



$K_{0.5}Na_{0.5}VO_3-SiO_2$ Co-sintering Agent of ceramic $Mg_{0.8}Ti_{0.2}O_3$ as Dielectric Material Candidate

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Info Article:

Sent:
January 11, 2023

Revision:
July 11, 2023

Accepted:
July 11, 2023

Keywords:

Sintering,
 $Mg_{0.8}Zn_{0.2}TiO_3$,
Solid state.

Abstract

This paper focuses on the characteristics of $K_{0.5}Na_{0.5}VO_3$ (KNV) and SiO_2 when added to $Mg_{0.8}Zn_{0.2}TiO_3$ (MZT) material to reduce the sintering temperature. Initially, a single phase of $Mg_{0.8}Zn_{0.2}TiO_3$ (MZT) was synthesized using the conventional solid-state reaction method at $850^\circ C$ for 4 hours. Subsequently, $(K_{0.5}Na_{0.5})VO_3$ was formed as a single phase at a temperature of $500^\circ C$ for 2 hours. The reduction of MZT sintering temperature was then carried out by adding $K_{0.5}Na_{0.5}VO_3$ and SiO_2 , and sintering at $950^\circ C$ for 4 hours. Adding $K_{0.5}Na_{0.5}VO_3$ and SiO_2 resulted in a maximum density value of 2.76 g/cc and an average grain size of 3 μm based on scanning electron microscopy (SEM) analysis. The optimal composition was found to be 0.7MZT-0.25KNV-0.05 SiO_2 . X-ray diffraction (XRD) characterization using the Rietveld method revealed the presence of three phases: $(Mg/Zn)TiO_3$, $(K/Na)VO_3$, and $MgTi_2O_5$. Based on the results, it can be concluded that the $(K/Na)VO_3-SiO_2$ material can be effectively utilized as a sintering agent for $(Mg/Zn)TiO_3$, reducing the sintering temperature of the material.

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INTRODUCTION

Dielectric materials are materials used in electronic applications[1] [2] [3] [4] [5]. One of the candidate dielectric materials is $MgTiO_3$. The dielectric material must meet characteristics such as a high dielectric constant (ϵ_r), and high-quality factor (Q_{xf}) because it is necessary to achieve high-frequency selectivity, stability in terms of transmitting and receiving components, as well as a temperature coefficient that is low or close to zero when the resonant frequency occurs (τ_f)[6] [7]. To be applicable, $MgTiO_3$ has characteristics such as a dielectric constant of ~ 17.4 , a temperature coefficient close to zero, and a Q_{xf} quality factor value of $\sim (10,000-30,000)$ at a frequency of 7 GHz[8]. The magnitude of the $Mg_{0.8}Zn_{0.2}TiO_3$ Lattice Parameter (x 0.1 nm), among others, a = b = 4.9851 and c = 13.7116[8].

Several exciting studies were conducted on $MgTiO_3$ material to improve its properties and characteristics[5] [9] [10] [11]. One of them is in the sintering process. Since the $MgTiO_3$ synthesis process requires a very high temperature $\sim 1300 C$ [8], many studies have been conducted to reduce the sintering temperature. The addition of a dopant to this material can be an alternative that can be done to reduce the sintering temperature[12] [13] [14] [15] [16] [17]. Ermawati et al. examined the $MgTiO_3$ material with zinc substitution to form $Mg_{1-x}Zn_x TiO_3$ composition using the wet solution mixing method, which resulted in a relative density of 90% at a temperature of $1300^\circ C$. Zinc serves to speed up the reaction, increase density, and increase relative permittivity[18].

