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LASER INTERFERENCE THERMOMETRY OF SOLIDS IN PLASMAS

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Abstract: Laser interferometric thermometry of semiconductor and dielectric plates is the most sensitive and reliable technique for measurements in plasma processing. This technique can be used within a wide range of experimental conditions, including roughness and thin films on the surface, non-parallelism of surfaces, almost any angle of light incidence. Some results on the temperature monitoring of silicon crystal heated in a plasma and on the end-point detection of exothermic plasma chemical reaction on the surface are presented.

Keywords: laser thermometry, plasma surface interaction

1 INTRODUCTION

Nowadays, about 10 techniques for laser thermometry of solids are known. These techniques are based on the active probing the investigated objects by laser beam to measure one of the temperature-dependent optical parameters. Physical principles, limitations and measurement characteristics of laser thermometry techniques have been discussed in detail in review [1]. The main reason for developing new techniques is the problem of surface thermometry in gas discharge plasmas, which are commonly used in semiconductor microtechnology. Traditional techniques are in general useless in plasma experiments. Laser thermometry has a number of advantages over traditional techniques: 1) a contact of probing laser beam and surface is evident and reliable in any conditions, which include high temperature, mechanical vibration, chemical activity of medium in plasma reactor; 2) complete immunity to electromagnetic noise over the entire spectral range from low and radio frequencies to optical ones. In this paper some characteristics of active thermometry and experimental results on transient temperatures measurement of semiconductor crystals in RF gas discharge plasma via laser interferometric technique are discussed.

2 LASER INTERFEROMETRIC THERMOMETRY

This technique is most frequently used for technological monitoring in plasma processing [2-6] and for research of plasma/surface interaction [7-9], since it is currently the most sensitive, accurate and disturbance-protected technique in comparison with conventional techniques and other methods of laser thermometry of solids. Plane-parallel plate made of semiconductor or dielectric is Fabry-Perot interferometer for laser beam in transparency spectral range of material (Fig.1).

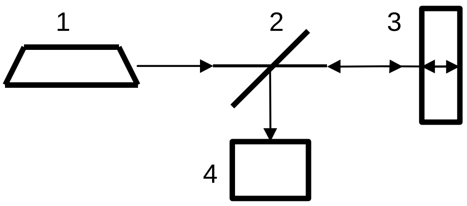

Figure 1. Simplified sketch of the laser interferometric thermometry. Laser (1), semi-transparent mirror (2), plate under study (3), photodetector (4).

Contrary to any two-beam interferometers which are often used to investigate transparent objects (flames, plasmas, etc.) with nonreflective boundaries, the laser interferometric thermometry does not involve the reference light beam, since a solid plate under study has two reflected surfaces forming an optical cavity with multiple internal reflections of light. Interferometric thermometry of metal plates is impossible due to their optical opacity.

When the temperature (T) of transparent or semi-transparent plate changes its refractive index (n) and thickness (h) change too. The change of optical thickness (nh) leads to oscillations of intensity of both reflected and transmitted light. For example, the reflectance at normal incidence is described by well known expression

$$R = [A - B\cdot\cos(2nkh)]/[C - B\cdot\cos(2nkh)] \tag{1}$$

where $A = R_1 + R_2 \cdot \exp(-2\alpha h)$, $B = 2(R_1 \cdot R_2)^{1/2} \cdot \exp(-\alpha h)$, $C = 1 + R_1 \cdot R_2 \cdot \exp(-2\alpha h)$; R_1 and R_2 are the reflection coefficients of surfaces, light incidences on the surface signed by subscript 1; α is the absorption coefficient; $k = 2\pi/\lambda$ is the wavenumber, λ is the wavelength. When the phase $\varphi \equiv 2nkh = \pi m$, m=0,1,..., the value of R reaches its extrema:

$$R_{min} = (A-B)/(C-B) \text{ at } m = 0,2,..., \qquad R_{max} = (A+B)/(C+B) \text{ at } m = 1,3,..., \tag{2}$$

The plate temperature in heating or cooling mode may be calculated in real time using the sequence of Fabry-Perot resonances (so-called interferogram) shown in Fig.2-3. Near 10^2 interferograms in plasma can be registered and processed per day, even though each experiment is performed using new plate. The 5-mW laser beam after reflection from plate is easily detected against the plasma emission to reach a signal-to-noise ratio of the order of 10^3.

