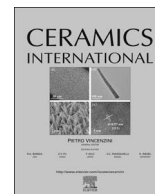




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Local structure, electrical and magnetic properties of Fe-doped $\text{Sr}_2(\text{Ni},\text{Mo})\text{O}_6$ double perovskite

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ABSTRACT

Double perovskite material of 10 mol% Fe-doped $\text{Sr}_2(\text{Ni},\text{Mo})\text{O}_6$ was prepared by solid-state reaction. The NiMoO_4 precursor was first synthesized as a starting material with calcination temperature at 1100 °C in air. Effects of FeO , Fe_2O_3 and Fe_3O_4 dopant on phase formation, morphology, electrical and magnetic properties of the specimens were investigated and discussed. The XRD technique was used to analyze the phase formation of the samples. It can be clearly seen that phase formation of pure $\text{Sr}_2(\text{Ni},\text{Mo})\text{O}_6$ indicated the cubic phase structure, while the tetragonal phase was found with FeO , Fe_2O_3 and Fe_3O_4 dopant. All XANES spectra were collected at the ambient temperature and atmospheric pressure. The result demonstrated that the oxidation state of Fe is 3^+ , indicating the occurrence of $\gamma\text{-Fe}_2\text{O}_3$ phase for all samples. In addition, the room temperature ferroelectric and ferromagnetic properties were determined using a simple Sawyer–Tower circuit (at fixed measuring frequency of 50 Hz) and Vibrating Sample Magnetometer (VSM), respectively. The maximum ferroelectric and ferromagnetic properties were seen with Fe_2O_3 dopant. Interestingly, the ferroelectric and ferromagnetic properties of $\text{Sr}_2(\text{Ni},\text{Mo})_{0.9}\text{Fe}_{0.1}\text{O}_6$ ceramics were affected by iron oxide doping.

1. Introduction

Recently, the promising double perovskite materials have been attracted considerable interest for many technological applications due to its good electronic conductivity, high catalytic activity and thermal stability [1,2]. Theoretically, double perovskites multiferroic materials have the general formula $A_2BB'O_6$, where A is typically a rare earth, alkali, or alkaline earth metal, while B and B' are typically transition metal. Normally, double perovskites materials such as $\text{Sr}_2\text{MnMoO}_6$ [3,4], $\text{Sr}_2\text{CoMoO}_6$ [5] and Sr_2NiWO_6 [6], show the magnetic and electrical properties. However, the crystal structure of double perovskites materials are known to competing bonding preferences of the A , B and B' species, is often unstable [7,8]. As well known, the crystal defects and the structural changes are often intimately linked to the physical, electrical and also magnetic properties of materials.

The double perovskite $\text{Sr}_2\text{NiMoO}_6$ was initially reported to have $I4/$

mmm tetragonal symmetry with cell parameters $a = 3.932 \text{ \AA}$, and $c = 3.943 \text{ \AA}$. The structural phase transition was reported at 503 K from tetragonal to a cubic phase with cell constant, $a = 7.878 \text{ \AA}$ [9]. Previous works by Gagulin et al. [5] studied on the conductivity and dielectric properties of $\text{Sr}_2\text{CoMoO}_6$ and $\text{Sr}_2\text{NiMoO}_6$ compounds, resulting in the semiconducting behavior with the conductivity characterization and their dielectric permittivity's show peaks attributable to phase transitions at 530 K and 463 K, respectively.

In our previous studies, the solid solution of $\text{Sr}_2\text{NiMoO}_6$ can be synthesized by both conventional and precursor routes. For conventional route, the starting materials of SrCO_3 , NiO and MoO_3 were mixed together and then calcined at 1300 °C in air. The phase structure of this route presented the tetragonal phase and their some electrical properties [7,8]. However, the impurity phase of SrMoO_4 ceramics is easily to found [7,8,10]. Therefore, the NiMoO_4 precursor reacts with SrCO_3 route is another way to clearly that impurity phase as well.

