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# THE THERMOELASTIC STRESSES DURING LASER ANNEALING OF TITANIUM DIOXIDE ON A SAPPHIRE SUBSTRATE

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**Abstract.** In order to analyze the technique of laser annealing of titanium dioxide films on sapphire substrates and to optimize the film properties, a thermomechanical model of this technique has been considered. The model allowed us to monitor and vary the values of thermoelastic stresses in the film-substrate structures caused by changes of film annealing technological parameters such as the laser power, the film thickness, the pulse duration, the speed of laser emission, etc. The temperature field under the laser beam was simulated and then the stresses were analyzed using the thermomechanical finite element model. The simulation results showed an important role of the $TiO_2$ film-to-substrate thickness ratio. The optimal combination of technological parameters was selected to prevent formation of cracks and other defects in the films.

**Keywords:** film, thermoelastic property, laser annealing, substrate, numerical method, sapphire substrate

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# ТЕРМОУПРУГИЕ НАПРЯЖЕНИЯ ПРИ ЛАЗЕРНОМ ОТЖИГЕ ДИОКСИДА ТИТАНА НА САПФИРОВОЙ ПОДЛОЖКЕ

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**Аннотация.** С целью анализа технологии лазерного отжига пленок оксида титана на сапфировых подложках и оптимизации свойств пленок рассмотрена термомеханическая модель этого отжига. Она позволяет контролировать и варьировать значения термоупругих напряжений в структурах пленка-подложка, вызванных изменением технологических параметров отжига пленок, таких как мощность лазера, толщина пленки, длительность импульса, скорость лазерного излучения и др. Температурное поле под лазерным лучом моделировали с последующим анализом напряжений с помощью термомеханической модели конечных элементов. Результаты моделирования показали важную роль соотношения между значениями толщины пленки и подложки. Подобрано оптимальное сочетание технологических параметров, позволяющее предотвратить образование в пленках трещин и других дефектов.

**Ключевые слова:** пленка, термоупругие свойства, лазерный отжиг, подложка, численный метод

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## Introduction

The possibility of obtaining thin films on substrates is important for the design of functional devices such as photoelectric converters or sensitive elements of gas sensors. Modern semiconductor gas sensors use thin films of metal oxides ($TiO_2$, $Fe_2O_3$, $V_2O_5$, $SnO_2$, $WO_3$, ZnO) as sensing elements. These materials are popular because of their workability, low cost, high chemical stability, mechanical strength, and high adhesion to sapphire. As the result, semiconductor gas sensors are characterized by small sizes, high sensitivity, and reliability [1, 2].

Traditional technologies of microelectronics, such as vacuum deposition and photolithography, can be used for creating thin films. Application of more sophisticated technologies increases the performance of gas sensor and decreases its cost and power consumption [3].

Laser radiation (LR) for obtaining thin films on sapphire substrates increases the performance of gas element production, provides stability of film parameters and higher oxide quality. Decreased duration of film laser annealing (LA) on sapphire surface excludes the necessity to provide vacuum conditions or special inert atmosphere to prevent uncontrolled surface impurities [4 – 8]. The main benefits of gas sensor development on a sapphire substrate with titanium oxide ($TiO_2$) film are high selectivity to detectable gases, reduced operating temperature, and increased stability over time.

The mismatch of the lattice parameters and the thermal expansion properties between thin film layers and substrate materials is the main cause of the cross-layer defect development and stress generation. Thermoelastic stresses in a thin film often hamper its performance. Therefore, it is important to be able to control the stress formation in thin film.

The Stoney formula [9] is commonly used for estimation of stresses in the thin film systems. This formula has been extended to calculate stresses in multi-layered thin films deposited on a substrate exposed to non-uniform misfit strains, which provides a way to experimentally determine stresses in such systems [10]. Analytical and experimental methods, including the substrate curvature and X-ray methods, are also useful but they cannot provide the stress distribution in thin film systems when manufacturing and across individual film layers.

