Thesis for the Degree of Master of Interdisciplinary Program of
Biomedical Mechanical and Electrical Engineering

Temperature-Insensitive Pressure Measurement Using Field-Induced Oscillation in Vanadium Dioxide Thin Film

by

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Temperature-Insensitive Pressure Measurement Using Field-Induced Oscillation in Vanadium Dioxide Thin Film
(바나듐 이산화물 박막의 전계유도 발진을 이용한온도에 무관한 압력 측정 대한 연구)

Advisor: Prof. Yong-Wook Lee

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VO_2: Vanadium Dioxide

FIO: Field-Induced Oscillation

MIT: Metal Inslator Transition

SPT: Structural Phase Transition

NDR: Negative Differential Resistance

f: Frequency

P: Pressure

A: Amplitude

T: Temperature

Al_2O_3: Aluminum Oxide or Sapphire

PLD: Pulsed Laser Deposition

바나듐 이산화물 박막의 전계유도 발진을 이용한온도에 무관한 압력 측정 대한 연구

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추상

센서부로 사용되어진 VO_2 thin film을 기반으로 하는 두 단자 장치에서 생산되는 FIO를 사용함으로써 온도에 민감하지 않은 압력 측정 장치가 새롭게 제안되었다. 전압 oscillation을 발생시키기 위해 VO2 기반의 장치는 저항이 직렬로 연결되었으며 DC 전압이 가해졌다. 압력이 장치에 가해지면 oscillation 진폭과 주파수는 증가하였고 이 두 값은 온도에도 의존하였다. 이 온도에 대한 cross-sensitivity는 oscillation의 압력과 온도 반응 사이의 차이를 분명히 바로잡아서 극복할 수 있었다. 측정된 압력 민감도는 0–9 MPa에서 ~93 kHz/MPa로 R-square 값은 0.9965 였고, 온도 민감도는 0–50도 사이에서 ~18 kHz/°C로 R-square 값은 0.9952 였다. 그러나 50 °C 이상의 온도에서는 oscillation을 관찰할 수 없었다.

Study on Temperature-Insensitive Pressure Measurement Using Field-Induced Oscillation
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Abstract

A temperature-insensitive pressure measurement by incorporating the field-induced oscillation (FIO) created in a two-terminal device based on a vanadium dioxide (VO_2) thin film, which was employed as a sensing head is newly proposed. A DC voltage was applied to the VO_2–based device serially connected with a resistor to generate voltage oscillation across the resistor. The oscillation amplitude and frequency increased with pressure applied to the device but also depended on temperature. This cross-sensitivity to temperature could be overcome by harnessing clear difference between pressure and temperature responses of the oscillation parameters. The measured pressure sensitivity was ~93 kHz/MPa over 0–9 MPa and temperature sensitivity was ~18 kHz/°C over 0–50 °C with R-square value of 0.9965 and 0.9952 respectively. However, we observed no oscillation when the temperature increases above 50 °C.

1. Introduction

Vanadium dioxide (VO_2) is a representative strongly correlated material showing a reversible phase transition (PT) between an insulating state and a metallic state near 340 K.1) It is well known that this PT can be induced by temperature,1,2) pressure,3) light,4) electric field,5) etc. By harnessing the filed-induced PT of a VO_2 thin film, a variety of switching applications have been explored which includes electrical switches,5,6) varistors,7) THz metamaterials,8) among others. In particular, in a two-terminal electrical device based on a VO_2 thin film, field-induced oscillation (FIO) was realized by using negative differential resistance (NDR)9) leading to the steep increase of electrical current flowing through the device.10-12)

The two terminal device based on VO_2 device has been used in measuring various physical quantities like temperature13-17), pressure18), bending19-20), stress point21-22), etc. In many areas especially, pressure sensor can be used, a good example of this is an electrical measurements of a VO_2 thin film under a high pressure of 25 GPa which has been reported using a load-controllable point-contact structure as a method, thereby attracting much interest for device application. However the load-controllable point-contact system is one simple and usable tool for studying the physical properties of VO_2 thin film under high pressure. Furthermore, to measure the same physical properties of VO_2 thin film for low pressure at 10 kHz, another method needs to be implemented however until this present, no report on temperature-insensitive pressure measurement using FIO in VO_2 thin film has been found. Then considering the unique electro-mechanical properties of the FIO in VO_2 thin film, an electric FIO-based pressure sensor is expected to be a good sensor with acceptable sensitivity.

