

**THE EFFECT OF TiO<sub>2</sub> THIN FILMS ON THE SENSITIVITY,  
REPEATABILITY AND CURRENT DENSITY OF THE DIELECTRIC  
BOLOMETER Ba<sub>0.6</sub>Sr<sub>0.4</sub>TiO<sub>3</sub> AS A DISTANCE SENSOR**

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**ABSTRACT**

Sol-gel method has been used for the preparation of titanium as a buffer layer in the dielectric bolometer Ba<sub>0.6</sub>Sr<sub>0.4</sub>TiO<sub>3</sub>. The TiO<sub>2</sub> films were prepared onto RuO<sub>2</sub>/SiO<sub>2</sub>/Si substrate and annealed at various temperatures. The X-ray diffraction (XRD) results showed that TiO<sub>2</sub> thin film annealed at 300 and 350 oC were amorphous, and transformed into the anatase form at 400 oC, and mix phase between anatase and the brookite phase at 450 oC. The field emission scanning electron microscope (FE-SEM) results showed that the grain size of TiO<sub>2</sub> thin films increased as the temperature increased. All the sensitivity, repeatability and current density of the sensors decreased with the increased grain size of TiO<sub>2</sub> thin films after annealed above 350 oC. Our result shows that all of the sensors except sensor with TiO<sub>2</sub> film annealed at 550 oC can act as a sensor even though without the IR source.

**INTRODUCTION**

Recently, dielectric bolometer barium strontium titanate (BST) has been developed and commercially available for INFRARED (IR) sensors [1]. The dielectric bolometer BST is different from the thermal-type IR sensors, i.e. thermopile, pyroelectric (PE) sensor and resistive bolometer. It does not need thermal stabilizer as well as the IR chopper (in spell out mode) [2]. It also offers lower cost, faster response times than ultrasonic distance sensor [3], highly sensitive and compact IR sensors compared to those of the other type.

TiO<sub>2</sub> is a well-known n-type semiconductor that possesses a high dielectric property. TiO<sub>2</sub> thin films have been widely investigated for various applications, e.g., photocatalysts [4-6], photoelectrodes [7], gas sensors [8], electrochromic display devices [9,10] and as a buffer layer in the dielectric bolometer [11,12]. Natural polymorphs of TiO<sub>2</sub> are known to exist as tetragonal rutile, anatase, and brookite. According to Zhang and Banfield [13], based on isothermal and isochronal reactions, the transformation sequence and thermodynamic phase stability of the TiO<sub>2</sub> thin films depend on the initial particle sizes of anatase and brookite. For small particle size (<35nm) anatase is more stable than brookite [14].

In this paper, we report the effect of TiO<sub>2</sub> thin films on the sensitivity, repeatability and current density of the dielectric bolometer Ba<sub>0.6</sub>Sr<sub>0.4</sub>TiO<sub>3</sub> as a distance sensor. The TiO<sub>2</sub> thin films were prepared by sol-gel process using titanium butoxide as a starting

material and HCl as a catalyst (see Figure 1). The prepared TiO<sub>2</sub> thin films were characterised using XRD and FE-SEM. The current density of the distance sensor was measured using an Electrometer Model Keithley 2602 System SourceMeter. The arrangement as shown in Figure 2b was employed in order to measure the sensitivity and repeatability of the sensor.

## EXPERIMENTAL

P-type silicon substrates (12 mm x 20 mm x 1.2 mm) were used as the support substrates. It was carefully cleaned by ultrasonic cleaner with acetone, methanol and distilled-water as the clearing agents. SiO<sub>2</sub> and RuO<sub>2</sub> were deposited by electron-gun evaporation with thickness of 1500 Å and 1000 Å, respectively. The TiO<sub>2</sub> thin films were prepared onto RuO<sub>2</sub>/SiO<sub>2</sub>/Si substrate. Figure 1 shows the flow chart of the TiO<sub>2</sub> thin films preparation process.

