Faculty of Industrial and Manufacturing Technology and Engineering

EFFECT OF MANGANESE DOPANT ON THE STRUCTURAL, MORPHOLOGICAL AND PIEZOELECTRIC PROPERTIES OF POTASSIUM SODIUM NIOBATE THIN FILMS

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Master of Science in Manufacturing Engineering

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DECLARATION

I declare that this thesis entitled "Effect of Manganese Dopant on the Structural, Morphological and Piezoelectric Properties of Potassium Sodium Niobate Thin Films" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature :

Name : MUHD. AFIQ HAFIZUDDIN BIN AZMAN

Date : 25th September 2025

APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Manufacturing Engineering

Signature :..

Supervisor Name: Prof. Madya Ts. Dr. Umar Al-Amani bin Haji Azlan

Date : 25th September 2025

DEDICATION

I am deeply grateful to my supervisor, who has been an unwavering pillar of support and guidance throughout my journey. Your commitment to my growth and development has been truly inspiring. Your expert insights, constructive feedback, and constant encouragement have not only shaped my professional skills but also instilled in me a sense of confidence and resilience. I am fortunate to have a mentor like you, who always goes the extra mile to ensure that I reach my full potential.

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ABSTRACT

The urgent need to replace toxic lead-based piezoelectric, particularly $Pb(Zr,Ti)O_3$ (PZT), has intensified research into lead-free alternatives such as potassium sodium niobate (KNN), though practical applications remain limited by poor densification, volatilization of light cations, porosity, and reduced domain mobility compared to Pb-based systems. Manganese (Mn) doping has been proposed to address these challenges by refining grains, suppressing oxygen vacancies, stabilizing ferroelectric domains, and enhancing polarization, though its effects are strongly concentration dependent. The main aim of this research was to enhance the structural, morphological and piezoelectric properties of KNN thin films by incorporating Mn dopants of varying concentrations (0.1 mol, 0.3 mol, 0.5 mol, 0.7 mol, and 0.9 mol) through the sol–gel method. Specifically, the objectives were: (a) to fabricate and validate KNN thin films with different MnO concentrations using the sol–gel method, (b) to analyze and evaluate the structural, morphological, and electrical properties of Mn-doped KNN thin films, and (c) to determine and justify the optimal Mn doping concentration that maximizes piezoelectric performance. KNN thin films with Mn concentrations ranging from 0.1-0.9 mol were synthesized and systematically characterized to achieve these objectives. X-ray diffraction (XRD) confirmed that all films crystallized into a stable orthorhombic perovskite phase without secondary phases, while high-resolution transmission electron microscopy (HRTEM) and selected area electron diffraction (SAED) revealed clear lattice fringes and sharp diffraction rings, confirming high crystallinity. Field-emission scanning electron microscopy (FESEM) demonstrated that pure KNN films exhibited irregular and porous grains, whereas 0.3-0.5 mol Mn-doped films displayed dense, uniform grains with reduced porosity, consistent with atomic force microscopy (AFM) roughness measurements that showed stabilization at 0.5 mol. Electrical resistivity analysis revealed a non-linear correlation with Mn doping, reaching an optimal minimum of 0.05 MΩ at 0.3 mol before increasing at higher concentrations due to defect accumulation and porosity effects. Piezoelectric force microscopy (PFM) measurements demonstrated substantial improvements over pure KNN, with stable coefficients between 0.3-0.7 mol and a maximum at 0.9 mol, though the latter was accompanied by increased roughness, resistivity, and structural degradation. By integrating these findings, 0.3 mol Mn doping was identified as the optimal concentration, providing the best balance of densification, resistivity reduction, roughness stabilization, and consistent piezoelectric response, while 0.5 mol also exhibited promising results. Overall, this study demonstrates that Mn doping in the range of 0.3-0.5 mol optimally enhances the piezoelectric performance of KNN thin films, offering a viable lead-free alternative to toxic PZT systems and providing insights into the growth mechanism by which Mn influences grain structure, defect chemistry, and polarization dynamics for improved electromechanical properties.