KNaVO_3 material is included in the perovskite structure. Based on the ionic radius of Na^+ is 0.102 nm, similar to the K^+ radius of 0.138 nm. Na^+ can replace the position of the K^+ ion because of the similarity in atomic radius [19] [20]. In addition, from previous studies, vanadium is a catalyst capable of increasing the catalytic activation of titanium silicalite material. This doping aims to make the KNV material a co-sintering agent material. The V_2O_5 material has a low melting point, around 650°C [21]. In another study, V_2O_5 material was used to dope ceramic materials ($\text{Zr}_{0.8}\text{Sn}_{0.2}$) TiO_4 . However, the results obtained were still too high at the sintering temperature of 1300°C with adding 1 wt% V_2O_5 [22]. Akinori Kan's research uses $\text{Mg}_4(\text{Nb}_{2-x}\text{V}_x)\text{O}_9$ material, where V will replace Nb. With the composition $x = 0.0625$, the sintering temperature can be reduced to 1025°C . V_2O_5 material is a good candidate for lowering the sintering temperature [23]. In Liu's research, V_2O_5 was synthesized with copper (Cu) to become CVO. The CVO material will be doped with $(\text{Zn}_{0.7}\text{Mg}_{0.3})\text{TiO}_3$ so that the best results say the sintering temperature drops to 950°C when 0.2% by weight of CVO is added [14]. In another research, adding V_2O_5 can lower the sintering temperature of $\text{Zn}_{0.8}\text{Mg}_{0.2}\text{TiO}_3$ at 1100°C for 4 hours to get a density of 90% [21]. Previous studies also stated that KNV can work at temperatures of 500°C [24].

This paper discusses the characteristics of $\text{K}_{0.5}\text{Na}_{0.5}\text{VO}_3$ (KNV) and SiO_2 added to $\text{Mg}_{0.8}\text{Zn}_{0.2}\text{TiO}_3$ (MZT) material in terms of densification, structure, and microstructure to reduce its sintering temperature. Provision of SiO_2 doping glass, which has complex properties. Due to this hard nature, SiO_2 will reinforce the MZT-KNV material.

RESEARCH METHODS

The materials used in this study include synthetic MgO , ZnO , and TiO_2 powders (>95% Merk) for synthesizing $\text{Mg}_{0.8}\text{Zn}_{0.2}\text{TiO}_3$ materials. Meanwhile, for the synthesis of $\text{K}_{0.5}\text{Na}_{0.5}\text{VO}_3$, synthetic powders of K_2CO_3 , Na_2CO_3 , and V_2O_5 are needed. Other ingredients are synthetic SiO_2 powder, distilled water, 96% alcohol, and acetone. The solid reaction method synthesized the $x\text{MZT}-y\text{KNV}-(1-x-y)\text{SiO}_2$ material with variation $x=0.6-0.9$. The Variation of sample such as 0,9 MZT-0,05 KNV-0,05 SiO_2 ; 0,8 MZT-0,15 KNV-0,05 SiO_2 ; 0,7 MZT-0,25 KNV-0,05 SiO_2 ; 0,6 MZT-0,35 KNV-0,05 SiO_2 . $\text{Mg}_{0.8}\text{Zn}_{0.2}\text{TiO}_3$, $\text{K}_{0.5}\text{Na}_{0.5}\text{VO}_3$, and SiO_2 powders were weighed according to the calculated stoichiometric ratios. Then all the powders are mixed, put in a jar, and added with alcohol as a milling medium. This powder mixture was milled with a custom rotary attritor at 600 rpm for 6 hours. Furthermore, the alcohol content is removed using a rotary evaporator. The dry $x\text{MZT}-y\text{KNV}-(1-x-y)\text{SiO}_2$ material was then crushed with a mortar to form a powder. The $x\text{MZT}-y\text{KNV}-(1-x-y)\text{SiO}_2$ powder was pelleted and sintered at 950°C .

Characterization using X-Ray Diffraction (X-ray X'Pert Diffractometer (Philips) using $\text{CuK}\alpha$ radiation in a two θ range from 15 to 65 and step size of 0.02) is carried out to find out what phases are present in the material. This test was carried out as powder samples on MZT and KNV materials after being calcined. At the same time, the synthesized material MZT-KNV- SiO_2 was tested in disk form after sintering. The results of the XRD characterization are in the form of a graph between intensity and diffraction angle. To determine the phase of the material, an analysis of the XRD results was carried out using the Match! Software. From the XRD data, it is also possible to obtain the materials' lattice parameters, which are analyzed through refined, calculated, and measured patterns using the Rietica program based on the Rietveld method—density testing using the geometric approach. Where the dimensions of the sample are first measured to get the diameter and height of the selection, in addition, the mass of the model was weighed. Observation of the material's microstructure using Scanning Electron Microscopy (SEM). Before the sample was characterized by SEM, the sample's surface in the form of pellets was polished with velvet cloth and Al solution. Then the model is in an ultrasonic cleaner and followed by an etching process (heated) at a temperature of 925°C for 20 minutes.