The numerical simulation is an efficient technique for studying complex systems, including studies in thermoelastic stress analysis of the thin film-substrate system. Multiphysics package as ANSYS is very suitable for such investigations. It allows to perform the thermomechanical analysis for both temperature and stress estimation. The simulation gives the opportunity to analyze the generation and evolution of stresses in thin film structures and processing conditions on the stress distribution. A more comprehensive investigation was carried out by A. Pramanik and L. Zhang [11]; they used anisotropic material properties and a three-dimensional finite element (FE) model to investigate the residual stresses in the thin film and substrate. E. Citirik et al. [12] used the FE analysis to simulate the residual stresses developed in density modulated silicon (Si) thin films, which incorporate alternating low- and high-density layers. H. A. Tinoko et al. [13] showed a methodology to estimate mechanical parameters of thin films by means of a bulge test and a numerical approach. Although many studies in stress evolution during thin film production have been carried out, some questions related to underlying mechanisms remain unanswered, and it remains unknown how the stresses are stipulated by specific material structures and manufacturing conditions, influence of material nonlinearity properties in temperature range, etc.

The goal of our research was to investigate the thermoelastic stresses in the thin $TiO_2$ films on substrates obtained by LA and to analyze occurrence and evolution of thermoelastic stresses and defects in the film-substrate structure and to determine the conditions that prevent cracks formation.

It is necessary to find optimal conditions for manufacturing $TiO_2$ films on sapphire substrates for microelectronics application and to improve not only substrate quality but also films characteristics (i. e., reduce defects, improve oxide quality as well as reproducibility and stability of film parameters over time). The influence of the technological process on thermoelastic stresses in $TiO_2$ films has been studied experimentally and with simulation. We have investigated the effects of material properties, thin film structure and processing conditions on the distribution of stresses.

## Computational approach

The localization of the thermal effect during $TiO_2$ films LA on a sapphire substrate leads to a large temperature gradient in the zone of the laser beam influence, which results in thermomechanical stresses and possible defects. The scheme of LR of a $TiO_2$ film 10 μm thick on the sapphire surface 430 μm thick is presented in Fig. 1.

Numerical methods allow to carry out simulations and determine the optimal parameters of the films' LA. Two sequential steps have to be performed for the thermomechanical stress analysis: (1) a pure thermal analysis which provides temperature and heat flux distributions in space and time, (2) a thermoelastic analysis to compute the mechanical stresses due to the temperature gradients.

To calculate the temperature fields, we used a three-dimensional heat equation:

$$C\rho\frac{\partial T}{\partial t}-\left(\frac{\partial}{\partial x}k\frac{\partial}{\partial x}T+\frac{\partial}{\partial y}k\frac{\partial}{\partial y}T+\frac{\partial}{\partial z}k\frac{\partial}{\partial z}T\right)=q, \tag{1}$$

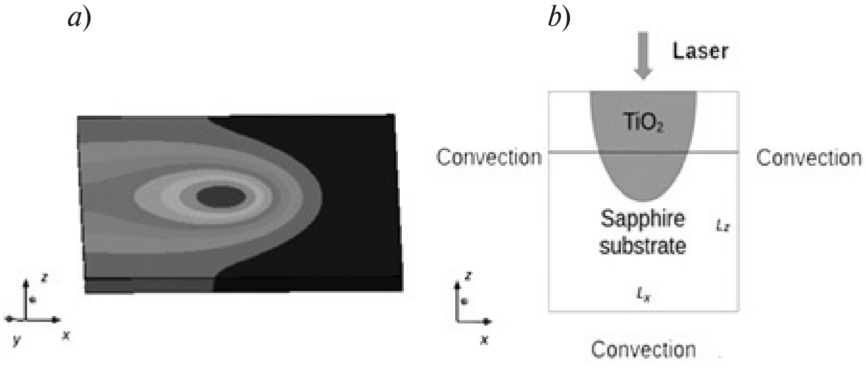

Fig. 1. Scheme of laser annealing of the titanium oxide – sapphire system (*b*) and the temperature distribution in the system's cross-section at time instant *t* (*a*);
$L_x$, $L_z$ are the width and height of the sample, the laser moved from left to right

where $C$ is the heat capacity; $\rho$ is the mass density; $T$ is the temperature; $t$ is the time; $k$ is the thermal conductivity coefficient and $q$ is the power density of the heat source.

