# **Effective Time of Nanogratings Formation on Sol-Gel Films**

# by Two-Beam Laser Interference

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The paper discusses the analytical modelling of periodic nanostructure formation on thin sol-gel films (SiO<sub>2</sub> and AlZnO) by picosecond laser pulses utilizing a two-beam interference technique. The results obtained are in good correspondence with data of direct laser interference patterning of AlZnO films. The effective time, which includes the pulse duration, material heating, and cooling, is estimated for both cases. The heating time for films is determined by the thermodynamic equilibrium between the electron gas and atomic lattice. Heat outflow from the irradiation region results in cooling the films. As the first approximation, the effective time of nanograting formation is determined by thermal properties of a film, as well as by the period of the interference pattern and temperature distribution of a heat source. The nanogratings formation finishes during the film cooling and takes less than 5 ns from the start of irradiation. The formation time also depends on the interference pattern period and can be reduced by its decrease.

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## 1. Introduction

Periodic nanogratings formed on composite sol-gel films are mostly considered as elements in photonics, microanalytical and photovoltaic systems. In photonics, subwavelength gratings containing nanoparticles (NPs) are actively used for optical filters [1, 2]. Such structures recorded on semiconductor TiO<sub>2</sub> films containing Ag/Au NPs demonstrate dichroic and interference properties and are in demand for data protection [3] and holographic data storage [4]. The grating with periodically distributed NPs serves as SERS sensors for Raman spectroscopy [5]. Plasmonic properties of such materials are useful for photodetection enhancement [6]. A unique combination of electrical properties of a sol-gel film and optical characteristics of nanogratings is used in microanalysis [7] and photocatalytic applications [8].

Currently, the practical use of gratings on sol-gel film containing NPs requires a detailed study of their formation mechanisms and characteristics. Laser processing has become one of the most successful ways of recording subwavelength gratings on sol-gel films [1], metal films [9], transparent materials [10]. Moreover, film processing in the interference field of short and ultrashort laser pulses of UV radiation has become the most widespread [2, 11, 12] to fabricate structures with a period from 250 to 550 nm, which is equal to the applied wavelength. Further development of such an approach provides opportunities for creating devices based on metasurfaces [13].

The effect of laser interference pattern with picosecond pulse duration on thin films is complex and understudied. The film melting and evaporation with the corresponding NPs modification are the main laser regimes of nanostructure formation based on multiphoton absorption and electron-phonon interactions [14]. The effective time of nanogratings formation is the time from pulse start to thermodynamic equilibrium between the electron gas and atomic lattice. In this case, the matrix temperature achieves the maximum value. The effective time determination remains a controversial issue.

On the one hand, various models describe the laser processing of thin sol-gel films. For example, a twotemperature model is successfully applied to describe the absorption of laser radiation and material heating by ultrashort laser pulses [15]. On the other hand, the bulk metals processing with two-beam interference can be analyzed by numerical simulation [16]. However, these models have the following features:

(i) the two-temperature model does not consider the cooling process making impossible the estimation of the total time for nanogratings formation;

(ii) the numerical simulation of bulk materials interference patterning gives the results, which are difficult to analyze.

Under experimental conditions, the preference is given to analytical models, which in the first approximation consider such material features as thickness, composition, physical properties, and laser processing regimes. The development of analytical models is crucial for novel composite films as they allow for optimal conditions to form nanogratings with the required properties.

This paper gives an analytical model, which describes the processing of sol-gel films by picosecond laser pulses utilizing a two-beam interference technique. The modelling results are presented and discussed to estimate the effective time, which includes the pulse duration, material heating, and cooling. At the same time, based on the modelling results, the optimal laser processing regimes of nanocomposites are predicted. The features of nanogratings during evaporation or melting of the sol-gel films are theoretically studied.

#### 2. Model conditions

In the current model, we applied AlZnO (5% Al) film (thickness of  $h_f = 200$  nm), deposited on a fused silica substrate, and pure SiO<sub>2</sub> medium for comparison. A typical source for interference patterning is a picosecond laser duration with high pulse energy and with the ability to select shorter radiation wavelength by choosing 3rd or 4th harmonics. In particular, our team has applied a two-beam interference technique with picosecond laser pulses to fabricate nanogratings on SiO2 sol-gel film contained silver NPs [2]. The developed model is supported with details, parameters, and compared to the mentioned experiment.

A confocal imaging system was applied to realize the interference field in the processing region [2]. Two diffracted beams are overlapping beneath the aspherical lens providing an interference pattern in their intersection area. In the model, a single picosecond laser pulse ( $\lambda = 355$  nm,  $\tau = 25$  ps) forms a heat source, which leads to the film melting (Fig. 1, a) or evaporation (Fig. 1, b). The pulse energy,  $E_p$ , was gradually changed from 3 to 10 µJ.



