Design, Fabrication and Validation of High-permittivity Low-loss Microwave Material for Biomedical Sensor

Jasmin Gasi
Abstract

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The purpose of this task is to synthesize a dielectric substrate material through a sintering process, which can be used for noninvasive physiological sensor development. Low loss, high dielectric constant ceramic material is used. Sintering process is employed to ensure stable structure and homogeneous dielectric properties of the substrate. Samples were prepared with TiO2 and in combination with CuO and Al2O3. All samples were measured and validated in 500 MHz to 20 GHz frequency range. Characterization measurements were performed with a Vector Network Analyzer, FieldFox N9918A, and connected to Keysight, open ended coaxial probe and performance probe. Reflection based measurement method was used due to its simplicity, speed and requirement of wideband data. The dielectric measurement results of developed samples show non-frequency dispersive behaviour, high dielectric constant and data was also selected at 2.45 GHz in aligned to the industrial, scientific and medical band. The resulting measurements show the highest dielectric constant of 16.6 at 2.45 GHz with a very low loss behaviour.

Keywords: Biomedical, high dielectric constant, low loss dielectric, sensors, sintering, open ended coaxial probe, open ended circular waveguide, titanium dioxide.
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Abstract

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Abbreviations

4G – 4th Generation
BDAS – Bone Density Analysis System
BT – Bluetooth
CAD – Computer-Aided Design
COMFORT – Complex Fracture Orthopedic Rehabilitation
DI – De-Ionized
EM – Electromagnetic
HFSS – High Frequency Simulation Software
ISM – Industrial Scientific Medical
kN – kilo Newton MUT –
Material Under Test
NFC – Near-Field Communication
OECP – Open Ended Coaxial Probe
OECW – Open Ended Circular Waveguide
PEG – Polyethylene Glycol
SMA – SubMiniature version A
VNA – Vector Network Analyzer
WiFi – Wireless Fidelity

Constants

\( c_0 = 2.99792458 \cdot 10^8 \text{ m/s}, \) ........................................ Speed of light in vacuum
\( \varepsilon_0 = 8.854187817 \cdot 10^{-12} \text{ Vs/Am}, \) ......................... Permittivity in vacuum
\( \mu_0 = 4\pi \cdot 10^8 \text{ As/Vm}, \) ........................................... Permeability in vacuum
1. Introduction

1.1 Background
Nowadays sensors are integrated in many kinds of electronic devices. Sensors can be used to continuously measure and collect (log) data of the environment, such as temperature, pressure, and humidity. Electromagnetic (EM) sensors are based on fields, which occur from interactions between either static (electrostatics) or dynamic (electromagnetics) charges. As shown in Fig 1, microwave band of the EM spectrum are commonly used in several application, such as; 4 Generation mobile communication network (4G), Wireless Fidelity (Wi-Fi), Bluetooth (BT), Near-Field Communication (NFC), and Radio Detection and Ranging (RADAR). Microwaves have been used over decades in home environment and for mobile communication. Furthermore, microwaves have been considered for medical application involving the detection of tissue variation and for intra body communication. Hence, physical and physiological factors related to tissue variation and EM properties in human body, can be utilized to monitor ongoing biomedical tissue progression. This tissue properties have been studied extensively for instance in ossification (Bone Density measurement Analysis) [2], muscle atrophy or rehabilitation after lower extremity trauma (Complex Fracture Orthopedic Rehabilitation Trauma) [3], and degree of skin burns (SENSEBURN) [4].

Fig. 1. Electromagnetic spectrum [1]
Microwaves in Medical Engineering Group at Uppsala University propose a new approach to diagnose diseases or monitor treatment of patients via non-invasive microwave sensors. These sensors are developed and optimized to be considered to efficiently operate in the reactive near field region. Sensors used in medical devices are evolving; they are excessively developed in different environments like hospitals, rehabilitation centers and in home-care to continuously monitor and follow-up health conditions. To enable such applications, these sensors require being interlock with a specific dielectric medium for optimal operation.