Laser energy absorbed by the structure is described by the Bouguer – Lambert – Beer expression

$$q = a(1-R)I_0(t)e^{-az}, \tag{2}$$

where $R$, $a$ are the reflection coefficient and the absorption one, respectively, and $I_0(t)$ is the laser power (LP) density.

The spatial distribution of the laser power density while pulse duration follows a Gaussian distribution, which was approximated using the equation:

$$I_0(t) = I_0 e^{\frac{-(x^2+y^2)}{r}}, \tag{3}$$

where $r$ is the radius of laser beam; $x$, $y$ are the spatial coordinates.

The initial condition for the heat Eq. (1) is $T\,(x, y, z, t = 0) = T_0$.

The boundary conditions are set to simulate convection on all boundaries:

$$-k\frac{\partial T}{\partial n} = \beta(T - T_0),$$

where $\beta$ is the convection heat transfer coefficient.

The Neumann boundary conditions (flux-type) were used on the top surface for the calculation of heat transfer from the film to the substrate. The boundary condition on the top layer can be described as

$$-k\frac{\partial T}{\partial z}\Big| L_z = q_{i,j,k},$$

where $q_{i,j,k}$ is the part of flux in the nodal point $i$, $j$, $k$.

The boundary condition between the layers (with the film thickness $L_1$ and substrate thickness $L_2$) defines the equivalent temperatures in both layers:

$$T_1(z = L_1 - 0, t) = T_2(z = L_1 + 0, t).$$

After laser annealing, the cooling takes place in the air. The laser scan speed was varied for the simulation. Due to large temperature gradients, thermal stresses appear during the heating-up and the cooling-down processes. The material strains can be described as [14]:

$$\varepsilon_{tot} = \varepsilon_{el} + \varepsilon_{th}, \tag{4}$$

where $\varepsilon_{tot}$ is the total strain, $\varepsilon_{el}$ is the elastic strain, $\varepsilon_{th}$ is the thermal strain.

The thermal strain is described by linear temperature dependence:

$$\varepsilon_{th} = \alpha(\Delta T). \tag{5}$$

The elastic stress is determined by the Hooke's law [14]:

$$\sigma = [D]\{\varepsilon_{th}\}. \tag{6}$$

As consequence of Eqs. (4) – (6), the strain-stress relationship for isotropic material can be described as

$$\begin{aligned} \varepsilon_{xx} &= \frac{1}{E}\left(\sigma_{xx} + \nu(\sigma_{yy} + \sigma_{zz})\right) + \alpha(\Delta T), \\ \varepsilon_{yy} &= \frac{1}{E}\left(\sigma_{yy} + \nu(\sigma_{xx} + \sigma_{zz})\right) + \alpha(\Delta T), \end{aligned} \tag{7}$$

$$\varepsilon_{zz} = \frac{1}{E}\left(\sigma_{zz} + \nu(\sigma_{xx} + \sigma_{yy})\right) + \alpha(\Delta T),$$
$$\varepsilon_{yz} = \frac{2(1+\nu)\sigma_{yz}}{E}, \tag{7}$$
$$\varepsilon_{zx} = \frac{2(1+\nu)\sigma_{zx}}{E},$$

where $E$ is the Young's modulus, $\nu$ is the Poisson's ratio, $\alpha$ is the coefficient of linear thermal expansion, $\Delta T$ is the local rise of temperature.

The support on the three adjacent faces is assumed frictionless.

The thermophysical properties adopted for both substrate and film are displayed in Table [2].

Table

**Thermophysical properties of the titanium dioxide and sapphire substrate**

| Parameter | Parameter value | |
|---|---|---|
| | Titanium dioxide | Sapphire |
| Mass density, kg·m$^{-3}$ | 4260 | 4000 |
| Heat capacity, J·kg$^{-1}$·K$^{-1}$ | 690 | 1430 |
| Thermal conductivity, W·m$^{-1}$·K$^{-1}$ | 85 | 5 |
| Coefficient of thermal expansion, K$^{-1}$ | $9.19 \cdot 10^{-6}$ | $8.8 \cdot 10^{-6}$ |
| Young's modulus, GPa | 282 | 350 |
| Poisson's ratio | 0.27 | 0.27 |

Three different thicknesses of the substrates whose sizes are assumed to be 10 × 10 × 0.43 mm, 10 × 10 × 1 mm, 10 × 10 × 1.5 mm have been investigated. The film thickness is varied from 1 to 30 μm.