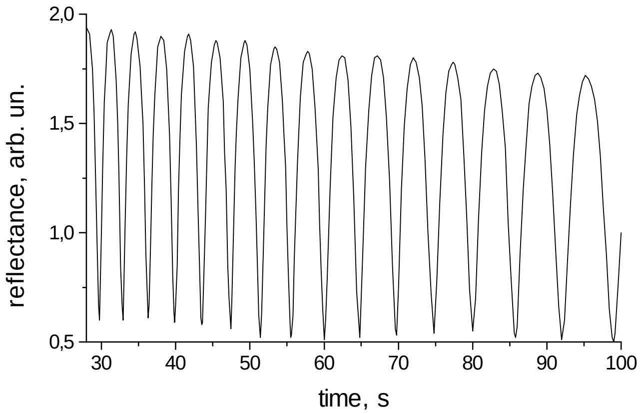

Figure 2. Light reflectance oscillations in heating the 0.38-mm-thick single silicon crystal in oxygen plasma at gas pressure of 50 Pa and applied RF power of 140 W. He-Ne laser beam at wavelength of 1152 nm is 0.5 mm in diameter. The angle of light incidence on the surface is $\approx 5^0$ relative to the normal incidence. Temperature interval between two neighbouring minima equals to $\Delta T \approx 6.8\ ^0C$. Interference contrast $V_R \approx 0.5$ is less than the greatest possible one $(V_R)_{max} = 1$ due mainly to non-parallelism of two opposite reflecting surfaces.

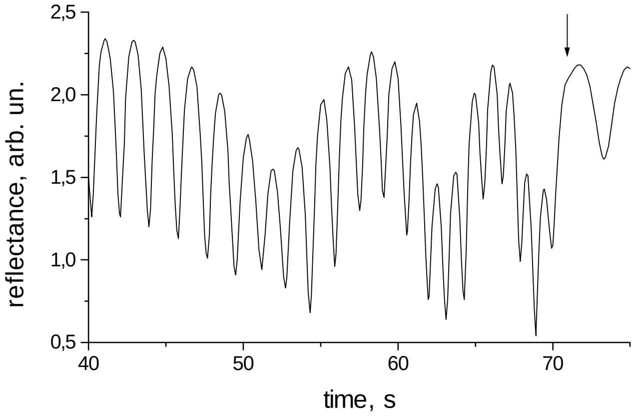

Figure 3. Interferogram (in reflected light) of single silicon crystal covered with thin polymer film in oxygen plasma. When a film thickness decreases in time due to chemical reaction, strong modulation of both R_1 and resulting interferogram is observed. Each modulation period corresponds to thickness decrease $\Delta h = \lambda/2n$. Since the reaction is exothermic, self-acceleration of polymer etching takes place. The arrow indicates the end of etching at the moment of complete removal of the film from the surface.

Technique for data handling has been discussed in review [10]. It should be remembered that the transient temperature only may be measured by interferometric technique with the constant wavelength. For measuring the steady-state temperature some spectral tuning over a range $\Delta\lambda$ is necessary ($\Delta\lambda \geq \lambda^2/2nh$) to measure the wavelength interval between two interference extrema. The initial temperature T_0 of plate is necessary to be known. Temperatures corresponding to interference extrema can be determined using known expression $n(T)\cdot h(T) = 0.25\lambda m$, where λ is the wavelength, m = 0,1,2..., even numbers correspond to maxima of transmittance, and odd numbers to maxima of reflectance. When the temperature is time-constant, the reflectance of plate remains at a steady level. Laser thermometer is ready for measurement immediately after the next plate is loaded into the reactor. Such productivity of measurements is unattainable for contact thermometry.

