Preparation and characterization of spinel type Zn$_2$TiO$_4$ nanocomposite

Alima Mebrek$^{a,b}$, Safia Alleg$^{a,*}$, Sihem Benayache$^{a,b}$, Mohamed Benabdeslem$^{c}$

$^a$ Laboratoire de Magnétisme et Spectroscopie des Solides (LM2S), Département de Physique, Université Badjı Mukhtar Annaba, B.P. 12, Annaba 23000, Algeria
$^b$ Centre de Recherche en Technologies Industrielles (CRTI), B.P. 64, Route de Dely Ibrahim, Chéraga, 16014 Alger, Algeria
$^c$ LESIMS, Département de Physique, Université Badjı Mukhtar Annaba, B.P. 12, Annaba 23000, Algeria

ABSTRACT

Zinc orthotitanate Zn$_2$TiO$_4$ spinel structures have been prepared by solid state reaction in two stages. First, a mixture of ZnO and TiO$_2$ (67% anatase + 33% rutile) in a molar ratio of 2:1 was mechanically milled for 6 and 18 h, at room temperature, in a high energy planetary ball mill under argon atmosphere. Next, the ball milled powders were calcined at 900°C for 2 h, pressed into pellets and then sintered for 4 h at 1100°C in air. Phase formation, microstructure, surface morphology and optical properties were characterized by X-ray diffraction, Raman scattering spectroscopy, Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy coupled with energy dispersive X-ray spectroscopy, atomic force microscopy and UV-visible spectrophotometry. The mechanical milling process for 6 h gives rise to the formation of nanocrystalline orthotitanate Zn$_2$TiO$_4$ (15.5%, \( < \lambda > = 13.2 \text{ nm} \)) in addition to unreacted rutile TiO$_2$, anatase TiO$_2$ and ZnO structures. As the milling process progresses up to 18 h, the volume fraction of Zn$_2$TiO$_4$ increases to about 44.5%. The sintered pellets exhibit a composite structure where about a small amount of rutile nanograins are dispersed into the Zn$_2$TiO$_4$ matrix. FT-IR and Raman results confirm the biphasic character of the sintered pellets. The band gap energy is milling time dependent. It varies from 3.22 for pellet 6 h to 3.45 for pellet 18 h.