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# Cerium and Titanium Oxide Interdigitated Capacitors for Pressure Sensing Applications

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## Abstract

Pressure measurement is an essential tool in understanding environmental and biological processes. However, devices for such applications need to be small in size and easy to adhere to a wide range of surfaces. It is also preferable that the data gathered by such a device be transferred wirelessly to a receiver, remote from the sensing environment, for the safety of personnel or patient comfort. With this in mind, the pressure sensing properties of thick film cerium oxide ($CeO_2$) and titanium oxide ($TiO_2$) interdigitated capacitors were investigated. The devices were fabricated on flexible polymer substrates so that they could be attached to both planar and cylindrical surfaces. Each sensor was then placed under hydrostatic pressure ranging from 0 – 17 kPa. It was found that sensors based on $TiO_2$ were most sensitive. It is thought that this is due to differences in the materials particle size.

**Keywords**: cerium oxide, titanium oxide, interdigitated capacitors, pressure sensors

## 1 Introduction

The ability to remotely measure physical and chemical changes in difficult to reach areas has a number of applications in both biological and environmental monitoring [1]. There is a particularly high demand for reliable and cost-effective pressure sensitive devices as they are considered a useful tool for clinical studies in such areas as cardiology, neurology and rehabilitation [2].

To date, MEMS sensors have been the preferred method of creating miniature sensors, particularly for biomedical applications [3, 4]. Typically, these devices are based on the capacitive principle as they are more sensitive and have lower power consumption than piezoresistive sensors [5]. However, the presence of moving parts makes packaging, which is particularly important for biomedical sensors, a difficult task [6]. A further disadvantage is the fact that MEMS are only cost-effective if $10^5$ – $10^6$ devices are produced per year.

Thick film technology offers a cost effective and reliable alternative to sensor fabrication, which is capable of meeting the demand for medical sensors [5]. The most common method of thick film sensor fabrication is screen-printing. This technique can achieve high line resolution with economical use of paste thus lowering production costs [7]. In addition, capacitors with no moving parts and high sensitivity can be fabricated [8]. A large number of materials are suitable for use and as a result, devices with the desired physical properties can be achieved [9].

Previous investigations into the properties of interdigitated capacitors with a polyvinylidene fluoride (PVDF) dielectric layer have shown high sensitivity to pressure [10]. Interdigitated electrodes with a polymer based dielectric layer have also been popular in the development of strain gauges [11].

In this work, interdigitated capacitors were fabricated using either cerium oxide ($CeO_2$) or titanium oxide ($TiO_2$) as the dielectric layer. Cerium oxide has unique electrical properties, which make it useful for applications involving microelectronic and optoelectronic devices [12]. On the other hand, $TiO_2$ is a popular material for use in medical applications, as it is biocompatible due to its chemical stability [13].

Both materials were prepared and deposited on interdigitated electrodes by screen-printing. Each material was then examined using SEM in order to gain an understating of their morphology and particle size. Their sensitivity was tested by placing the devices under hydrostatic pressure ranging from 0 – 17 kPa.

## 2 Experimental Procedure

To fabricate interdigitated capacitors, the conductive and insulative layers were deposited by screen-printing onto polymer substrates. DuPont 4929 conductive paste was used for the electrodes, while the $CeO_2$ and $TiO_2$ dielectric pastes were developed in house.

To form the dielectric paste, the functional material (either $CeO_2$ or $TiO_2$, supplied by Sigma Aldrich and Riedel-De Haen Ag Seelze-Hannover respectively) was mixed with 7 wt.% of binder, which in this case is polyvinyl butyral (PVB). Finally, the solvent ethylenglycolmonobutylether was added in order to form a paste of suitable consistency.

Interdigitated electrodes are essentially two electrodes, which form a comb like structure. Traditionally, this has been popular for use in chemical sensing, as lumped elements for microwave integrated circuits and for dielectric studies on thin films [14-16]. However, more recently they have also found use in pressure and strain sensing applications [10, 11]. Modelling the capacitance of interdigitated structures is not a simple task, as analytical methods can only provide solutions to traditional electrode shapes such as parallel-plate and cylindrical [17].

