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Structural and Dielectric Properties of Strontium doped Magnesium Titanates

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Abstract: In this paper, we produced Strontium-doped Magnesium titanate ceramics using a solid-state reaction method while maintaining a consistent molar ratio. We explored the impact of different Strontium compositions on the material properties. Characterization techniques included X-ray diffraction (XRD) for structural analysis, scanning electron microscopy (SEM) paired with energy-dispersive X-ray spectroscopy (EDAX) for microstructural evaluation, and dielectric measurements conducted with a HIOKI 3532-50 LCR Hitester. The findings revealed that the ceramic samples displayed an ilmenite hexagonal structure, with particle sizes determined through XRD analysis. Furthermore, dielectric measurements provided valuable information regarding the dielectric constant and loss of the materials.

Keywords: Solid State Reaction Route, Dielectric measurement, Thermal studies, XRD, SEM, EDAX.

I. INTRODUCTION

$MgTiO_3$ ceramics exhibit an ilmenite structure, which is derived from a hexagonal framework. This structure arises from the presence of equal proportions of divalent and tetravalent cations, which are systematically arranged at the octahedral sites and alternate along the c-axis of the unit cell. Due to their low-loss dielectric properties, materials with the ilmenite structure, such as $MgTiO_3$, have garnered significant attention for high-frequency applications and the stability of resonant frequencies in microwave telecommunication systems. Notably, $SrTiO_3$ and $MgTiO_3$ share a cubic perovskite and trigonal crystal structure characterized by a specific space group, and they possess similar ionic radii (1.44Å for Sr^{2+} and 0.72 Å for Mg^{2+}). This similarity enables the substitution of zinc and manganese ions, resulting in the formation of a (Sr, Mg) TiO_3 solid solution that has the potential to enhance thermal stability and dielectric properties.

Magnesium Titanate has garnered significant interest due to its robust absorption capabilities in the visible spectrum, which could enhance solar energy applications. The addition of metal ions to titanate ceramics can notably broaden the absorption range of photo-catalysts into the visible region, as doping alters the equilibrium concentrations of electrons or holes. In industrial and commercial electronic systems, components such as microwave resonators, filters, oscillators, and capacitors are essential, necessitating materials with a high dielectric constant, low dissipation factor, and minimal temperature coefficient of the dielectric constant, particularly at high and hyper frequencies. This study aims to investigate how milling conditions affect the structural and dielectric properties of sintered Strontium-doped MgTiO3 across various compositions, with characterization performed using X-ray diffraction (XRD), scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDAX), and a HIOKI 3532-50 LCR Hitester.

II. EXPERIMENTAL DETAILS

A. Materials

Strontium dioxide (TiO_2) powder (P-25), Strontium Oxide (SrO) and Magnesium Carbate ($MgCO_3$) were purchased from Aldrich Chemical (USA) with purities above 99.9%. Poly Vinyl Alcohol (PVA) as binder to prepare the pellets.

B. Preparation of Strontium SrTiO3 samples

Ceramic samples were synthesized using the traditional Solid State Reaction Route from high-purity oxide powders exceeding 99.9%. The initial materials were combined in ethanol and subjected to Ball Milling for 24 hours with Zirconia balls in polyethylene containers, followed by drying at 150°C. The resulting mixed powders were then calcined at 1200°C for 24 hours. Afterward, the grained powders were blended with a 2.5 wt% Polyvinyl Alcohol (PVA) solution as a binder and pressed into discs with a diameter of 10 mm and a thickness of 2 mm under a hydrostatic pressure of approximately 10 Kg/cm². Finally, the prepared pellets were sintered at 1250°C for 6 hours.

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C. Characterization Techniques

The structural properties of the materials were analyzed using X-ray diffraction (XRD), while the surface morphology was assessed through scanning electron microscopy. To explore the phase formation in Strontium-doped $MgTiO_3$, differential scanning calorimetry (DSC) measurements were conducted using an HT-DSC (SDT Q 600 from TA Instruments). Porosity was evaluated via the Archimedes method, employing water immersion techniques. Additionally, the dielectric constant, loss tangent, and AC conductivity were measured with a HIOKI 3532-50 LCR HiTester, imported from Japan, across varying temperatures and frequencies up to 1 MHz.

