Synthesis and Characterisation of Zinc Niobate Nanopowders via a Rapid Vibro-Milling Method

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ABSTRACT

In this study, an approach to synthesize single-phase ZN nanopowders with a mixed oxide synthetic route via a rapid vibro-milling technique has been developed. The formation of columbite $ZnNb_2O_6$ phase in the calcined powder has been investigated as a function of calcination conditions by TG-DTA and XRD techniques. Morphology, particle size and chemical composition have been determined via a combination of SEM and EDX techniques. The potentiality of a vibro-milling technique as a significant time-saving method to obtain single-phase ZN nanopowders at low temperature was demonstrated. It is seen that single-phase ZN powders were successfully obtained from a calcination condition of 600°C for 0.5h with heating/cooling rates of 30°C/min. In addition, by employing an appropriate choice of the milling time, an average particle size in the range of 50–300 nm could be achieved.

Key words: Phase formation , Calcination ,Vibro-milling , Zinc niobate , Columbite

INTRODUCTION

Zinc niobate ($ZnNb_2O_6$, ZN) material has been shown to be a good candidate for microwave dielectric applications (Maeda et al., 1987; Lee et al., 1996; Kim et al., 2001). The columbite-structured $ZnNb_2O_6$ is also an attractive B-site precursor for the preparation of lead zinc niobate ($Pb(Zn_{1/3}Nb_{2/3})O_3$ or PZN)-based ferroelectric ceramics, used for high performance in electromechanical actuators and transducers and piezoelectric ultrasonic motors (Jaffe et al., 1971; Xu, 1991; Park and Shrout, 1997). This is significant because it is very difficult to synthesize those compounds via the conventional solid-state reaction process using oxides as starting materials (Gururaja et al., 1987; Sekar et al., 1996). $ZnNb_2O_6$ powders were usually prepared by a solid-state reaction process (Lee et al., 1997). Recent work by Vittayakorn et al., (2003) has shown promise in producing phase-pure columbite ZN powders with the conventional mixed-oxide ball-milling method, while Kong et al., (2002) has successfully synthesized ZN powders via high-energy ball-milling or mechanochemical processing.

The present work is aimed at synthesizing single-phase zinc niobate nanopowders. The conventional mixed oxide synthetic route via a rapid vibro-milling technique has been developed with one-step reaction of all starting materials. The rapid vibro-milling technique is employed for the first time in this work as a significant time-saving method to obtain single-phase ZN nano-sized powders at low temperature.