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Effect of Zinc in the Microwave Dielectric Properties of $(Ca_{1-x}Zn_x)TiO_3$ (x = 0, 0.5) Ceramics

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Authors' contributions

This work was carried out in collaboration among all authors. Authors Asad Ali, DK and MH designed the study, performed the statistical analysis, wrote the protocol, and wrote the first draft of the manuscript. Authors MHJ, MQH, AK, Abid Ahmad managed the analyses of the study. Author AZ managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Calcium Titanium Oxide or Calcium Titanate ($CaTiO_3$-CT) and Zinc doped calcium titanium oxide (Ca1-xZnxTiO_3-CT:Zn) Powder in a concentration of ($0 \leq x \leq 0.5$ molar ration of Zinc) were obtained by a simple method called solid-state reaction. Ceramics are based on $CaTiO_3$-CT and $(Ca_{0.5}Zn_{0.5})$ TiO_3 solid solutions synthesized and characterized in order to study microwave dielectric properties. X-ray spectra analysis, FT-IR Spectra, and SEM studies shown that the synthesized powders were contained nano-crystallite shape are Orthorhombic. The suitable shift in the XRD peaks for x=0.5 composition towards comparatively lower angles gradually with the increase of "x" values, which indicates that the lattice parameters of Ca1-xZnxTiO_3 decreases after

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substituting. The SEM produces the images of ($CaTiO_3$-CT) and ($Ca_{0.5}Zn_{0.5}$) TiO_3 by scanning the surface with a focused beam of electrons. The common grain size for x=0.5 has the highest value equal to 2.08 µm, and usually, the grain size has an increasing trend by increasing zinc content from 1.214 to 2.08 µm. In Fourier Transform Infrared Radiation spectroscopy (FTIR), IR radiations pass through the ($CaTiO_3$-CT) and ($Ca_{0.5}Zn_{0.5}$)TiO_3, which determine the quality consistency of the above mention samples. It can also identify the unknown materials and determine the number of components in a given mixture. The dielectric properties of the samples $CaTiO_3$-CT and Ca1-xZnxTiO3-CT:Zn were studied at room temperature. The dielectric constant (εr) is found to decrease with an increasing frequency range from 2 to 2.50.

Keywords: Ceramics; dielectric properties; sintering; CaTiO3; (Ca0.5Zn0.5)TiO3.

1. INTRODUCTION

Economic communication system based on broadcasting electromagnetic waves(wireless) applications emanate in the late 1970s, disclose in the 1980s, and increased hurriedly through the1990s. Infinite systems are quickly filling the 400MHz – 20GHz band. At the beginning of the last decade Cellular Telephone (400MHz – 1GHz), Television Receiver Only (TVRO, 2GHz – 5GHz), Direct Broadcasting (DBS, 11GHz to 13GHz) and wonderfully Satellite Communication become dispose all over the world. Nowadays, the highest important applications are wireless cable, gigantic definition, and interactive Television (TV), collision abstention, global position, cellular satellite, and Personal Communication Systems (PCS) of many types. Among a few factors, industrial evolution has been inspired by the development of advanced ceramics, and they are commercialized as low-cost, high-volume products.

Advanced ceramics are easily integrated into radio frequency (Microwave) circuits. They function as a capacitor, frequency filter signals-distributing elements, and inductors [1]. The quick gain of the latest telecommunication has managed the improvement of Microwave (radio frequency) dielectric ceramics used for a duplexer, bandpass filter, and resonator. In regard to the applications in millimeter-wave frequency, these advanced ceramics with Perovskites structure require three main characteristics, which are given below:

i. A high-quality factor (Q) to Maximize frequency selectivity.
ii. A large dielectric constant (ϵ_r) to minimize the size of a circuit.
iii. And the third one is a near-zero temperature coefficient of the resonant frequency (τ_f) to assure large-temperature [2,3]

Microwave (Radio Frequency) materials having dialectic losses contain intrinsic losses, which are the essential losses of multi-photon origin in on ideal crystalline material and extrinsic losses caused by lattice imperfection [4,5]. The disconnection of intrinsic and extrinsic microwave lasses using normal Microwave (MW) measurements is very difficult. The best-talented approach to this problem is to analyze a higher frequency dielectric response containing the whole gigahertz and infrared (IR) range [6].