Here, we show the temperature–insensitive pressure measurement by incorporating the FIO created in a two-terminal device based on a VO_2 thin film. We demonstrated how the FIO method can be utilized for the temperature-insensitive pressure measurements on the electrical properties of VO_2 thin film. Firstly, a DC voltage was applied to the VO_2 thin film device with a serially connected resistor to generate oscillation across the resistor. The oscillation frequency and amplitude increased with pressure being applied to the sensor head. Secondly, considering the sensor head under increased temperature, the oscillation frequency increases while the oscillation amplitude decreases. This cross-sensitivity to temperature could be overcome by harnessing clear difference between pressure and temperature responses of the oscillation frequency and amplitude. The measured pressure sensitivity was ~93 kHz/MPa over 0–9 MPa with R-square value of 0.9965 and under the effect of temperature, the sensitivity was ~18 kHz/°C, over 0−50 °C with R-square value of 0.9952. However, we observed no oscillation when the temperature increases above 50 °C. To the best of our knowledge, there has been no report on a temperature-insensitive pressure sensor based on the FIO in VO_2.

2. Fabrication Process

Over the time, various deposition techniques have been employed to grow high quality VO_2 thin films, such as magnetron sputtering or reactive ion beam sputtering, and pulsed laser deposition technique$^{23-25)}$. For the growth of the VO_2 thin film, the sensor heads were grown on Al_2O_3 substrates by employing pulsed laser deposition (PLD) methods with gold (Au) interdigitated electrodes pre-patterned. The average film thickness of the grown films was measured as ~100 nm. The grown film was etched to form current channels by ion beam milling, and Au electrodes were deposited on the etched film by using photolithographic technique.

Figure1. (a) Shows an optical microscope plane view of fabricated VO_2 oscillation device and a schematic of an experimental setup to measure the current – Voltage (I-V) characteristic of the device. The electrode separation length (L) and the exposed film width (W) of the fabricated device were 3 and 5 μm respectively.

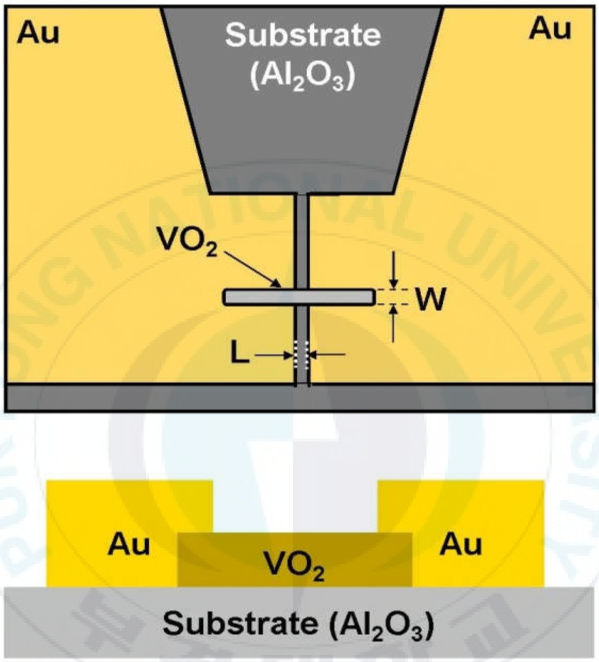

Fig. 1. (a) Plane-view optical microscope image of fabricated VO2 oscillation

For the measurement, we made use of microprobe station (micromanipulator) with tungsten probes and parameter analyzer (HP 4156C). The ohmic contact resistance of the device was <5Ω, which was < 0.05% compared with the resistance across the device at room temperature. The *I-V* properties of the fabricated VO_2-based on two-terminal device measured with constant applied voltage (V-mode) and current (I-mode) swept by the parameter analyzer, respectively. Figures 1 (b) and (c) shows the *I-V* hysteresis loops measured in voltage-controlled mode (V-mode) and current-controlled mode (I-mode) at room temperature and the electrical current flowing through the device was limited to 10 mA to prevent excess current. Thereafter, the threshold voltage and current of the device, i.e., limiting factors defining an oscillation window beyond which the FIO could not be generated, were measured as ~5.0 V and ~2.60 mA in figures 1 (a) and 1 (b), respectively.