1.2 Aim

The aim of this work is to develop a dielectric material, which has specific material properties adapted for a particular Open Ended Circular Waveguide (OECW) design. This design is compatible to be used in biomedical applications. Recently, the OECW design was developed using Computer-Aided Design (CAD) and simulated with the commercially available High Frequency Simulation Software (HFSS) [5]. The fabricated material should be considered non-frequency dispersive, high dielectric constant $\varepsilon'$, and low loss tangent (tan $\delta$) in the lower microwave frequency spectrum. The characterization/target frequency spectrum for the dielectric material are from 500 MHz - 20 GHz and used in the OECW to operate in ISM-band (2.40 – 2.50 GHz). Fig. 2 schematically represents the CAD development of the final OECW.

![Fig. 2. The development of OECW sensor. (a) representing the exterior,](image-url)
Materials, which have low loss tangent, but high dielectric constant in microwave spectrum, are of high interest for these sensors. The frontend coupling medium acts as an interface between the sensor and the body. For optimal operation condition, the sensor has to be impedance matched to the skin. Great advantage of a material, with high dielectric and low loss tangent, for use as a sensor is that the higher dielectric properties enable a smaller physical size. Ultimately the goal of this work is to achieve, through sintering, a solid and homogeneous material, which is nonfrequency dispersive, good mechanical structure, time-stable, and to achieve a dielectric constant (30 \(<\varepsilon'\ <40\)) with associated low loss gent (\(\tan\delta\ \leq \ 0.001\)).

Characterized in the microwave spectrum, in the range of 500 MHz - 20 GHz.

1.3 Approach

The Material Under Test (MUT), will be characterized and fabricated under various parameters such as pressure, temperature, time, and composition of material. Characterization of the material will be done using an open-ended coaxial method and/or other methods, such as perturbation method, which can increase the reliability of the result. The development and fabrication process consists of, calculations, powder compression, sintering, pellet post processing, measurements, and validation. The fabricated dielectric material will later on be implemented/inserted in the OECW and used in clinical trials.
1.4 Significance

In general, the purpose to develop high dielectric constant and low loss tangent material is to reduce physical dimensions and increase the bandwidth of the sensors. In this project, the motivation is mainly to have an impedance matched coupling medium for higher EM transmission through human skin interface [6]. Low loss material is targeted to achieve an efficient sensor for increased penetration and could lead to develop non-invasive means of monitoring variation of tissue properties.
2. Theory of electromagnetics

2.1 Maxwell theorems and microwaves
Maxwell equations is one way in how electromagnetic theory is commonly explained. The differential form of Maxwell equations are [1], [7-8]:

\[ \nabla \cdot \mathbf{D} = \rho_f \quad (1) \]

\[ \nabla \times \mathbf{E} = (-\partial \mathbf{B})/\partial t - \mathbf{M} \quad (2) \]

\[ \nabla \cdot \mathbf{B} = 0 \quad (3) \]

\[ \nabla \times \mathbf{H} = \mathbf{J}_f + \partial \mathbf{D} / \partial t \quad (4) \]

Where \( \mathbf{D} \) is electric flux density,
\( \rho_f \) is the function of electric charge density,
\( \mathbf{E} \) is electric field intensity,
\( \mathbf{B} \) is the magnetic flux density,
\( \mathbf{M} \) is magnetic current density (which is just illusory and its purpose is to stand as a mathematical suitability),
\( \mathbf{H} \) is magnetic field intensity,
\( \mathbf{J}_f \) is the function of the electric current density.

