



**FABRICATION AND CHARACTERISATION OF
POLYTETRAFLUOROETHYLENE-BOROSILICATE AND
POLYTETRAFLUOROETHYLENE-SODA LIME SILICA GLASS
COMPOSITES FOR MICROWAVE SUBSTRATE APPLICATION**

By

ALHAJI IBRAHIM ABUBAKAR

**Thesis Submitted to the School of Graduate Studies, Universiti Putra
Malaysia, in Fulfillment of the Requirement for the Degree of Philosophy**

June 2022

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DEDICATION

To my brothers in this journey, do not despair. Know that the promise of the Gatherer is certain. Be patient. Khairan zahiran, amin summa amin.



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the Degree of Doctor of Philosophy

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June 2022

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Faculty : Science

Rapid and unprecedented developments in the information technology and telecommunication industry demands high-speed, light and low-cost microwave substrates for high-frequency applications. This work presents the preparation of low-cost composites using borosilicate (BRS) and soda lime silicate (SLS) glasses. The glasses were used as fillers in the polytetrafluoroethylene (PTFE) matrix as a replacement for woven fiberglass and ceramics to reduce the cost and improve the performance of PTFE-based composites for microwave substrate application. The BRS and SLS glass powders were recycled using glass waste through ball milling. The dependence of the complex permittivity on BRS and SLS grain sizes was determined. A 63 μm grain size fillers were selected to fill the PTFE matrix through a dry powder processing technique. Surface morphology and material composition were analysed using X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray (EDX). Tensile strength, coefficient of thermal expansion (CTE), density, and moisture absorption of the composites were studied with respect to filler size and content.

The dielectric properties were characterised from 8 GHz to 12 GHz for a rectangular waveguide (RWG) and from 1 GHz to 12 GHz for an open-ended coaxial probe (OCP). In addition, the characterisation was conducted at room temperature using a vector network analyser. Similarly, the RWG was used to measure the magnitude of reflection ($|S_{11}|$) and transmission ($|S_{21}|$) coefficients. The results of the OCP and RWG methods indicated that the dielectric properties of all composites increased with filler content, however, the RWG method exhibited higher values than the OCP method due to the presence of air gaps in the latter method. At 10 GHz frequency, the dielectric constant (ϵ'), loss factor (ϵ'') and loss tangent ($\tan\delta$) of PTFE/BRS and PTFE/SLS composites

varied from 2.11, 0.0022, 0.0011 and 2.11, 0.0022, 0.0011 to 2.24, 0.0029, 0.0013 and 2.57, 0.0031, 0.0012, respectively, for the OCP method. For the RWG method, the ϵ' , ϵ'' , and $\tan\delta$ had a respective change from 2.16, 0.0035, 0.0015, and 2.18, 0.0035, 0.0016 to 2.31, 0.0042, 0.0018 and 2.57, 0.0047, 0.0018 when filler content was varied from 5 wt.% to 25 wt.%. In addition, the results of $|S_{11}|$ and $|S_{21}|$ were used to calculate the power loss (dB) for the different volume fractions of PTFE/BRS and PTFE/SLS composites. The power loss at 10 GHz frequency was found to be from 3.33 dB and 3.48 dB to 3.89 dB and 3.85 dB in that order. Furthermore, the electric field distribution through the dielectric composites in the RWG was visualised for various filler content and then used to calculate the attenuation of the electric field intensity. The attenuation was found to increase from 3.67 dB and 3.70 dB to 3.81 dB and 3.77 dB for the PTFE/BRS and PTFE/SLS composites, respectively, when the filler content was adjusted from 5 wt.% to 25 wt.%.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

**FABRIKASI DAN PENCIRIAN KOMPOSIT KACA
POLITETRAFLUOROETILENA-BOROSILIKAT DAN
POLITETRAFLUOROETILENA- SODA KAPUR SILIKA BAGI APLIKASI
SUBTRAT GELOMBANG MIKRO**

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Perkembangan pesat dalam teknologi maklumat dan industri telekomunikasi memerlukan substrat gelombang mikro berkelajuan tinggi, ringan dan murah untuk aplikasi frekuensi tinggi. Dalam kajian ini, penyediaan komposit kos rendah menggunakan kaca borosilikat (BRS) dan soda kapur silika (SLS). Kaca digunakan sebagai pengisi dalam matriks politetrafluoroetilena (PTFE) sebagai gantian kepada kaca gentian teranyam dan seramik untuk mengurangkan kos dan meningkatkan prestasi komposit berasaskan PTFE bagi aplikasi substrat gelombang mikro.. Serbuk kaca BRS dan SLS dikitar semula menggunakan sisa kaca melalui pengisaran bebola. Kebergantungan ketelusan kompleks pada saiz butiran BRS dan SLS telah ditentukan. Pengisi bersaiz butiran 63 μm dipilih untuk mengisi matriks PTFE melalui teknik pemprosesan serbuk kering. Morfologi permukaan dan komposisi bahan dianalisis menggunakan belauan sinar-X (XRD), mikroskopi elektron pengimbasan (SEM), dan penyebaran tenaga sinar-X (EDX). Kekuatan tegangan, pekali pengembangan haba (CTE), ketumpatan, dan penyerapan kelembapan bagi komposit telah dikaji dengan ukuran dan kandungan pengisi.