The capacitance of interdigitated structures generally depends on the electrode gap (G), finger length (L) and width (W), the spatial wavelength ($\lambda = 2(W+G)$) and metallization ratio ($\eta = 2W/\lambda$) [16]. These terms are characteristic of the electrodes and do not change during operation. As a result, the sensitivity of the device is dependant on the changing properties of the dielectric layer deposited over the electrodes.

In this study, electrodes with 25 fingers, of length 6 mm and width 0.2 mm were used. The spacing between the electrodes was 0.2 mm. The thickness of the $CeO_2$ and $TiO_2$ layers were measured by profilmetry and found to be 35.87 µm and 22.5 µm respectively. Their baseline capacitance was measured to be 9 pF and 7.5 pF respectively.

Once the capacitors were formed, their AC electrical properties were examined from 1 Hz – 10 MHz using a Solatron S1 1260 Impedance Gain/Phase Analyser. Devices were adhered to planar and cylindrical surfaces. Their response to hydrostatic pressure in the range 0 – 17 kPa was investigated. This pressure range was chosen as typical pressures in the GI tract range from 6 – 16 kPa, while arterial pressure lies between 13 kPa and 18 kPa and intracranial pressure is in the region of 1 kPa. The sensor was protected using a thin waterproof polymer membrane. Changes in capacitance with pressure were converted to a frequency, which was wirelessly transmitted to an external receiver, using the block-diagram shown in figure 1.

At the receiver end of the circuit, the frequency is converted to a voltage, which is the final output. This voltage (*Vo*) is related to the sensor capacitance (*C*) according to equation 1

$$C = \frac{1}{(V_o \times k_{VCO})(R_2 + 2R_1)\ln 2} \qquad (1)$$

where, $k_{VCO}$ is the measured response from the voltage controlled oscillator (slope of frequency vs. voltage = 13.1 kHz/V) and $R_1$ and $R_2$ are the timer resistors which are used to control the transmitted frequency.

Using this approach, the response of each sensor was recorded. In addition, their hysteresis was measured to be the maximum difference between loading and unloading cycles as a percentage of full-scale. Their repeatability was measured to the maximum difference between calibrating cycles and is also expressed as a percentage of full-scale.

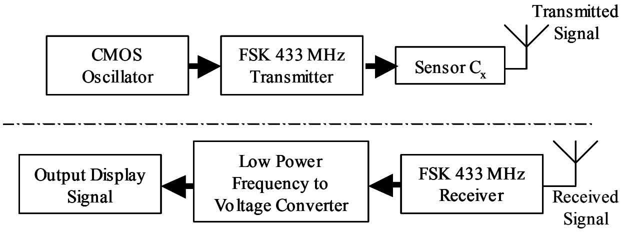


**Figure 1:** Interface, transmitter and receiver circuit using for hydrostatic pressure measurements

To determine the short-term stability of each device on a planar surface, they were left under a static pressure of 7 kPa for a twenty-four hour period so that any creep in the output capacitance could be recorded.

The effect of temperature on each device was studied using a specially designed test rig based on the peliter effect. Using this device, the temperature was varied from 10 °C – 60 °C and the change in capacitance was recorded.

Finally, an Olympus microscope was used to examine the surface of the $CeO_2$ and $TiO_2$ films when placed on planar or cylindrical surfaces.

# 3 Results and Discussion

## 3.1 Scanning Electron Microscopy (SEM)

A Joel JSM-840 SEM was used to examine the particle size and morphology of the polymer thick films. Cerium oxide films were viewed at a magnification of 5000, as shown in figure 2.


**Figure 2:** SEM of $CeO_2$ film at 5000 magnification

It can be seen that the film consists of flake like particles of $CeO_2$ with a diameter of approximately 5 µm. In contrast, SEM images of the $TiO_2$ film at a magnification of 20000 reveal a film with spherically shaped particles of diameter 200 nm. This can be seen in figure 3.