KESAN DOPAN MANGAN TERHADAP STRUKTUR, MORFOLOGI DAN SIFAT PIEZOELEKTRIK FILEM NIPIS KALIUM NATRIUM NIOBAT

ABSTRAK

Keperluan mendesak untuk menggantikan bahan piezoelektrik berasaskan plumbum yang toksik, khususnya $Pb(Zr,Ti)O_3$ (PZT), telah meningkatkan minat penyelidikan terhadap bahan alternatif bebas plumbum seperti kalium natrium niobat (KNN). Walau bagaimanapun, aplikasi praktikal KNN masih terhad disebabkan oleh masalah kepadatan rendah, ketidakstabilan akibat pemejalwapan kation ringan, keliangan, serta mobiliti domain yang berkurangan berbanding sistem berasaskan plumbum. Pendopingan mangan (Mn) telah dicadangkan bagi menangani cabaran ini dengan memperhaluskan butiran, menekan kekosongan oksigen, menstabilkan domain ferroelectrik, dan meningkatkan polarisasi, walaupun kesannya sangat bergantung kepada kepekatan doping. Tujuan utama kajian ini adalah untuk meningkatkan sifat struktur, morfologi dan piezoelektrik filem nipis KNN dengan memasukkan dopan Mn pada pelbagai kepekatan (0.1 mol, 0.3 mol, 0.5 mol, 0.7 mol, dan 0.9 mol) melalui kaedah sol–gel. Secara khusus, objektif kajian adalah: (a) menghasilkan dan mengesahkan filem nipis KNN dengan kepekatan MnO yang berbeza menggunakan kaedah sol–gel, (b) menganalisis dan menilai sifat struktur, morfologi, dan elektrik filem nipis KNN terdop Mn, serta (c) menentukan dan membuktikan kepekatan doping Mn yang optimum bagi memaksimumkan prestasi piezoelektrik. Filem nipis KNN dengan kepekatan Mn antara 0.1–0.9 mol telah disintesis dan dicirikan secara sistematik untuk mencapai objektif ini. Analisis pembelauan sinar-X (XRD) mengesahkan bahawa semua filem berjaya dikristalkan ke dalam fasa perovskit ortorombik yang stabil tanpa kehadiran fasa sekunder, manakala mikroskopi elektron transmisi resolusi tinggi (HRTEM) dan pembelauan elektron kawasan terpilih (SAED) menunjukkan jalur kisi yang jelas serta cincin pembelauan yang tajam, menandakan tahap penghabluran yang tinggi. Mikroskopi elektron imbasan pancaran medan (FESEM) menunjukkan bahawa filem KNN tulen mempunyai butiran tidak sekata serta berliang, manakala filem terdop Mn 0.3–0.5 mol mempamerkan butiran yang padat, seragam dan kurang berliang, sejajar dengan ukuran kekasaran melalui mikroskopi daya atom (AFM) yang menunjukkan kestabilan pada 0.5 mol. Analisis rintangan elektrik menunjukkan korelasi bukan linear dengan doping Mn, mencapai nilai minimum optimum 0.05 MΩ pada 0.3 mol sebelum meningkat semula pada kepekatan yang lebih tinggi akibat pengumpulan kecacatan dan kesan keliangan. Pengukuran piezoelektrik melalui mikroskopi daya piezo (PFM) menunjukkan peningkatan yang ketara berbanding KNN tulen, dengan pekali yang stabil antara 0.3–0.7 mol dan nilai maksimum pada 0.9 mol, walaupun pada tahap ini turut disertai peningkatan kekasaran, rintangan, dan kemerosotan struktur. Secara keseluruhannya, doping Mn pada 0.3 mol dikenal pasti sebagai kepekatan optimum kerana ia memberikan keseimbangan terbaik antara pemadatan butiran, pengurangan rintangan, kestabilan kekasaran, dan tindak balas piezoelektrik yang konsisten, manakala 0.5 mol juga menunjukkan prestasi yang menjanjikan. Kajian ini membuktikan bahawa doping Mn dalam julat 0.3–0.5 mol dapat meningkatkan prestasi piezoelektrik filem nipis KNN secara optimum, menawarkan alternatif bebas plumbum yang berdaya saing kepada sistem PZT yang toksik, serta memberikan kefahaman baharu mengenai mekanisme pertumbuhan di mana Mn mempengaruhi struktur butiran, kimia kecacatan, dan dinamik polarisasi untuk meningkatkan sifat elektromekanikal.

