**Microwave Properties of SrY2O4 Spinel Synthesized for Advanced Applications**

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This study investigated the microwave dielectric properties and microstructures of SrY2O4 ceramics fabricated using the conventional solid-state route. The investigation revealed that the dielectric constant (ε*r*) values exhibited saturation behavior, reaching a plateau within the range of 14-15. Notably, high quality factor (*Q*⋅*f*) values ranging from 64,500 to 92,000 (measured at 11 GHz) were achievable when sintering temperatures were maintained between 1480 °C and 1520 °C. Furthermore, the temperature coefficient of resonant frequency (τ*f*) demonstrated minimal sensitivity to variations in sintering temperature. Significantly, SrY2O4 ceramics sintered at 1520 °C for 4 h exhibited a desirable combination of microwave dielectric properties, including a high ε*r* value of 15, an exceptional Q⋅f value of 92,000, and a favorable τ*f* value of -6.2 ppm/°C. These findings suggest the potential of SrY2O4 ceramics, particularly under these specific sintering conditions, for applications in microwave devices.

Keywords: spinel, quality factor, temperature coefficient of resonant frequency, microwave, dielectric constant

# Introduction

The increasing demands placed on the microwave frequency range systems and devices by emerging communication standards, satellite broadcasting, and mobile systems necessitate the development of devices with enhanced functionality while maintaining high energy efficiency. Dielectric materials are crucial components for such applications. They act as insulators, influencing factors like signal propagation, power loss, and overall device performance. Thus, the development of novel high-quality dielectric materials is a key strategy for achieving enhanced functionality and energy efficiency in the microwave frequency range [1, 2].

Traditionally, microwave dielectric materials are characterized by several key parameters: high electromagnetic quality factor (*Q* or *Q*∙*f*, where *f* represents frequency), reasonably low dielectric constant (ε*r*) values enabling desired operational frequencies and component miniaturization, temperature stability of these parameters, and ideally, a near-zero temperature coefficient of resonant frequency (τ*f*). Increasing the *Q*-factor of the material allows us to compensate for the signal attenuation caused by the transition to the millimeter wave range. Low values of the dielectric constant reduce the signal-transmission time delay, which is proportional to the square root of the dielectric constant [3]. And reaching a temperature coefficient of the resonance frequency close to zero means compensation for the drift of the operating frequency caused by temperature fluctuations. Materials exhibiting this combination of properties are crucial for the development and production of microwave substrates, capacitors, resonators and antennas [4], filters [5].

Often, from the point of view of production, dielectric materials in the form of ceramics are sufficient. Ceramics are cheaper, easier to produce, chemically and mechanically resistant. Within the realm of ceramics, two specific material classes hold promise for significant advancements: ilmenites and spinels [6]. These materials exhibit exceptionally high *Q*-factors [7]. And **spinels** with a general formula of AB2O4, where A and B can be various metal cations [8], are interesting because their characteristics are easily modified by changing their chemical composition, which makes it possible to obtain materials with various functionalities and nature.

Recent research (within the past 3 years) reports about high-*Q* titanates and yttrates with the spinel structure, high *Q*-factor and low dielectric constant. For examples, studies have reported the successful synthesis of Mg3Ga2SnO8 spinel exhibiting a high quality factor *Q*∙*f* = 189052 GHz, a temperature coefficient of resonant frequency τ*f* = −34.25 ppm/°C, and a dielectric constant ε*r* = 9.3 after sintering at 1470°C [9]. Similarly, Zn3Ga2SnO8 ceramics demonstrated *Q*∙*f* = 88725 GHz, τ*f* = −25.1 ppm/°C, and ε*r* =10.6 after sintering at a lower temperature = 1320°C [10]. In the work [11] *Q*∙*f* = 84090 GHz, ε*r* = 14.78, τ*f* = −14.98 ppm/°C were achieved for the SrY2O4 ceramics after sintering at 1475°C. These studies show the potential of spinel materials for 5G communication and other microwave applications.

SrY2O4-based dielectrics are widely used for fuel cells, displays, coatings, high-quality microwave ceramics, and phosphors [12]. SrY2O4 is an inter oxide in SrO and Y2O3 pseudo-binary phase diagram. It has attracted immense interest as a promising host lattice owing to its thermal and chemical stability. It should be noted that SYO is promising the main lattice for doping with various rare and non-rare earth elements elements. This ensures chemical and thermal stability, the prestigious main lattice in the AB2O4 oxide family. SYO is synthesized by various routes, such as solid-state, sol-gel, combustion, microemulsion and aldo-keto methods. SYO on doping from various elements give different radiation spectra at different excitation wavelengths in UV and vacuum UV excitation [13–19]. In addition, SYO phosphors are more attractive due to high water resistance and unique glow characteristics [20]. On the other hand, recent works report a decrease in the temperature coefficient of the dielectric constant at the cost of the Q factor value [3] by adding CaTiO3.