Where relation between electric flux density and electric field intensity is:
\[ \mathbf{D} = \mathbf{E} \quad (5) \]

Similarly, relation between magnetic flux density and magnetic field intensity:
\[ B = \mu H \]  
\[ = \mu_r \mu_0 \]  
(6) Where permittivity is:
\[ \varepsilon = \varepsilon_0 \]  
(7) and permeability of the medium is:
\[ \mu = \mu_r \mu_0 \]  
(8)

giving the phase velocity in the medium, (dielectric \( \mu_r = 1 \)):
\[ v = \{\text{in general}\} = \frac{1}{\sqrt{\varepsilon \mu_0}} \]  
(9)

wavelength in the medium (dielectric):
\[ \lambda = \frac{c_0}{f \sqrt{\varepsilon}} \]  
(10)

As previously described in Fig. 1, all signals in electromagnetic spectra respond to different frequency and wavelength. Microwaves are classified in frequency between 300 MHz and 300 GHz with wavelength between 1 m and 1 mm in free space \( r \approx 1 \) or vacuum \( r = 1 \),

\[ v = \{\text{Non}Non -- magnetic\text{dielectric} \mu_r = = \]  
\[ 1\} = \frac{1}{\sqrt{\varepsilon \mu_0}} = c_0 \]

2.2 Measurement and characterization

In this work, characterization is performed using an open ended coaxial probe from Keysight, Fig 4, which is used from 500 MHz to 20 GHz in frequency range. An alternative could be perturbation theory in a L-, S-, C-, X- and Ku-band, but requires more effort and will be in consideration only if time is given.

Fig. 4. Keysight open ended coaxial probe [12]
Fig. 5. Performed measurements (a) Calibration settings for coaxial probe, (b) calibration equipment (c) measurement setup, (d) Material Under Test
3. Theory of sintering

Sintering, or calcination, is a thermal treatment process of a powder or powder compact at an elevated temperature below the melting temperature. Since the powder is not melted, the final product will contain void with sintered part having a lower density than cast part of the same material. Generally, the mechanical strength of sintered part is greater as the final density approaches that of continues solid, special application have taken advantages of the porosity that sintered part process. The porous spaces between the material's particles are minimized during the sintering process as the material is squeezed together under high temperature and pressure. This increases some of the material's properties, including:

- Electromagnetic dielectric properties
- Thermal and electrical conductivity
- Material strength
- Translucency

There are endless variations in heating the single material even with few physical parameters, time, pressure and temperature. These parameters can be controlled with sufficient accuracy, but having a significant effect on sintering result. A defined requirement, in the sintering process, is that the temperature has to be lower than melting point of the material.

Process of sintering used in this work will be explained more in next chapters, fabrication of sintered material.
**Fig. 6.** Powder pressing process. (a) Powder (loose), (b) powder partially pressed, (c) powder fully pressed, (d) green compact

**Fig. 7.** Overview of sintering steps from preheating to cooling down processes [11]
Fig. 8. Overview of pressing and sintering equipment for fabrication of samples, where (a) is Ingot -pressing tools, (b) presents high hydraulic pressing tool (0-400 kN) and (c) Furnace MTI VBF-1200X

4. Fabrication process

In the fabrication process, different parameters were considered such as packing pressure, pellet diameter size (before and after sintering), heating duration time and mixture composition. First step is to weight decided powders and mix them either dry or using solvents. Next is to fill the cavity, Fig 9a, of the pressing tool with a defined weight of powder and compress with associated piston, as seen in Fig 9b. After exposing the powder to pressure, the sample is removed out of the pressing tool, Fig 9c. The sample is then placed in the furnace, Fig 9d, for high temperature exposure. The exposure temperature and time profile can be defined from a temperature graph and associated function T(t) in the oven program, Fig. 8c. After sintering, the pellet is taken out, Fig 9e, from the furnace, Fig 9d. To achieve flat surface, post processing is performed, such as grinding, Fig. 9f. The sample is now ready for measuring and validation.
Fig. 9. Step by step process of sintering for sample fabrication (a) filling the powder, (b) powder under pressure, (c) out measurement, (d) pellet placed in the furnace, (e) sintered samples, (f) grinding sample

In the fabrication process, there are many critical steps where things can result in sample failure. In order to fill the pressing tools with the powder, the material should be scaled, and all results documented to optimize and improve next experiment. Failure can easily happen directly while removing or moving the sample to furnace. To achieve sufficient compressed powder without binder is a hard step in sintering process. Either too low or too high packed powder can decrease the possibility to achieve aimed result. Validation and control measurement of sintered samples is important, hence in that way you can determine shrinkage or expansion of material, which was sintered. Several samples were fabricated to achieve valuable results. Positive correlation was shown between increased dimension and sample failure.
5. Results