**Fig. 1** Illustration of film processing by interferences spot of the laser pulse. a – partial evaporation of the film, b – film melting.

Two aspherical lenses can be applied to provide an interference pattern with the various period,  $\Lambda$ . The lens (f = 8 mm, NA = 0.5 and L = 6.1 mm) utilized by us previously provided  $\Lambda = 500 \text{ nm}$  [2]. The laser incident angle between beams, in this case, was  $\theta = 41.74^{\circ}$ . and  $\Lambda = 300 \text{ nm}$  (lens 2, f = 3.1 mm, NA = 0.68 and L = 4.6 mm). Lens with f = 3.1 mm, NA = 0.68 and L = 4.6 mm). Lens with f = 3.1 mm, NA = 0.68 and L = 300 nm, while  $\theta = 73.14^{\circ}$ . In the model, we considered the smallest beam spot diameter equals  $2r = 5 \mu m$  for both cases. Moreover, an increase in size would only lead to a decrease in the calculation efficiency. The number of fringes in an interference pattern J =  $2r\Lambda$  is 10 and 17 for the interference pattering periods 500 nm and 300 nm, respectively.

#### 3. Model description

The following approximations are considered to describe the thermal conduction model for utilized films processing by two-beam interference pattern with the picosecond laser pulse: - the laser fluence is absorbed by the sol-gel film, without light penetration in the substrate;

- if the heat inflow duration,  $t_h$ , is higher than the relaxation time of the electron-phonon interactions,  $t_{ep}$ ,  $(\tau > t_{ep})$ . Thus,  $t_h$  equals the pulse duration  $\tau$ , otherwise,  $t_h$  equals  $t_{ep}$  ( $\tau < t_{ep}$ ); - the heat outflow duration from the heated region depends on the thermal diffusivity of the film and substrate, and began after  $t_h$  time;

- the relaxation time  $t_{ep}$  is determined by the material properties, and does not depend on laser processing regimes; - the heat outflow from the interference pattern during the film heating is not considered, due to ultrashort time  $t_{ep}$ ;

- substrate heating on the interface with film starts at the film cooling.

A periodic heat source in the film as a result of interference action is described as a heat inflow into each interference fringe during the relaxation time  $t_{ep}$  for the case  $\tau < t_{ep}$ , or to the end of the pulse if  $\tau > t_{ep}$ . The total heat inflow is described by the sum of the integrals in the form of the following expression:

$$dT_{in} = \sum_{u=0}^{J} \left[ F(t,\tau) \int_{0}^{t_{h}} T(u,\xi) d\xi \right], \ t < t_{h}$$
(1)

where *t<sub>h</sub>* is the time of heating determined as the duration of heat inflow:

$$t_h = \begin{bmatrix} t_{ep}, \ \tau \leq t_{ep}, \\ \tau, \ \tau > t_{ep}. \end{bmatrix}$$

The function  $T(u, \zeta)$  describes the shape of the heat source in space from one interference fringe. The total heat outflow from a periodic source is also described by the sum of the integrals:

$$dT_{out} = \sum_{u=0}^{J} \left[ \int_{0}^{t} T(u,\xi) d\xi - \int_{0}^{t-t_{h}} T(u,\xi) d\xi \right], \ t \ge t_{h}$$
(2)

The function  $T(u, \xi)$  in (1) and (2) is determined, as

$$T(u,\xi) = \frac{8a \cdot \exp\left(-X_s^2 - Y_s^2 - Z_s^2\right)}{\left(p^2 \Lambda^2 + 8a\xi\right)\sqrt{\xi}},\tag{3}$$

where

$$X_{s} = \frac{x + (0.5J - u)\Lambda}{\sqrt{0.5p^{2}\Lambda^{2} + 4a\xi}}, \quad Y_{s} = \frac{y}{\sqrt{0.5s_{y}r^{2} + 4a\xi}},$$
$$Z_{s} = \frac{z}{\sqrt{0.5h_{c}^{2} + 4a\xi}}.$$

