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INVESTIGATION OF INTERNAL THERMOELASTIC STRESSES IN TIO_2 FILM ON SAPPHIRE SUBSTRATE

Yu. V. Klunnikova
Southern Federal University, 347928, Taganrog, Nekrasovsky, 44, Russia

S. P. Malyukov
Southern Federal University, 347928, Taganrog, Nekrasovsky, 44, Russia

A. V. Sayenko
Southern Federal University, 347928, Taganrog, Nekrasovsky, 44, Russia

Yu.N.Biyatenko
Southern Federal University, 344090, Rostov-on-Don, Milchakova 10, Russia

ABSTRACT

We investigate thermoelastic stresses in titanium dioxide films on a sapphire substrate that arise during thermal annealing. The effect of thermal processes on thermoelastic stresses in titanium dioxide films has been studied experimentally. The obtained experimental results are in good agreement with the theoretical calculations of mechanical stresses in a titanium dioxide film.

Keywords: Devices, Film, Sapphire Substrate, Thermal Annealing, Thermoelastic Stresses.

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1. INTRODUCTION

The possibility of reception thin films on dielectric and semiconductor substrates gives wide opportunities for the design of functional devices (photoelectric converters and sensitive elements of gas sensors). Nowadays the interest in studying the properties of thin films has increased in micro- and nanoelectronics. There are the most widely spread gas sensitive materials such as oxides of titanium (TiO_2), tin (SnO_2), tungsten (WO_3), zinc (ZnO), indium (In_2O_3), copper (Cu_2O, CuO), iron (Fe_2O_3), their combinations and others. One of the important features of gas sensors based on titanium dioxide (TiO_2) is the possibility of their operation at high temperatures due to the film high chemical stability [1-4].

2. METHOD OF TITANIUM DIOXIDE FILMS FORMATION

The formation of titanium dioxide films on sapphire substrate is accompanied by the appearance of thermoelastic stresses due to the difference in thermal expansion coefficients of the film and substrate, which can cause destruction of the film and affect the operation of the devices functional elements based on this film.

The purpose of this work is to study the formation of thin films of titanium dioxide and to estimate the level of thermoelastic stresses arising in the structures of titanium dioxide (TiO_2) film – sapphire substrate. The mechanisms of the film structures formation play an important role in the processes of the appearance and relaxation of internal thermoelastic stresses.

The centrifugation method and then annealing in a muffle furnace were used for titanium dioxide film formation on sapphire substrate [3-4]. We clean sapphire substrate in an ultrasonic bath for 15 minutes in water and then in isopropanol. The titanium dioxide layer was deposited from 0.3 M titanium diisopropoxidebis (acetylacetonate) (75 wt. % in isopropanol) in 1-butanol (99.8 %, Sigma-Aldrich) on sapphire substrate with 0.43 mm thick by centrifugation (SPIN NXG-P1 centrifuge, rotor speed 4000 rpm, deposition time 40 seconds). Titanium dioxide film drying was carried out in furnace at 125 ^{0}C for 5 minutes and then annealed at 500 ^{0}C in muffle furnace for 30 minutes. Figure 1 shows the technological process of film annealing in a muffle furnace (heating and cooling). The titanium dioxide film on sapphire substrate was held at 200 ^{0}C for 10 minutes.