RESULTS AND DISCUSSION

The results of XRD can be analyzed using Match! 2. From the results of the Match! 2 analysis, three phases were identified in the synthesis of the $x\text{MZT}-y\text{KNV}-(1-x-y)\text{SiO}_2$ material, namely the MgTiO_3 phase with entry number #000790831, the KNaVO_3 phase with entry number #000871120,

and the MgTi_2O_5 phase with entry number #000790833. The MgTi_2O_5 phase is the secondary phase of MgTiO_3 . These three phases exist in every variation of the $x\text{MZT-yKNV}-(1-x-y)\text{SiO}_2$ material starting from variations $x=0.9;0.8;0.7;0.6$. In Figure 1, it can be seen that there are three phases in each variation. This phase was identified in $x\text{MZT-yKNV}-(1-x-y)\text{SiO}_2$ with a sintering temperature of 950°C .

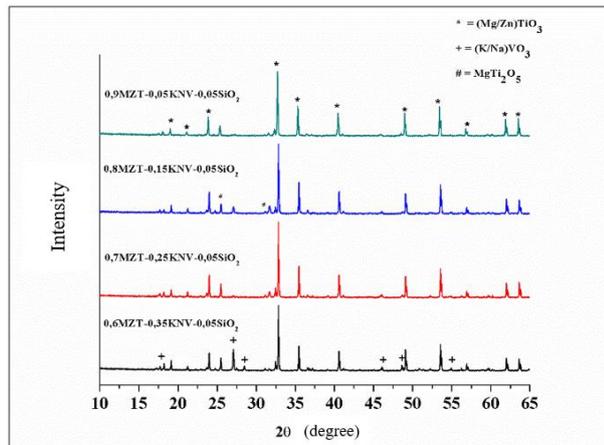


Figure 1. X-ray diffraction pattern on $x\text{MZT-yKNV}-(1-x-y)\text{SiO}_2$ material with sintering temperature of 950°C

Table 1. Density

Composition	Density (gr/cc)
0.9 MZT-0.05 KNV-0.05 SiO ₂	1.90
0.8 MZT-0.15 KNV-0,05 SiO ₂	2.26
0.7 MZT-0.25 KNV-0,05 SiO ₂	2.76
0.6 MZT-0.35 KNV-0,05 SiO ₂	2.26