III. RESULTS AND DISCUSSION

A. XRD.

The structural properties of the samples were analyzed using X-ray diffraction (XRD) plots, illustrated in Fig. 1, across various Strontium compositions (0.1, 0.3, 0.5). The data revealed that the peak intensity reached its maximum at a Strontium composition of 0.3, with a noticeable shift towards higher angles as the Strontium content increased. This shift is attributed to the substitution of magnesium ions by Strontium ions in the lattice structure. Additionally, the particle sizes decreased from 8.07 to 6.18 as smaller Sr^{2+} cations were introduced, leading to a reduction in both lattice parameters and volume with increasing strontium composition. The XRD patterns indicate that the sample exhibits octahedral ilmenite structure

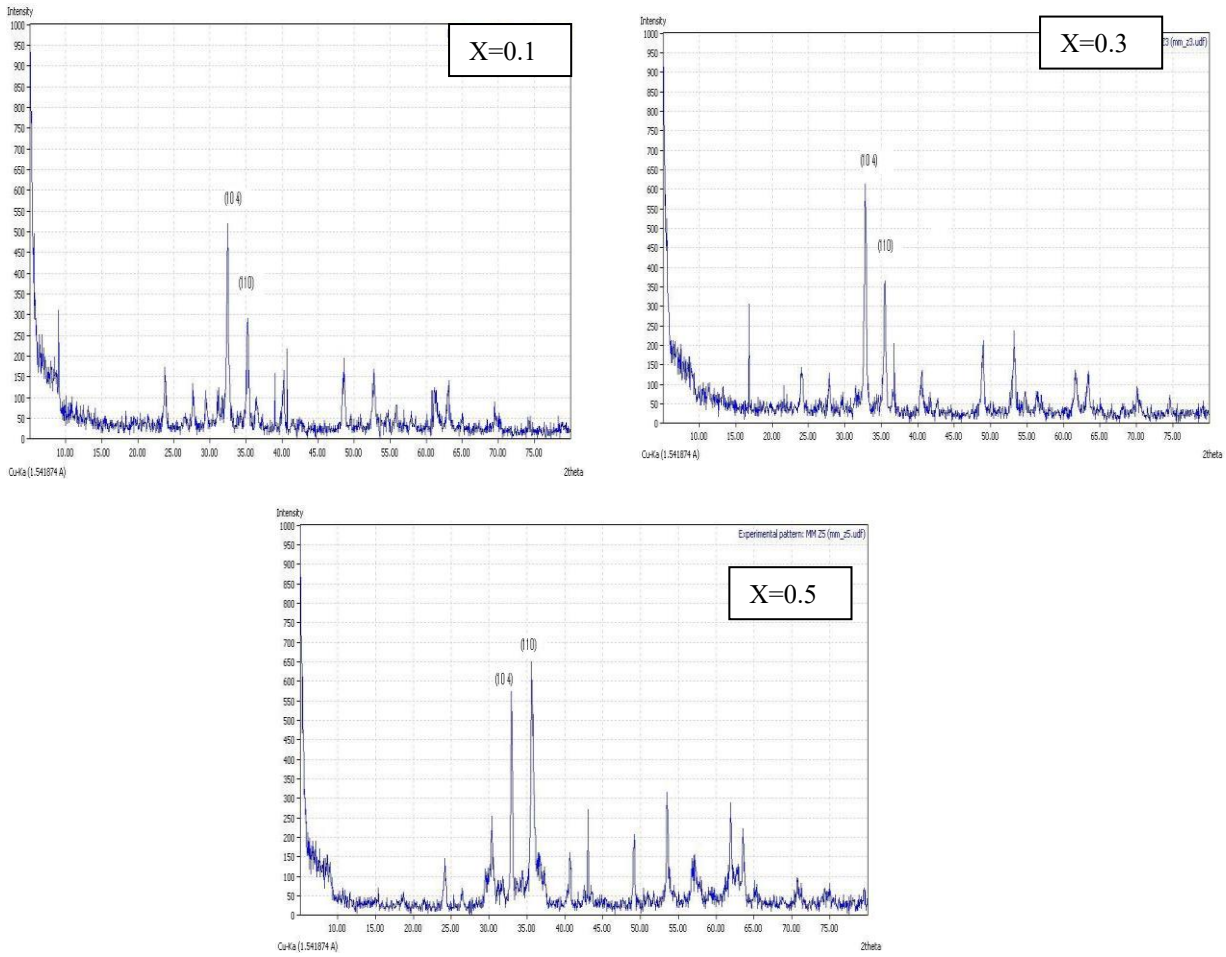

Figure.1. Shows the XRD plots for the $Sr_xMg_{1-x}TiO_3$ at x=0.1, 0.3, 0.5

B. SEM & EDAX

Figure 2 illustrates the scanning electron micrographs of Strontium-doped Magnesium Titanate at various Strontium compositions, sintered at 1250°C. Notably, the grain size is largest at a Strontium composition of x=0.5, attributed to the similar atomic radii of Strontium and Magnesium. The average grain size ranges from 1 to 2 µm and tends to increase with higher Strontium content. Additionally, the density of the compound rises with increasing Strontium composition, while porosity analysis via scanning morphology reveals lower porosity at higher densities. The incorporation of

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Strontium into the perovskite lattice structure facilitates grain growth. EDAX analysis confirms the stoichiometric balance of the elemental composition in the samples.