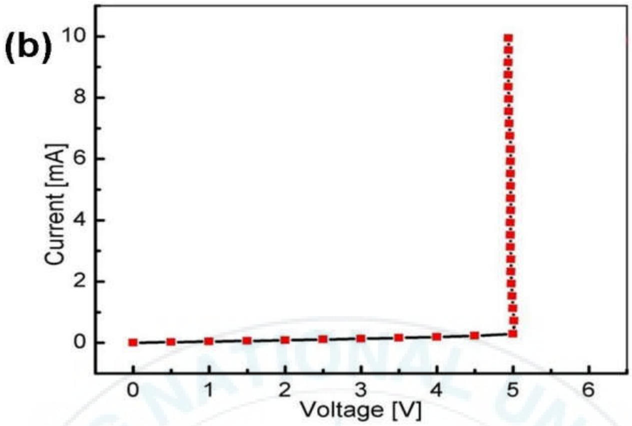

Fig. 1. (b) I-V hysteresis loops measured in fabricated VO_2 thin film with V-mode

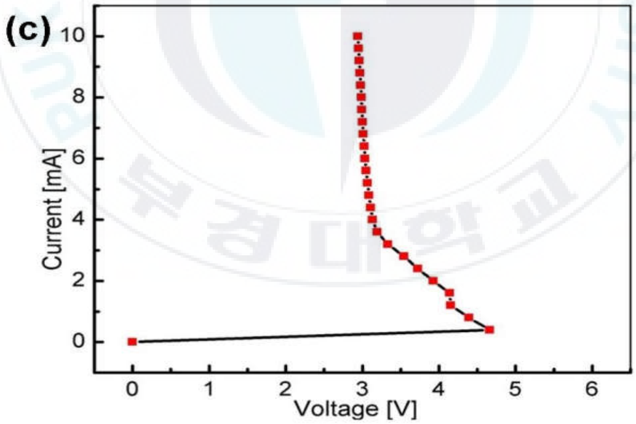

Fig. 1. (c) I-V hysteresis loops measured in fabricated VO_2 thin film with I-mode.

Since it is difficult to directly measure the pressure by applying force directly onto the VO_2-based device, it is better and easier to use a pressure chamber to quantify a required pressure in the range we want for our experiment. One should also be careful whether the output electrical oscillation that we observe on oscilloscope changes or not when we increase the applied pressure.

Figure 2 shows the schematic diagram of the experimental setup for realizing the measuring applied pressure by oscillation waveform observation. In order to measure the temperature-insensitive pressure using FIO in VO_2 thin film, the schematic diagram was built up as shown in figure 2. The sensor head was placed inside the chamber, closed hermetically while the cables were connected to the two ends of the Au. Interdigital electrodes of the device were set outside the chamber.

