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**Transition metal oxides with metastable phases: a way towards superior ferroic properties (TransFerr)**

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Multiferroics have been known as materials exhibiting ferromagnetic and ferroelectric properties at the same time, which have exhibited interesting physical properties as well as possibility of practical applications. The rhombohedrally distorted simple perovskite structure of BiFeO3 is one of the representative multiferroic materials and has been much interested due to the antiferromagnetic behavior with a relatively high Neel temperature and the ferroelectric behavior with a high curie temperature. Multiferroic materials, owing to the coexistence of ferroelectricity, ferromagnetism and even ferroelasticity in the same phase, have shown promising applications in nonvolatile information storages, spintronic devices and magnetoelectric sensors. Among the multiferroic materials studied so far, BiFeO3 (BFO) is known to have a rhombohedrally distorted perovskite structure with a R3c symmetry. It has two order parameters at room temperature: a ferroelectric ordering with a high Curie temperature TC of 1103K, and a antiferromagnetic ordering of the G-type with a magnetic transition temperature TN of 643K. As the only one single phase multiferroic material which simultaneously possesses the ferroelectric and ferromagnetic properties at room temperature, BFO has been one of the most interesting materials studied. At present, the ceramics and thin films of BFO have been extensively investigated. Although rhombohedral BiFeO3 (BFO R-phase) has been studied extensively since first discovery in 1960s, electrical properties of the pure BFO R-phase have been rarely reported due to its high conductivity, which may originated from uncertain oxygen stoichiometry, high defect density and poor sample quality. In order to understand the properties of multiferroic BFO, it is very important that the fabrication of pure BFO R-phase should be established. If temperature and oxygen parsial pressure were not controlled accurately during crystallization of the BFO R-phases, the kinetics of phase formation always lead to other impurity phases in Bi-Fe-O system such as Bi2Fe4O9, Bi2O2.75 and Bi46Fe2O72. Wet chemical methods are a promising route to prepare fine and homogeneous powder. Various wet chemical methods such as hydrothermal, co-precipitation, combustion synthesis, molten-salt method, thermal decomposition, and sol–gel process have been developed and designed to prepare pure BiFeO3 nanopowder. Recently, acid-assisted gel strategy has been proved to be an effective way to synthesize metastable BiFeO3 nanopowder. Pure BiFeO3 phase could be obtained by leaching out the minor Bi2O3 phase using diluted nitric acid. Pure BiFeO3 powder can be directly synthesized through the acetic acid-assisted or the tartaric acid-assisted sol-gel method. However, BiFeO3 powder synthesized by the organic acid-assisted sol-gel method maybe have relatively low purity resulting from the easy formation of bismutite phase during calcining. Therefore, mineral acid should be considered as an adjuvant to prepare BiFeO3 nanopowder.

Two versions of the sol-gel method were used to synthesize the BiFeO3 powders.

1. FexBiyOz-citrate-based powder was synthesized using citric acid, ethylenediamine and nitric acid salts of Fe and Bi. Ethylene glycol was used as a solvent. In the beginning, the nitric acid salts of Fe and Bi were dissolved in ethylene glycol without addition of water. Then, citric acid was added to form Fe and Bi citrate. After that, the pH of the solution was adjusted to a value of 7-8 by neutralizing excess citric acid with ethylenediamine. The last neutralization step should be carried out with constant stirring, dropwise adding ethylenediamine and waiting for a constant pH to be established before the next drop is added. After homogenization of the resulting solution, ethylene glycol was added thereto. The solution was stirred for 30 minutes and then dried at 100 ° C until the gel formed and condensed, and then at a temperature of 250 ° C until a powder was formed. The resulting powder was annealed in air at a temperature of 550 ° C. (3 hours and 10 hours).

2. Synthesis of BiFeO3 powder used nitrate salts of Fe and Bi, water, HNO3, and citric acid as a solvent. The basic compounds were dissolved in HNO3 acid, which was then heated on a hot plate at 80-90 ° C to form a gel (about 4-5 hours). The resulting gel was then heated in an oven at a temperature of 180 ° C for 2 hours. The aim is to evaporate the water and remove the elements C, N and H. The resulting powder is then sintered in an oven at a temperature of 550 °C for 10 hours, respectively.

The best results from the XRD data was given by method 2. Therefore, it was used to synthesize sol-gel powders of composition Bi0,9La0,1FeO3. The concentration of lanthanum was constant. The annealing temperature was 550 °C (during 10 hours), 600 °C (during 3 hours), 700°C (during 3 hours), 800°C (during 3 hours). In addition, samples of sol-gel powder annealed at a temperature of 180 ° C for 3 hours and at 600 ° C for 3 hours without preheating were prepared.

According to the XRD data, the maximum content of the required phase was in powder 2 (BiFeO3).

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