Sifat dielektrik dicirikan dari 8 GHz hingga 12 GHz untuk pandu gelombang segi empat tepat (RWG) dan dari 1 GHz hingga 12 GHz untuk kuar sepaksi terbuka (OCP). Di samping itu, pencirian dilakukan menggunakan penganalisis rangkaian vektor pada suhu bilik. RWG juga digunakan untuk mengukur pekali magnitud pantulan ($|S_{11}|$) dan pekali pemancaran ($|S_{21}|$). Hasil kaedah OCP dan RWG menunjukkan bahawa sifat dielektrik bagi semua komposit meningkat dengan kandungan pengisi, bagaimanapun, kaedah RWG menunjukkan nilai yang lebih tinggi daripada kaedah OCP kerana adanya ruang udara dalam kaedah OCP. Bagi kaedah OCP, pada frekuensi 10 GHz, ketelusan relatif (ϵ'), faktor kehilangan (ϵ'') dan tangen hilangan ($\tan\delta$) bagi komposit PTFE/BRS dan

PTFE/SLS bebezbeza dari 2.11, 0.0022, 0.0011 dan 2.11, 0.0022, 0.0011 hingga 2.24, 0.0029, 0.0013 dan 2.57, 0.0031, 0.0012, masing-masing. Untuk kaedah RWG, ϵ' , ϵ'' , dan $\tan\delta$ mempunyai perubahan masing-masing dari 2.16, 0.0035, 0.0015, dan 2.18, 0.0035, 0.0016 hingga 2.31, 0.0042, 0.0018 dan 2.57, 0.0047, 0.0018 apabila kandungan pengisi bervariasi dari 5 wt. % hingga 25 wt.%. Di samping itu, keputusan $|S_{11}|$ dan $|S_{21}|$ telah digunakan untuk mengira kehilangan kuasa (dB) untuk pecahan isipadu yang berbeza untuk komposit PTFE/BRS dan PTFE/SLS. Kehilangan kuasa pada frekuensi 10 GHz didapati dari 3.33 dB dan 3.48 dB hingga 3.89 dB dan 3.85 dB dalam urutan tersebut. Selanjutnya, sebaran medan elektrik melalui komposit dielektrik dalam RWG digambarkan untuk pelbagai kandungan pengisi dan kemudian digunakan untuk mengira pelemahan keamatan medan elektrik. Pelemahan didapati meningkat dari 3.67 dB dan 3.70 dB menjadi 3.81 dB dan 3.77 dB untuk komposit PTFE/BRS dan PTFE/SLS, masing-masing, apabila kandungan pengisi dilaraskan dari 5 wt. % hingga 25 wt.%.

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LIST OF ABBREVIATIONS

PTFE	Polytetrafluoroethylene (Teflon)
BRS	Borosilicate
SLS	Soda lime Silica
OCP	Open-ended coaxial probe
RWG	Rectangular waveguide
EF	Electric field
XRD	X-ray diffraction
SEM	Scanning Electron Microscope
EDX	Energy dispersive x-ray
ICSD	Inorganic crystal structure database
CTE	Coefficient of thermal expansion
FEM	Finite element method
FDTD	Finite difference time domain
VNA	Vector network analyser
TRL	Thru, Reflect, Line
RF	Radio frequency
ECAL	Electronic calibration
TR	Transmission/Reflection
S_{11}	Reflection coefficient
S_{21}	Transmission coefficient
ϵ^*	Complex permittivity
ϵ'	Dielectric constant/relative permittivity
ϵ''	Loss factor