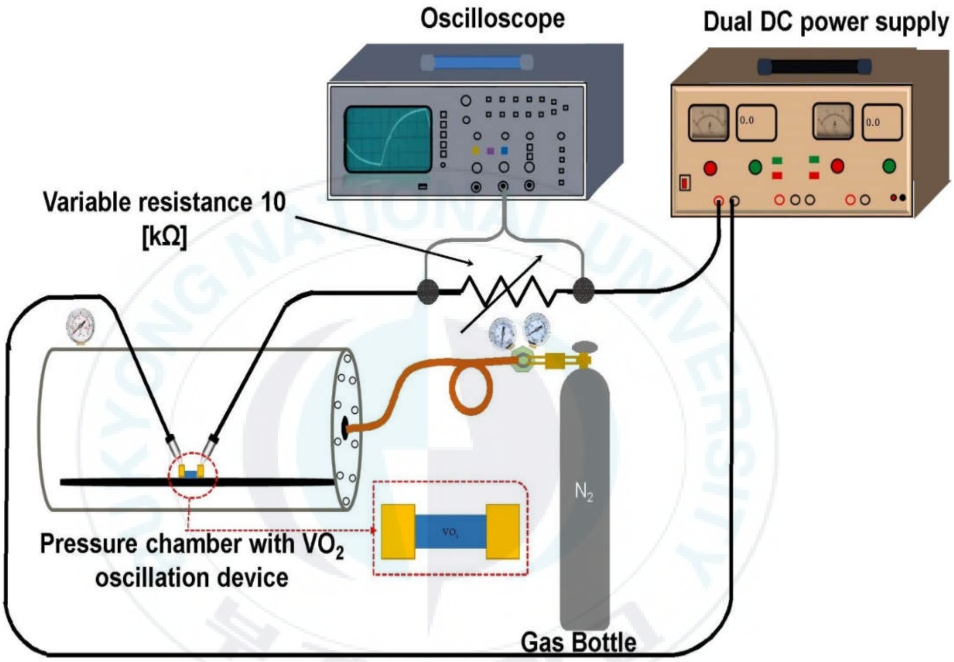

Fig. 2. Schematic chart of experimental setup to measure applied pressure by observing oscillation waveforms.

Before the test, we connected to the right combination these cables with other equipment as standard resistor of resistance R = 10 KΩ, a dual DC power supply (model: HC 2330AD) and an Oscilloscope (Tektronix TDS 2022C). Earlier applying pressure into the chamber, we ensured the air inside was set loose to the outside via the outlet valve. At standard atmospheric pressure, the oscillation curve was found by varying the voltage and resistance to the point of getting oscillation which is viewed via the oscilloscope. Then, the manufacturing process of sensing head and detection of pressure are explained following these three (3) steps: 1) The VO_2 oscillation device was introduced into the chamber and closed hermetically. Two cables were connected on both Au electrode of the VO_2 device. The cables were set outside of the chamber and make a right connection to the other equipment's needed for our experiment. 2) The output signal of an oscilloscope was obtained at the point of getting oscillating wave forms without pressure. 3) The nitrogen (N_2) gas cylinder was operated to apply pressure inside the chamber, and the oscillation curve began to change, the period (T) and amplitude (A) observed on the figure changes. We started to measure the signal at different pressure in the range of 0 to 9 MPa.

It is know that the VO_2, being a monoclinic (MoO_2 type) structured semiconductor at room temperature, has a structural phase transition temperature of 340 K, above which it becomes tetragonal (rutile type) structured with metallic conductivity. An electrical measurement of a VO_2 thin film under hog pressure of 25 GPa, has been reported using the load-controllable point-contact system, attracting much interest for the device. In this research, the sensor head present inside the chamber is subjected to a various pressures, then the applied pressure is located not only in one point but it is the same pressure on every point on our device, contrary to the load-controllable point-contact system where the pressure it is high in the localized area, the stress occurs only in this place and will be distributed toward all the direction in the material.

3. Results and discussion

Moving to the experimental results, involving oscillation curve, Figure 3 Shows the oscillatory electrical waveform measured with the VO_2 oscillation device. In this figure, we noticed two parameters such as amplitude and period. As indicated on the figure, the amplitude is the height from the center line to the peak and the period goes from one peak to the next (or from any point to the next matching point). A 38 μs electrical pulse with a peak-to-amplitudes of the oscillation waveforms were measure at ~5.2 Volts. Considering one period of the oscillation, we determined the frequency that is how often something happens per unit of time. So, $T = 1/f$ or $f = 1/T$. If the limiting factors as voltage and current are varied by certain external stimuli like pressure, temperature, etc., the oscillation curve can be switching between the appearance and disappearance of the oscillation curve, and oscillation properties such as frequency and amplitudes can also be changed. Therefore, the oscillation properties can be modified by external control parameters.