The simulation parameters are the following: the laser power is 30 – 90 W; the scan speed is 1 – 25 mm/s; the laser scan length is 8 mm; the laser beam radius is 1.25 mm.

The FE package ANSYS was used to perform the thermophysical analysis. The temperature history calculation was used as an input for structural analysis. The transient thermal analysis was performed for the temperature distribution in the $TiO_2$ film on the sapphire substrate.

A challenge arises from meshing the thin layers in order of good element quality. The number of elements used in our ANSYS simulation for the case with substrate thickness 1 mm is 5720 in which 44.5 % are the 10-node SOLID291 elements, 3.93 % is the 20-node SOLID279 element, 18.69 % 8-node contact elements CONTA174 and 32.88 % are thermal surface elements SURF152. The minimum orthogonal quality is 0.04 (more than 0.01), which is the evidence of adequate meshing. The average element surface area is $3.6683 \cdot 10^{-5}$ m$^2$. The mesh density is finer in the laser path trajectory which gives us the opportunity to concentrate the elements in regions with large temperature gradients, obtaining increased computational efficiency.

## Materials and methods

For the experiments a thin film of tetraethoxytitanium $Ti(OC_2H_5)_4$ was brought up onto a sapphire substrate with a thickness of 430 μm by centrifugation (centrifuge SPIN NXG-P1, rotor rotation speed of 2000 – 3000 rpm, application time of 30 s). Sapphire supports to promote high adhesion strength to the gas sensitive material and has a high melting point, high chemical and radiation resistance, high hardness and transparency, which leads to the quality and stability improvement of the gas sensitive material [15, 16].

After pre-drying in the oven at 100 – 120 °C for 15 – 20 min (the solvent and hydrolysis products have been removed from the film before) LA is carried out using the radiation of a pulsed solid-state Nd:YAG laser with a wavelength of 1064 nm (film temperature of 500 – 600 °C, laser beam scanning rate of 1 – 20 mm/s, laser power of 30 – 90 W). With that treatment the crystalline structure is modified and defects are reduced in order to improve the quality and stability of the gas sensitive material. The use of LA makes it possible to shorten the processing time to obtain the gas sensitive material in comparison with traditional methods (annealing in a muffle furnace) [4 – 8].

The phase composition of the thin film structure was investigated by powder X-ray diffractometry [17]. The diffractometer ARLX'TRA, Thermo ARL was used to perform X-ray phase analysis of obtained thin films. A qualitative analysis of the phase composition was performed using an open database (card index) COD (Crystallographhy Open Database) and the Match program. The X-ray roentgenogram of the obtained film, reflexes of the standardized roentgenogram and Miller indices are shown in Fig. 2, *a*. We have chosen the X-ray roentgenogram for titanium oxide with the structure of rutile (card No. 99-207-1134) for reference. It can be seen from the obtained data that the reflexes of the standard sample coincide with the reflexes of the resulting film. Therefore, the material has a phase composition like the rutile modification of titanium oxide. Fig. 2, *b* shows SEM image of the surface morphology of the titanium dioxide film.

We carried out experimental studies to measure thermal stresses in thin $TiO_2$ films on the Tencor FLX-2320 (Japan) in the laboratories of "Piezopribor" Research Center (Rostov-on-Don, Russia).