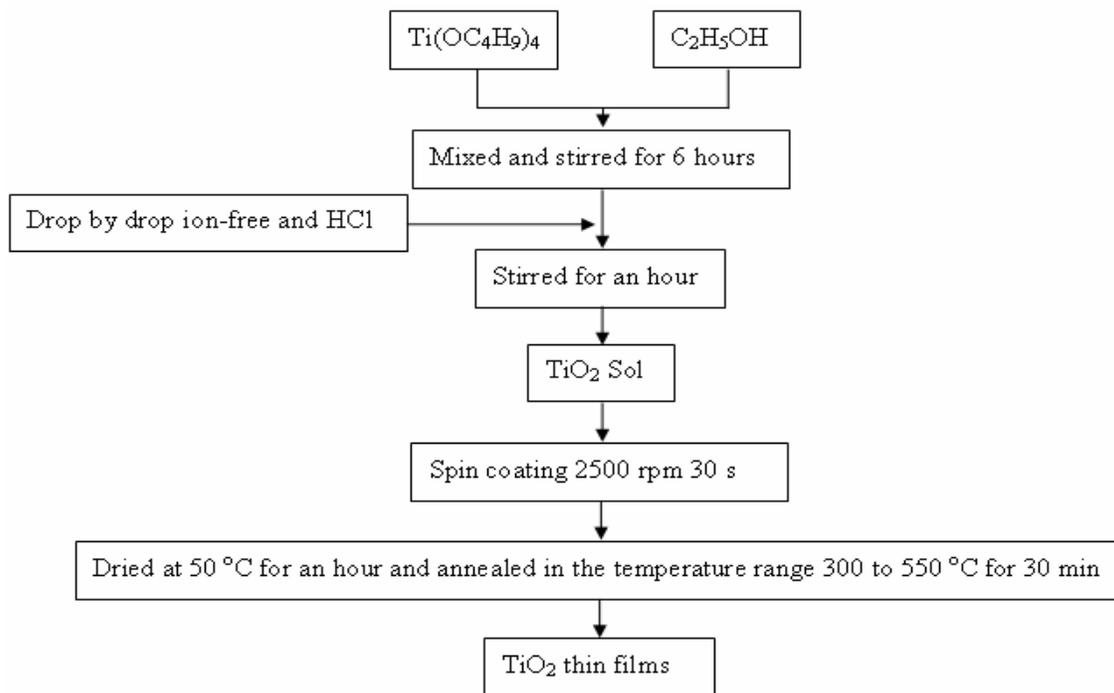


Figure 1: The process of fabrication of the TiO<sub>2</sub> thin films

Titanium butoxide (1.001 ml) was dissolved in ethanol (5.076 ml). After stirring vigorously for 6 hours at room temperature, the ion-free water (0.300 ml) and hydrochloric acid (0.432 ml) was added to the above solution during constant stirring. Then, the solution was vigorously stirred for an hour at room temperature. The obtained solution was transparent at room temperature. The chemical composition of starting butoxide solution were Ti(OC<sub>4</sub>H<sub>9</sub>)<sub>4</sub>: C<sub>2</sub>H<sub>5</sub>OH: H<sub>2</sub>O: HCl = 1:26.5:1:1 in molar ratio. Using a spin coater at 2500 rpm for 30 seconds, the gel was transferred into film form.

The TiO<sub>2</sub> films prepared on RuO<sub>2</sub>/SiO<sub>2</sub>/Si were used for polymorphs and grain size characterisation. All of the films were dried at 50 °C in air for an hour to evaporate the solvent then the films were annealed at the temperature range of 300 to 550 °C with increasing temperature rate of 5 °C/min for 30 minutes in furnace. X-ray diffraction was used for crystal phase identification and field emission scanning electron microscope (FE-SEM) for measured the grain size of the TiO<sub>2</sub> thin films. To get a configuration of the dielectric bolometer Ba<sub>0.6</sub>Sr<sub>0.4</sub>TiO<sub>3</sub> as a distance sensor, the BST thin films was prepared by sol-gel process onto TiO<sub>2</sub>/RuO<sub>2</sub>/SiO<sub>2</sub>/Si substrate [11]. After that, aluminium (1000 Å) layer was deposited onto the samples by electron-gun evaporation. These will give us the capacitor with configurations of Al/BST/TiO<sub>2</sub>/RuO<sub>2</sub>/SiO<sub>2</sub>/Si (Figure 2a).