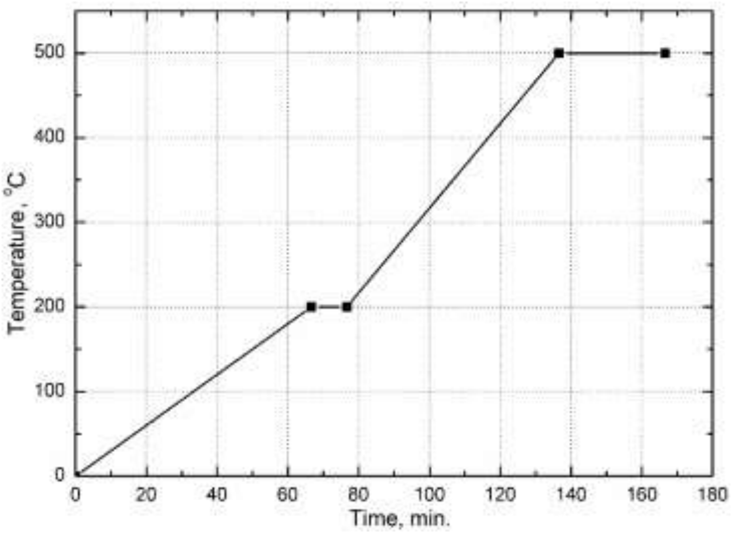
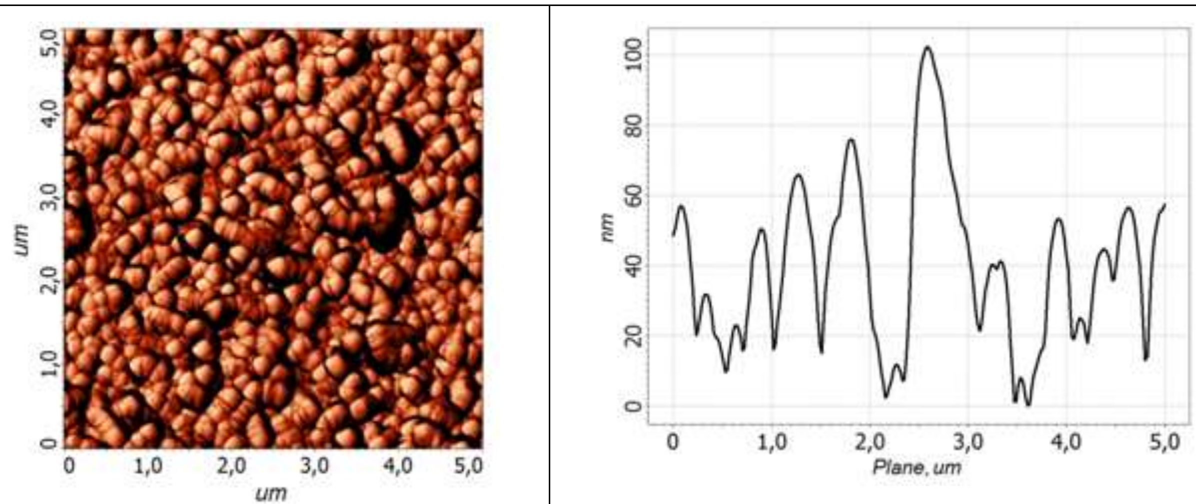


Figure 1 Technological process of titanium dioxide film annealing in a muffle furnace

The use of sapphire substrate allows further annealing of the gas sensitive material, because the sapphire has the high adhesion strength to the gas sensitive material, the high melting point, chemical and radiation resistance, high hardness and transparency, which leads to improved quality and stability of the gas sensitive material [5-8].

3. INVESTIGATION OF TITANIUM DIOXIDE FILMS PROPERTIES

The obtained films of titanium dioxide on sapphire substrate were investigated by atomic force microscopy (AFM). The statistical processing of AFM data was performed using a software package Image Analysis 3.5. The results of the studies (Figure 2) showed that the obtained titanium dioxide films on sapphire substrate are uniform, while the crystal diameter values are in the range up to 200 ± 250 nm.



A

B

Figure 2AFM image (phase contrast) of titanium dioxide film on sapphire substrate

We carried out the experimental studies to measure thermal stresses in thin TiO_2 films on the Tencor FLX-2320 (Japan) in the laboratories of «Piezopribor» research center (Russia, Rostov-on-Don). The basis of this study is the technique for measuring the surface stresses of thin films by estimating the change in the radius of curvature of the substrate caused by the influence of a thin film formed on it. The deflection of titanium dioxide thin film on sapphire substrate is shown in Figure 3. The calculation results of thermoelastic stresses obtained from these studies are shown in Figure 4. 3D map of thermoelastic stresses of titanium dioxide thin film on sapphire substrate is presented in Figure 5.

Thermoelastic stresses depend on the parameters and conditions of the film formation process (Figure 1). Their value can be varied to the required one by changing various parameters and factors of film formation process (annealing temperature, centrifugation speed and time).