The temperature sensitivity of registered signal may be defined by different ways. For example, an absolute value of derivative $|dR/dT|$ may be obtained from (1) to characterize the sensitivity. The value of sensitivity determined by this manner changes over the interference period and it is inconvenient to use. It is conveniently to define the mean sensitivity (S) which is averaged over the interference period by the follow way for reflectance mode:

$$S(\%K^{-1}) = 400[(R_{max} - R_{min})/(R_{max} + R_{min})]\cdot(\Delta T)^{-1} = 200\Phi\cdot V_R\cdot\pi^{-1} \qquad (3)$$

where $\Delta T = \lambda/2nh[n^{-1}\cdot(\partial n/\partial T)+h^{-1}\cdot(\partial h/\partial T)]$, $\Phi = 2\pi\cdot(\Delta T)^{-1}$, $V_R = (R_{max} - R_{min})/(R_{max} + R_{min})$. The value of S shows what is signal relative change in percent when the temperature variation is equal to 1 K. Using this definition one may compare the temperature sensitivities for any techniques, including thermocouples, etc. The values of S for 1-mm-thick plates made of different materials at T=300 K are shown in Table 1. The value of ΔT is independent on the surface conditions and slightly decreases with temperature. Sensitivity is proportional to the plate thickness; the temperature interval ΔT varies in inverse proportion to the thickness. Both thermocouple and resistance thermometer have the sensitivity which is two order of magnitude less as compared to semiconductors plates in laser interferometric thermometry.

Table 1. Temperature interval ΔT between two neighbouring interference minima and period-averaged sensitivity S for 1-mm-thick semiconductor and dielectric plates.

Material (wavelength, μm)	Fused SiO_2 (0.633)	ZnS (0.633)	GaP (0.633)	GaAs (1.15)	Si (1.15)	Ge (2.0)
ΔT, K	31.4	4.5	1.6	2.1	2.6	2.1
S, $\%K^{-1}$	13	89	253	187	153	190

The dependences of S on wavelength λ and temperature T have been discussed previously [10]. Sensitivity S increases with shortening the wavelength of probing light as long as an optical absorption plays the negligible role ($\alpha h \ll 1$). Since the sensitivity increases with the plate thickness, thermometry of thin films (~1÷10 μm in thickness) is of little promise. But if the film is deposited on the transparent wafer, laser thermometry of wafer itself gives the opportunity to study the interaction between plasma and this film made of practically any material.

There are several reasons for decreasing the interference contrast V and therefore the sensitivity S. First, a deviation from ideal parallelism of two opposite surfaces results in contrast drop. Second, the difference between R_1 and R_2 leads to decrease of V. Three, the contrast falls off to zero, when the plate thickness, beam diameter (D) and angle of incidence (i) are connected by expression

$$D/h \leq (\sin 2i)/(n^2 - \sin^2 i)^{1/2} \qquad (4)$$

This absence of interference is due to complete spatial separation of light beams on the photodetector. Nevertheless, laser interferometric thermometry may be used within a broad experimental conditions, including roughness and thin films on the surface, non-parallelism of surfaces (up to $3\cdot10^{-4}$ rad), almost any angle of light incidence [11]. It is important for technological temperature control, that the interferometric technique does not require both sides of the semiconductor crystal to be polished, contrary to common a priori opinion. Experiments show that a backside roughness of single silicon crystals does not prevent the temperature measurement.

Maximal interference contrast V and temperature sensitivity S are reached when the reflectances of both opposite surfaces are identical. When a surface is under plasma processing, this results in temporal variation of reflectance through a number of physical and chemical processes (surface erosion, thin film deposition, etc.). But only one of two surfaces is exposed to chemically active plasma. The value of S decreases in time with increasing difference in reflectances of two surfaces. As the value of ΔT does not depend on the surface conditions, temperature measurement is performed only for the moments which correspond to the interference extrema.