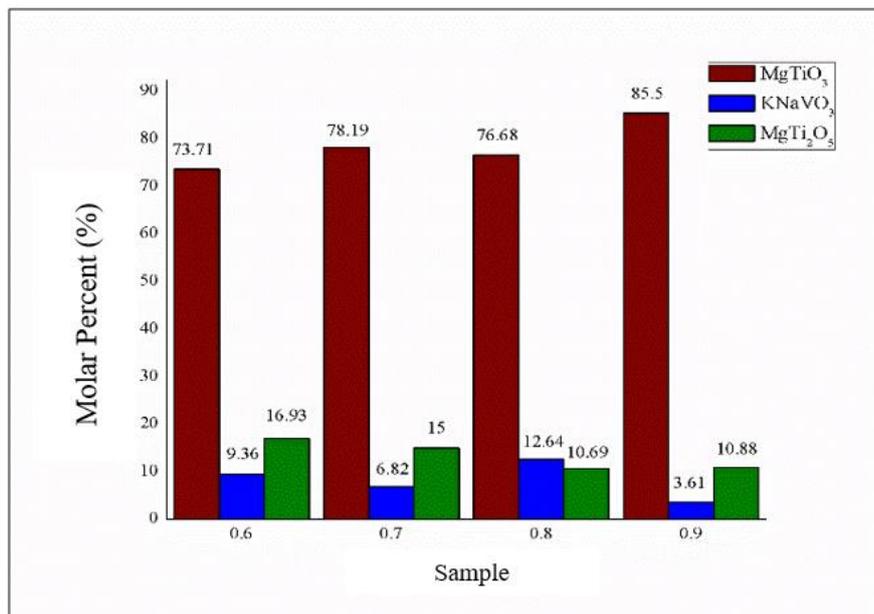


Figure 2. Molar percent ratio of the mixed MZT molar composition

The density of sample measurement shows that the variation $0.7\text{MZT-}0.25\text{KNV-}0.05\text{SiO}_2$ is a stable variation; this variation also has the most shrinkage at the sintering temperature of 950°C (Table 1). The density test in this study used geometry testing, which refers to the dimensions of the pellets after the sintering process. Quantitative analysis results were analyzed using the Rietveld method. In Figure 2, it can be seen that the increase in the molar percentage of MZT does not affect the molar ratio of KNV or MgTi_2O_5 . The $0.7\text{MZT-}0.25\text{KNV-}0.05\text{SiO}_2$ variation is stable because when the MZT percentage increases, the MgTi_2O_5 and KNV percentage decrease. It only occurs in the 0.7MZT-

0.25KNV-0.05SiO₂ variation, while the percentage of KNV and MgTi₂O₅ phases fluctuates in other variations. In the interpretation of 0.7MZT-0.25KNV-0.05SiO₂, the molar ratio for each stage is 78.19 ± 1.58 at MZT, 6.82 ± 0.77 at KNV, and 15.00 ± 0.62 on MgTi₂O₅.

As a comparison, the variation compared to the 0.7 MZT-0.25 KNV variation sintered at 950°C. is aimed at seeing the role of SiO₂.

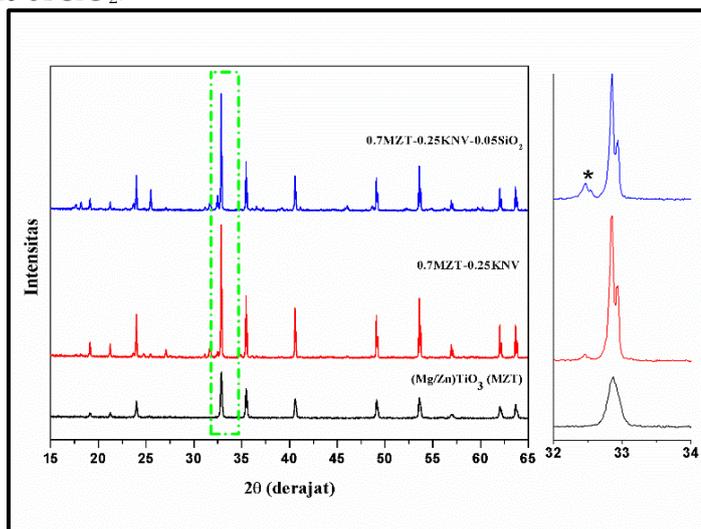


Figure 3 The results of the XRD characterization varied with the addition of SiO₂ and without SiO₂ sintered at 950°C.

In Figure 3, it can be seen that there is a higher secondary phase growth in the variation with the addition of SiO₂. Indicates the role of SiO₂ in accelerating and increasing grain growth. If you look at the lattice parameters of the three samples, it shows a shift. The results of the Rietveld analysis for the samples in Figure 3 are presented in Table 2.

Table 2. Rietveld analysis for the samples

Sampel	Density (gr/cc)	Fasa	Parameter Kisi (Å)			Molar (%)
			a	b	c	
0,7 MZT-0,25 KNV-0,05 SiO ₂	2.76	MZT	5,059002	5.059002	13.906686	78,19 ±
			± 0,000128	± 0.000128	± 0.000650	1.58
		KNV	10,575791	10.048646 ±	5.828188	6,82 ±
			± 0,004500	0.003554	± 0.001607	0.77
		MgTi ₂ O ₅	3.739849	9.749272	10.020041	15,00 ±
			± 0.000298	± 0.001105	± 0.00112	0,62
0,7 MZT-0,25 KNV	2.72	MZT	5,058496	5,058496	13,905257	85,6
			± 0,000115	± 0,000115	± 0,000583	± 2,54
		KNV	10,574344	10,070513	5,806014	10,94
			± 0,004197	± 0,003986	± 0,001770	± 1,49
		MgTi ₂ O ₅	9,753399	10,020373	3,742960	4,00
			± 0,002607	± 0,002396	± 0,000664	± 0,050
(Mg/Zn)TiO ₃		5.058949	5.058949	13.909819	-	
		± 0.000282	± 0.000282	± 0.000993		