The Electrometer Model Keithley 2602 System SourceMeter was used to measure the current density of all of the sensors. The sensitivity and repeatability of all of the sensors have been characterised with and without infrared radiation from a LED (NEWPORT model 505B laser diode driver) with a peak wavelength at 830 nm (Figure 2b). The sensors were connected to circuit with supply voltage 1.8 volts. The LED driver was switched to functions generator as a chopper. A final output signal was observed with an oscilloscope (Hitachi VC-6545 digital storage oscilloscope). The IR source position was fixed and the sensor was moveable.

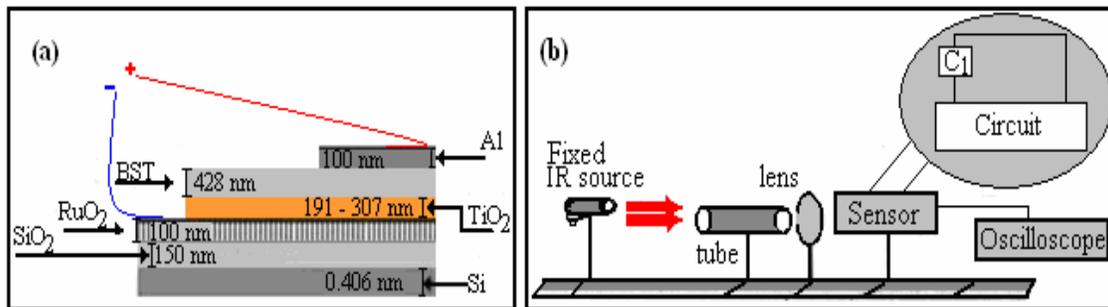


Figure 2: (a) Schematic structure of the BST sensors and (b) The set-up for measuring the sensitivity and repeatability of the sensor

## RESULTS AND DISCUSSION

Figure 3 shows the XRD patterns of the TiO<sub>2</sub> films annealed at 300 to 550 °C for 30 minutes in air. Our results shown that the film annealed at 300 to 350 °C have amorphous structure. Increasing the temperature to 400 °C crystallized the films into anatase phase. These results agree well with the results reported by Yu et al [21] and Kim et al [22] who used sol-gel technique to prepare their TiO<sub>2</sub> thin films. In this work, mixed phase between anatase and brookite was obtained as the temperature increased from 450 to 550 °C. These increments also improve the crystallinity as indicated by the increment of the peaks intensities.