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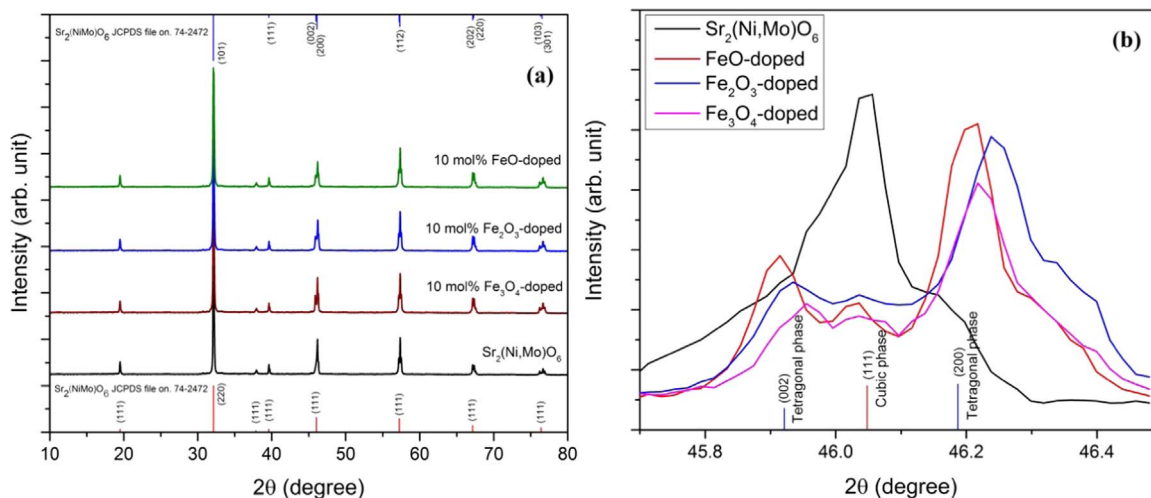


Fig. 1. (a) X-ray diffraction pattern of $\text{Sr}_2\text{NiMoO}_6$ ceramics with different iron oxide dopant for 10 mol% and (b) XRD pattern at $2\theta = 45.7 - 46.5$.

However, the disadvantage of $\text{Sr}_2\text{NiMoO}_6$ precursor has low electrical and magnetic properties due to the cubic phase structure was presented. In the search for structural and the electrical properties improvement, Kumar et al. were studied and concluded that the La^{3+} doping has effect on structural and the electrical of $\text{Sr}_2\text{NiMoO}_6$ compound. However, there has no systematic studied on other metal oxide dopant in $\text{Sr}_2\text{NiMoO}_6$ system. Therefore, the main purpose of this work is to investigate effects of different iron oxide family (FeO , Fe_2O_3 and Fe_3O_4) doping on phase formation, ferroelectric and ferromagnetic properties of ceramic in $\text{Sr}_2(\text{Ni},\text{Mo})_{0.9}\text{Fe}_{0.1}\text{O}_6$ system.

2. Material and methods

2.1. Samples preparation

Double perovskite ceramics in $\text{Sr}_2\text{NiMoO}_6$ system with different iron metal oxide dopant as FeO , Fe_2O_3 and Fe_3O_4 were fabricated and characterized. Raw materials of SrCO_3 , NiO , MoO_3 , FeO , Fe_2O_3 and Fe_3O_4 with > 99% purity were used to prepare samples. The NiMoO_4 precursor was first synthesized with calcination temperature of 1100°C for 2 h and introduced into the batch calculations with SrCO_3 according to $\text{Sr}_2\text{NiMoO}_6$ system by the solid-state reaction technique. After drying, all products were calcined in an alumina crucible at a temperature of 1300°C for 9 h with heating/cooling rate of $5^\circ\text{C}/\text{min}$. The calcined powders were uniaxial pressed at 300 psi into disc-shaped pellets with a diameter of 10 mm and a thickness of 1–2 mm, with 3 wt% polyvinyl alcohol (PVA) added as a binder. Following binder burnout at 500°C , the pellets were sintered at 1400°C for 2 h at a heating/cooling rate of $5^\circ\text{C}/\text{min}$.