Calcium titanium oxide ($CaTiO_3$-CT) is an excellent candidate for microwave (MW) dielectric since it has a large permittivity ε_r=160 and an admissible quality factor, Q= 8000 at 1.5GHz. But unfortunately, it has a high positive temperature coefficient of the resonant frequency τ_f = +850ppm K^{-1} [7]. The crystallized in the orthorhombic space group Pbnm with four formula units per unit call at room temperature. An Infrared (IR) spectroscopic study on calcium titanate was repeated by Železný et al. [7-9].

An alternative $MgTiO_3$ceramics material has attracted huge contemplation due to its good microwave dielectric properties and much low cost. ϵ_r=17, $Q \times f$ = 160,000GHz and a τ_f = -50ppm/^{o}C have been achieve for $MgTiO_3$ceramics [10]. Interchanges at A- and B-sites enhanced the $Q \times f$ value of $MgTiO_3$. The doping A-site, with elements like Ni, Co, and Zn, led to enrichment in the $Q \times f$ value of $MgTiO_3$(180,000 GHz to 364,000 GHz) [11,12]. $Mg_{0.95}Ni_{0.05}TiO_3$reported to have an admirable dielectric property, i.e., $Q \times f{\sim}1.8 \times 10^5 GHz \epsilon_r{\sim}$ 17.2 and $\tau_f{\sim}$-45 ppm/C [13] but the high negative of make $Mg_{0.95}Ni_{0.05}TiO_3$ useless for feasible applications to the τ_f value through zero, it is required to alternate advisable cations or conjoin with materials having large positive τ_f values.

For $0.19Nd_{0.5}Na_{0.5}TiO_3$-$0.81Mg_{0.95}Ni_{0.05}TiO_3$ ceramics, $\epsilon_r = 25.61$, $Q \times f = 69{,}000$ GHz and $\tau_f = -6$ ppm/C were obtained [14,15]. Addition of $La_{0.5}Na_{0.5}TiO_3$ceramics tuned the value $\tau_f = 2.8$ ppm/C, and as a result $Q \times f = 86{,}000$ GHz and $\epsilon_r \sim 23.22$ were obtained for $0.13La_{0.5}Na_{0.5}TiO_3$-$0.81Mg_{0.95}Ni_{0.05}TiO_3$ ceramics. For other related system, $\epsilon_r = 22.9$,

$Q \times f = 92{,}000$ GHz and $\tau_f = -5.4$ ppm/^{o}C were achieved for $0.1Ca_{0.8}Sm_{0.4/3}TiO_3$-$0.9Mg_{0.95}Ni_{0.05}TiO_3$ sintered ceramics [16]. Mini doping of Zr let to $Q \times f \sim 195{,}000$ GHz, $\epsilon_r \sim 17$ and $\tau_f \sim -46$ ppm/C for $Mg_{0.95}Ti_{0.98}Ni_{0.05}Zr_{0.02}O_3$ [17]. However, the higher negative value of τ_f make $Mg_{0.95}Ti_{0.98}Ni_{0.05}Zr_{0.02}O_3$ unworkable for application. The value τ_f of $Mg_{0.95}Ti_{0.98}Ni_{0.05}Zr_{0.02}O_3$ was tuned to zero by mixing of $SrTiO_3$ and $Q \times f \sim 85{,}000$ GHz, $\epsilon_r \sim 20$ and $\tau_f \sim -3$ ppm/^{o}C were attained for $0.04SrTiO_3$-$0.96Mg_{0.95}Ti_{0.98}Ni_{0.05}Zr_{0.02}O_3$ ceramics [18]. $Q \times f \sim 3600$ GHz, $\epsilon_r \sim 170$ and $\tau_f \sim 800$ ppm/^{o}C were obtained $CaTiO_3$ [19]. Since $CaTiO_3$ ceramics holds larger $Q \times f$ value than $SrTiO_3$, therefore, $CaTiO_3$addition may be concluded in temperature stable ceramics material with much larger $Q \times f$ than $SrTiO_3$ [18].