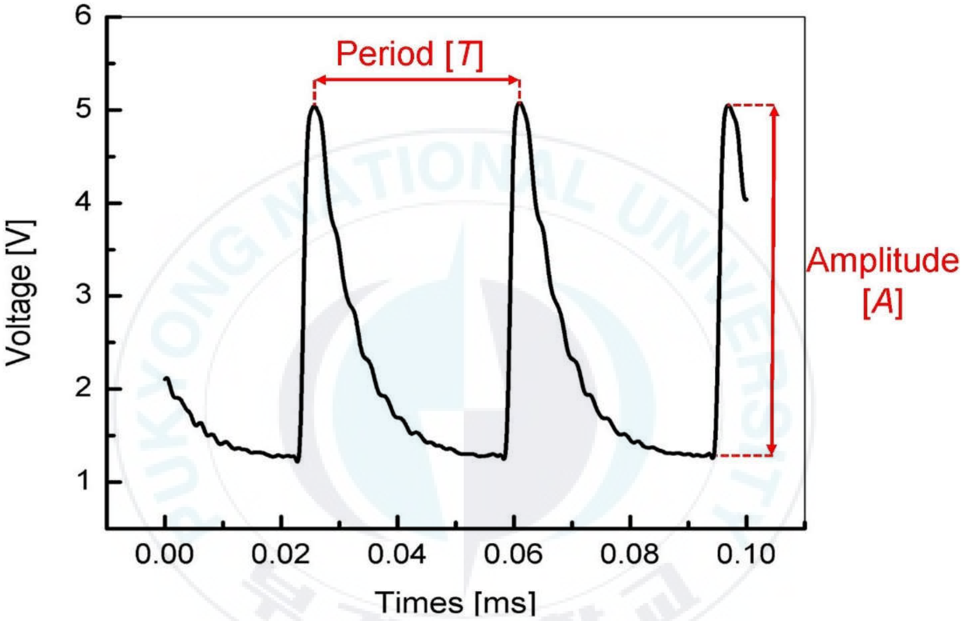

Fig. 3. Oscillatory electrical waveform obtained by measure on VO_2 Oscillation

Another experiment was conducted with the same VO_2 oscillation devices to show the clear difference between temperature and pressure response of the oscillation parameters. Figure 4 shows a test electrical circuit used to investigate the temperature response of the FIO. For this, the simple schematic diagram of an electrical circuit for generating the MIT oscillation and controlling the frequency was constructed. The single-loop circuit for the varied temperature is composed of an oscilloscope (Tektronix TDS 2022C) that recoded the oscillatory electrical responses, with a standard resistor of resistance equal to 10kΩ which was connected in series with the two-terminal of VO_2 thin film. In order to apply rectangular voltage pulse, a dual DC power supply (model: HC 2330AD) was utilized.

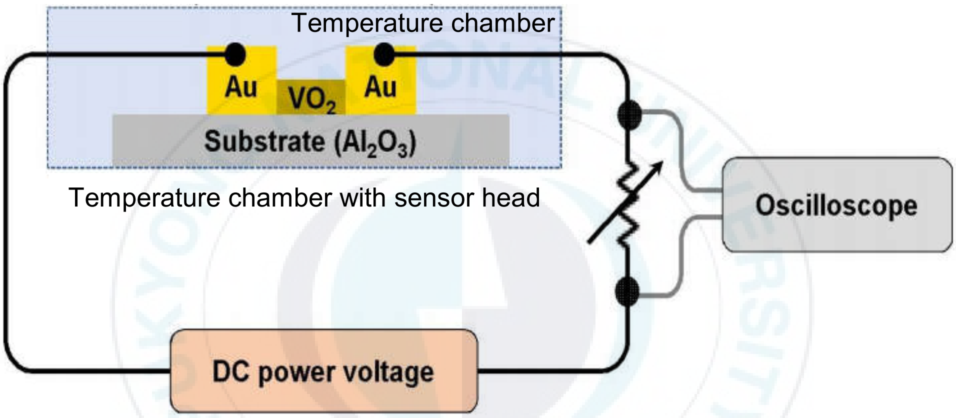

Fig. 4. The test electrical circuit used to examine the temperature sensor response on the VO_2 oscillation.

The temperature was experimentally investigated with respect to fabricated VO_2 thin film devices. The process of sensing head and detection of temperature can be described as thus; the sensor head was put on a hot plate in accordance to the right connection between all the equipment and both terminal cables which was connected to the two electrodes of VO_2 thin film. Firstly, without temperature, we obtained the oscillation by varying the voltage and resistance. When the oscillation appears, we applied the temperature which was increased in the range of 0 °C to 55 °C .The changes on the oscillation curve was observed as the temperature increases, the period become short and the amplitude decrease as shown in figures 5 (b) and (d). This is because, the sensor head is rapidly heated up with the increasing temperature, and the insulating VO_2 grains in the device whose temperature exceeds the critical temperature (~68 °C) are changed into the metallic VO_2 grains through the photo-thermally induced PT. Even at low voltage, the field-induced PT can readily occur, and the device current can increase rapidly due to the considerable reduction of the device resistance, induced by this PT. This is the same phenomena that we observed on photo-assisted electrical gating26), where the increase of the temperature decreases the threshold voltage.