Here, *a* is the thermal diffusivity of the heating material,  $s_y = l_y/r = 5$  is the relative between the spot radius in the *Y* and *X* axis, p = 0.45 is the proportionality coefficient between fringe width and their distance. The T function in equation (3) is the integrand and it describes the heat inflow and outflow in the irradiated region with interference fringe shape. The  $X_s$  and  $Y_s$  parameters represent the heat source shape along and across the fringes, respectively. The  $Z_s$  parameter represents the heat source thickness. Thermal diffusivity depended on *Z* axis and can be described by the following equation:

$$a = \begin{cases} a_{air}, \ z < -0.5h_f, \\ k_m / (\rho c_m), \ -0.5h_f \le z \le 0.5h_f, \\ a_{sub}, \ z > 0.5h_f, \end{cases}$$
(4)

where  $k_m$ ,  $c_m$ , and  $\rho$  are thermal conductivity, heat capacity, and density of the films (Table 1),  $a_{air} = 2.107 \cdot 10^{-5} \text{ m}^2/\text{s}$ , and  $a_{sub} = 5.956 \cdot 10^{-7} \text{ m}^2/\text{s}$  are thermal diffusivity of air and fused silica (substrate).

Table 1 Characteristics of sol-gel films

Parameter	$SiO_2$	AlZnO
Heat capacity $c_m$ , J/(kgK)	732 [17]	494 [18]
Therm. conductivity $k_m$ , W/(mK)	0.96 [17]	5.40 [19]
Density $\rho$ , kg/m <sup>3</sup>	2201 [17]	5700 [18]
Electron concentration $n_e$ , cm <sup>-3</sup>	$1.0 \cdot 10^{16}$	$9.2 \cdot 10^{19}$ [20]
Bandgap / Fermi energy $E_F$ , eV	8.70 [17]	3.53 [21]
Extinction <i>k</i> <sub>ext</sub>	10 <sup>-5</sup> [22]	0.0135
Sound speed $v_s$ , $\mu$ m/ns	5.76	6.59 [21]

The function of  $F(t,\tau)$  in (1) represents the increase of the film temperature during heat inflow up to cooling that is determined as:

$$F(t,\tau) = \begin{cases} 1 - \exp\left[-B_{1}\frac{t}{t_{ep}}\sin(2\pi\nu_{p}t)^{2} - \\ -B_{2}t/(\nu_{p}t_{ep}^{2}) \\ \sqrt{t/\tau}, \ \tau > t_{ep}, \end{cases}, \quad (5)$$

where  $B_1$  and  $B_2$  are the coefficients that determine the amplitude and rate growth of the temperature, as the kinetic energy of the phonons,  $v_p$  is the frequency of phonons oscillation in the matrix. For  $\tau \leq t_{ep}$  (ultrashort pulses), the temperature increase in the relaxation time is represented as harmonic oscillations of phonons in the matrix. Otherwise, the temperature increase is approximated as a square root of the relative time (short pulses).

Temperature change in space over time is described as:

$$T_{\rm 3D}\left(x, y, z, t\right) = Q\left(dT_{in} + dT_{out}\right) + T_K, \qquad (6)$$

where  $T_K = 273$  K, Q is the source of the absorbed energy from a single interference fringe in the laser spot. The following function Q is used in this modelling:

$$Q = \frac{1}{J+1} \frac{A_b E_p}{c_m \rho \tau \sqrt{\pi a}}, \quad -0.5h_f \le z \le 0.5h_f,$$
(7)

where  $A_b$  is the absorption coefficient including linear and nonlinear absorption. The film thickness limitation is determined by the light penetration depth into transparent medium or a thin film, when  $\lambda/(4\pi k_{ext}) >> [\tau \cdot k_m/(\rho c_m)]^{0.5}$ . In this case, the maximum thickness of the heat source modeled is determined by equation  $h_{max} = -\lambda/(4\pi k_{ext}n)\ln(A_b - e^{-1})$ . Here *n* is the refractive index,  $e \approx 2.72$ . The AlZnO film with  $A_b = 0.35$  possesses  $h_{max} \approx 0.7 \ \mu m$ ,  $SiO_2$  film with  $A_b = 0.25$ possesses  $h_{max} \approx 100 \ \mu m$ . Thus, the model is applicable for films with AlZnO thickness up to 0.7  $\mu$ m, and SiO<sub>2</sub> thickness up to 100  $\mu$ m.