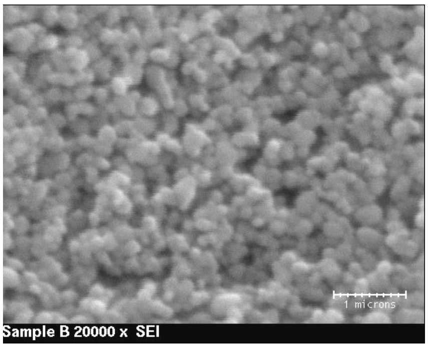


**Figure 3:** SEM of $TiO_2$ film at 20000 magnification

### 3.2 AC Properties

As capacitive sensors are excited by AC signals, the frequency dependant behaviour of the $CeO_2$ and $TiO_2$ devices was investigated [8]. The change in capacitance with frequency ranging from 1 Hz – 10 MHz for both materials is shown in figure 4.

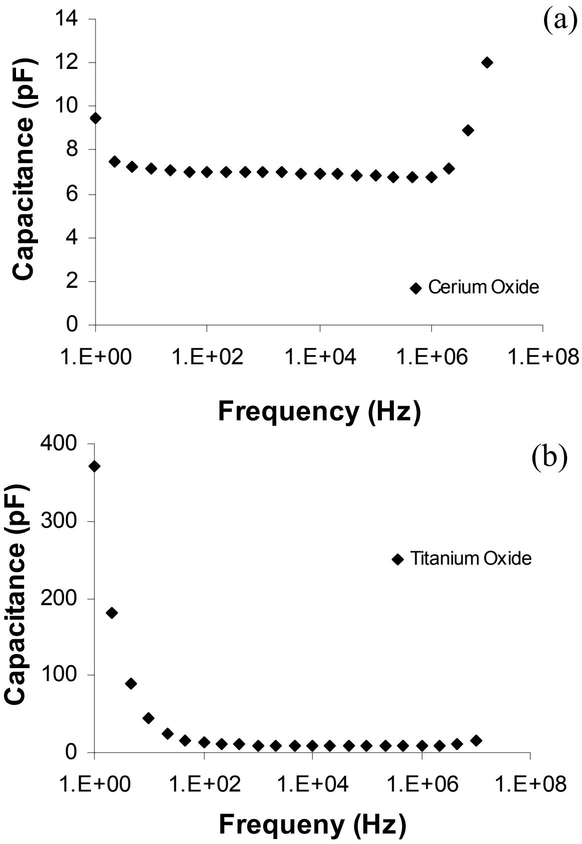


**Figure 4:** Variation in capacitance with frequency for (a) $CeO_2$ and (b) $TiO_2$

It can be seen that $CeO_2$ is stable up to 1 MHz, after which the capacitance is seen to increase rapidly. For $TiO_2$ devices, strong frequency dependence has been observed with the capacitance decreasing rapidly between 1 and 100 Hz. From 100 Hz to 1 Mz, the capacitance remains essentially stable, beyond this point, the capacitance can be observed to increase further.

It can be concluded from these results, that the optimum frequency for each device lies in the region of 100 Hz – 1 MHz. This is important when designing suitable interface circuitry for the devices.

### 3.3 Sensitivity of Interdigitated Capacitors to Hydrostatic Pressure

#### 3.3.1 Devices on Planar Surfaces

Interdigitated capacitors with dielectric layers based on $CeO_2$ and $TiO_2$ were interfaced with the circuit shown in figure 1 and tested in the range 0 – 17 kPa. The change in capacitance of each sensor was represented as a corresponding change in voltage at the receiver end of the circuit. Each sensor was tested under hydrostatic conditions as biomedical applications involve the measurement of pressure in fluids [2]. The response of both devices is shown in figure 5. It can be seen that $TiO_2$ capacitors are most sensitive to pressure variations, with the change in voltage over the entire pressure range being recorded as 44.5 mV, while for $CeO_2$ devices it is 24.4 mV.