CHAPTER 1

INTRODUCTION

The last decade has witnessed a rapid and unprecedented development in information technology and telecommunication industries driven by military and consumer markets (Manu et al., 2013; Hao Wang et al., 2020; C. Xie et al., 2017). This sudden change creates a high demand for high-speed, light and low-cost microwave substrates. A microwave base substrate that meets specific criteria, supports the microwave circuits (Ren et al., 2018; Tan et al., 2022; Varghese et al., 2015). Microwave substrates are dielectric materials with moderate permittivity and low loss tangent at microwave frequencies (Li et al., 2021). The substrate candidate materials should possess a low dielectric constant to reduce signal propagation delay, a small magnitude of thermal expansion coefficient (CTE) to avoid a mismatch between the conductor and the substrate material, a high thermal conductivity (TC) to transport the heat generated away from the circuits at high frequencies (> 1 GHz) and good mechanical strength (Murali et al., 2009; Tan et al., 2021). Polymers are widely used for substrate applications due to their low dielectric constant and chemical inertness, but they have a high thermal expansion coefficient and low thermal conductivity (Han et al., 2020; Thomas et al., 2016). However, polytetrafluoroethylene (PTFE) is the most widely used among polymers due to its low dielectric loss and low moisture absorption, but it has a high melting point, making it difficult to process. PTFE is crystalline and has a broad range of applications as an excellent insulator from high electrical power to defence industries (Venkateswarlu et al., 2015; Zhou et al., 2020). Generally, the properties of polymers depend on their microstructure and composition as such suitable fillers of varying sizes and content should be used to fill and strengthen the polymers (Thomas et al., 2016). Several studies have been conducted to improve PTFE's thermal, mechanical and dielectric properties (Han et al., 2020; Shamsuddin et al., 2020; Varghese et al., 2015; C. Xie et al., 2017; Jie Yang et al., 2020). To enhance the thermal and mechanical properties of PTFE without affecting its electrical properties, a filler with a low dielectric constant and CTE and high tensile strength is required. Most practical demands to control the properties of the PTFE can be addressed by incorporating glass fillers such as borosilicate (BRS) and soda lime silica (SLS) glasses as they have high thermal conductivity and tensile strength, low CTE, and moderate dielectric constant (Ashby, 2012; Bruns et al., 2020a; Karasu et al., 2020; Khzaalah et al., 2022).

1.1 Problem Statement

Commercial fibreglass and ceramic are the most commonly used fillers to produce PTFE-based microwave substrates due to their good electrical, thermal and mechanical properties (Luo et al., 2018; H. Wang et al., 2020). However, the increased demand for high-speed and low-cost microwave substrates demands alternative lower-cost filler materials to sustain the industry's advancement and reduce the cost of microwave substrates for consumers. Therefore, this research

proposes a new technique to reduce the cost of polymer-based composites by using recycled BRS and SLS glass waste in combination with a PTFE matrix. The BRS and SLS glasses have comparable dielectric and mechanical properties to the commercial fillers. However, the glasses possess better rigidity and lower CTE. Furthermore, the technique involves extracting glass powders from the BRS and SLS waste and following optimisation of the dielectric properties by reducing the particle size to micrometre sizes through ball milling. The relationship between grain size and the dielectric properties of the BRS and SLS powders was then determined. The BRS and SLS powders with optimum dielectric properties could reduce the cost of inorganic fillers for microwave substrates and further eliminate environmental pollution without compromising performance.

PTFE is a carbon-based, fluorinated synthetic polymer (Kecerdasan & Ikep, 2017). It has outstanding dielectric and moisture absorption properties that make it the most preferable among polymers for microwave substrate application (Zhang et al., 2022). However, its high CTE, lack of rigidity, and high processing temperature pose a challenge to its handling for practical application (Q. Li et al., 2021). The inclusion of BRS and SLS fillers into the PTFE matrix through a simple dry powder processing method could easily remedy the processing obstacles and produce composites with well-adjusted dielectric, mechanical, and thermal properties suitable for microwave substrate application. Also, the BRS and SLS particle sizes could affect the dielectric, mechanical, and thermal properties of the PTFE/BRS and PTFE/SLS composites, which have to be examined. Moreover, composites must have low dielectric properties for high-speed data transmission. Therefore, an optimum filler size of 63 μm for the BRS and SLS was fed into the PTFE/BRS and PTFE/SLS to enhance the mechanical and thermal properties without adversely affecting the dielectric properties of the PTFE matrix.

The conventional measurement technique used to characterise the dielectric properties of the PTFE/BRS and PTFE/SLS composites was the open-ended coaxial probe (OCP). The technique has the advantage of being simple to implement and easy to prepare a sample for measurement (Aydinalp et al., 2022). However, the technique suffers from the wrong assumption that samples are homogeneous and isotropic within the volume of the measurement probe (Brace & Etoz, 2020). Moreover, the technique is one-port, thus, it could not characterise the scattering parameters (S_{11} and S_{21}) of the composites. The S_{11} and S_{21} are fundamental to determining the power loss in the composites due to absorption. In this research, a rectangular waveguide (RWG) measurement technique was used, in addition to the OCP technique, to measure the dielectric properties as well as the S_{11} and S_{21} of the PTFE/BRS and PTFE/SLS composites. The dielectric results from the RWG technique were used as inputs to the COMSOL software using the finite element method (FEM) to calculate the S_{11} and S_{21} and visualised the electric field distribution in the composites.

