

Microwave-Hydrothermal Processing of Niobium- and Tantalum-Based Electronic Ceramics

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Chemical reactions responsible for producing high value-added products are, in most cases, also responsible for generating by-products and pollutants. New chemical and biochemical approaches are providing new reaction concepts. As in the development of traditional chemical and petrochemical processes, reaction engineering, broadly defined as the field that quantifies the engineering aspects of chemically reactive systems, is providing enabling tools that accelerate the development of environmentally friendly processes [1,2]. Among the so-called high value-added products, the nanostructured materials have generated great excitement and expectations in the last few years [3]. These materials contain a large fraction of interfacial component, leading to outstanding characteristics that bulk materials do not possess. In fact, an immense academic interest together with recent technological advances in the fabrication, characterization and manipulation of nanostructures will impact in the next years the chemical, energy, electronic and space industries [4].

Nowadays, the technology for the preparation of nanopowders with superior characteristics is the basis on which conventional industries such as painting, coating, detergent and cosmetics have obtained innovations, and on which rising industries such as information recording media, advanced ceramics and electronics are promoted. There are many technologies currently employed for the production of nanostructured powders, with prominence for the hydrothermal technology. The advantages and benefits include a high degree of chemical homogeneity achieved on the molecular scale, the use of mild temperatures and pressures, the "single-step" production of nanocrystalline powders, and the elimination of high-temperature calcination and milling procedures to react and remove aggregates [5]. In the beginning of the 21st century, the hydrothermal technology represents the most promising route for environmentally friendly and low-cost production of advanced ceramic materials, either in batch reactors as well as in continuous reactors [6]. A wide range of crystalline, single and multi-component oxide materials can be produced by hydrothermal technology [7], and it is also possible to synthesize transition metal compounds with unusual oxidation states, low-temperature phases, and metastable compounds [8]. A recent innovation in this technology was the introduction of microwaves into the reaction vessels to produce ceramic materials more rapidly [9]. It offers many advantages over conventional autoclave heating, including rapid heating to crystallization temperature, homogeneous nucleation, fast supersaturation by the rapid dissolution of precipitated hydroxides, which leads to lower crystallization temperatures and shorter crystallization times [10].

Our group works with hydrothermal processing of electroceramics for applications as magnetics, piezoelectrics and relaxors [11,12]. These materials include dielectric (linear and non-linear) and conductive (super-conductors, conductors and semiconductors)

ceramics currently employed as capacitors, memories, sensors and actuators in microelectronics and communication components and devices for production control, environment monitoring, biomedical applications, and more recently as solid oxide fuel cell cathodes [13]. It is well known that the study of electroceramics is application driven and technology centered, with foundations in materials science, chemistry, and solid-state physics. The tools of the electroceramist include the majority of the periodic table, inorganics and organo-metallics, solid state chemistry, crystallography and other structural and chemical characterization techniques, experimental physics, modeling and device engineering.

In this paper, we report the microwave-hydrothermal synthesis of Nb- and Ta- based electroceramics, such as $\text{Ba}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3$ and $\text{Bi}_2\text{SrTa}_2\text{O}_9$. The first ceramic material is an important candidate material for applications as dielectric resonators in microwave and millimeter wave technologies due to their high permittivities and low dielectric losses [14]. The second material has been extensively studied in the last decade because of their ferroelectric and structural flexibility suitable for ferroelectric integrated devices [15]. The most distinguished technological application for SBT is the so-called ferroelectric random access memories (FeRAM). In this work, the fundamental issues relating to the hydrothermal processing conditions (temperature, pressure, time and pH) on the control of the phase behavior as well as of the morphological properties of these electroceramics are discussed in detail. X-ray diffraction, gas adsorption, scanning and transmission electron microscopies, Fourier-transform infrared and Raman spectroscopies, and complex impedance spectroscopy were employed in the characterization of the nanostructured powders and sintered ceramics.

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