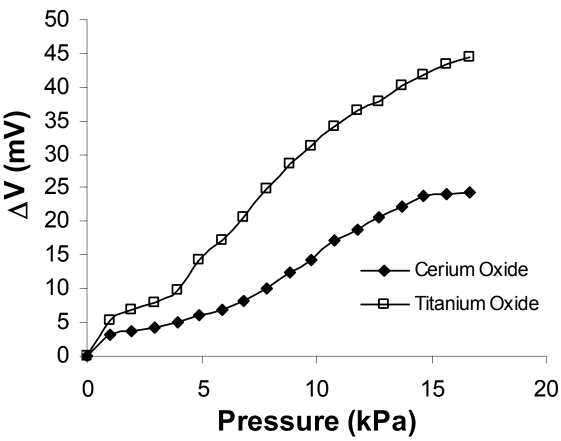


**Figure 5:** Response of $CeO_2$ and $TiO_2$ capacitors on planar surfaces to pressure

It is thought that the higher sensitivity to pressure displayed by the $TiO_2$ capacitor is a result of the smaller particle size (200 nm), recorded by SEM as shown in figure 3. The non-linear response can in part be attributed to the characteristics of the phase locked loop, which is part of the circuit on the receiver end of the system. It is thought that the inflection point, which occurs at approximately 4 kPa, may be caused by the presence of the protective polymer film.

It was also observed that $TiO_2$ capacitors displayed lower hysteresis, when compared to the $CeO_2$ devices. Previously measured values for polymer thick film devices range from 6 % to 30 % [8, 18]. The cause is generally attributed to friction and structural changes within the material [19]. Figure 6 shows the hysteresis for $CeO_2$ and $TiO_2$, which was measured to be 7 % and 4 % respectively.

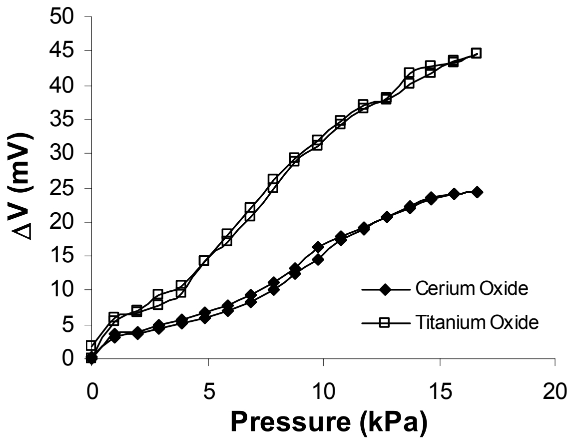


**Figure 6:** Hysteresis displayed by $CeO_2$ and $TiO_2$ capacitors on planar surface

The repeatability of each capacitor was measured by cycling the measurements a number of times. The results for each device over five cycles are shown in figure 7.

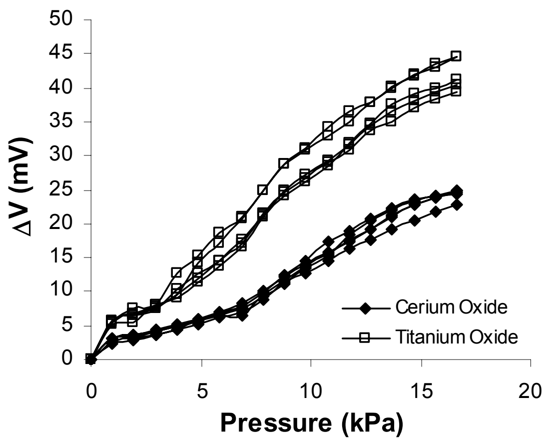


**Figure 7:** Repeatability measured for $CeO_2$ devices on planar surface

Devices based on $CeO_2$ showed a repeatability of 14 %, while for those using a $TiO_2$ dielectric layer a value of 17 % was recorded. The results compare well with those previously obtained for thick film sensors [18]. For polymer materials, the most likely cause of repeatability errors is material plasticity, although other causes include thermal noise and charge build-up [19].

To determine the long stability of each device, they were left under a static pressure of 7 kPa for 24 hours. The results are shown in figure 8. It can be seen that the capacitance changed by 3 % and 4 % for $CeO_2$ and $TiO_2$ devices respectively. It is thought that changes in capacitance over time are caused by viscoelastic behaviour, displayed by the polymer component of the oxide thick films [20] .