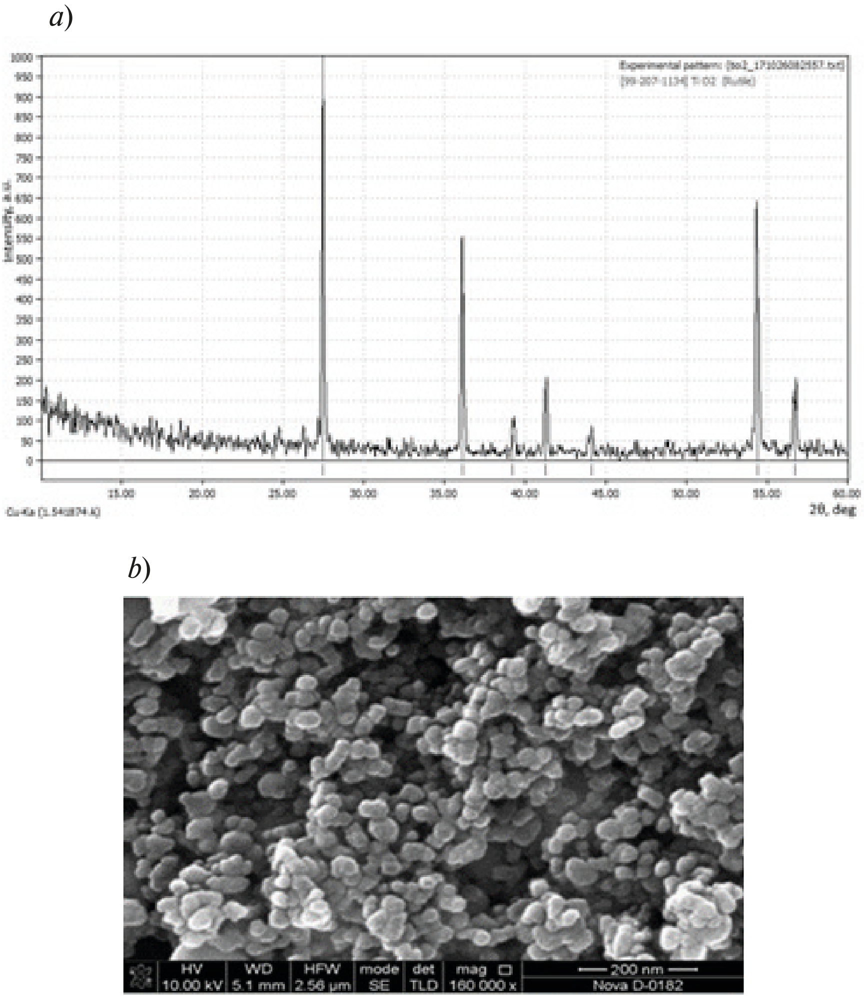

Fig. 2. The X-ray roentgenogram of the $TiO_2$ film obtained by laser annealing (*a*) and the SEM image of the surface morphology of the film on sapphire substrate (*b*); laser wavelength is 1.064 μm

## Results and discussion

Different calculation scenarios for TiO$_2$ film laser annealing on sapphire substrate were considered. The experimental results showed that the temperature for TiO$_2$ film structure formation was about 500 °C [18]. Thermal analyses showed a temperature peak on the film surface.

We calculated the stress distribution in the TiO$_2$ film – sapphire structure caused by LR. The general purpose FE code ANSYS was used to simulate TiO$_2$ film – sapphire LA with variation of laser parameters such as the its speed and power. We investigated the scenarios with different substrate thicknesses (0.43, 1.00, and 1.50 mm). The influence of laser power, substrate thickness and laser beam speed on maximum temperature for TiO$_2$ laser annealing on sapphire substrate is presented in Fig. 3. Increasing the speed of the laser leads to a decrease in maximum temperature on the sample surface. This decrease can be explained by the fact that the heating and cooling require less time and the material gets less energy. The cooling rate significantly influenced the formation of TiO$_2$ film on sapphire substrate.

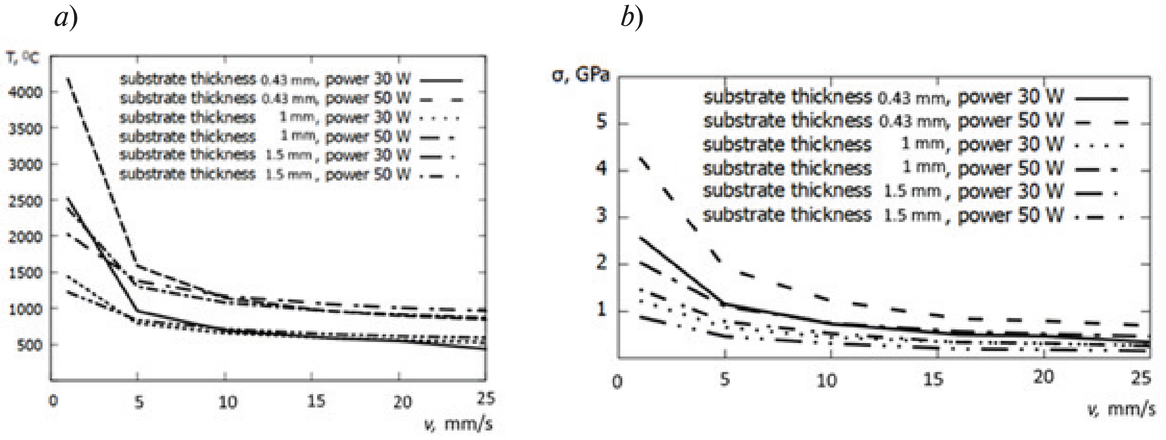
*a*) *b*)