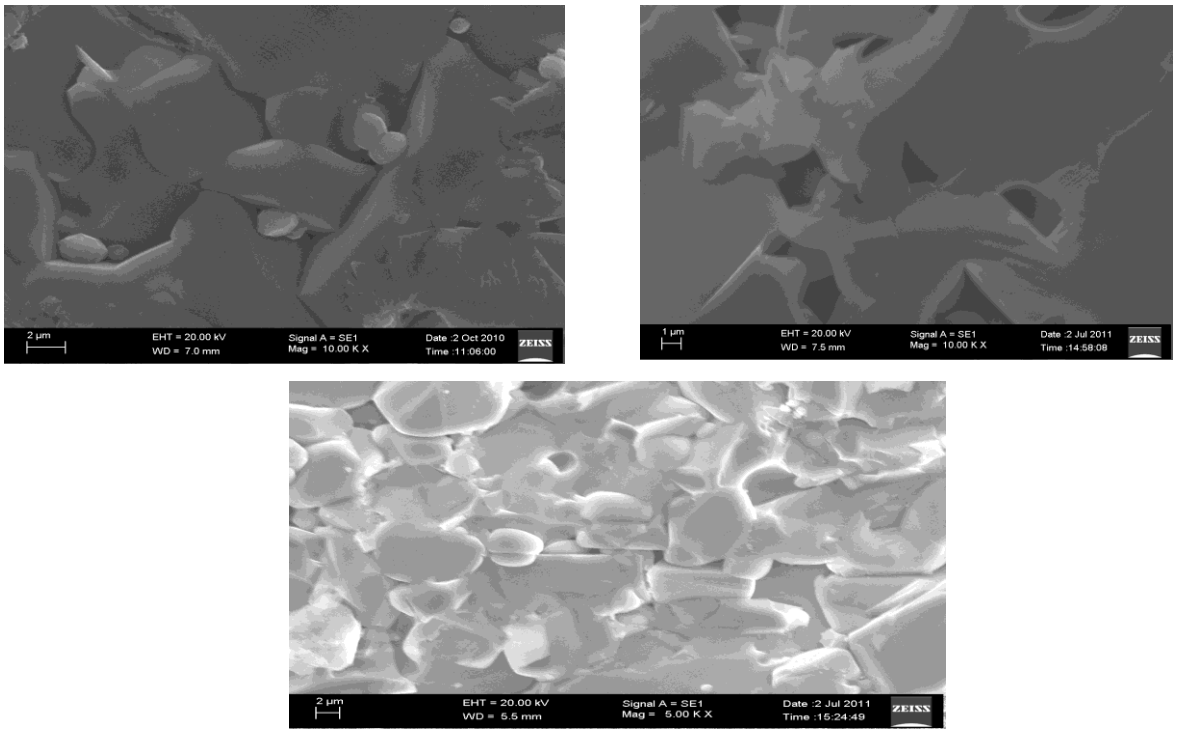

Figure 2. Shows the scanning electron micrographs of $sr_xmg_{1-x}tio_3$ at x=0.1, 0.3, 0.5

C. **Dielectric Measurements**

To perform dielectric measurements, the sample was ground into a fine powder over a period of 24 hours. Subsequently, 1 gram of this powder was mixed with a small quantity of polyvinyl alcohol (PVA) to serve as a binder. The resulting mixture was then shaped into pellets using a pellet-making machine, producing pellets with a diameter of 1.2 cm and a thickness of 2 mm, which were compressed under a hydraulic pressure of 10 tons per square centimeter. These pellets were then sintered at a temperature of 1250°C for 6 hours. After this treatment, the pellets were used for dielectric measurements with the HIOKI 3532-50 LCR Hitester, across a range of frequencies and temperatures. As shown in Figure 3, the dielectric constant varied with frequency, reaching its maximum at a composition of x=0.3, after which it decreased across all frequencies. This reduction may be explained by the lower ionic polarizability of magnesium in comparison to that of strontium.