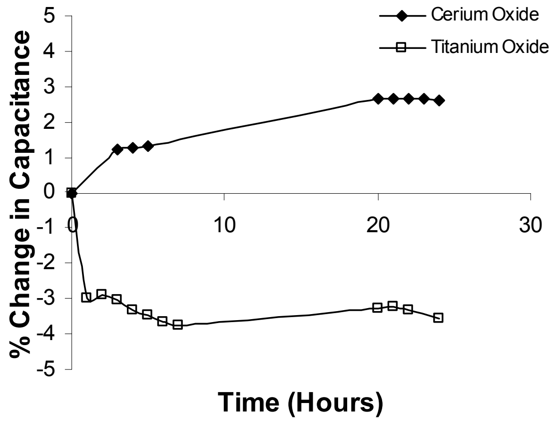


**Figure 8:** Creep measured over 24 hours for $CeO_2$ and $TiO_2$ devices

From the results presented here it can be seen that capacitors using $TiO_2$ for the dielectric layer are more sensitive to pressure changes. However, it is also important to quantify the sensitivity of both the $CeO_2$ and $TiO_2$ devices to temperature, in order to develop stable interface circuitry. Each sensor was tested in temperature range 10 $^oC$ – 60 $^oC$ and the Temperature Coefficient of Capacitance (TCC) was calculated according to equation 1,

$$TCC = \frac{C_{t_1} - C_{t_2}}{C_{t_1}\Delta T}10^6 \quad (1)$$

where, $C_{t1}$ is the capacitance at temperature 1, $C_{t2}$ is the capacitance at temperature 2 and $\Delta T$ is the change in capacitance. It was found that both devices were extremely sensitive to temperatures less than 20 $^oC$, with the TCC in region 1 (10 $^oC$ – 20 $^oC$) measured to be 92371 and 99902 ppm/$^oC$ for $CeO_2$ and $TiO_2$ respectively. The TCC for region 2 (20 $^oC$ – 60 $^oC$) was 500 and 2680 ppm/$^oC$ respectively, which is well within the range considered normal (up to 3000 ppm/$^oC$) for thick film capacitors [8].

### 3.3.2 Devices on Cylindrical Surfaces

Devices on flexible substrates can easily be adhered to surfaces with more complex geometries. This can be an advantage in biomedical applications. For example, strain gauges are often attached to limb implants in order to asses their performance [21]. As a result, devices on flexible substrates were adhered to cylindrical surfaces of diameter 7 mm. Their sensitivity, hysteresis and repeatability were then measured.

The change in voltage for pressures in the range 0 – 17 kPa was measured to be 20 mV and 34 mV for $CeO_2$ and $TiO_2$ devices respectively. The results are shown in figure 9.

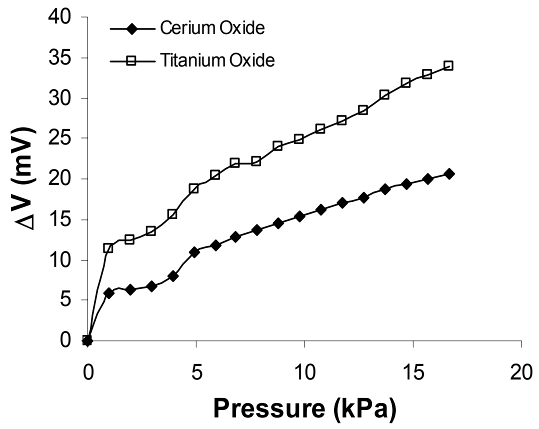


**Figure 9:** Response of $CeO_2$ and $TiO_2$ devices on cylindrical surfaces to pressure.

In this study, the hysteresis was measured to be 11 % and 10 % for $CeO_2$ and $TiO_2$ devices respectively, as shown in figure 10.

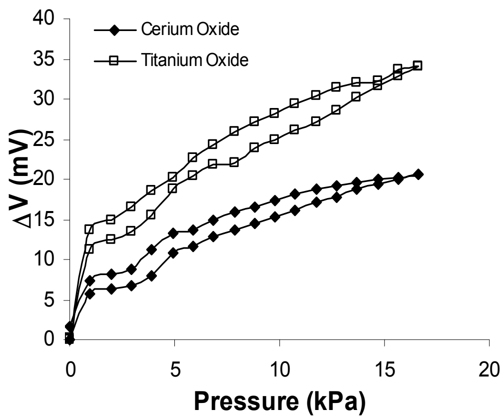


**Figure 10:** Hysteresis measured for $CeO_2$ and $TiO_2$ devices on cylindrical surfaces.

The repeatability of the $CeO_2$ and $TiO_2$ devices was measured to be 17 % and 15 % respectively. The results over five cycles are shown in figure 11.