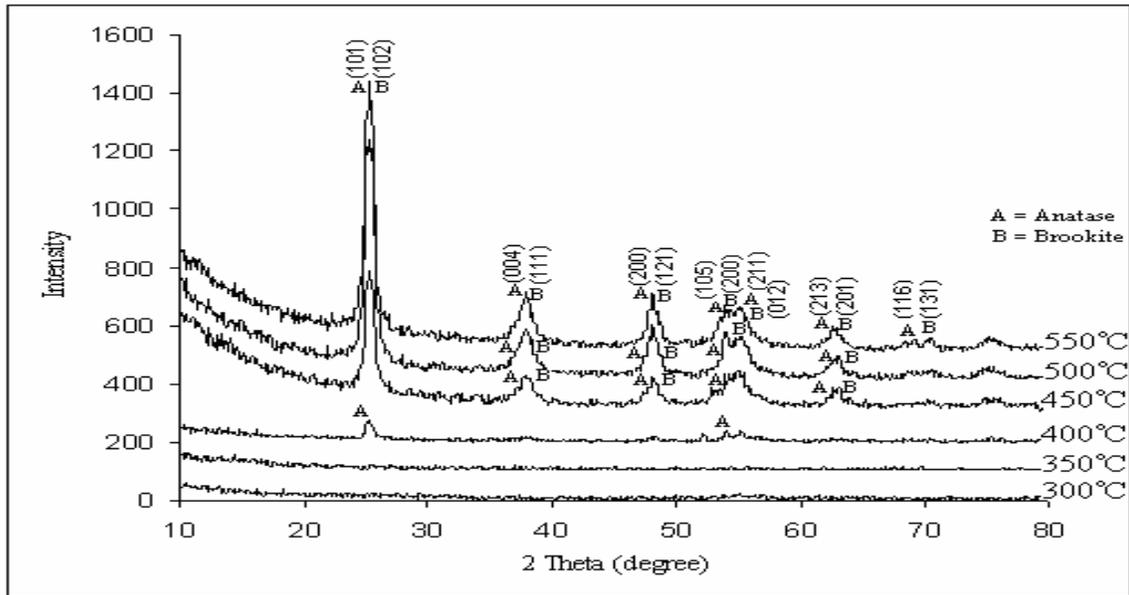


Figure 3: XRD patterns of the TiO<sub>2</sub> thin film annealed at various temperatures.

Figure 4 shows the FESEM micrographs of the surface of the films annealed at various temperatures. As the annealing temperature increased, the grains sizes were also increased. The increment of the average grain size was from 17.0 to 27.5 nm.

As the grain size of the TiO<sub>2</sub> increased, the space between grains also increased this causing the increment of the current leakage in the TiO<sub>2</sub> thin films. Besides the crystallinity of the TiO<sub>2</sub> thin films, the grain size of the TiO<sub>2</sub> thin films are also very important to the next process in the sensor fabrication as the upper ferroelectric BST which directly deposited on the TiO<sub>2</sub> thin films. The grain size of TiO<sub>2</sub> thin films also affect to the interface between BST and RuO<sub>2</sub>. If the grain size of TiO<sub>2</sub> is bigger, then the interface between BST and RuO<sub>2</sub> will be larger.

Figure 5 shows the results of the current density of the dielectric bolometer Ba<sub>0.6</sub>Sr<sub>0.4</sub>TiO<sub>3</sub>. The measurements were done in the voltage range from 0 to 3 volt at room temperature. The  $J_c$  increased as the annealing temperature of the TiO<sub>2</sub> thin films increased. Above 350 °C, all the  $J_c$  of the sensors decrease as the annealing temperature of the TiO<sub>2</sub> films increased. This could be due to the effect of anatase-brookite phase and the increment of the grain size of the TiO<sub>2</sub> thin films which then increased the space between the grains in the TiO<sub>2</sub> films hence the leakage current also increased.