## 4. Results and discussion

## 4.1 Time of film heating

Film heating by an ultrashort laser pulse (from 10 fs to 100 ps) is well described in terms of the two-temperature model

[15]. Free electron relaxation time or  $t_{ep}$ , is considered by this model, which determines the heating time of the material after pulse action. In this approximation, the electron gas is heated instantly, reaching the maximum temperature at the end of the pulse. The hot electron gas begins the interaction with the cold atomic lattice. The system aspired to a thermal equilibrium as a result of this interaction, and the matrix is heated. The atomic lattice oscillates during these interactions. The increase of the amplitude and kinetic energy or temperature of such oscillations is described by the equation (5) in the first approximation. When heat inflow from the electron gas to the atomic lattice equals heat outflow due to thermal conductivity,  $k_m$ , the relaxation time,  $t_{ep}$ , is achieved. In the first approximation, this time can be determined by the equation [23]:

$$t_{ep} = \frac{c_e}{\beta} \ln \left[ 1 + \frac{\beta}{k_m} \frac{c_e}{c_a} \left( \frac{\lambda}{4\pi k_{ext}} \right)^2 \right],\tag{8}$$

where  $c_e$  and  $c_a$  are heat capacities of the electron gas and the atomic lattice,  $\beta$  is the coefficient of heat transfer between the electron gas and the atomic lattice,  $k_{ext}$  is the extinction of a film. Heat capacity  $c_e$  can be determined as an ideal gas at high temperature ( $T_e$  about  $10^4 - 10^5$  K). In this case,  $c_e = (3/2)n_ek_B$ , where  $k_B$  is the Boltzmann constant,  $n_e$  is free electron concentration. The main equation of  $c_e$  has the following form [15]:

$$c_{e} = \pi^{2} n_{e} k_{B}^{2} T_{e} / (2\rho E_{F}), \qquad (9)$$

where  $E_F$  is the bandgap or Fermi energy of free electrons (Table 1). Heat capacity of the electron gas has the values:  $c_e = 3.067 \cdot 10^{-5}$  J/(kg·K) for SiO<sub>2</sub> and  $c_e = 0.269$  J/(kg·K) for AlZnO. Atomic lattice heat capacity approximately equals film heat capacity or  $c_a \approx c_m$  (Table 1). The value  $t_{ep}$  with  $\beta \sim 9.35$  MW/(m<sup>2</sup>K) is  $t_{ep} = 100$  ps for SiO<sub>2</sub> and  $t_{ep} = 383$  ps for AlZnO.

The frequency of phonon oscillations in the atomic lattice is determined as [15]:

$$v_{p} = (3/4\pi)^{1/3} v_{s}/s_{p}, \qquad (10)$$

where  $v_s$  is the sound speed in the film,  $s_p = (n_e)^{-1/3}$  [24] is the average interatomic spacing (Table 1). The frequency  $v_p$  had the following values:  $v_p = 1.846$  THz for SiO<sub>2</sub> and  $v_p = 0.077$  THz for the AlZnO film. Coefficient  $B_1$  in the equation (5) is equal to 1.0 in both cases. The second coefficient  $B_2 = 770$  is used for SiO<sub>2</sub> and  $B_2 = 135$  for AlZnO. The approximation of atomic lattice oscillations is applied for film heating (Fig. 2). Thus, the cooling curve starting after  $t_{ep}$  (Fig. 2) is described as a standard heat outflow from the heated regions by Eq. 2.

### 4.2 Results of modelling and discussion

The periodic nanostructures formation in the film begin when the temperature in the center of the interference fringe exceeds the phase transformation temperature. For AlZnO film, fusion temperature is equal to 1668 K and annealing (sintering and crystallization at 870 K). In addition, for the films contained silver NPs, the destruction (1400 K) and growth (700 K) temperatures of NPs are the main parameters (Fig. 2a). For SiO<sub>2</sub> film, these temperatures differ, the decomposition starts at 2270 K, fusion at 1883 K and softening at 1000 K (Fig. 2b). The effective time,  $t_{eff}$ , of a nanograting formation is the sum of heating time,  $t_h$  and the cooling time up to fusion temperature.



Fig. 2 Temperature of AlZnO (a) and SiO<sub>2</sub> (b) films on the surface (red line) and the substrate (blue line) in interference fringe center.

The modelling result shows the melting of AlZnO film surface takes ~193 ps when T > 1668 K (Fig. 2a). The annealing duration of AlZnO film extends over 490 ps at T > 870 K. The NPs growth is activated only at cooling when the temperature decreases from 1400 K to 700 K that continuous ~255 ps. Thus, the nanograting formation at temperatures below evaporation and melting is caused by the change in optical properties of the film as a result of NPs growth/destruction. This modelling case suggests the gradient nanograting formation.

The partial decomposition of the SiO<sub>2</sub> film is shown in the second case (Fig. 2b). The modelling result shows the decomposition time is equal to 18 ps. The fusion time is about 73 ps, which is twice as fast compared to AlZnO film fusion. This difference is due to the film's thermal properties and time  $t_{ep}$ . The time of NPs growth in SiO<sub>2</sub> film is 293 ps that correspond to the values obtained for AlZnO film. The nanograting is formed at the thermal decomposition of SiO<sub>2</sub> films. As a result, the shape of the future grating is determined by the maximum temperature obtained, its distribution, and the time  $t_{ep}$ .