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LIST OF ABBREVIATIONS

MnO	-	Manganese oxide
KNN	-	Potassium sodium niobate
PZT	-	Lead zirconate titanate
XRD	-	X-ray diffraction
FESEM	-	Field Emission Scanning Electron Microscopy
LCR	-	Inductance, Capacitance, Resistance
AFM	-	Atomic Force Microscopy
PFM	-	Piezo response Force Microscopy
HRTEM	-	High-Resolution Transmission Electron Microscopy
CH_3COOK	-	Potassium acetate
CH_3COONa	-	Sodium acetate
$Nb(OC_2H_5)_5$	-	Niobium Ethoxide
$(KNa)NbO_3$	-	Potassium Sodium Niobate
T_C	-	Curie Temperature
CSD	-	Chemical Solution Deposition
PLD	-	Pulsed Laser Deposition
Bi	-	Bismuth
Sr	-	Strontium
Zn	-	Zinc
Pt	-	Platinum
AC	-	Alternating Current
2-MOE	-	2-Methoxyethanol
RTP	-	Rapid Thermal Processing
$C_3H_8O_2$	-	Propylene glycol

$C_5H_8O_2$	-	Acetylacetone
EDX	-	Energy-Dispersive X-ray
SE	-	Secondary electron
BSE	-	Back-scattered electron
EHT	-	Electron high tension
Rh	-	Rhodium
SAED	-	Selected Area Electron Diffraction
TEM	-	Transmission Electron Microscopy
Au	-	Gold
Nb	-	Niobium
MEMS	-	Microelectromechanical systems
R_q	-	Root mean square roughness
R_a	-	Average roughness

LIST OF SYMBOLS

$wt.\%$	-	weight percentage
mol	-	SI base unit for amount of substance
d_{33}	-	piezoelectric coefficient
d_{31}	-	piezoelectric coefficient
kp	-	electromechanical coupling factor
pC/N	-	Pico Coulombs per Newton
g/mol	-	gram per mol
rpm	-	revolutions per minute
µl	-	microlitre
λ	-	lambda
Å	-	Angstrom
kV	-	kilovolt
µA	-	microampere
µm	-	micrometre
Pa	-	Pascal
mA	-	milliampere
kHz	-	kilohertz
mol%	-	mol percentage
MΩ	-	Mega Ohm
h*	-	hole vacancy
mV	-	millivolt

LIST OF APPENDICES

LIST OF PUBLICATIONS

The followings are the list of publications related to the work on this thesis:

Azman, M.A.H., Abd Rashid, M.W., Azlan, U.A.A., Shamsuri, S.R., Dom, A.H.M., Kassim, S.R., Harttar, M.A.M. and Hatta, M., 2024. Effect of Manganese Dopant on The Structure and Electrical Properties of Potassium Sodium Niobate Thin Film. *Malaysian Journal of Microscopy*, *20*(1), pp.317-327.

Afiq, H.A.M., Warikh, A.R.M., Azlan, U.A.A., Akmal, M.M. and Moriga, T., 2024. Synthesis and Characterization of Mn-doped Sodium Potassium Niobate (KNN) Thin Film. *Journal of Advanced Manufacturing Technology (JAMT)*, *18*(2).

Hafizuddin Azman, M. A., Abd Rashid, M. W., Haji Azlan, U. A.A., Mohamad, N., Abd Rahim, T., Mat Harttar, M. A., & Moriga, T., (2025). Characterization on Structural and Electrical Properties of Manganese-doped Potassium Sodium Niobate (KNN) Thin Film by Sol-gel Method. *Journal of Advanced Research in Micro and Nano Engineering*, *29*(1), pp.36–45.