2.2. Sample characterizations

The phase formation of the ceramics was analyzed by X-ray diffraction (XRD; Philips XPert Pro). Archimedes technique was used to measured density of the specimens. The microstructures of the sintered samples were examined using scanning electron microscopy (SEM; JEOL JSM-840A). The room temperature ferroelectric and ferromagnetic properties were determined using a simple Sawyer–Tower circuit (at fixed frequency of 50 Hz) and a Vibrating Sample Magnetometer (VSM), respectively. X-ray Absorption Near-Edge Structure (XANES) measurements were conducted at ambient temperature the X-ray absorption spectroscopy beamline (BL-8) of the Siam Photon Laboratory, Synchrotron Light Research Institute (SLRI), Thailand (electron energy of 1.2 eV, beam current 80–120 mA). The double-crystal monochromator was operated with a pair of Ge (220)

crystals for scanning the energy of the synchrotron X-ray beam with energy step 0.2 eV to excite the electrons in Fe K-edges. Standard Fe foil was used for photon energy calibration. The experiments were performed in transmission mode and the incident. The transmitted signals were recorded by the ionization chamber (filled with Ar gas). Finally, the data were processed using the ATHENA program.

3. Results and discussion

Phase formation behaviors of ceramics in $\text{Sr}_2(\text{Ni},\text{Mo})_{0.9}\text{Fe}_{0.1}\text{O}_6$ system with different iron oxide dopants are examined by the XRD technique, as shown in Fig. 1(a). The results show a single phase perovskite structure without the secondary phase for all samples. From the XRD patterns, the major cubic phase with $Fm\bar{3}m$ symmetry (JCPDS file No. 74-2472) was found for a pure $\text{Sr}_2\text{NiMoO}_6$ ceramics, corresponding to our previous work [8]. The crystal structure of $\text{Sr}_2\text{NiMoO}_6$ has been affected from the synthesis routes (conventional and precursor), that resulted in the cubic phase from NiMoO_4 precursor route and tetragonal phase from the conventional route. For the conventional route, the numerous of cations are competing in bonding as a result of reduce symmetry of the structure, the tetragonal phase was presented, whilst precursor route has more thermodynamically stable, cubic phase was presented [11].

However, the major tetragonal phases were presented with 10 mol % of FeO -, Fe_2O_3 -, and Fe_3O_4 -doped in $\text{Sr}_2\text{NiMoO}_6$ ceramics related to $P4mm$ symmetry (JCPDS file No. 74-2466). The samples did not show the secondary phase of iron oxide or iron compounds. It can be seen that a major effect of iron doping seems to be a change in crystal structure from cubic to tetragonal phase. For a clear evident, XRD peak profiles of the (002) and (200) peaks at $2\theta = 45.7 - 46.5$ are shown in Fig. 1(b). The results indicated the tetragonal phase for all iron dopants; the c/a ratio was used to reveal the tetragonal phase of the specimens, which have c/a ratio of 1.0051, 1.0055 and 1.0044 for FeO -, Fe_2O_3 -, and Fe_3O_4 -doped, respectively. This phenomenon indicates a reduction in the lattice constant with Fe doping, which is expected as smaller Fe ions are incorporated into the Ni or Mo sites of the $\text{Sr}_2\text{NiMoO}_6$ lattice ($\text{Fe}^{2+} = 0.75 \text{ \AA}$, $\text{Fe}^{3+} = 0.69 \text{ \AA}$, $\text{Ni}^{2+} = 0.83 \text{ \AA}$, $\text{Mo}^{6+} = 0.73 \text{ \AA}$), corresponding with Fe-doped TiO_2 and ZnO [12–14], suggesting a significant lattice-defect formation by Fe doping.

The measured and first derivative of Fe K-edge XANES spectra of iron oxide doping in $\text{Sr}_2\text{NiMoO}_6$ system compared with FeO , Fe_2O_3 and Fe_3O_4 standard are shown in Fig. 2(a) and (b), respectively. Generally, the feature of XANES spectrum can be used as a finger print for investigating the formation of secondary phase in the sample. It can be clearly seen that

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