The temperature distribution in the AlZnO film is modeled after the influence of two-beam laser interference with a period  $\Lambda = 500$  nm (Fig. 3). We consider two cases for AlZnO (Fig. 3a) and SiO<sub>2</sub> (Fig. 3b) films. As we have estimated, *tep* corresponds to 383 ps and after 650±100 ps the temperature is not enough to modify the film. The periodic heat source disappeared in the film occurred much later when the film almost cooled. According to the modelling it takes ~5 ns. Rapid film heating (3.83 K/ps) and cooling (3.9 K/ps) prevent a noticeable heat transfer to the substrate and the environment (Fig. 3). However, the film fusion and annealing occur strongly in the central part of the interference fringes, where the maximum temperature is reached. NPs growth takes place at the edge of interference fringes, where the temperature is lower in the range of 1400-700 K. Here, let us mention that such a perfect periodic of the temperature profile is formed due to the beam profile approximation by top-flat fringes. The temperature profiles are discontinuous at the interfaces because the model considers laser energy absorption only in the film. The substrate and environment heating at the interfaces with the film are due to heat transfer.



Fig. 3 The temperature profile in films: AlZnO (a) and SiO<sub>2</sub> (b)) simulated for different times (0.1 ns and 5 ns) since the beginning of the laser pulse. The interference period  $\Lambda = 500$  nm.

Then we decrease the period up to  $\Lambda = 300$  nm to show the difference (Fig. 4). A decrease in the interference fringe period led to the convergence of heat sources. The connection time of thermal fronts from the neighboring sources is decreased at a smaller distance between interference fringes. As a result, we obtain the almost complete disappearance of the periodic heat source upon 5 ns after the beginning of the laser pulse. This is due to the localization of heat sources and an increase in heat outflow into the film during cooling. Similar dynamics of periodic heat source relaxation is observed in the case of the SiO<sub>2</sub> film heating. For both cases, the decomposition occurs only in the center of interference fringes, while fusion and softening occur at the edge of interference fringes.

The effective time of film heating ranged from 100 to 400 ps that is too short for the NPs growth in the  $SiO_2$  matrix. Such heating activates phase transformation of the film matrix and destruction of NPs. The thermal diffusion of NPs growth and organization into periodic structures require a lower temperature and a longer heat exposure time. Such conditions are achieved at the stage of film cooling. The time of NPs growth increased with an increase in the period of the interference pattern. Thus, silver NPs in SiO<sub>2</sub> film grow at a

temperature in the range of 1400 - 700 K that corresponds to the cooling time  $500 \pm 100$  ps (Fig. 2b). The estimated time is insufficient for the growth of large nanoparticles; therefore, according to the experiment [2], their size does not exceed 3-10 nm.



Fig. 4 The temperature profile in the film region (AlZnO) modification at various times, the interference period is 300 and 500 nm.

The thermal field modelling shows that the field periodicity in the case of  $\Lambda = 500$  nm, remains strictly period during *t<sub>eff</sub>*. The periodicity disappears after 5 ns, which is much longer than *t<sub>eff</sub>*, not exceeding 0.65±0.1 ns for both cases. The further pulse duration increasing ( $\tau > 5$  ns) will preserve the periodicity of the heat source, however, the temperature between the interference fringes will exceed the allowable value.

The modelling results obtained are in good correspondence with data of direct laser interference patterning of metal [16]. The experimental results obtained by our team about SiO<sub>2</sub> sol-gel films contained Ag NPs interference patterning are also correlated with the simulated results revealed in this paper [2].

## 5. Conclusion

The analytical model describing the nanograting formation on sol-gel films (SiO<sub>2</sub> and AlZnO) by two-beam interference technique with picosecond laser pulses is proposed. In the study, the following results are obtained:

- time of film heating depends on electron-phonon interactions in the film matrix and is about 100 ps for the SiO<sub>2</sub> film and  $\sim$ 383 ps for the AlZnO film;

- effective time of grating formation with a period of 500 nm is less than 1 ns. In particular, this time is equal to 0.15 - 0.40 ns for the SiO<sub>2</sub> film and 0.5 - 0.7 ns for the AlZnO film;

- periodicity of heat source is maintained within the interference fringes and remained at cooling up to the full formation of the grating;

- time of the heat source disappearance depends on the interference period and thermal properties of the film.

The modelling results obtained opens new opportunities for precise laser fabrication of nanogratings on thin semiconductor and dielectric films. The fabrication of such gratings is particularly promising for numerous applications in photonics.

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