To validate and characterize all fabricated samples a handheld Vector Network Analyzer (VNA), FieldFox N9918A, Fig. 10a, from Keysight, was used. A performance probe from Keysight was used as a dielectric probe to measure dielectric properties of each sample. The VNA and probe was connected to a semi-rigid coaxial cable, Fig. 10b. The benefit of using performance probe is higher accuracy, larger footprint, and more robust structure, Fig. 10c, compare to Keysight’s other probes.

Keysight requirements for performance probe is:

Maximum recommended \(\varepsilon'\): < 100
Minimum recommended loss tangent > 0.05
Not recommended for low loss (loss tangent < 0.5) materials with \(\varepsilon'\) > 5. [12]

**Fig. 10** Equipment for measuring dielectric constant and loss tangent of sample: (a) Field Fox, (b) KillerBee\textsuperscript{TM} test cable DC – 26.5 GHz, and (c) performance probe kit.
**Fig. 11.** Measurement setup by using performance probe to measure dielectric constant and loss tangent of samples: (a) OECP probe, (b) MUT and (c) probe kit, setup equipment and associated peripherals

**Fig. 12.** Box with sintered samples.

**Table 1.** Sintered samples and determined parameters

<table>
<thead>
<tr>
<th>Sample</th>
<th>Material</th>
<th>Pressure [kN]</th>
<th>Temperature [°C]</th>
<th>Duration [h]</th>
<th>Dimensions (diameter x height) [mm x mm]</th>
<th>Weight [g]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>TiO₂</td>
<td>40</td>
<td>1100</td>
<td>3</td>
<td>13.2 x 2.74</td>
<td>0.8481</td>
</tr>
<tr>
<td>2</td>
<td>TiO₂</td>
<td>30</td>
<td>1100</td>
<td>3</td>
<td>13.24 x 7.58</td>
<td>2.4587</td>
</tr>
<tr>
<td>3</td>
<td>TiO₂</td>
<td>40</td>
<td>1100</td>
<td>3</td>
<td>13.28 x 9.16</td>
<td>2.8766</td>
</tr>
<tr>
<td>4</td>
<td>TiO₂</td>
<td>40</td>
<td>450</td>
<td>3</td>
<td>14.03 x 8.78</td>
<td>2.9</td>
</tr>
<tr>
<td>5</td>
<td>TiO₂</td>
<td>40</td>
<td>450</td>
<td>3</td>
<td>14.01 x 2.46</td>
<td>1.497</td>
</tr>
<tr>
<td>6</td>
<td>TiO₂</td>
<td>50</td>
<td>778</td>
<td>8</td>
<td>24.93 x 4.8</td>
<td>NA</td>
</tr>
<tr>
<td>7</td>
<td>TiO₂+Al₂O₃+CuO</td>
<td>50</td>
<td>778</td>
<td>8</td>
<td>24.97 x 7.07</td>
<td>NA</td>
</tr>
<tr>
<td>8</td>
<td>TiO₂</td>
<td>50</td>
<td>778</td>
<td>8</td>
<td>20.07 x 14.04</td>
<td>NA</td>
</tr>
</tbody>
</table>
Sample 9 | TiO₂+Al₂O₃+CuO | 50 | 778 | 8 | 20.07 x 9.42 | NA
--- | --- | --- | --- | --- | ---
Sample 10 | TiO₂+PEG400 | 50 | 850 | 10 | 25.00 x 42.2 | 36

5.1 Sample measurements

All measured data are gathered in, Table 1, including associated fixed parameters and variables. Before each measurement, the equipment was calibrated using the normal three standard calibration (usually open air, short circuit and De-Ionized (DI) water and performed at the tip of the probe at ambient temperature 22 °C. Fig. 12 shows a characteristic of dielectric constant and loss tangent of DI water.