Recently studied two ceramics $CaTiO_3$ and $Mg_{0.95}Ti_{0.98}Ni_{0.05}Zr_{0.02}O_3$ were separately prepared by using a simple method of solid-state reaction. The microwave dielectric properties and phase of (1-x)$Mg_{0.95}Ti_{0.98}Ni_{0.05}Zr_{0.02}O_3$- x$CaTiO_3$ advanced ceramics system were examined by using the cavity method with the help of vector network analyzer and X-ray diffraction technique in order to touch the temperature stable and low loss ceramics. For x=0 $Mg_{0.95}Ti_{0.98}Ni_{0.05}Zr_{0.02}O_3$, was major/leading phase along with $Mg_{0.95}Ti_{0.98}Ni_{0.05}Zr_{0.02}O_3$ phase that was formed as a small secondary phase, microwave (MW)/ radio frequency (rf) properties $Q \times f$ of 105,855 GHz$\varepsilon_r \sim$ 17.1 τ_f= -46.3ppm / ^{0}c were achieved for the composition with x=0, Because of the positive value of τ_f calcium titanium oxide ($CaTiO_3$-CT), its inclusion to the combination with $x = 0$ turned the $\tau_f$value to zero. A closely linear change in all properties was scrutinized as a result of $CaTiO_3$ addition. Mixing of $CaTiO_3$ change the value of (τ_f) of $Mg_{0.95}Ti_{0.98}Ni_{0.05}Zr_{0.02}O_3$ ceramics to the value near to zero and $Q \times f \sim 108{,}775$ GHz, $\varepsilon_r \sim 28.4$ and $\tau_f \sim$ 3.1 ppm/C over obtained for x=0.15 [10].

Another promising example of advanced ceramics (1-x)$CaTiO_3$–x$LaAlO_3$ $(0 \le x \le 1)$. The dielectric constant, "ϵ_r" a decrease from 47.83 to 28.25 as the $LaAlO_3$ contented in the CTLA ceramics increased from 0.3 to 0.7 because the polarizability alteration decreases from 1.74 to 5.0% by the increase of $LaAlO_3$, the value of temperature coefficient of the $\tau_f$decreases from 17.77 to -20.42 ppm /C due to the increasing of A-site valence from 1.9240 to 2.4695 and $Q \times f$ increase from 30,000 to 42,000GHz because the order degree of B-sites ions increased and cation rattling decreased. At $x = 0$ .5 a structural transformation obtained, which debilitated $Q \times f$ value and temperature coefficient of the resonant frequencyτ_f [11].

In this research work, the $Ca_{1-x}Zn_xTiO_3$-CT:Zn (0$\le x \le 0.5$) ceramics were prepared by using of simple two-step solid-state reaction method and examine densification, microwave dielectric properties and microstructure analysis of the ceramics system using X-ray Diffraction (XRD) Scanning Electron Microscope (SEM), Fourier Transform Infrared spectroscopy (FTIR) and the microwave dielectric measurement at room temperature.