1.2 Research Objectives

This research prepares, fabricates and characterises PTFE/BRS and PTFE/SLS on their structural, morphology and dielectric properties. The specific objectives of the study are:

- I. To fabricate PTFE/BRS and PTFE/SLS composites with different filler sizes to investigate the effect of the filler size on the mechanical, thermal, and dielectric properties of the composites.
- II. To fabricate PTFE/BRS and PTFE/SLS composites with different filler volume fractions to examine the effect of filler content on the structural, mechanical, thermal, and dielectric properties of the composites
- III. To examine the effect of recycled BRS and SLS fillers on the S_{11} , S_{21} and power loss due to absorption for the PTFE/BRS and PTFE/ SLS composites. The experimentally measured $|S_{11}|$ and $|S_{21}|$ would be compared with those calculated from the FEM implemented in COMSOL software.
- IV. To visualise the electric field distribution across dielectric samples and calculate the attenuation of the field intensity due to filler content.

1.3 Scope of the Study

This research prepares recycled BRS and SLS powders from glass waste. The powders will be reduced into five micro sizes using a high ball milling technique for twenty-four hours. The complex permittivity of the recycled glass powders will be measured in the microwave frequency range of 1–12 GHz. The first set of composites will be fabricated using recycled glass powders of different sizes at a 5 wt.% fixed concentration. The fabrication will be conducted via the powder dry processing technique to determine the optimum filler size. This filler size then will be used to fabricate the final composites. The percentage weight of the glass powders to fill the matrix will be varied from 5 wt.%-25 wt.% with a 5 wt.% increment. The structural, morphological, and compositional characterisation will be conducted using techniques of XRD, SEM, and EDX. Tensile strength, CTE, moisture absorption, and the composites' density will also be determined.

The influence of the recycled fillers on the complex permittivity of PTFE/BRS and PTFE/SLS composites will be studied using rectangular waveguide and open-ended coaxial probe techniques at 8.2 GHz- 12.4 GHz and 1 GHz -12 GHz, respectively. Similarly, the effect of the fillers on the scattering parameters will be determined using the rectangular waveguide technique. The study also calculates the scattering parameters using the finite element method (FEM) implemented in COMSOL software. Measured and computed S-parameters will be compared, and error analysis between them would be determined. The COMSOL software will also be utilised to visualise the interaction of electromagnetic waves with the dielectric samples. Attenuation of the

electromagnetic intensity of the composites will be calculated via the finite element method (FEM) using COMSOL software.

1.5 Thesis Layout

The thesis consists of six chapters. Chapter 1 presents the general overview of substrate materials and the interaction of microwaves with materials. The chapter highlights the problem statement and research objectives, followed by the scope of the study and the thesis layout.

Chapter 2 looks at the literature review of some principles and presents a dry powder processing technique. The properties of PTFE, BRS, and SLS glasses were presented, along with a review of the composite preparation methods. The chapter also reviews dielectric measurement techniques and FEM formulation for calculating S-parameters.

Chapter 3 discusses the theory behind mechanical alloying and dry powder processing techniques, and the chapter presents the dielectric properties and the nature of polarisation taking place. It also discusses the basic electromagnetic wave equation and its derivation. Finally, the theory for FEM analysis is presented.

Chapter 4 consists of the methodology used in this study. The preparations of BRS and SLS glass powders are discussed. PTFE/BRS and PTFE/SLS composites fabrication process is given in detail. The use of OCP, RWG, and FEM concerning dielectric characterisation is thoroughly discussed. The methods for characterising tensile and CTE are also provided. The structural, morphological, and compositional techniques are also presented.

Chapter 5 gives the results of the entire characterisation carried out. Sections 5.1 and 5.2 discuss the results for XRD, SEM, and EDX. This is followed by a discussion on the results from moisture absorption, density, tensile strength and CTE in sections 5.3, 5.4, 5.5, and 5.6. The complex permittivity results from the open-ended coaxial probe and rectangular waveguide techniques are discussed in sections 5.7 and 5.8. Section 5.9 discusses the S-parameters results calculated from RWG and FEM. Section 5.10 discusses the power loss due to material absorption, while section 5.11 discusses electrical field distribution and its attenuation.

Chapter 6 summarises the study and recommends future work.

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