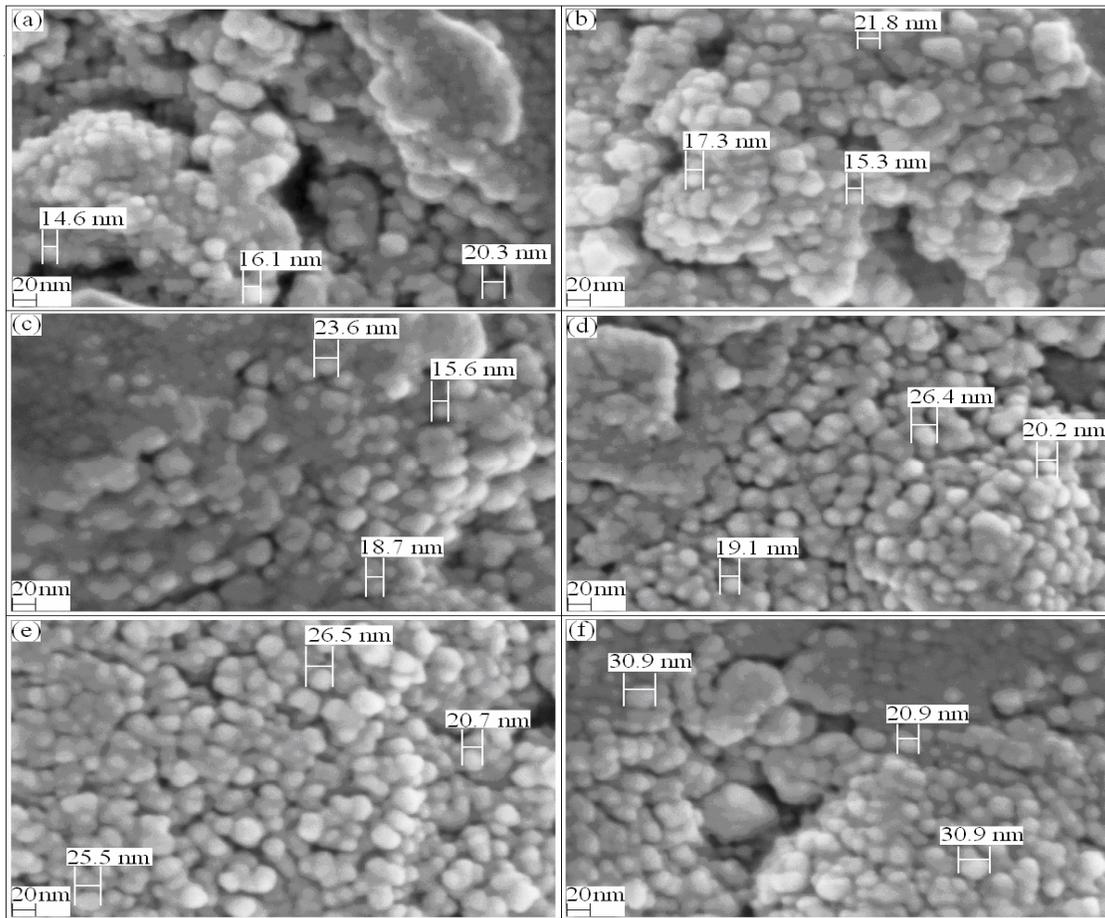


Figure 4: FESEM micrographs photographs of the surface of TiO<sub>2</sub> thin films (a) 300 °C, (b) 350 °C, (c) 400 °C, (d) 450 °C, (e) 500 °C and (f) 550 °C

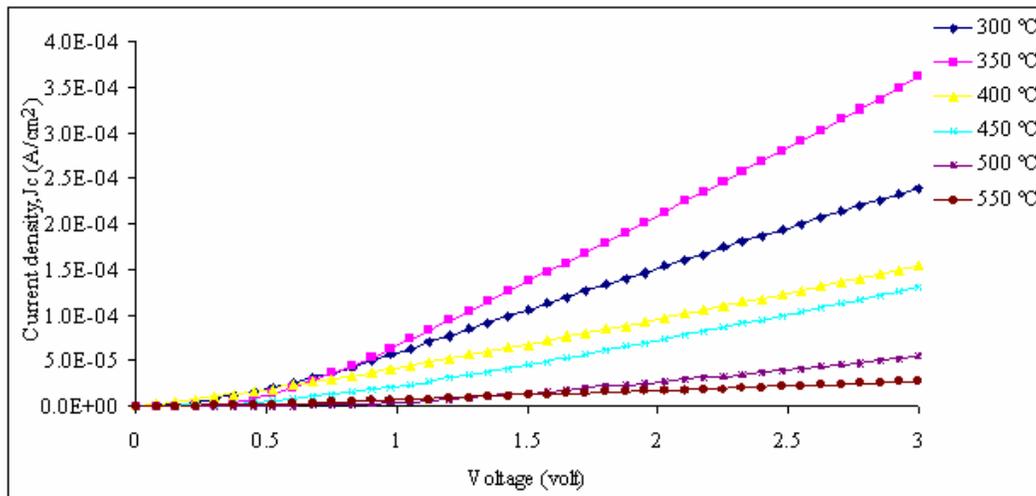


Figure 5: The current density, ( $J_c$ ) of the dielectric bolometer Ba<sub>0.6</sub>Sr<sub>0.4</sub>TiO<sub>3</sub>