2. EXPERIMENTAL PROCEDURE

2.1 Sample Preparation

The samples of this research work were synthesized by a simple two steps method called solid-state-reaction. Starting powders calcium carbonate ($CaCO_3$) (Aldrich 99.9%), titanium oxide (TiO_2) (Aldrich 99.9%), and zirconium oxide (ZnO_2) (Aldrich 99.9%) powders as the beginning materials were measured according to the compositions ($Ca_{1-x}Zn_xTiO_3$-CT:Zn) with $(0 \le x \le 0.5)$. For the mixing of materials, we used crucible made from ceramics materials. We fined the molecular weight of the calcium titanium oxide $CaTiO_3$ and Zinc doped calcium titanium oxide ($Ca_{1-x}Zn_xTiO_3$-CT:Zn) accurately. The mixed powders were then calcined at 800^{o}C for 2 hours. The calcined powders were pressed (at a pressure of 100MPa) into the pellets and sintered at temperature 1000^{o}C for 3 hours. The diameter (size) of the pellets of each sample was measured by 10mm.

2.2 Characterization and Theoretical Analysis

The phase and microstructure analyses were carried out by using the X-ray diffraction method (XRD) (JDX-3532, JEOL, Japan) with Cu, Kα radiation ($\lambda = 0.154$ nm) and scanning electron microscope (SEM) (JSM-5910, JEOL, Japan) respectively. The micro-dielectric properties of the fabricated ceramics ($CaTiO_3$) and ($Ca_{1-x}Zn_xTiO_3$-CT:Zn) pellets were measured by LCR meter (Agilent 4287A). The apparent densities of the centered ceramics pellets were by densitometer (MD 300s) using the Archimedes principle.

Due to lattice vibration in the real crystal structure of phonon may be absorbed or released. The fundamental interaction of lattice vibration with EM radiation (Electromagnetic waves) is found by the parameter of Infrared active phonons. Phonon dispersion can be obtained by the four-parameter factorized oscillator model [20], which are given below.

$$\epsilon(\omega) = \epsilon^{'}(\omega) - i\epsilon^{''}(\omega) = \epsilon_{\infty} \prod_{j=1}^{n} \frac{\omega^2{}_{LOj} - \omega^2 + i\omega^2\gamma_{LOj}}{\omega^2{}_{TOj} - \omega^2 + i\omega^2\gamma_{TOj}} \quad (1)$$

Where $\omega^2{}_{LOj}$ and $\omega^2{}_{TOj}$ are the longitudinal and transverse frequency of the *j*th polar phonon mode, appropriately, γ_{LOj} and γ_{TOj} their respective damping constants and ϵ_{∞} The optical permittivity due to the electronic polarization processes. In the small-frequency limit $(\omega \ll \omega_{TOj})$ equation (1) yields losses which are dependent on frequency linearly, $\epsilon^{''} \propto \omega$ and frequency, not dependent on permittivity. However, the oscillator model equation (1) describes the one phonon absorption processes, whereas the dielectric losses in small-frequency limits or obtained almost entirely by two phonon absorption or larger order processes. Therefore, formally the oscillator exemplary in not accurate in the Microwave (MW) range. A microscopic theory is more complicated [21]. It has to be used to calculate the temperature and frequency behavior of Microwave (MW) losses. However, this theory concern with microscopic parameters, and therefore it gives only qualitative predictions. In the small frequency limit, the theory [21] predicts in maximum crystal symmetries the same linear behavior$\epsilon^{''} \propto \omega$, which comes from the oscillator model. The calculated far-infrared (IR) reflectivity spectra have been fitted by the oscillator exemplary (equation (1)) using the relation for the reflectivity under normal incidence.

$$R(\omega) = \left|\frac{\sqrt{\epsilon(\omega)}-1}{\sqrt{\epsilon(\omega)}+1}\right|^2 \quad (2)$$

However, it is known that minimum errors in the reflectivity may lead to manifest errors in the fitted permittivity and especially in the losses. Therefore the fitting has been carried out, taking into account the complex gigahertz permittivity, which allowed a crucial advancement in the quality of the fits.