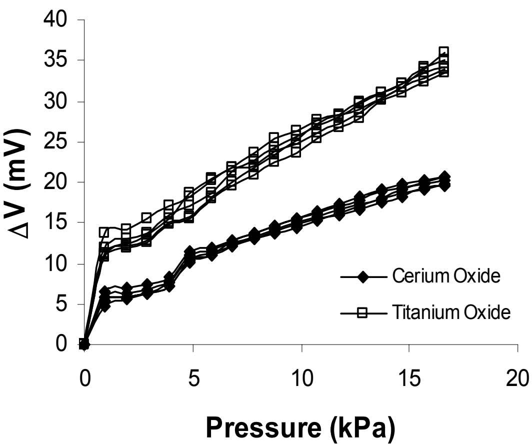


**Figure 11:** Repeatability as measured for $CeO_2$ on cylindrical surfaces

Previous investigations into the pressure sensing properties of polyvinylidene fluoride (PVDF) interdigitated capacitors showed that when they are adhered to cylindrical surfaces their sensitivity increases [10]. This is also accompanied by an increase in hysteresis, caused by the formation of cracks in the polymer thick film.

In this study, an increase in hysteresis was recorded for both $CeO_2$ and $TiO_2$ devices. However, unlike the PVDF devices, there was a marked decrease in sensitivity for both materials.

A comparison of the surface of PVDF, $CeO_2$ and $TiO_2$ thick films, by optical microscopy is shown in figure 12. It can be seen that the PVDF surface shows evidence of cracking which is increased through bending of the polymer substrate. On the other hand the $CeO_2$ film surface shows no evidence of cracking and the $TiO_2$ film shows a small amount of cracking around the electrodes when the substrate is placed on a cylindrical surface.

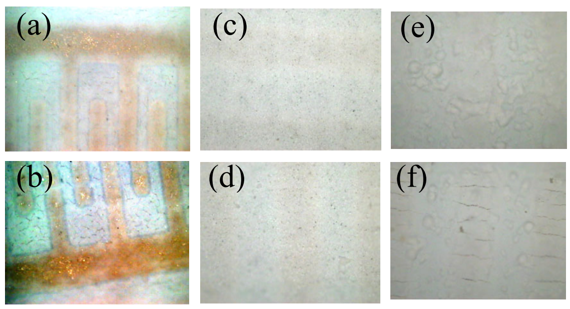


**Figure 12:** Images of (a) PVDF film on planar surface, (b) PVDF of a cylindrical surface, (c) $CeO_2$ on a planar surface, (d) $CeO_2$ on a cylindrical surface, (e) $TiO_2$ on a planar surface and (f) $TiO_2$ on a cylindrical surface.

## 4 Conclusions

In this work the response of $CeO_2$ and $TiO_2$ thick films to pressure ranging from 0 – 17 kPa was examined. The devices were fabricated on flexible substrates and then adhered to planar and cylindrical surfaces. The morphology of each film was examined using SEM and it was found that $TiO_2$ films had a significantly smaller particle size (200 nm) when compared to $CeO_2$ films (5 µm). This may be used to explain the performance of each sensor under hydrostatic pressure. The results show that capacitors with a $TiO_2$ dielectric layer were the most sensitive.

It was also seen that devices on cylindrical surfaces showed a marked decrease in sensitivity, in contrast to the increase observed for PVDF films. After comparison of the films through optical microscopy it can be seen that the $CeO_2$ and $TiO_2$ films show little or no cracking, unlike the PVDF film. This may be the cause of the reduced sensitivity. It can be concluded that both materials show high sensitivity to pressure and could be used for wireless biomedical pressure sensing applications.

## 5 Acknowledgements

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