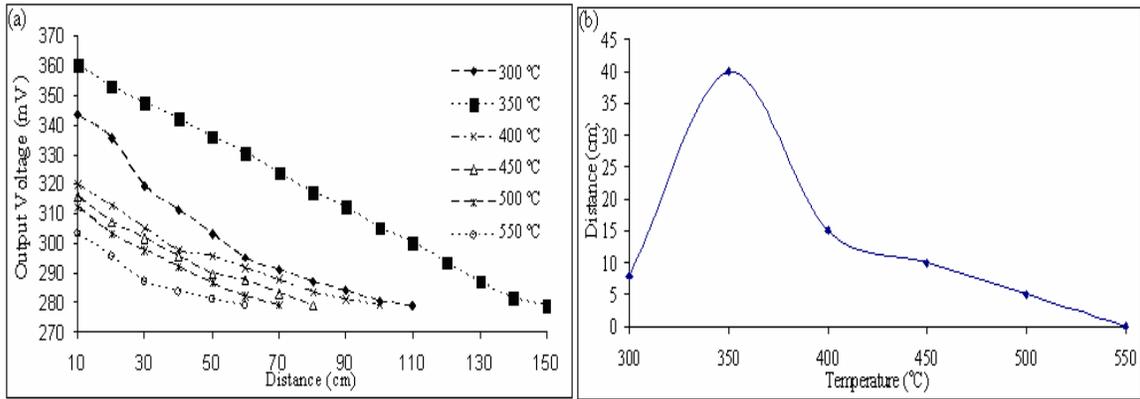


Figure 6: (a) Voltage change versus distance, (b) The maximum distance which can be detected by sensor without IR source.

For the measurements of the sensitivity (Figure 6) and repeatability (Figure 7), the sensors were moved in a straight line with the respect to the IR source. Figure 6a shows the results for the sensitivity of all sensors. Above 350 °C, the sensitivity of all sensors decreased with the increment of the distance and annealing temperature. Our results also show that without IR LED driver (IR source) all sensors except the sensor with TiO<sub>2</sub> annealed at 550 °C could detect the distance (see in Figure 6b) meaning that they can be used as active sensors. As shown in the Figure 6b, beyond 350 °C, the response of all of the sensors without IR source decreased with the increment of the TiO<sub>2</sub> annealing temperature. The summary of the results are listed in Table 1.

To check the repeatability of all of the sensors the experiment was repeated up to four cycles. As shown in the Figure 7, the sensor with TiO<sub>2</sub> thin films annealed at 300 and 350 °C has better repeatability than others. These results show that all the sensitivity and repeatability of the dielectric bolometer are influenced by the crystallinity and grain size of the buffer layers ie. TiO<sub>2</sub>.

Table 1: The sensitivity, repeatability and current density of the BST sensor and average grain size of the TiO<sub>2</sub> thin films.

TiO <sub>2</sub> (°C)	Maximum respond distance with IR source (± 2 cm)	Maximum respond distance without IR source (± 2 cm)	Repeatability with IR source	Current density, J <sub>c</sub> at 3 volt. (10 <sup>-4</sup> A.cm <sup>-1</sup> )	Average grain size (±0.1 nm)
300	110	8	Better	2.397	17.0
350	140	40	Better	3.615	18.133
400	100	10	Good	1.541	19.3
450	80	10	Good	1.299	21.933
500	70	5	Good	5.439	24.2
550	50	0	Good	2.718	27.566