3 EXPERIMENT

3.1 Goals and problems of measurements in plasmas

Temperature measurements can be the main way to get some information concerning the plasma surface interaction. Surface temperature is one of the controlling parameters in plasma processing due to essential temperature dependencies of deposition and etch rates. Contributions of each kind of particles colliding the surface into the total heat flux from plasma can be determined using the transient temperature T(t) and its time derivatives after discharge ignition.

Optical constants of materials can be probably varied due to surface charging and electrical fields in plasmas. The role of this effect may be examined experimentally using the fact that the characteristic times of surface charging and plate heating are significantly different. The fictitious temperature jump immediately after discharge ignition is indicative of the role of field effect. But there are no observations such phenomena in low-pressure plasmas when the laser interferometric thermometry is used.

3.2. Experimental setup

The studying a temperature kinetics of silicon and glass plates was performed in a cylindric quartz reactor 20 cm in diameter and 45 cm in length. The RF discharge (13.56 MHz) is excited by external semi-cylindric electrodes at the gas pressure 10 to 300 Pa in different gases (He, Ar, N_2, O_2 and CF_4). RF power applied to the discharge is 50 to 300 W. This reactor and optical scheme of laser thermometer were described in detail previously [7]. A plate is placed normally to the reactor axis on the holder which consists of two quartz rodes 1.5 mm in diameter.

4 RESULTS AND DISCUSSION

A regular study of temperature regimes of glasses and semiconductors in plasmas has became possible only after development of laser thermometry of solids. Over the past ten years in our team about 3000 interferograms were registered during plates heating in gas discharge plasmas. Temporal dependencies of plates temperature were derived from those interferograms to understand kinetics and mechanisms of heat transfer from plasmas to surfaces.

The nature of thermal phenomena in interaction between plasmas and surfaces may be understand when the data are presented not only as time dependencies of plate temperature T(t) (shown in Fig.4) but also as temperature dependencies of heating rate dT/dt (Fig.5).

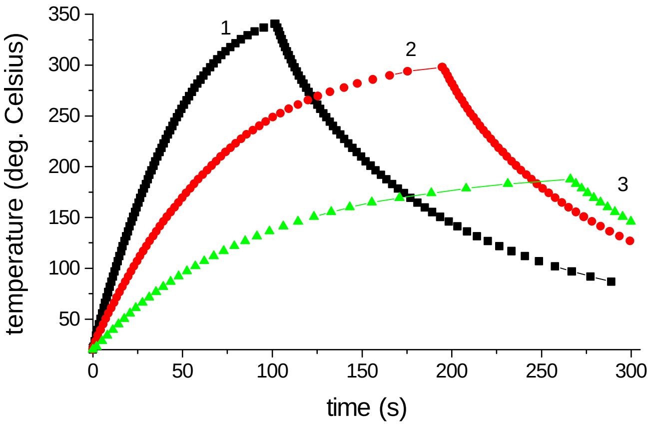

Figure 4. Temperature of 0.5-mm-thick silicon single crystal (3x3 cm) after RF discharge ignition and shutdown in nitrogen at 50 Pa and applied power (W): 340 (1), 230 (2), 140 (3).

Differentiation of T(t) with respect to time is the direction to perform the separation of the phase trajectory (dT/dt versus T) into linear components with different slopes and nonlinear components. The trajectory plotted in temperature - heating rate coordinate plane (Fig.5) is similar to phase trajectory in coordinate-momentum plane widely used in mechanics. By choosing such variables one can observe trajectory regions having linear form. Linear dependence of dT/dt on the temperature of chemically and catalytically inert surface provides a way to detect the presence of the other heat transfer mechanisms by registering the deviation of dT/dt from linear form. For example, some deviations downwards from linearity at high temperatures in Fig.5 are connected with radiative cooling of crystal. It is necessary to improve in nearest future data processing toward more precise determination of the second (d^2T/dt^2)

derivative by the use of smoothing of dT/dt. Linear dependence of dT/dt on T is typical for simple relaxation processes, in which the sole mechanism of heat transfer from plasma operates all the time.

