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Microwave dielectric properties of neodymium tin oxide

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Abstract

The microwave dielectric properties of Nd₂Sn₂O₇ ceramics were investigated with a view to their application in mobile communication. Nd₂Sn₂O₇ ceramics were prepared by the conventional solid-state method with various sintering durations. A maximum density of 7.11 g/cm³, a dielectric constant (ε_r) of 17.02, a quality factor (*Qf*) of 33,100 GHz, and a temperature coefficient of resonant frequency (τ_f) of -55 ppm/°C were obtained when Nd₂Sn₂O₇ ceramics were sintered at 1550 °C for 9 h. © 2013 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

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Keywords: Nd₂Sn₂O₇; Dielectric constant; Quality factor; Temperature coefficient of resonant frequency

1. Introduction

The advantages of using complex perovskite ceramics $A(B'_{0.5}B''_{0.5})O_3$ ($A=Me^{2+}$, Me^{3+} ; $B'=Me^{2+}$, Me^{3+} ; $B''=Me^{4+}$, Me^{5+} , Me^{6+}) are reportedly associated with their excellent microwave dielectric properties [1–3]. Extensive research into $Ln(Mg_{0.5}Ti_{0.5})O_3$ (Ln=La, Sm, Nd, Da, Y) ceramics and related ceramic systems have focused on their potential application in resonators, filters and antennas in modern communication systems, including radars and global positioning systems (GPS), which are operated at microwave frequencies [4,5]. $Ln(Mg_{0.5}Ti_{0.5})O_3$ ceramics exhibit a high dielectric constant ($\varepsilon_r \sim 22-27$), a high quality factor ($Q \sim 3770-7550$ at 10 GHz), and an adjustable temperature coefficient of resonant frequency.

Recently, numerous studies of Nd(Mg_{0.5}Sn_{0.5})O₃ ceramics have been undertaken [6–10]. Nd(Mg_{0.5}Sn_{0.5})O₃ ceramics that were sintered at 1550 °C for 4 h have been obtained with a dielectric constant of 19.3, a *Qf* of 43,300 GHz, and a τ_f of -57 ppm/°C. A dielectric constant of 18.9, a *Qf* of 32,300 GHz, and a τ_f of -52 ppm/°C were obtained for 0.25 wt% B₂O₃-doped Nd(Mg_{0.5}Sn_{0.5})O₃ ceramics, sintered at

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1500 °C for 4 h. A dielectric constant of 21.1, a Of of 50,000 GHz, and a τ_f of $-60 \text{ ppm/}^{\circ}\text{C}$ were obtained for Nd (Mg_{0.5}Sn_{0.4}Ti_{0.1})O₃ ceramics that were sintered at 1550 °C for 4 h. A dielectric constant of 19.2, a *Qf* of 68,900 GHz, and a τ_f of -67 ppm/°C were obtained for Nd(Mg_{0.45}Co_{0.05}Sn_{0.5})O₃ ceramics that were sintered at 1550 °C for 4 h. A dielectric constant of 19.5, a Qf of 129,200 GHz, and a τ_f of -66 ppm/ $^{\circ}C$ were obtained when the Nd(Mg_{0.4}Zn_{0.1}Sn_{0.5})O₃ ceramics were sintered at 1500 °C for 4 h. The dielectric constant increased from 31.8 to 47.7, the Qf decreased from 54,200 to 42,800 GHz, and the τ_f increased from -43 to +41 ppm/ °C as y increased from 0.5 to 0.7 when (1-y)Nd(Mg_{0.4}Zn_{0.1}Sn_{0.5})O₃-yCa_{0.8}Sr_{0.2}TiO₃ ceramic system sintered at 1600 °C for 4 h. A 0.5 wt% B_2O_3 -doped 0.4Nd $(Mg_{0.4}Zn_{0.1}Sn_{0.5})O_3-0.6Ca_{0.8}Sr_{0.2}TiO_3$ ceramic system that was sintered at 1350 °C for 4 h had a dielectric constant of 38.3, a *Qf* of 35,000 GHz, and a τ_f of -4.8 ppm/°C.

Neodymium tin oxide $(Nd_2Sn_2O_7)$ was found as a second phase in the composites [6–10]. The formation of this Sn-rich second phase was attributed to the loss of MgO upon ignition. However, no technical information on the microwave dielectric properties of $Nd_2Sn_2O_7$ ceramics is available in the published literature. This fact motivates this investigation of the microwave dielectric properties of $Nd_2Sn_2O_7$ ceramics. In this work, $Nd_2Sn_2O_7$ ceramics were synthesized using the conventional mixed-oxide method. The effects of the sintering duration on

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the microwave dielectric properties of $Nd_2Sn_2O_7$ ceramics sintering at 1550 °C were explored. The microwave dielectric properties of the $Nd_2Sn_2O_7$ ceramics varied with the sintering duration. These microwave dielectric properties were further analyzed by densification, X-ray diffraction (XRD) patterns, and observation of their microstructures.