![Dielectric properties of DI Water](image1)

![Tangent Loss of DI Water](image2)

**Fig. 12. Measured characteristic of DI water at 22°C (a) dielectric constant (b) loss tangent**

During the first sintering trial, the material was heated to 1100 °C and then cooled down, Fig. 13. Three samples were prepared and sintered for 3 hours and cooled down for an hour. After cooling time, each sample was measured and results documented, Table 2. Measurements including slim probe were performed on one sample and all samples are measured with performance probe, Fig. 14, 15, 16.
Fig. 13. Temperature curve of sintering over time

Table 2. Results of measurement at frequency 2.45 GHz

<table>
<thead>
<tr>
<th>Frequency (2.45 GHz)</th>
<th>Slim probe ($\varepsilon_r$)</th>
<th>Performance probe, ($\varepsilon_r$)</th>
<th>Slim probe, (tan $\delta$)</th>
<th>Performance probe, (tan $\delta$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>4.97246</td>
<td>16.4838</td>
<td>-0.01904</td>
<td>-0.0064</td>
</tr>
<tr>
<td>Sample 2</td>
<td>NA</td>
<td>12.5743</td>
<td>NA</td>
<td>-0.0096</td>
</tr>
<tr>
<td>Sample 3</td>
<td>NA</td>
<td>12.10484</td>
<td>NA</td>
<td>-0.0088</td>
</tr>
</tbody>
</table>
Fig. 14. Sample 1, (a) dielectric constant ($\varepsilon_r$), (b) loss tangent ($\tan \delta$)

Fig. 15. Sample 2, (a) dielectric constant ($\varepsilon_r$), (b) loss tangent ($\tan \delta$)

Fig. 16. Sample 3, (a) dielectric constant ($\varepsilon_r$), (b) loss tangent ($\tan \delta$)
During the second sintering trial, samples were heated to 450 °C for 180 minutes and then cooled down for an hour, Fig. 17. After cooling period samples were measured, Table 3, one sample was measured with a slim probe and both samples were measured with performance probe, Fig. 18, 19.

**Table 3. Results of measurement at frequency 2.45 GHz**

<table>
<thead>
<tr>
<th>Frequency (2.45 GHz)</th>
<th>Slim probe, ( \varepsilon_r )</th>
<th>Performance, probe, ( \varepsilon_r )</th>
<th>Slim probe, ( \tan \delta )</th>
<th>Performance probe, ( \tan \delta )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 4</td>
<td>4.33098</td>
<td>16.6556</td>
<td>0.0346</td>
<td>0.0151</td>
</tr>
<tr>
<td>Sample 5</td>
<td>NA</td>
<td>8.7038</td>
<td>NA</td>
<td>0.0049</td>
</tr>
</tbody>
</table>
Fig. 18. Sample 4, (a) dielectric constant ($\varepsilon_r$), (b) loss tangent ($\tan \delta$)

Fig. 19. Sample 5, (a) dielectric constant ($\varepsilon_r$), (b) loss tangent ($\tan \delta$)

Sintering 3

Temperature in °C

0 100 200 300 400 500 600 700 800 900

Time (min)

0 200 400 600 800 1000 1200 1400
In the third sintering trial, sintering program is prolonged where highest temperature was 778 °C almost 8 hours, Fig. 20. After sintering, the samples were cooled for 3 hours. Sample 6 and 8 were dry mixed and only contained TiO$_2$ in powder form. Sintered samples were measured with performance probe, Table 4.

Table 4. Results of measurement at frequency 2.45 GHz.

<table>
<thead>
<tr>
<th>Frequency (2.45 GHz)</th>
<th>Performance probe, ($\varepsilon_r$)</th>
<th>Performance probe, (tan $\delta$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 6</td>
<td>5.6005</td>
<td>0.0897</td>
</tr>
<tr>
<td>Sample 8</td>
<td>5.89122</td>
<td>0.0551</td>
</tr>
</tbody>
</table>

Fig. 21. Sample 6, (a) dielectric constant ($\varepsilon_r$), (b) loss tangent (tan $\delta$)
Sample 7 and Sample 9 were dry mixed with TiO$_2$, CuO, and Al$_2$O$_3$ and pressed and sintered in the third sintering trial, Fig. 20. Sintered samples were measured with performance probe, Table 5.

