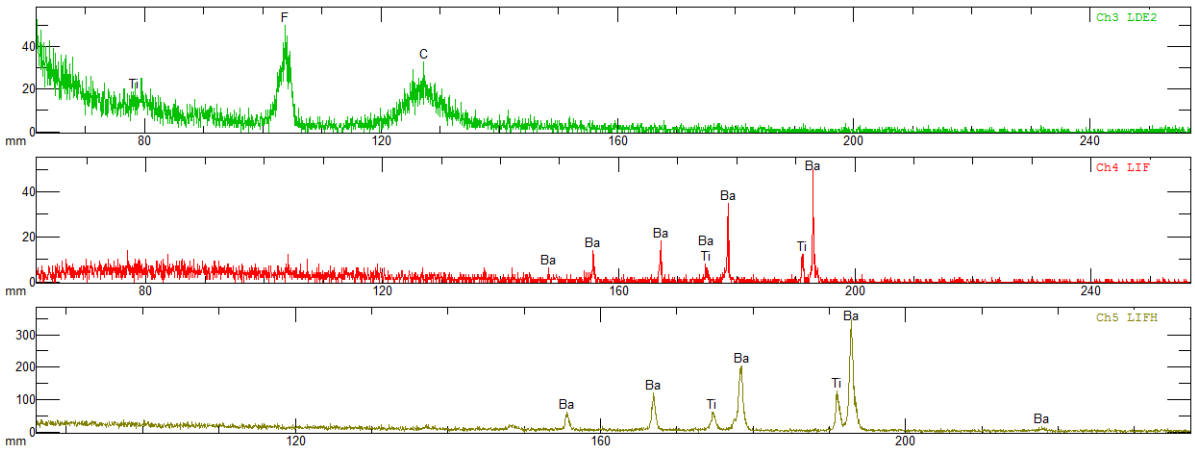

Figure 2. WDS spectra of the $BaXF_6$ nanowires obtained using different crystals.

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