2. Experimental procedure

The Nd₂Sn₂O₇ ceramics were prepared by the conventional mixed-oxide method. The starting raw chemicals were Nd₂O₃ (99.99%) and SnO₂ (99.0%) powders. The raw materials were mixed in a manner consistent with the stoichiometric proportions of the Nd₂Sn₂O₇ ceramics. The powders were ball-milled in alcohol for 12 h and dried. The Nd₂Sn₂O₇ was then calcined at 1200 °C for 4 h. The calcined powder was re-milled for 12 h using PVA solution as a binder. The obtained powder was then crushed into fine particles and sieved through a 200 mesh. This very fine powder thus obtained was then axially pressed at 2000 kg/cm² into pellets with a diameter of 11 mm and a thickness of 6 mm. These pellets were then sintered at 1550 °C for 8–10 h in air. Both the heating rate and the cooling rate were set to 10 °C/min.

Following sintering, the phases of the samples were investigated by X-ray diffraction. An X-ray Rigaku D/MAX-2200 with $CuK\alpha$ radiation (at 30 kV and 20 mA) was utilized along with a graphite monochromator in the 2θ range of 10–70°. Scanning electron microscopy (SEM; JEOL JSM-6500F) and energy dispersive X-ray spectrometry (EDS) were utilized to examine the microstructures of the specimens. Their apparent densities were measured by Archimedes' method in distilled water. The microwave dielectric properties of the specimens were measured using the postresonator method developed by Hakki and Coleman [11]. This scheme adopted a cylindrical specimen of diameter D and length L. The specimens whose microwave dielectric property was measured had an aspect ratio, D/L, of approximately 1.6, which is in the permitted range that was determined by Kobayashi and Katoh [12]. A cylindrical resonator was sandwiched between two conducting plates. Two small antennas were positioned close to the specimen to couple the microwave signal power into or out of the resonator. The other ends of the antennas were connected to an Agilent N5230A network analyzer. The resonance characteristics depended on the size and dielectric properties of the specimen. The microwave energy was coupled using electric-field probes. The TE_{011} resonant mode was optimal for obtaining the dielectric constant and the loss factor of the specimen. An Agilent N5230A network analyzer was utilized to identify the TE_{011} resonant frequency of the dielectric resonator, and the dielectric constant and quality factor were calculated. The value of τ_f was measured by the same method as the dielectric constant. The test cavity was placed in a chamber in which the temperature was increased from 25 to 75 °C. The τ_f value (ppm/°C) was determined from the change in resonant frequency,

$$\tau_f = \frac{f_2 - f_1}{f_1 (T_2 - T_1)},\tag{1}$$

where f_1 and f_2 are the resonant frequencies at T_1 and T_2 , respectively.

3. Results and discussion

Fig. 1 displays the X-ray diffraction patterns of the $Nd_2Sn_2O_7$ ceramics sintered at 1550 °C for 8–10 h. The X-ray diffraction patterns of $Nd_2Sn_2O_7$ ceramics did not vary significantly with sintering durations. The spectral angles of the X-ray diffraction peaks were the same following sintering at 1550 °C for 8–10 h.

Clearly, $Nd_2Sn_2O_7$ was the main crystalline phase, which was accompanied by small amount of Nd_2O_3 as the second phase. $Nd_2Sn_2O_7$ with a cubic crystal structure (ICDD-PDF #87-1220) and Nd_2O_3 with a hexagonal crystal structure (ICDD-PDF #74-1147) were identified.

Fig. 2 shows the microstructures of $Nd_2Sn_2O_7$ ceramics, following sintering at 1550 °C for 8–10 h. Comparing the microstructures of $Nd_2Sn_2O_7$ ceramics that were sintered for different durations indicated that the average grain size increased with the sintering duration. To identify the composition of the second phase, an energy-disperse spectroscopy (EDS) analysis was carried out on the grains of the $Nd_2Sn_2O_7$ ceramics that were sintered at 1550 °C for 9 h, as shown in Fig. 2(b). The quantitative analysis, presented in Table 1, reveals that grains A and B were $Nd_2Sn_2O_7$ and the grain C was Nd_2O_3 .

Fig. 3 displays the amounts of the main phase and apparent densities of the $Nd_2Sn_2O_7$ ceramics that were sintered at 1550 °C for 8–10 h. The amount of the main phase was evaluated from strongest lines of both main and second phases,

$$Nd_2Sn_2O_7 (vol\%) = \frac{I_{A(2\ 2\ 2\ 2)}}{I_{A(2\ 2\ 2)} + I_{B(0\ 1\ 1)}} 100$$
(2)

where I_A and I_B are the strongest lines of Nd₂Sn₂O₇ (2 2 2) and Nd₂O₃ (0 1 1), respectively. The amount of the main phase sintering at 1550 °C remained stable as sintering duration increased from 8 to 9 h and increased from 87.31% to 89.77% as sintering duration increased from 9 to 10 h. The formation of the second phase of Nd₂O₃ affected the apparent density and microwave dielectric properties of Nd₂Sn₂O₇ ceramics. The apparent densities of the Nd₂Sn₂O₇ ceramics sintered at



Fig. 1. X-ray diffraction patterns of $Nd_2Sn_2O_7$ ceramics sintered at 1550 °C for 8–10 h.

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