The presence of MIT independent of any SPT was experimentally demonstrated by many experiments such as infrared transmission and reflectivity for $V_{1-x}Cr_xO_2$ under high pressure, Raman scattering with temperature, careful study of coherent phonons, micro-X-ray diffraction, programmable critical-temperature sensor, Raman-scattering under high pressure, electron diffraction, and strain-induced MIT in single-domain nanobeams. A 0 °C, we observed an oscillation with 0.2 MHz of oscillation frequency corresponding to the period of oscillation equal to ~38 µs and is determined by the thermal inertia of VO_2-based device, threshold temperatures and latent heat of the phase transition. When the temperature increase inside the chamber, the VO_2 thin film change from insulator to metallic state, the resistivity decrease and the period of oscillation decreases, means that the frequency increased proportionally with increasing temperature. The temperature oscillations are accompanied by periodical switches between insulator and metallic states of VO_2.

Then from figure 3, with the relation between the frequency and period that we spoke about previously, we determined the close approach between the pressure and temperature based on their parameters. The measured parameters frequency shift (Δf) and amplitude variation (ΔA) can be known by the matrix equation (1)

$$\begin{bmatrix} \Delta f \\ \Delta A \end{bmatrix} = \begin{bmatrix} a & b \\ c & d \end{bmatrix} \begin{bmatrix} \Delta P \\ \Delta T \end{bmatrix} \quad (1)$$

Where *a* and *c* are the pressure sensitivities of the oscillation frequency and amplitude respectively, *b* and *d* are their thermal sensitivities. With some calculations, e.g. to obtain the frequency value, equation (2) below can be used.

$$\Delta f = a\Delta P + b\Delta T \ , \quad (2)$$

Then by identification, the exact value of a, b, c, and d can be obtained by these processes:

$$\frac{\Delta f}{\Delta P}\Big|_{\Delta T \to 0} = a; \frac{\Delta f}{\Delta T}\Big|_{\Delta P \to 0} = b; \frac{\Delta A}{\Delta P}\Big|_{\Delta T \to 0} = c; \frac{\Delta A}{\Delta T}\Big|_{\Delta P \to 0} = d; \quad (3)$$

The value for the different slope curve is as follows; *a = 92.95 kHz/MPa; b = 0.46 kHz/*°C*; c = 18.18 V/MPa; d = -0.03 V/*°C.

Where ΔF = frequency shift; ΔP = pressure variation; ΔA= amplitude variation; and ΔT= temperature variation.

As shown, the changes between the time and amplitude for temperature and pressure response can be investigated by comparison. For pressure sensor response, the range was between 0 – 9 MPa while for temperature sensor response, the range was between 0 – 50 °C. For our investigation, we only took two of oscillatory response curves for each pressure and temperature sensor response with respect to different range.