3. RESULTS AND DISCUSSION

3.1 Phase Analysis

Fig. 1 shows the room temperature XRD patterns of $Ca_{1-x}Zn_xTiO_3$ ceramics sintered at 1300°C for 3hrs in air. The suitable shift in the XRD peaks for x=0.5 composition towards comparatively lower angles gradually with the increase of "x" values, which indicates that the lattice parameters of $Ca_{1-x}Zn_xTiO_3$ decreases after substituting. It is mainly attributed that the values may be due to the incorporation of slightly larger (r=1.34 Å) Ca ions for the smaller (r=1.04 Å) Zn ions [22]. The diffraction peaks in the XRD patterns can be indexed with an orthorhombic phase structure that belonged to the space group (Pnma) and (Pbnm) matching with pdf card # (22-153) and (82-232) respectively and the composition which also illuminates that an orthorhombic phase structure was obtained for all the cases. The variation in the lattice parameters 'a', 'b', and 'c' of the $Ca_{1-x}Zn_xTiO_3$ ceramics with the increase in Zn^{2+} content is shown in Table 1. The lattice parameters 'a' and 'c' abruptly increases when content x=0.5. This is consistent with the observed change from the orthorhombic space group structure (Pnma) to the orthorhombic space group structure (Pbnm) [23,24].

Table 1. Structural Data of $Ca(Zn_xTi_{1-x})O_3$ ceramics from XRD Analysis sintered at 1000°C

Content (x)	a(Å)	b(Å)	c(Å)	Z	Structure	S.G
0.0	5.4405	7.6436	5.3812	4	Orthorhombic	Pnma
0.5	5.4750	5.4863	7.7579	4	Orthorhombic	Pbnm

X= Zn^{2+} content, Z= No. of atom per unit cell, S.G= Space group

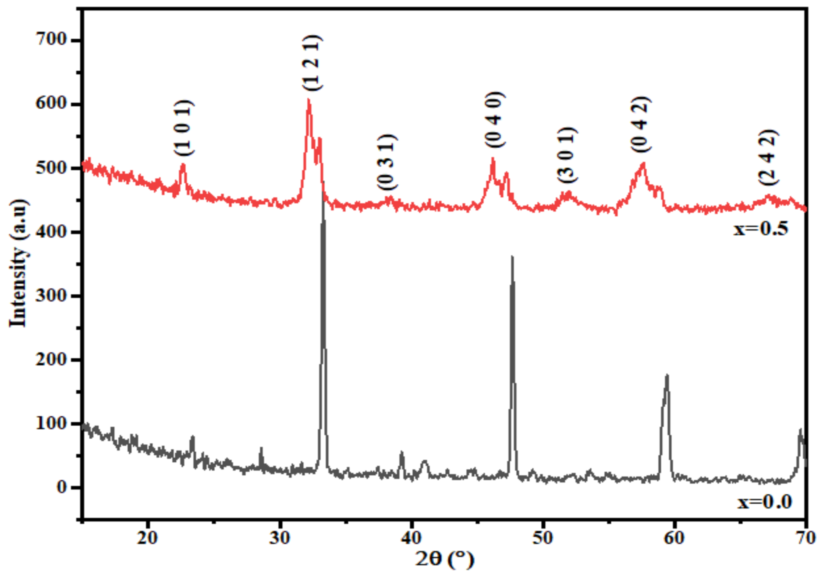

Fig. 1. XRD of $Ca(Zn_xTi_{1-x})O_3$ ceramics sintered at 1000°C

3.2 Microstructure Analysis

Fig. 2 represents the SEM micrographs of the specimens using $Ca(Zn_xTi_{1-x})O_3$ ceramics were observed at different contents 'x' sintered at 1000°C (x=0.0 & 0.5) for 3hrs in air. As seen from the SEM micrographs, it comprises of round-like and large rod-shaped grains showed up with little pores. The common grain size for x=0.5 has the highest value equal to 2.08 µm, and usually, the grain size has an increasing trend by increasing zinc content from 1.214 to 2.08 µm; this form of morphology has been antecedently rumored for $CaTiO_3$ ceramics [25]. The increasing trend could also be because of the various distributions of B-site Zr^{4+} and Ti^{4+} ions [26].

