A Mixed Oxide Synthetic Route to $Mg_4Nb_2O_9$ Nanopowders in a Corundum-like Phase

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ABSTRACT

An approach to synthesize single-phase magnesium niobate ($Mg_4Nb_2O_9$) nanopowders with a mixed oxide synthetic route is developed via a rapid vibro-milling technique. The powders were characterized by TG-DTA, XRD, SEM and EDX techniques. The effect of calcination temperature on phase formation characteristic of the powders was examined. The calcination temperature was found to have a pronounced effect on the phase formation of the calcined magnesium niobate powders. It was also found that the minor phases of unreacted MgO and Nb_2O_5 precursors and a columbite $MgNb_2O_6$ tended to form together with the corundum $Mg_4Nb_2O_9$ phase, depending on calcination temperatures. Furthermore, it was observed that optimisation of vibro-milling time and calcination temperature could lead to a single-phase $Mg_4Nb_2O_9$ with particle size of less than 100 nm.

Key words: Magnesium niobate, Nanopowder synthesis, Phase formation, Calcination

INTRODUCTION

Magnesium niobate ($Mg_4Nb_2O_9$; MN) is one of the binary niobate compounds which has been investigated as a potential candidate for the synthesis of microwave dielectric materials (Kan and Ogawa, 2004). It is also a room-temperature photoluminescent material and a suitable buffer layer material for fabricating ferroelectric memory devices (You et al., 1994-b). Recent work on the preparation of a single-phase relaxor perovskite $Pb(Mg_{1/3}Nb_{2/3})O_3$ or PMN (Lu and Yang, 2001) which is becoming increasingly important for electrostrictor, actuator and capacitor applications (Moulson and Herbert, 2003), has also shown that $Mg_4Nb_2O_9$ is a better precursor than the columbite $MgNb_2O_6$ (Ananta et al., 1999).

It is known that various compositions are possible in the Mg-Nb-O system (Norin et al., 1972). To date, four possible magnesium-niobium oxides have been identified: $Mg_4Nb_2O_6$, $Mg_4Nb_2O_9$, $Mg_5Nb_4O_{15}$ and $Mg_{2/3}Nb_{11(1/3)}O_{29}$ (Pagola et al., 1997). You et al., (1994a) reported that $MgNb_2O_6$ and $Mg_4Nb_2O_9$ are the only stable phases that exist at room temperature. It is known that synthesis of $Mg_4Nb_2O_9$ phase by the conventional method, i.e., by reacting individual oxides, generally results in varying amounts of the columbite $MgNb_2O_6$ phase alongside the corundum phase (Pagola et al., 1997; Sreedhar and Pavaskar, 2002). Thus, a number of chemical routes using expensive precursors, for example, polymerised complex (Camargo et al., 2000) and precipitation (Kan and Ogawa, 2004) methods, have been developed as alternatives to the conventional solid-state reaction of mixed oxides. All of these techniques are aimed at reducing the preparation temperature of the compound, even though they are more involved and complicated than the mixed oxide route. Generally, the