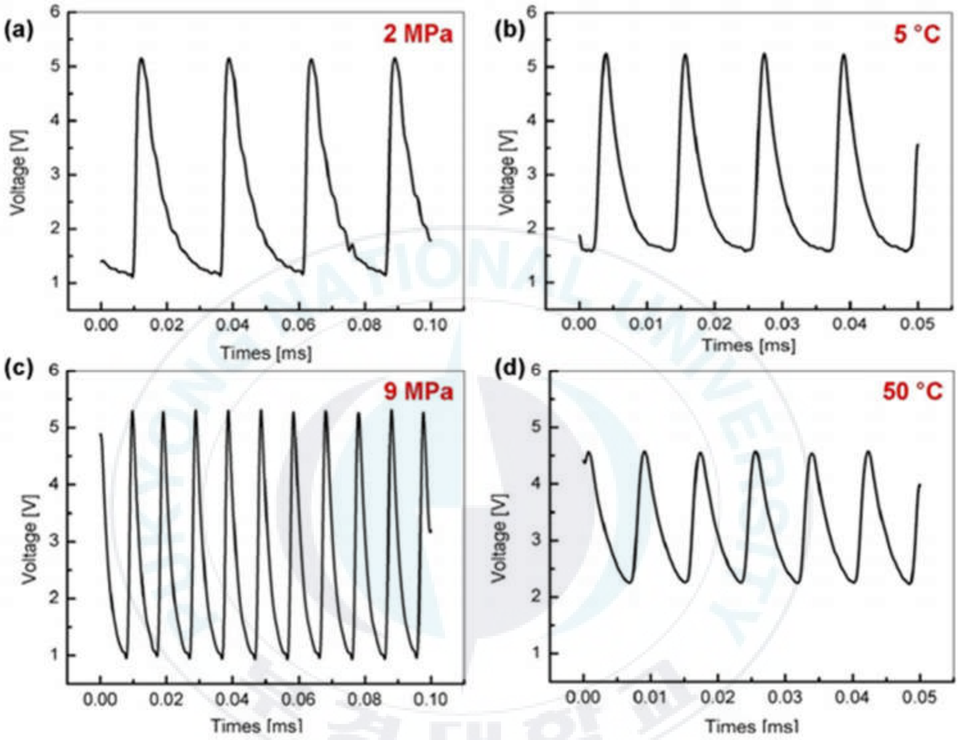

Fig. 5. The oscillatory electrical waveform measured with the sensor head by applying pressure in the range of (a) 0 MPa and (b) 9MPa, and temperature range (c) 5 °C and (d) 50 °C.

Figures 5 (a) and 5 (b) shows the oscillatory electrical waveform measured with the sensor head by applying pressure in the range of 2 MPa and 9 MPa, respectively. When the applied pressure increases, the amplitude also increases and the period becomes short. Using the relation which states that the frequency is the number of occurrences of a repeating event per unit of time, we deduced that when the period becomes short, the frequency increases with increasing pressure according to these figures 5 (a) and (b). The frequency had been observed to increase with voltage and the period under pressure was shorter than that at standard atmospheric pressure, and the corresponding sensitivity (S) of the pressure measurement in our experiment was ~93 kHz/MPa. This sensitivity indicates that there are current changes significantly with the pressure, and there is a potential to develop a new class of stable and sensitive vacuum pressure sensor based on VO_2 thin film. Equation (4) below was derived with the position x as a function of time.

$$x(t) = A\cos\left(\frac{2\pi f}{T}\right) = A\cos\left(2\pi f\right), \tag{4}$$

The value of *f* increases when the value of *A* increases too according to the relation between the frequency and Amplitude shown in equation (4). Likewise when the pressure is high, the amplitude is thus high and the period reduces. The amplitude slowly increased with varying applied pressure of 5.20, 5.25, 5.28, 5.37, 5.42, 5.5, 5.60, and 5.6 Volts for 0, 2, 3, 5, 6, 7, 8, and 9 MPa, respectively at a specific threshold voltage of ~7.4 volts. The period under temperature was shorter than that at room temperature, and the corresponding sensitivity (S) of the temperature measurement in our experiment was ~18 kHz/°C. On the other hand, the temperature range presented in fig. 5 (c) at 5 °C and fig. 5 (d) at 50 °C shows the oscillation electrical waveform measured with the same sensor head than pressure by applied temperature. This second experiment was carried out without pressure but only with applied temperature. Here, we observe that with a very low temperature (5 °C), the amplitude start to decrease when the applied temperature increases, indicating that our sensor head reacts with the temperature. The amplitude reaches ~3.4 V with respect to a temperature variation of 50 °C. Then according to the same equation (4), we can conclude that the higher the applied temperature, the amplitude becomes lower and the period becomes short which invariably means that the frequency increases.

In order to measure the transient response of the pressure and temperature through the sensor head, both test circuit discussed earlier was constructed and tested. The device current was calculated from voltage measured across the resistor with an oscilloscope using Ohm’s law.