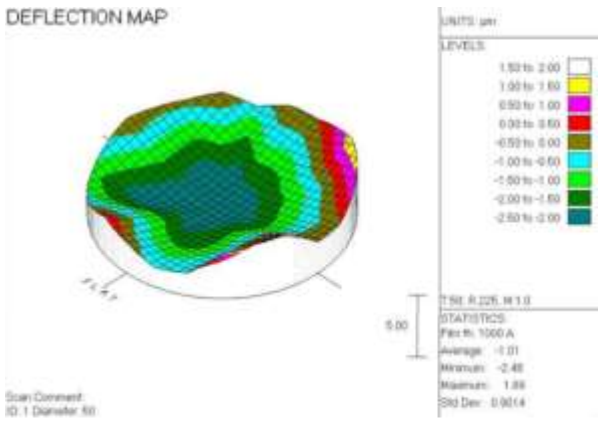


Figure 3 3D map of the deflection of titanium dioxide film with a thickness of about 100 nm on sapphire substrate (0.43 mm thick)

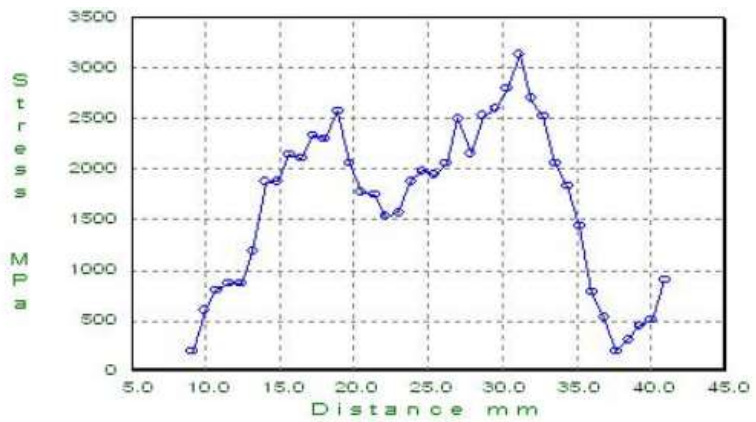


Figure 4 Study of thermoelastic stresses in titanium dioxide films with a thickness about 100 nm on sapphire substrate (0.43 mm thick)

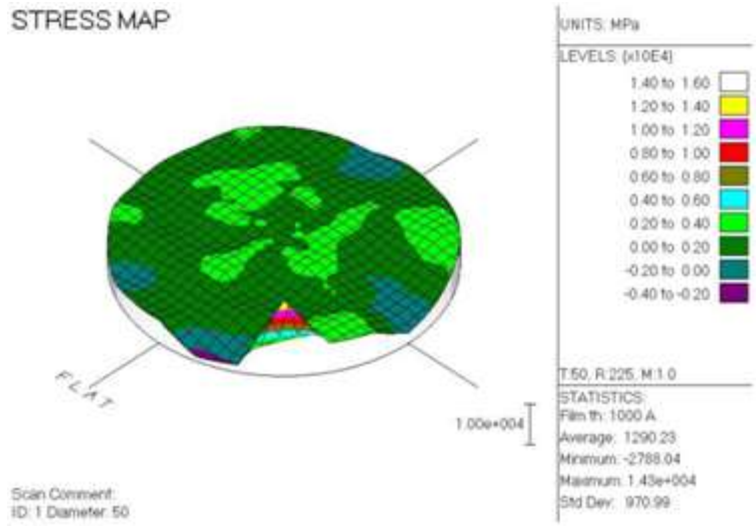


Figure 53D map of thermoelastic stresses of titanium dioxide thin film on sapphire substrate

The temperature coefficient of linear expansion of titanium dioxide films is $7{,}4\cdot10^{-6}$ $1/^{0}C$, which is higher than sapphire crystal one [4]. So the thermal stresses in titanium dioxide on sapphire substrate are tensile.

Investigations of the temperature effect on thermoelastic stresses in titanium dioxide films on sapphire substrate showed that when we heat the structure to 40 ^{0}C, the value of the thermoelastic stress is at the same level (about 1.1 GPa) (Figure 6). A further temperature increase leads to growth of the value of thermoelastic stresses in titanium dioxide films on sapphire substrate, which may have a negative effect on the characteristics of devices functional elements based on these films.