Fig. 3. The dependences of the sapphire substrates' temperature $T$ (*a*) and its stress σ (*b*) on the laser beam speed at different laser power values and the thicknesses of sapphire substrates; the thickness of a TiO$_2$ film is 5 μm

It can be seen from Fig. 3 and 4 that the large temperature gradients are the source of thermal stresses on the film surface. The heated part of material expands but the rest material restrains the movement of heated one. So, we have the compressive region in the under irradiated zone. During the cooling, compressive stresses appeared in irradiated zone, but the rest is in tensile state.

Fig. 4 illustrates the equivalent (von-Mises) stresses during TiO$_2$ film LA on sapphire substrate. The maximum temperature for that case was about 530 °C at a laser scan speed of 25 mm/s, substrate thickness was 1 mm and laser power was 30 W. When the laser beam begins to scan a path (Fig. 4, *a* – *c*), the stresses increase, but the low temperature of the surrounding material restricts the heated zone expansion causing the formation of compressive stresses (equivalent (von-Mises) stress is 1.3 GPa at 0.08 s of LA). When cooling, the temperature decreases and the material is exposed to lower stresses (equivalent (von-Mises) stress is 8.42 MPa at 8 s, the laser treatment time is 0.32 s).

The behavior of stresses in the processed material strongly depends on the temperature gradients. The maximum principal stress (see Figs. 3, 4) indicates the overall stress state of material. When the largest principal stress exceeds the uniaxial tensile strength 30, a crack might be initiated. The tensile strength limit of TiO$_2$ film on sapphire substrate is 333 MPa. If the maximum principal stress is less than the tensile strength limit for titanium dioxide, no cracks formation is expected.

The simulation results indicate that the thickness ratio between the TiO$_2$ film and the sapphire substrate plays an important role in the LA. The capability of the substrate to diffuse the heating from the film enables a proper temperature distribution inside the film, avoiding the overheating of the surface. It has significant influence on defects like cracks formation.