From fig. 3, the dielectric constant variation with frequency and we found that the dielectric constant is maximum at composition x=0.3 and the decreased at all frequencies because which might be the smaller ionic polarizability of magnesium than that of strontium.

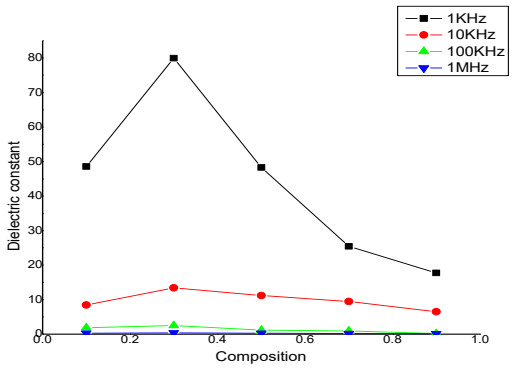

Figure.3. Shows the dielectric constant variation with composition of strontium in $mgtio_3$

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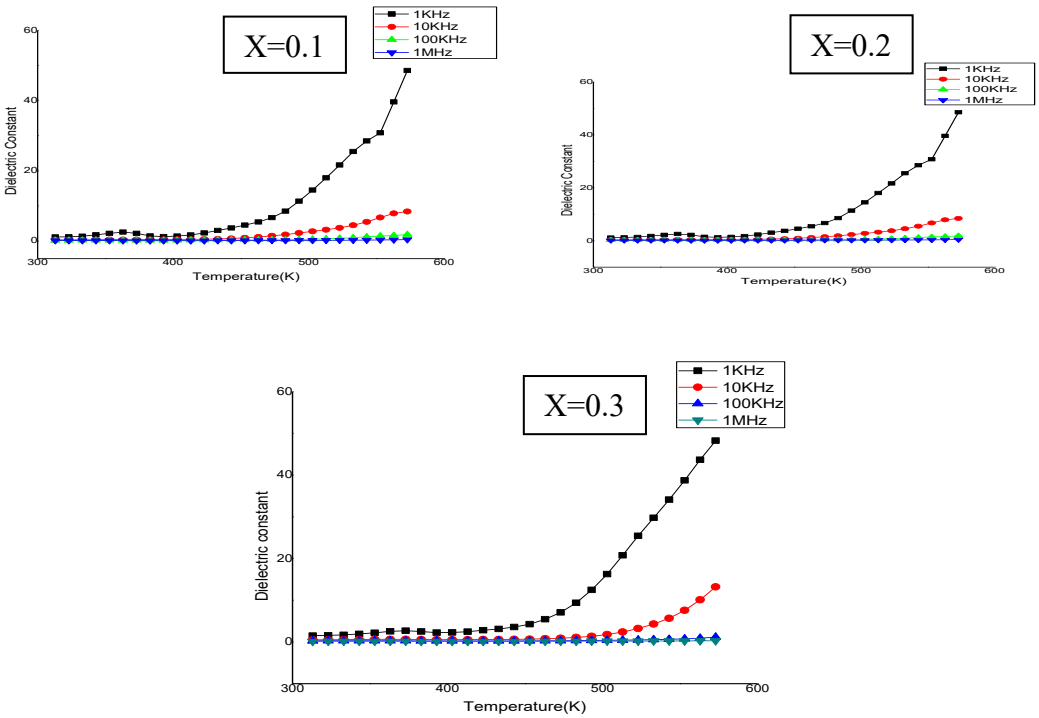
Figure 4 illustrates how the dielectric constant of $Sr_xMg_{1-x}TiO_3$ (with x values of 0.1, 0.3, and 0.5) varies with temperature across different frequencies. The findings reveal that the dielectric constant increases with rising temperature, while a decrease in frequency results in a lower dielectric constant. This phenomenon is linked to the off-center displacement of Sr^{2+} ions at the sites of Mg^{2+} ions within the magnesium titanate ceramics.



IV. CONCLUSION

Strontium-doped magnesium titanate ceramic samples were produced through the solid-state reaction method, with a sintering temperature of 1250°C. Structural analysis indicated that the samples displayed an octahedral ilmenite structure, with particle sizes diminishing as the strontium content increased. The addition of strontium improved the thermal stability of the magnesium titanate ceramics, which could be beneficial for applications involving strontium-based titanate microwave ceramics. The dielectric constant rose with increasing strontium content up to x=0.3, after which it started to decrease. Furthermore, changes in the loss tangent were noted with variations in temperature and frequency, revealing that both the dielectric constant and dielectric loss increased with temperature, while both parameters decreased as the measured frequencies increased.

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