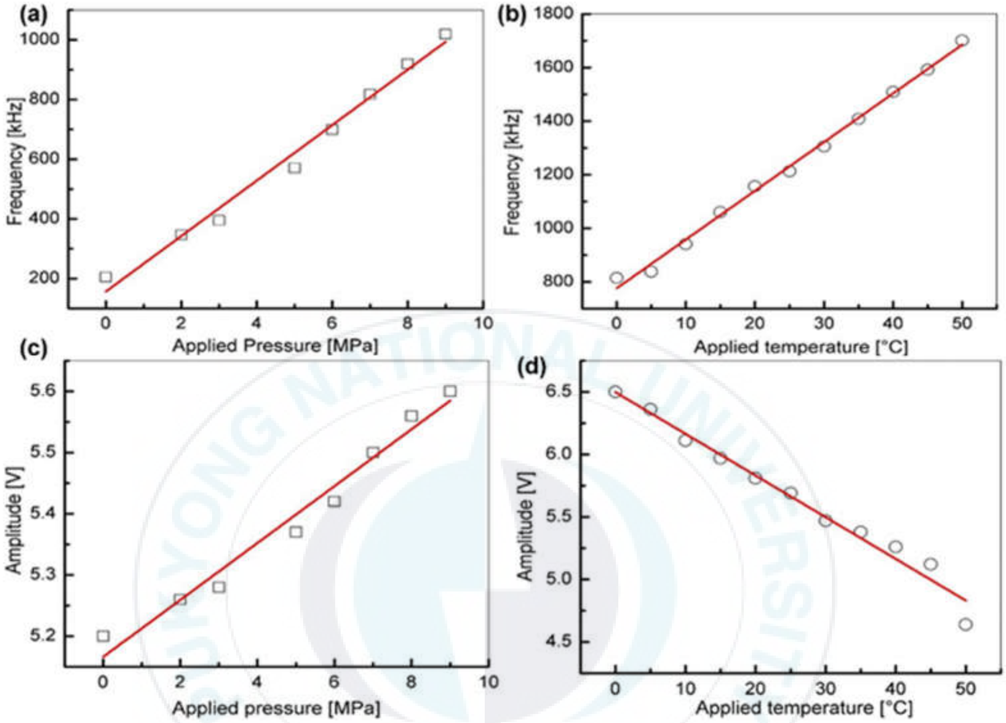

Fig. 6. Transient response of pressure and temperature sensor, the frequency and amplitude oscillation response curve were made to show the different responses of sensor head in different (a) and (b) applied pressure (c) and (d) applied temperature.

Figure 6. (a), (b) and 6. (c), (d) show the transient responses of pressure and temperature of the oscillation parameters, respectively, the frequencies up to 1 MHz and 1.8 MHz and amplitude up to 5.6 V and 6.5 V, with maximum applied pressure and temperature of 9 MPa and 50 °C respectively. The oscillation parameters response curves were made such that they show the different responses of sensor head. On a high pressure, the sensitivity of the oscillation frequency was measured as ~93 kHz/MPa, showing a good linearity with R-squared values of 0.9965 and 0.9687 which correspond to figures 6 (a) and (b) respectively. In the same approach, the turning sensitivity of the oscillation frequency on high temperature was measured as ~18 kHz/°C with R-squared values of 0.9952 and 0.9753 which correspond to figures 6.(c) and (d) respectively.

4. Conclusion

In summary, a temperature-insensitive pressure measurement using FIO in VO_2 thin film was experimentally investigated with respect to fabricating the VO_2 thin film device by PLD method, through applying a DC voltage to the VO_2 thin film of the device linked with the circuit for generating the FIO. With the increase of pressure to the device, the oscillation amplitude and frequency increases with temperature dependent. This was because, with increase temperature to the device, the frequency amplitude still increases however the oscillation amplitude decreases. The measured pressure sensitivity of our sensor head was ~93 kHz/MPa under a high pressure over 0–9 MPa. On the other hand, the measured temperature sensitivity was ~18 kHz/°C under high temperature. Base on the transient responses of our device, we show a good linearity with R-squared value of 0.9965, 0.9687, 0.9952, and 0.9753 for fig. 6 (a), (b), (c), and (d), respectively. We clearly show a close relation between the pressure and temperature sensor base on FIO in a VO_2 thin film using the variation of frequencies and amplitudes oscillation curves. We notice that the temperature applied to the device cannot exceed 50 ºC (>50 ºC), i.e. above this value we cannot obtain any oscillation. The applications of VO_2 oscillation are many and it is a good material for sensing pressure and detecting temperature which seems to be promising. However, more work is still needed.

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