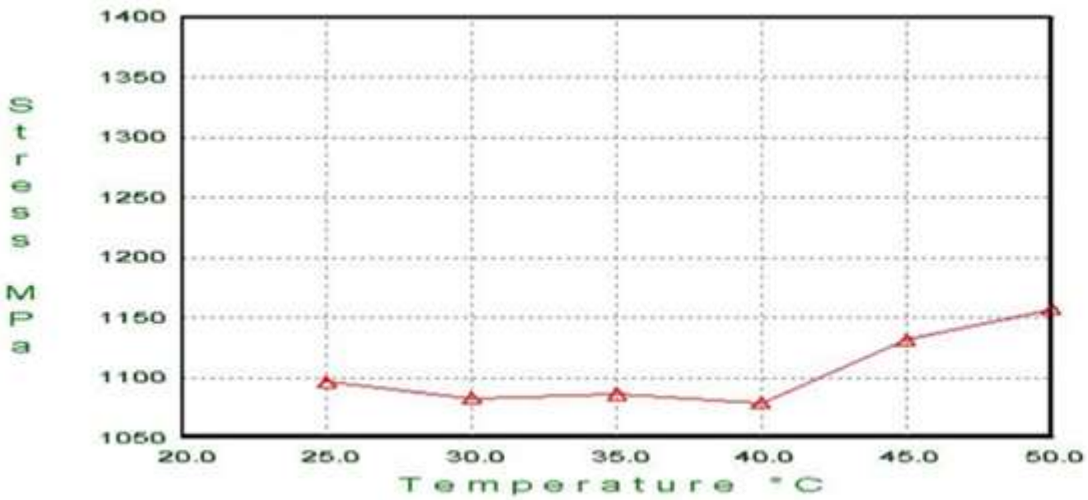


Figure 63D map of thermoelastic stresses of titanium dioxide thin film on sapphire substrate

The resulting thermal stresses σ_T for a biaxial symmetric stressed state are determined by the relation [9]:

$$\sigma_T = \frac{E_f}{1-v_f}\left(a_f - a_s\right) \cdot \Delta T, \qquad (1)$$

whereE_f is the Young's modulus of film; v_f is the Poisson's ratios of film; a_f, a_s are the coefficients of linear expansion of the film and substrate; ΔT is the temperature change.

The calculation results of thermoelastic stresses in titanium dioxide films on sapphire substrate are shown in Figure 7.

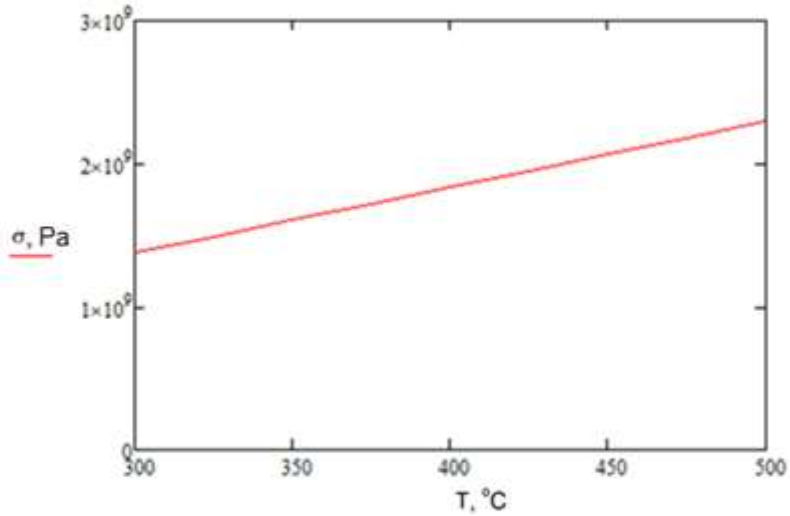


Figure 7 Dependence of thermoelastic stresses on temperatures in titanium dioxide films

The stresses in titanium dioxide films with 100 nm thick on sapphire substrate were about 1 – 3 GPa from the results of measurements and calculations.

4. CONCLUSION

The titanium dioxide thin films on sapphire substrate were obtained by centrifugation using a precursor diisopropoxide titanium bis (acetylacetonate), which can find wide application for gas sensitive sensors and photoelectric converters. We investigate of thermoelastic stresses in thin TiO_2 films on sapphire substrate arising because of the difference in values of film and substrate thermal expansion coefficients. Theoretical and experimental studies have shown that tensile thermal stresses are formed during titanium dioxide film obtaining on sapphire substrate. We study the temperature influence on thermoelastic stresses in titanium dioxide films. It is determined that at a heating temperature of the structure up to 40 ^{0}C the thermoelastic stresses value is at the same level (about 1.1 GPa).

The results of experimental studies of thermoelastic stresses in titanium dioxide films on sapphire substrate accord with theoretical calculations for film thickness about 100 nm.

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