Further research is needed to remove the limitations of temperature stability for spinel-structured microwave dielectrics, but before reaching a near-zero temperature coefficient of the resonant frequency in-depth study of the synthesis of high-*Q* SrY2O4 is necessary. It includes the optimization of synthesis conditions, particularly sintering temperature and duration. In addition, the effect of sintering regime on the density of ceramics requires further research, since density is known to have a significant effect on the final density, porosity, energy loss and quality factor of the materials. Increased porosity, as well as the presence of defects (impurity atoms, dislocations), negatively affect the characteristics of materials, namely, increases dielectric losses. Porosity and defects act as energy dissipation sites, leading to elevated electrical losses. Schlomann's theory posits that dielectric loss tangent is primarily determined by anharmonic terms in the crystal's potential energy [21]. Lattice defects can dramatically increase dielectric loss by disrupting the crystal's charge periodicity. Studies on systems like Ba(Zn,Ta)O3–BaZrO3 and (Zr,Sn)TiO4 corroborate this theory, demonstrating a strong correlation between defect concentration and dielectric loss [22].

As the sintering process progresses, particle coalescence and interdiffusion occur at the contact points, leading to a progressive reduction in inter-particle void space [23]. This phenomenon translates to an increase in the overall packing efficiency and, consequently, a higher material density. Extending the sintering time allows for a more extensive progression of these bonding mechanisms. This extended duration facilitates a greater degree of particle rearrangement and pore elimination, ultimately resulting in a denser final product with a lower residual porosity content. Porosity refers to the volume fraction of voids or empty spaces present within the material matrix. However, it is crucial to acknowledge the existence of an optimal sintering time window. Excessive sintering durations can induce detrimental effects such as grain coarsening and exaggerated atomic diffusion. These phenomena can lead to a phenomenon known as over-sintering, which can paradoxically decrease the final density and induce pore formation. Therefore, meticulous control and optimization of the sintering time are paramount to achieving the desired balance between density, porosity, and other material properties.

In this work, the influence of sintering time and temperature on the densification and microwave dielectric properties of SrY2O4 ceramics was investigated. The effect of substitution on microwave characteristics in the Sr and Y sublattices with Sm and Ca was explored.

# Experimental

SrY2O4, Sr0.5Ca0.5Y2O4, SrYSmO4 ceramics were prepared from high-purity raw materials of SrCO3, CaCO3, Sm2O3, and Y2O3 (≥99%). Since Y2O3 and Sm2O3 can easily absorb moisture in the air, it was preheated before weighing, and heated at 900 °C for 2 h. The ceramics were prepared by a traditional solid-phase reaction method [20], weighed according to the stoichiometric ratio. The stoichiometric quantities of reagents were introduced simultaneously with distilled water into a planetary Retsch PM100 with chalcedony balls and milled for 8 hours at a speed of 300 rpm. The extracted suspensions were dried in a drying oven at 100 °C and forced through a 200-mesh sieve and calcined at 1350 °C for 4 h. The calcined reagent was ground with distilled water into a fine powder for 12 h using a planetary Retsch PM100 with chalcedony balls and milled at a speed of 300 rpm. The fine powder, together with the 5% polyvinyl alcohol solution as a binder, was pressed into pellets with a thickness of 5 mm and diameter of 10 mm under a pressure of 500 kg/cm2. Finally, the ceramic samples were sintered for 2, 4 and 6 h in the temperature range of 1480–1560 °C. The heating rate and the cooling rate were both set at 200 ◦C/h.

X-ray powder diffraction was performed using a DRON-4-07 diffractometer (Cu Kα radiation, 40 kV, 20 mA) for the analysis of phases formed during the synthesis. The unit cell parameters of the samples were calculated using the Le Bail procedure and the FullProf software [24].

Surface of ceramics was determined using a scanning electron microscope JEM 10CX II (JEOL) and scanning electron microscope SEC miniSEM SNE 4500MB equipped with EDAX Element PV6500/00 F spectrometer. Using the Fiji [25] calculations by the method of the equivalent circle diameter were performed [26]. Sampling was performed in three different regions based on at least 50 grains in each region.