**Table 5. Results of measurement at frequency 2.45 GHz**

<table>
<thead>
<tr>
<th>Frequency (2.45 GHz)</th>
<th>Performance probe, ($\varepsilon_r$)</th>
<th>Performance probe, (tan $\delta$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 7</td>
<td>4.44512</td>
<td>0.0661</td>
</tr>
<tr>
<td>Sample 9</td>
<td>3.7206</td>
<td>0.3325</td>
</tr>
</tbody>
</table>
Fig. 24. Sample 9, (a) dielectric constant ($\varepsilon_r$), (b) loss tangent ($\tan \delta$)
Sample 10 was sintered in a longer sintering process where temperatures and time were slightly prolonged, Fig 25. Material was sintered for 8 hours and cooled down for 6 hours. Material was mixed with PEG 400 (Polyethylene Glycol molar mass 400) and pressed to 50 kN.

**Table 6. Results of measurement at frequency 2.45 GHz**

<table>
<thead>
<tr>
<th>Frequency (2.45 GHz)</th>
<th>Performance probe, ($\varepsilon_r$)</th>
<th>Performance probe, (tan $\delta$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 10</td>
<td>5.74382</td>
<td>-0.0044</td>
</tr>
</tbody>
</table>

**Fig. 25. Temperature curve of sintering 4 over time**

**Fig. 26. Sample 10, (a) dielectric constant ($\varepsilon_r$), (b) loss tangent (tan $\delta$)**
6. Conclusion and discussion

In this work, 10 samples were successfully sintered and measured. From the result, one clear observation is that a sufficient high dielectric constant was not yet achieved in the trials but all samples had very low loss tangent. Furthermore, the results can be used as a guide for designing alternative, improved approaches for future work. One approach would be to gradually higher the sintering temperature and to increase the sintering time. As the best results were seen within the first sintering trial where the used temperature was 1100°C, future trials could be initiated from this temperature and time parameters. Sintering large pieces of 25 mm in diameter and 50-60 mm in height was a very challenging task.

Fig. 27. Dielectric with good physical characteristics and $\varepsilon_r = 5$

A correlation was noticed between 25 mm and 20 mm samples where the larger pieces have lower dielectric constant compared to the smaller ones. This could be the consequence of gradually inhomogeneity in material after sintering.

Considering the preparation and properties used in preparation of samples, specific changes could be applied when preparing a material before pressurizing:

- To mill the material to smaller particle size (10-15µm)
- To mix the powder with solvent, and evaporate it before putting it in the pressure mold
- To use PEG with the lower molar mass preferably PEG 200
• To use lubrication to facilitate removing the compact out of the mold

Subsequently it was shown that measurements with OECP technique might not to be the most suitable one for these kinds of low loss tangent specimens. Measurements were still done with this technique for the reason that it was the only available method at the time. From the results it was visible that the loss tangent was too low for these measurements to be validated due to limitations of the measurement equipment. Event thought the measurements were not optimal we could still make several important observations that give good estimations of how future trials should proceed and suggestions on more suitable measurement methods that should be done. Samples should be measured with cavity-perturbation method (measurement method used for solid materials) and compared to the received results. This comparison can give an insight in reliability of performance probe as a measurement method for solid samples. For the future works the achieved material could be used as substrate in Split Ring Resonator (SRR) to do an accurate single frequency measurement.
References


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Acknowledgements

I would like to thank you all, who helped me in this work. My counselor and examiner Nóra Masszi who advised me for so many years.
Special thanks to Associate Professor Robin Augustine and his Microwaves in Medical Engineering Group (MMG): Syaiful, Mauricio, Noor, Viktor, and Laya.
Special thanks to Jacob Velander, my supervisor, a long lost, and found mate.
Thank you for all the help and advice you gave me. Yeik!

Thank you, my family, for the support and patience you showed in this stressful period.

Uppsala, Jun 2018