Azman, M.A.H., Piah, H.M.A., Abd Rashid, M.W., Azlan, U.A.A.H., Harttar, M.A.M., Hatta, M. and Moriga, T., 2025. The comparison on structural, morphological and electrical analysis on Mn and Zn-doped KNN thin film by sol-gel method. *Malaysian Journal of Microscopy*, *21*(1), pp.114-123.

CHAPTER 1

INTRODUCTION

1.1 Background

Piezoelectricity is a remarkable phenomenon in which certain materials generate an electric charge when subjected to mechanical stress, such as compression, tension, or shear forces. This occurs because these materials have a crystal lattice structure that is non-centrosymmetric, meaning the arrangement of atoms within the crystal lacks a centre of symmetry. When mechanical stress is applied, the positions of the atoms within the material shift, causing an imbalance in the distribution of electric charges, which results in the generation of an electrical potential or voltage across the material's surface. The effect is reversible, applying an electric field to these materials can cause material to deform mechanically, a property that is widely exploited in various devices (Sekhar et al., 2021).

The most common piezoelectric materials include both natural and synthetic substances. Natural piezoelectric materials, like quartz, have been used for centuries due to their stable properties and predictable behaviour, making ideal piezoelectric materials for applications in watches, oscillators, and frequency control devices. Synthetic piezoelectric materials, such as piezoelectric ceramics like lead zirconate titanate (PZT), have been engineered to enhance the piezoelectric effect, allowing for more efficient and durable performance (Tiwari et al., 2021). These materials were used in a wide range of applications, from industrial sensors, actuators, and transducers to medical devices like ultrasound machines, where these materials generate and detect high-frequency sound waves for imaging purposes.

Piezoelectric materials are also critical in consumer electronics, such as in microphones and speakers, where these materials convert sound waves into electrical signals and vice versa. In addition to sensing and signal conversion, piezoelectric materials are employed in precision instruments, including accelerometers, vibration sensors, and inkjet printers, where precise mechanical movements are created by the application of an electric field. Piezoelectric components in these devices respond quickly and accurately, enabling controlled displacement and high-resolution performance essential for modern precision technologies. The ability of piezoelectric materials to convert mechanical energy into electrical energy also makes piezoelectric materials useful in energy harvesting applications, where ambient vibrations or pressure changes are captured and converted into electrical power to operate small devices, such as wireless sensors or medical implants. Piezoelectricity is also utilized in actuators for applications requiring fine motion control, such as in optical devices, robotics, and nanotechnology. However, despite the wide-ranging utility of piezoelectric materials, there are certain challenges, particularly with materials like PZT, which contains lead, a toxic substance (Akshayveer et al., 2025). This has led to research focused on developing lead-free piezoelectric materials that maintain high performance while being environmentally friendly.

Lead zirconate titanate (PZT) is one of the most widely used and highly efficient piezoelectric materials, making it a promising candidate in various advanced applications due to its exceptional electromechanical properties. PZT is a ferroelectric ceramic composed of lead (Pb), zirconium (Zr), and titanium (Ti) in a perovskite crystal structure, which allows it to exhibit strong piezoelectric behaviour, high dielectric permittivity, and excellent mechanical stability. The ability of PZT to convert mechanical energy into electrical energy and vice versa with high efficiency makes it invaluable in diverse applications, including sensors, actuators, transducers, and energy harvesting devices (Mahapatra et al., 2021). One of the key advantages of PZT is commonly used in ultrasonic transducers for medical imaging and industrial non-destructive testing, where its ability to generate and detect high-frequency sound waves enables detailed imaging and flaw detection in materials. In microelectromechanical systems (MEMS), PZT is employed for precision motion control in applications such as inkjet printers, scanning probe microscopes, and optical devices, where nanometre-scale accuracy is required.