The density of the sintered specimens, as a function of sintering temperature, was measured by the liquid Archimedes method using distilled water as the liquid. The dielectric constants (εr) and Q·f values at microwave frequencies were measured using Agilent PNA-L Network Analyser (model N5230A, Agilent, USA, 10 kHz–20 GHz) using the resonant cavity method. For temperature coefficient of resonant frequency (τf), the technique is the same as that of quality factor measurement. The test cavity is placed over a thermostat and the temperature range used is 125 °C to 180 °C. The τ*f* (ppm/°C) is calculated by noting the change in resonant frequency:

(1)

where f1 is resonant frequency at T1 and f2 is resonant frequency at T2.

# Results and discussion

X-ray diffraction (XRD) analysis revealed the formation of a homogeneous SrY2O4 phase with an orthorhombic Pnma space group in the ceramic sintered at 1520 °C for 4 h (**Fig. 1**). Lattice parameters obtained from the pattern were a = 10.085(3) Å, b = 11.914(4) Å, and c = 3.412(1) Å. The bulk density achieved 97.5 % of the theoretical X-ray density, indicating minimal porosity. Furthermore, the observed composition matched the stoichiometric SrY2O4 formula.

XRD patterns for SrY2O4 ceramics sintered at 1480–1520 °C for varying durations exhibited similar characteristics. No evidence of secondary phases was detected, suggesting their potential presence below the detection limit of the technique. Notably, the XRD patterns remained consistent irrespective of the sintering time within this range.

SrY₂O₄ crystallizes in the orthorhombic Pnma space group. Sr²⁺ cations occupy eight-coordinate sites, bonded to eight surrounding O²⁻ anions. Y³⁺ cations reside in two crystallographically distinct environments. In the first Y³⁺ site, six O²⁻ neighbors form a distorted octahedral coordination, with a mixture of corner- and edge-sharing YO₆ units. The second Y³⁺ site also exhibits octahedral coordination with O²⁻, but with a different arrangement involving both corner- and edge-sharing YO₆ octahedra (**Fig. 1**, inset).

Scanning electron microscopy (SEM) images of SrY2O4 ceramics (**Fig.2**) reveal the influence of sintering temperature and time on porosity and grain morphology. As the sintering temperature increases from 1480 °C (**Fig.2a**, **2b**, **2c**) to 1560 °C (**Fig.2g**, **2h**, **2i**), porosity diminishes. At 1520 °C for 4 h (**Fig.2e**), the microstructure becomes nearly non-porous due to grain growth. This sintering condition also promotes uniform grain size distribution. However, higher temperatures (>1520 °C, **Fig.2g, 2h, 2i**) or extended sintering times can lead to detrimental effects. Abnormal grain growth and a decline in grain uniformity become evident at elevated temperatures (**Fig.2g, 2h, 2i**), potentially impacting the microwave dielectric properties. Prolonging the sintering time at a constant temperature also promotes grain growth (**Fig.2f**, **2i**), as evidenced by the increased grain size observed when the sintering time extends from 2 to 6 h (**Fig.2d**, **2e**, **2f**).

Densification studies revealed that SrY2O4 ceramics consistently achieved densities exceeding 90% of the theoretical value across all sintering conditions (**Fig.3**). A clear dependence of density on both sintering temperature and time was observed. As the sintering temperature increased from 1480 °C to 1520 °C, the density exhibited a concomitant increase, reaching a maximum value at the latter temperature for 2, 4 and 6 hours (**Fig.3** curve 1, curve 2 and curve 3 respectively). This trend can be attributed to the diminishing porosity evident in the corresponding scanning electron microscopy (SEM) images (**Fig.2d**, **2e**, **2f**). However, exceeding this optimal temperature (1520 °C) can induce detrimental abnormal grain growth, ultimately leading to a reduction in density.

Sintering time also exerts a significant influence on the density of the ceramics. As illustrated in **Fig.** **3**, longer sintering durations generally promote grain growth, which initially contributes to an increase in density. However, excessively prolonged sintering times can lead to detrimental grain coalescence. This phenomenon results in a non-uniform distribution of grain sizes (**Fig.2f**, **2i**) and the formation of pores within the microstructure, ultimately causing a decrease in density.