To find out the role of SiO₂, a material with variations of 0.7 MZT-0.25 KNV was made and sintered at 950°C. From the results of the Match! The analysis found that the presence of SiO₂ accelerated and increased grain growth for lattice parameters, presented in Table 1. It can be seen that the shift in lattice parameters is only a little, namely in the range of 0.01. Indicates that no other materials react with MZT. When viewed from the molar percentage, the sample with the addition of SiO₂ produces a more dominant main phase. Meanwhile, with the addition of SiO₂, more secondary phases are formed.

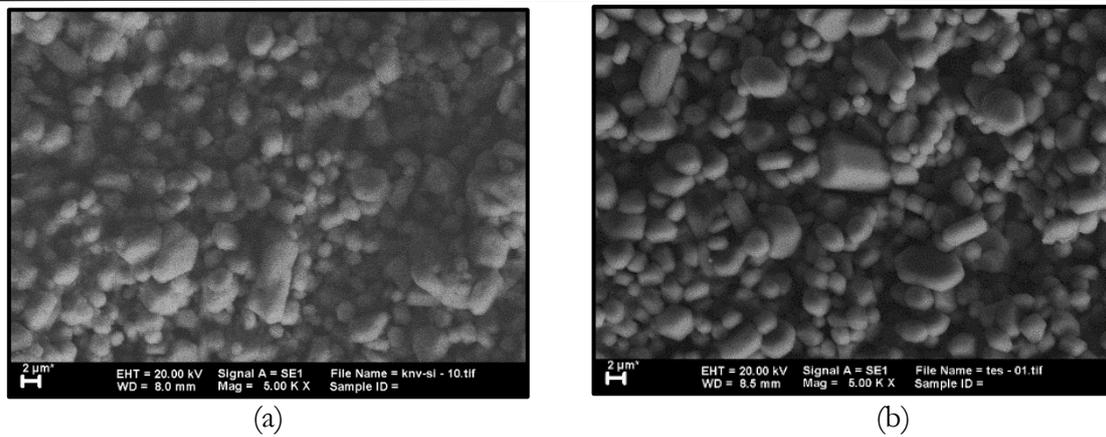


Figure 4. SEM test results (a) on a sample of 0.7 MZT-0.25 KNV-0.05 SiO_2 and (b) on a sample of 0.7 MZT-0.25 KNV sintered at 950°C for 4 hours.

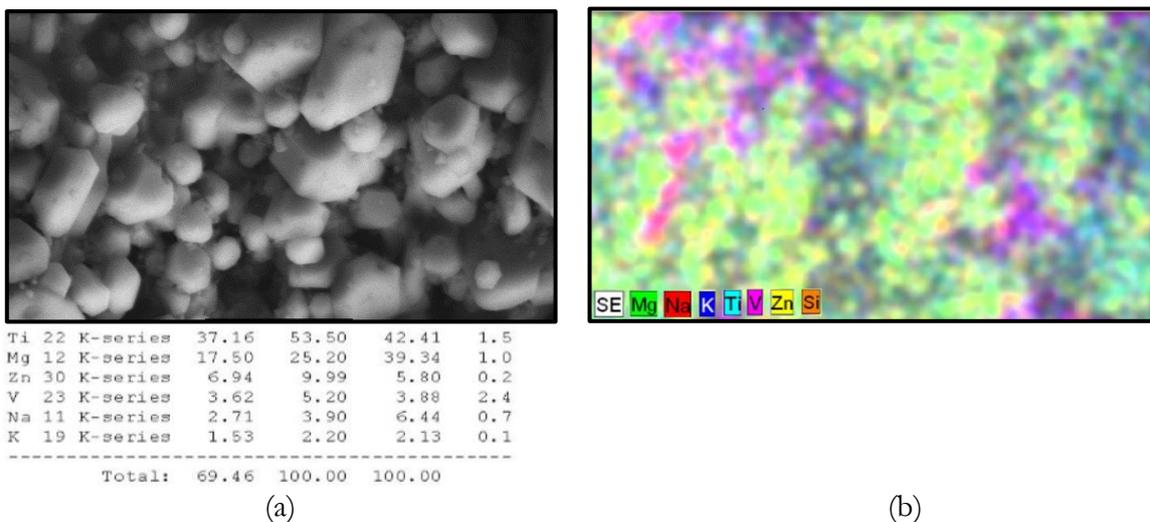


Figure 5. (a) SEM results and (b) EDX results at variations of 0.7 MZT-0.25 KNV-0.05 SiO_2 sintered at 950°C for 4 hours