Furthermore, its strong electromechanical coupling makes it ideal for piezoelectric actuators in adaptive optics, vibration damping systems, and fuel injectors in automotive engineering. PZT also plays a significant role in energy harvesting technologies, where ambient mechanical vibrations from machinery, human movement, or structural oscillations are captured and converted into electrical power. This harvested energy is used to power self-sustaining electronic devices, including wireless sensors and implantable medical devices (Zaszczyńska et al., 2020). However, despite its excellent performance, PZT faces challenges due to the presence of lead, which poses environmental and health concerns. Regulations such as the Restriction of Hazardous Substances (RoHS) directive and increasing global efforts to reduce toxic materials in electronic devices have prompted extensive research into lead-free alternatives, such as potassium sodium niobate (KNN), barium titanate ($BaTiO_3$), and bismuth ferrite ($BiFeO_3$) (Lv et al., 2020). Nonetheless, lead-free alternatives have yet to fully match the superior piezoelectric properties of PZT, making it difficult to replace in high-performance applications. Efforts to mitigate the environmental impact of PZT include developing lead-reduced formulations, recycling strategies, and integrating it with advanced nanostructures to enhance its efficiency while minimizing toxicity.

KNN has emerged as one of the most promising lead-free piezoelectric materials, offering a potential replacement for PZT due to its environmentally friendly composition and excellent piezoelectric properties. KNN is a solid solution of potassium niobate ($KNbO_3$) and sodium niobate ($NaNbO_3$), both of which exhibit perovskite structures similar to PZT, enabling it to maintain strong piezoelectric performance while eliminating toxic lead content. One of the key advantages of KNN is its relatively high Curie temperature (above 400 °C), which allows it to operate efficiently at elevated temperatures, making it suitable for applications in sensors, actuators, and high-temperature electronics (Tokay and Yazıcı, 2022). Additionally, KNN exhibits good electromechanical coupling and flexibility in its composition, allowing researchers to optimize its properties by doping it with elements such as lithium (Li), tantalum (Ta), and bismuth (Bi) to enhance its piezoelectric coefficient, dielectric properties, and mechanical stability (Li et al., 2024). These modifications have significantly improved KNN's competitiveness against PZT in applications such as ultrasonic transducers, medical imaging devices, energy harvesters, and precision actuators in MEMS.

In energy harvesting, KNN-based materials have demonstrated strong potential for converting mechanical vibrations into electrical energy, which can power wireless sensors and self-sustaining electronic systems. However, despite these promising attributes, KNN still faces challenges that must be addressed before it can fully replace PZT in commercial applications. One of the main limitations is its relatively lower piezoelectric coefficient compared to PZT, which affects its sensitivity and efficiency in certain high-performance applications. Furthermore, KNN is more challenging to process due to its volatility during high-temperature sintering, which can lead to difficulties in achieving dense and stable ceramic structures (Lu et al., 2024). Researchers are actively working on improving the sintering techniques, developing novel composite structures, and optimizing grain orientation to enhance KNN’s piezoelectric response and mechanical durability. Despite these challenges, ongoing advancements in material engineering and nano-structuring are steadily improving KNN's performance, making it a strong candidate for replacing PZT in applications where environmental safety and regulatory compliance are critical. As the demand for sustainable and lead-free piezoelectric materials continues to grow, KNN is expected to play a key role in the future of piezoelectric technologies, bridging the gap between high performance and environmental responsibility.

1.2 Problem Statement

PZT certainly is the most piezoelectric material used in several electronic applications like energy harvesting, owing to its exceptional ferroelectric, dielectric and piezoelectric properties (Zhang et al., 2017a). PZT also developed as the leading piezoelectric material with an estimated yearly worldwide trade of ten billion dollars. The superior performance of Pb-based piezoelectric materials, such as $Pb(Zr,Ti)O_3$ (PZT), originates from the stereochemically active $6s^2$ lone pair of Pb^{2+}. This lone pair induces asymmetric electron density, promoting strong off-centre lattice distortions that enhance ferroelectric polarization and facilitate polarization rotation under an applied electric field (Pyykkö, 2012; Rödel et al., 2009). Furthermore, Pb-based systems exhibit morphotropic phase boundaries (MPBs), where rhombohedral and tetragonal phases coexist, enabling large piezoelectric responses due to the ease of domain reorientation (Park & Shrout, 1997). These structural and electronic features give Pb-based ceramics outstanding piezoelectric coefficients and electromechanical coupling factors, surpassing most lead-free alternatives such as $BaTiO_3$ and KNN.