For SrY2O4 ceramics, both the apparent density and the corresponding relative density exhibited a positive correlation with increasing sintering temperature from 1480 °C to 1520 °C. The maximum relative density of 97.5% was achieved under the optimal conditions of 1520 °C sintering temperature and a sintering time of 4 h, **Fig.3** curve 2.

Sintering temperature significantly influenced the grain size of SrY2O4 ceramics, with grains increasing from 1.6 µm to 8.3 µm, from 2.0 µm to 11.0 µm, and 3.1 µm to 12.8 µm for sintering durations of 2 h, 4 h and 6 h (**Fig.4**, curves 1, 2, 3), as the temperature rose from 1480 °C to 1560 °C. Furthermore, **Fig.** **2** confirms a direct correlation between grain growth and sintering times.

A strong correlation was observed between the dielectric constant (ε*r*) and the sintering temperature of SrY2O4 ceramics (**Fig.** **5**), mirroring the previously established relationship between density and sintering temperature (attributable to the inverse relationship between density and porosity). ε*r* exhibited a monotonic increase with increasing sintering temperature, followed by a distinct decrease at temperatures exceeding 1520 °C for sintering durations of 2, 4 and 6 h (**Fig.5** curves 1, 2, 3). This initial enhancement in ε*r* can be ascribed to the densification of the ceramic with increasing temperature, leading to a reduction in porosity. However, extended sintering times (>4 h) can induce the formation of non-uniform grain structures, potentially leading to a degradation of the dielectric constant. Notably, well-sintered SrY2O4 ceramics displayed ε*r* values ranging from 14 to 15 within the temperature window of 1500–1520 °C. The maximum ε*r* of 15 was achieved for samples sintered at 1520 °C for 4 h, **Fig.5** curve 2, further emphasizing the critical role of density in governing the variation of ε*r*.

The quality-factor of the SrY2O4 ceramics *Q*·*f* exhibited a trend mirroring that of density, highlighting its dependence on both intrinsic and extrinsic factors (**Fig.** **6**). Dielectric loss in this material originates primarily from lattice vibrational modes, but also from pores and secondary phases [27]. The *Q*⋅*f* value demonstrated a rise with increasing sintering temperature, reaching a maximum at 1520 °C, followed by a decrease for sintering durations of 2, 4 and 6 h (**Fig.6**, curves 1, 2, 3). This behavior can be attributed to the presence of porosity at lower sintering temperatures, which diminishes the *Q*⋅*f* value. Additionally, extended sintering times (>4 h) can lead to a degradation of *Q*⋅*f* in SrY2O4 ceramics. As observed in other microwave dielectric materials, density plays a critical role in controlling dielectric loss. The observed consistency between the trends of *Q*⋅*f* and density suggest that density variation is the dominant factor influencing *Q*⋅*f*. Furthermore, a uniform grain morphology contributes to reduced dielectric loss, ultimately enhancing the *Q*⋅*f* value of the ceramics. Notably, a peak *Q*⋅*f* value of 92,000 GHz was achieved for samples sintered at 1520 °C for 4 h (**Fig.6** curve 2), highlighting the potential of SrY2O4 ceramics in practical microwave applications due to their low dielectric loss.

As expected, the temperature coefficients of resonant frequency (τf) of SrY2O4 ceramics exhibited minimal variation due to the consistent material composition and the absence of detectable secondary phases, which are known to significantly influence this parameter, **Fig.7** curves 1, 2, 3. The measured τ*f* values ranged from -5.4 ppm/°C to -7.2 ppm/°C, with a value of -6.2 ppm/°C obtained for SrY2O4 sintered at 1520 °C for 4 h (**Fig.7** curve 2). This minimal variation reinforces the control achieved over material composition and microstructure during the sintering process.

The impact of cation substitution on the sublattices within the spinel (AB₂O₄) of SrY₂O₄ was investigated, specifically focusing on its influence on microwave properties. Substitutions of both sites of the spinel structure were carried out.

In the case of A-site substitution strontium (Sr²⁺) was partially replaced with calcium (Ca²⁺) to form Sr₀.₅Ca₀.₅Y₂O₄. This substitution is expected to affect the lattice parameters and potentially the ionic polarizability within the unit cell. As shown in **Tab. 1**, such A-site substitution has been demonstrated to decrease the dielectric constant (ε*r*). This decrease in ε*r* can be beneficial for expanding the operational frequency range of microwave materials, as materials with lower ε*r* can be used at higher frequencies.