3.3 Dielectric Properties

The microwave dielectric properties of $Ca(Zn_xTi_{1-x})O_3$ ceramics sintered at 1000°C for 3hrs with different frequency ranges are shown in Fig. 3. The dielectric constant (ε_r) of $Ca(Zn_xTi_{1-x})O_3$ over a wide range of frequencies varying from 2.00GHz to 2.50GHz at room temperature. The dielectric constant (ε_r) is found to decrease with an increasing frequency range from 2 to 2.50. These trends of the curve are normal behavior for most dielectric ceramics attribute to the presence of strong dielectric relaxation as the rotation dipole becomes insufficient to align themselves to follow the oscillation of the applied ac electric field with increasing frequency [27-29].

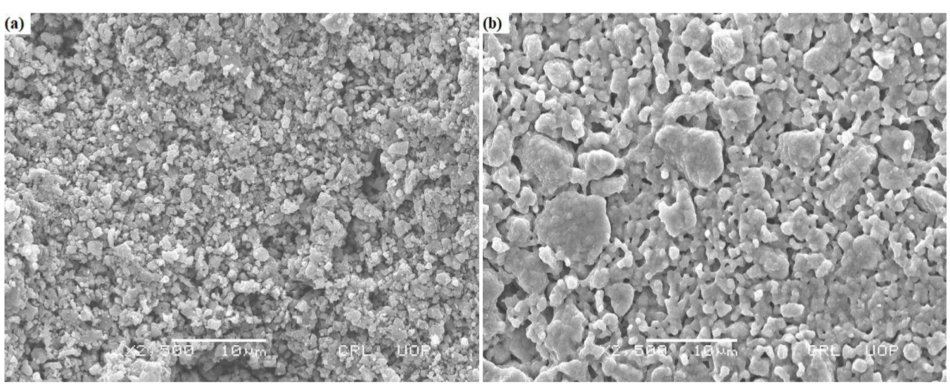

Fig. 2. SEM micrograph of the $Ca(Zn_xTi_{1-x})O_3$ ceramics sintered at 1000 °C for 3 h in air; (a) x = 0.0, (b) x = 0.5; indicating a slight change in grain sizes with increase in x

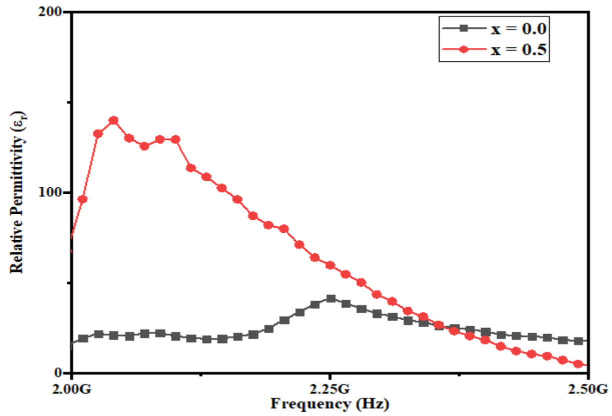

Fig. 3. Experimental plots of the dielectric constant versus frequency of $Ca(Zn_xTi_{1-x})O_3$ ceramics sintered at 1000°C for three h in air

4. CONCLUSION

The result of XRD showed that a suitable shift in the XRD peaks for x=0.5 composition towards comparatively lower angles gradually with the increase of “x” values, which indicates that the lattice parameters of Ca1-xZnxTiO3 decreases after substituting. SEM analysis showed that grain size for x=0.5 has the highest value equal to 2.08 μm, and usually, the grain size has an increasing trend by increasing zinc content from 1.214 to 2.08 μm. The dielectric constant (εr) is found to decrease with an increasing frequency range from 2 to 2.50Hz.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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