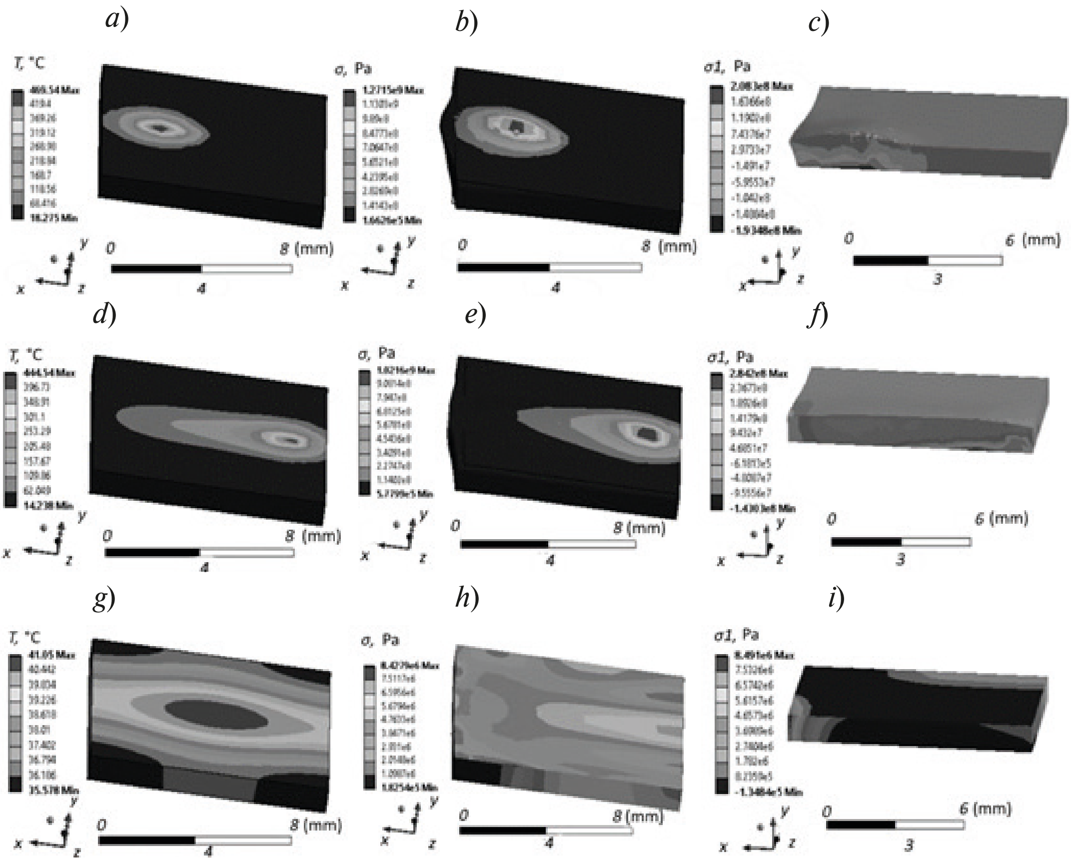

Fig. 4. The calculated distributions of maximum temperature (*a*, *d*, *g*), of equivalent von-Mises stresses (*b*, *e*, *h*), and of maximum principle stresses (*c*, *f*, *i*) over a vertical cross-section of the body at different time points in the laser processing, s: 0.08 (*a*, *b*, *c*), 0.32 (*d*, *e*, *f*) and 8.0 (*g*, *h*, *i*). These are criteria for crack initiation during $TiO_2$ film LA, under the following conditions: a laser scan speed is 25 mm/s; LP is 30 W; the film thickness is 5 µm, the substrate one is 1 mm; the duration of laser treatment is 0.32 s

An increase in the film thickness from 5 to 30 µm decreases the maximum principal stresses. The stress variation induced by an annealing is as much higher as the film thickness is low. We can see in Fig. 3 that the thicker substrate (1 mm) and the higher laser speed (more than 2 cm/s at laser power of 30 W) allow to obtain a film without cracking risk. It is possible to control and vary the value of thermoelastic stresses in the film-substrate structures due to changes of film annealing processing parameters: the substrate temperature, laser radiation power, film and substrate thicknesses, pulse duration, sample movement velocity, etc. Thereby one can optimize film properties for the task and device design.

## Conclusion

We have simulated $TiO_2$ thin films processing on sapphire substrates and conducted related experiments. We developed a three-dimensional model for an analysis of the stress distribution in the film-substrate structure. The films properties were investigated with atomic force microscopy method, scanning electron microscopy, and X-ray phase analysis.

The thermomechanical model for $TiO_2$ LA on sapphire substrate was implemented in ANSYS software. We investigated that the temperature on $TiO_2$ film (thickness of 5 µm) surface is about 500 – 600 °C at an average LR power of 30 W, which is a prerequisite for the growth of films on the substrate surface. It corresponds to the level of thermoelastic stresses (much less than the material elastic limit) such that the cracks formation is not expected. The morphology of the film structure can be varied by changing the laser power and temperature, which allows to reallocate defects in the structure and to improve the films quality for their usage in microelectronics and thin film optics.

Higher scan speed leads to lower temperatures and larger temperature gradient during the material heating and cooling. The film and substrate thickness play a key role in the formation of cracks and defects in film. The studies showed that cracks on the surface can be formed in thicker films. The thicker substrate (1 mm), the bigger laser scan speed (more than 20 mm/s), and the smaller laser power (30 W) allow to obtain the film without cracking risk. Thus, the $TiO_2$ film-to-substrate thickness ratio, LP, laser scan speed are the most important parameters to process a uniform film for their application in microelectronics.

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