**Table 1.** Dielectric constant, quality factor and temperature coefficient of dielectric permeability of ceramic materials with a spinel structure.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Parameter  Composition | ε | *Q*∙*f* | τ*f* | Density,  % | Sintering temperature,  °C |
| SrY2O4 | 15 | 92000 | -6.2 | 97.5 | 1520 |
| Ca0.5Sr0.5Y2O4 | 12.5 | 46000 | -19.8 | 92.8 | 1520 |
| SrYSmO4 | 15.5 | 42000 | -8 | 94.2 | 1480 |

In the case of B-site substitution yttrium (Y³⁺) was partially replaced with samarium (Sm³⁺) to form SrYSmO₄. B-site substitutions can influence factors like the bond lengths and bond strengths within the lattice. While the substitution of Y³⁺ with Sm³⁺ appears to have minimal impact on ε*r*, it has significant influence on the sintering temperature required to achieve optimal densification. It was shown that when the sintering temperature of materials containing samarium increases from 1420 to 1480 degrees, the density of ceramics increases from 90.7 to 94.2%. A further increase in the sintering temperature to 1520 degrees leads to a monotonous decrease in the relative density to 91.2%. Notably, SrYSmO₄, the material containing samarium via B-site substitution, achieved its maximum density at a sintering temperature of 1480 °C. This observed temperature represents a 40 °C decrease compared to the optimal sintering temperature of 1520 °C required for SrY₂O₄ (without samarium substitution). This finding suggests that B-site substitution with samarium potentially influences the densification kinetics of the material, allowing for the achievement of high density at a lower temperature. This decrease in sintering temperature could offer practical advantages by reducing energy consumption during the fabrication process.

# Conclusions

This study explored the microwave dielectric properties of SrY2O4 ceramics. Notably, all fabricated ceramics exhibited an orthorhombic crystal structure belonging to the Pnma space group. The investigation established optimal sintering conditions by analyzing sintered specimens. SrY2O4 ceramics sintered at 1520 °C for 4 h demonstrated exceptional microwave dielectric properties, characterized by a dielectric constant (ε*r*) of approximately 15, a high quality factor (*Q*⋅*f*) of around 92,000 (measured at 11 GHz), and a favorable temperature coefficient of resonant frequency (τ*f*) of -6.2 ppm/°C. These results highlight the potential of SrY2O4 as a suitable candidate material for wireless communication systems, owing to its desirable combination of properties crucial for microwave device applications.

# Acknowledgements

This work was supported in part by the National Research Foundation of Ukraine within the framework of the “Microwave Devices Based on Resonant Structures with Metamaterial Properties for the Life Protection and Information Security of Ukraine”, project ID 2021.01/0030.

# Declaration of interest statement

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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**Figure 1.** Experimental (dots) and calculated (lines) room-temperature powder X-ray diffraction patterns of SrY2O4 ceramic sample, sintered at 1520 °C for 4 h. Bars indicate the peak positions.

**Figure 2.** Scanning electron microscopy photographs of SrY2O4 ceramics sintered at 1480 °C for 2 (a), 4 (b) and 6 hours (c); 1520 °C for 2 (d), 4 (e) and 6 hours (f); 1560 °C for 2 (g), 4 (h) and 6 hours (i).

**Figure 3.** Relative density in SrY2O4 ceramics on sintering temperature, with sintering durations of 2 hours (curve 1), 4 hours (curve 2), and 6 hours (curve 3).

**Figure 4.** Dependence of grain size in SrY2O4 ceramics on sintering temperature, with sintering time of 2 h (curve 1), 4 h (curve 2), and 6 h (curve 3).

**Figure 5.** Dielectric constant on sintering temperature of SrY2O4 ceramics, with sintering time of 2 h (curve 1), 4 h (curve 2), and 6 h (curve 3).

**Figure 6.** Dependence of quality factor (*Q*·*f*) value on sintering temperature of SrY2O4 ceramics, with sintering time of 2 h (curve 1), 4 h (curve 2), and 6 h (curve 3).

**Figure 7.** Dependence of the temperature coefficient of the frequency (τ*f*) value on sintering temperature of SrY2O4 ceramics, with sintering time of 2 h (curve 1), 4 h (curve 2), and 6 h (curve 3).

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Fig. 1.

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Fig. 2.

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Fig. 3.

A graph of different colored lines

Description automatically generated

Fig. 4.

A graph of the temperature

Description automatically generated

Fig. 5.

A graph of different colored lines

Description automatically generated

**Fig. 6.**

A graph of different colored lines

Description automatically generated

**Fig. 7.**