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ScienceDirect

Ceramics International 35 (2009) 1139–1144

CERAMICS INTERNATIONAL

www.elsevier.com/locate/ceramint

Simultaneous synthesis and single-step sintering of lead magnesium niobate ceramic using mixed nanopowders

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Received 1 February 2008; received in revised form 15 March 2008; accepted 20 May 2008
Available online 19 July 2008

Abstract

A high density single-phase lead magnesium niobate ceramic with the highest peak dielectric constant reported so far, has been synthesized and sintered simultaneously via a modified mixed oxide route, using mixed oxide nanopowder and single-step sintering. The mixed nanopowder was sintered at 1200 °C in air and PbO atmospheres. By comparison, samples sintered in air, gained pyrochlore structure, while those samples sintered in PbO atmosphere had pure perovskite structure. Pellets sintered for 2.5 h exhibited best dielectric properties with peak dielectric constant of 18,672 at the frequency of 1 kHz at −13 °C. The dielectric properties, compressibility, phase formation, densification, and microstructure of the samples were investigated.

Keywords: Lead magnesium niobate; Mixed oxide; Pyrochlore; Dielectric constant

1. Introduction

Lead magnesium niobate, $Pb^{2+}Mg_{1/3}{}^{2+}Nb_{2/3}{}^{5+}O_3{}^{2-}$ (PMN), is one of the most widely investigated relaxor ferroelectric materials with a perovskite structure. Globally, there is a balance of positive/negative charges. But in this system, any particular unit cell has either Mg^{2+} or Nb^{5+} at its body-centre. There is thus a local deviation from the value +4 required for charge balance. This factor is responsible for the occurrence of relaxor behavior, leading to a ''Curie range'' of temperatures, rather than a single temperature for the occurrence of the ferroelectric transition [1]. The excellent relative permittivity of PMN (~20,000) over the operational temperature range, electrostrictive properties and low dielectric loss make it a promising electroceramic material for multilayer ceramic capacitors (MLCCs), sensors, electromechanical transducers, electro-optic applications, electrostrictive actuators and fuel injectors for automobile engines [2–5]. There has been a great deal of interest in the preparation of single-phase PMN powders as well as in the sintering and dielectric properties of PMN-based ceramics [6].

The mixed oxide synthetic route is probably one of the most fundamental, practical routine methods used in production of PMN, and it has been developed and modified in both scientific research and industrial mass production for many years [6–10]. This technique involves powder preparation (mixing, milling, drying, sieving and calcination), the forming of green bodies and densification, where heat is applied, either with pressure (HIP) or pressureless sintering [11,12]. Although many alternative *powder synthetic routes* such as co-precipitation [13], molten salt [14], sol–gel [15] as well as hydrothermal methods [16] have been introduced from time to time, widespread efforts have still been made on the modification and development of conventional mixed oxide methods.

In general, the overriding aim of any materials processing technique is to achieve a final product with consistent properties. In practice, the level of consistency obtained is often a matter of compromise, it being largely a consequence of the economics of fabrication and characterization.

The main problem in fabrication of pure perovskite PMN ceramics is formation of the unwanted pyrochlore phase with low dielectric constant (~200) which decreases the dielectric and electromechanical performances of the resulting material [17]. The pyrochlore phase is the major product at the initial

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doi:10.1016/j.ceramint.2008.05.010