Scanning Electron Microscopic (SEM) testing aims to determine the morphology of a sample. This tool utilizes a high-energy electron beam to scan an image on a model. The tested samples comprised 0.7 MZT-0.25 KNV-0.05 SiO_2 and 0.7 MZT-0.25 KNV, sintered at 950°C with a holding time of 4 hours. The results of the SEM test are shown in Figure 4. This test was carried out to compare the morphology of the samples with variations of SiO_2 addition and without addition. Based on Figures 4a and 4b, it can be seen that the morphological images of the two models are almost the same. The morphology of the first sample (figure 4. a) shows several grain types populations.

The largest population of grains is thought to show the MZT crystalline phase. In contrast, the small grain population is in the KNV phase. The existence of the MgTi_2O_5 phase is indicated by grains whose size is not much different from the grains of the main stage. KNV has a melting point of around 525°C, lower than Bi_2O_3 , which is 820°C. KNV grains grow around the MZT grain boundary. Thus, when the temperature is 950°C, the $x\text{MZT}-y\text{KNV}-(1-x-y)$, SiO_2 material has decreased porosity and is dense. If the two images (figure 4a and 4b) are compared, the number of grains is much more significant and evener with the addition of SiO_2 . SEM data analysis using the grain size cross-section method obtained an average grain size for non- SiO_2 samples of 2.5 μm . At the same time, the variation of the addition of SiO_2 produces an average grain size of 3 μm . The difference in grain size is due to the presence or absence of SiO_2 ; in this case, SiO_2 can accelerate grain growth, making the sizes larger and more numerous. From the EDX results (figure 5b), it was found that SiO_2 grew into grains around the MZT.

From this study, it can be stated that sintering temperature reduction is carried out to improve the properties and characteristics of the material. From this reduction in sintering temperature, it is the lowest energy that works in forming the phase. The sintering temperature reduction in this study was by adding KNV dopant material to the (Mg/Zn)TiO₃ material and adding SiO₂ to the host material, which aims to strengthen the fabric. This research has the same objective as the research conducted by Saukani (2015), namely, to lower the sintering temperature. Based on a study of dilatometric tests that had been carried out by Saukani (2015) and Rani (2016), the addition of 4 mol% Bi₂O₃ was able to reduce the MZT sintering temperature from 1400°C to 1100°C [15] [25]. However, adding KNV as a dopant material reduced the MZT sintering temperature from 1300°C to 950°C. So the addition of KNV is more effective in lowering the sintering temperature than the addition of Bi₂O₃. When viewed from the density value, the best results occurred in the 0.7MZT-0.25KNV-0.05SiO₂ variation with sintering at 950°C for 4 hours.

CONCLUSION

A single phase of Mg_{0.8}Zn_{0.2}TiO₃ (MZT) was obtained at a temperature of 850°C for 4 hours by synthesis using a conventional solid-state reaction. (K_{0.5}Na_{0.5})VO₃ was formed single phase at a temperature of 500°C for 2 hours. The MZT sintering temperature was reduced to 950°C for 4 hours by adding K_{0.5}Na_{0.5}VO₃ and SiO₂. A maximum density value of 2.76 gr / cc was achieved, and an average grain size based on SEM results was 3 μm at a composition of 0.7MZT-0.25KNV- 0.05SiO₂. XRD characterization using the Rietveld method obtained three phases: (Mg/Zn)TiO₃, (K/Na)VO₃, and MgTi₂O₅ phases. The results obtained are that the (K/Na)VO₃ –SiO₂ material can be used as a (Mg/Zn)TiO₃ sintering agent.

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