Despite increasing interest in lead-free piezoelectrics, their substitution for Pb-based systems often reveals critical disadvantages. KNN and $BaTiO_3$, for example, generally display lower piezoelectric constants and weaker electromechanical coupling due to the absence of Pb's lone pair effect (Wang et al., 2022; Xu et al., 2025). Additionally, PbO volatilization during sintering assists densification in Pb-based ceramics, a benefit not shared by lead-free systems, which frequently suffer from poor densification, porosity, and secondary phase formation. The lack of a wide and tunable MPB further limits the structural adaptability of lead-free systems, reducing their capacity for polarization rotation. Beyond piezoelectrics, Pb alternatives in batteries, shielding, or corrosion protection require thicker, heavier, or less durable designs to achieve equivalent performance (Shatish et al., 2021; Xu et al., 2006). These limitations underscore why Pb continues to dominate in performance-critical applications, despite environmental pressures to replace it.

The main challenge in this field lies in developing lead-free piezoelectric materials that can match the superior functional properties of Pb-based compounds while avoiding the environmental and health hazards associated with Pb. Lead is a toxic heavy metal that accumulates in biological systems, raising serious concerns about its continued use in consumer electronics, medical devices, and energy systems (Rödel et al., 2009). International regulations, such as the EU's Restriction of Hazardous Substances (RoHS), are accelerating the push to eliminate Pb, making it essential to identify eco-friendly alternatives. Despite its exceptional electrical characteristics, PZT has a key disadvantage. It has more than 60 wt% of lead. As per the Comprehensive Environmental Response, Compensation and Liability Act, 1980, lead is included in the priority list of harmful substances which potentially leading to damage the nervous system and spermatozoid production due to its carcinogenicity (Ibn-Mohammed et al., 2016).

However, the intrinsic structural and electronic advantages of Pb^{2+} remain difficult to replicate in other elements, meaning that lead-free systems often compromise between performance and sustainability. This creates a pressing research demand for defect engineering, dopant modification, and novel compositional design strategies to close the performance gap while ensuring environmental compliance.

The study and growth of KNN have been revisited in the past few years because of its attractive piezoelectric properties and numerous application possibilities. Though in theoretical KNN exhibits good piezoelectric performance comparable to that lead-based material, the realization of KNN to compete commercial PZT is still a challenge and remains intriguing issues due to volatilization of light cations. While others gave intense focus on reducing volatility effect to enhance piezoelectric properties, very little research work has focused on improving the piezoelectric properties by embedding new material into KNN mainly manganese that has unique ferroelectric and piezoelectric properties (Zhang et al., 2023).

Improper selection of manganese doping can lead to high leakage current, which disrupts the polarization switching and poling process by preventing effective dipole alignment. Additionally, poor insulating properties result in excessive electrical conductivity, leading to energy loss and reduced reliability. These factors significantly degrade the performance of piezoelectric materials, making such materials unsuitable for practical applications in sensors, actuators, and transducers. Hence, the appropriate concentration of manganese in KNN matrix should be identified for proper dispersion in KNN matrix. KNN base compounds are expected to have excellent electric properties, particularly piezoelectric properties by embedding manganese in KNN thin films (Cui et al., 2020).

The concentration of manganese was a decisive factor because its effects are strongly dependent on doping level. At low concentrations, Mn can act as an acceptor dopant, suppress oxygen vacancies, improve resistivity, and stabilize ferroelectric domains. However, excessive Mn incorporation often promotes secondary phase formation (e.g.,