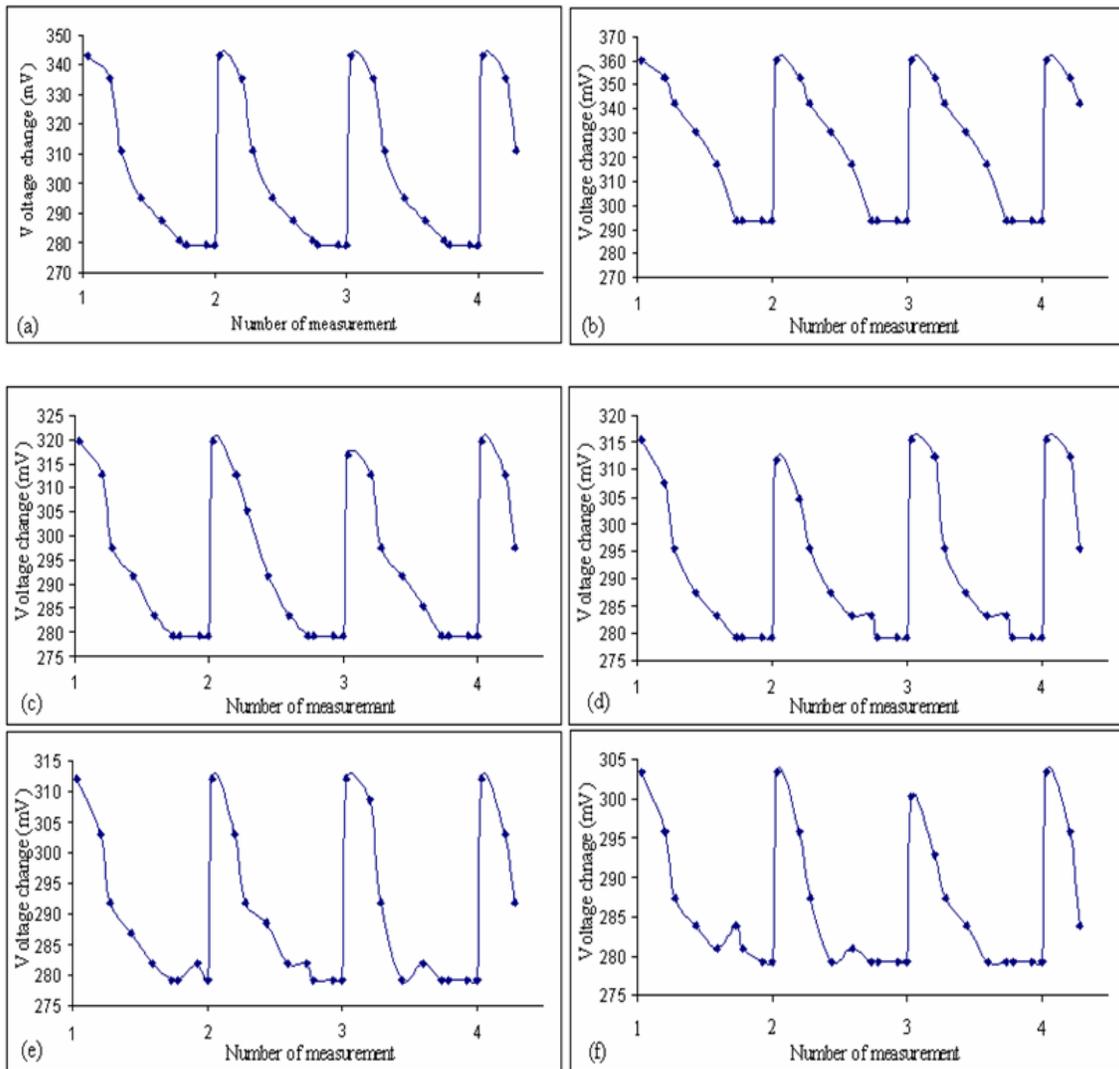


Figure 7 Voltage versus number of measurement for BST sensor with TiO<sub>2</sub> films were annealed at various temperatures for 30 minutes (a) 300 °C, (b) 350 °C, (c) 400 °C, (d) 450 °C, (e) 500 °C and (f) 550 °C (measurement were carried out using IR source).

### CONCLUSION

TiO<sub>2</sub> thin films which act as buffer layers in the dielectric bolometer Ba<sub>0.6</sub>Sr<sub>0.4</sub>TiO<sub>3</sub> have been successfully prepared by using sol-gel process. The TiO<sub>2</sub> films were annealed at various temperatures. The sensitivity, repeatability and current density of the sensors which contain TiO<sub>2</sub> films annealed at 300 and 350 °C increased with the temperature. Opposite results were obtained for the sensors which contain TiO<sub>2</sub> films annealed  $\geq$  400 °C. Our result shows that all of the sensors except sensor with TiO<sub>2</sub> film annealed at 550 °C can act as a sensor even though without the IR source. All the sensitivity, repeatability and current density of the BST sensor are heavily depended on the

microstructure, crystallinity and grain size of TiO<sub>2</sub> thin films. For further study, the dielectric properties of the sensor dielectric bolometer BST without and with TiO<sub>2</sub> buffer layers prepared by sol-gel and electron-gun evaporation techniques are still in progress.

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