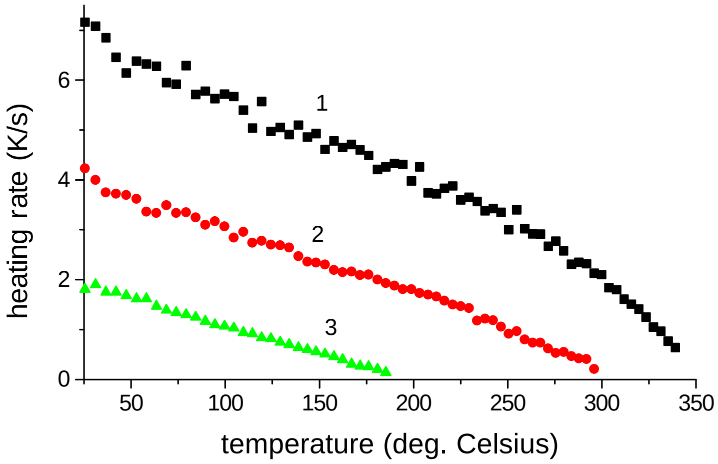

Figure 5. Temperature dependencies of heating rate of silicon crystal. Conditions and designations as in Fig.4.

Temperature kinetics of crystal essentially differs from simple relaxation curves when a chemical reaction takes place on the surface immerced to plasma. The rate of reaction depends on the temperature as exp(-ΔE/kT) where ΔE is the activation energy, k is the Boltzmann constant. Due to such strong dependence, heat release in exotermic surface reaction results in increasing the heating rate with temperature. In Fig.6 temperature kinetics of crystal covered with thin polimer film is shown when the film is etched in oxygen plasma and a polymer is transformed into volatile compounds. There is a direct connection between etching rate and heat release rate in chemical reaction.

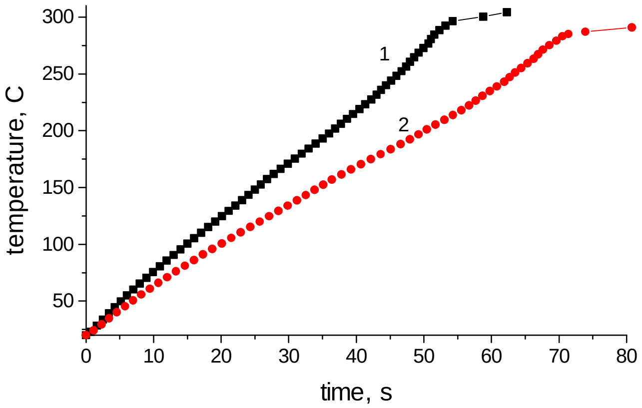

Figure 6. Temperature of silicon crystal covered with 1.5-μm-thick polymer film after discharge ignition in oxygen at 40 Pa. Applied power (W): 300 (1) and 200 (2). Duration of film etching is equal to 54 s at 300 W and 72 s at 200 W.

When the film is completely removed from the surface, a drastic decrease in the heating rate of crystal is observed (Fig.7). Since an etching rate over the crystal surface is non-uniform, the decrease of heating rate in the end of etching takes place in a some time interval (≈3÷5 s). This decrease has very short duration (≤1 s) when the etching rate is uniform over the entire surface. It is clear that the novel technique for end-point detection in plasma etching may be developed on the base of described phenomenon using noncontact temperature measurement of wafer [12].

So, the laser interferometric technique has overcame successfully some basic obstacles which were insuperable for developing the thermal diagnostics of plasma chemical reactions on the surfaces under plasma conditions using thermocouple and infrared pyrometry.

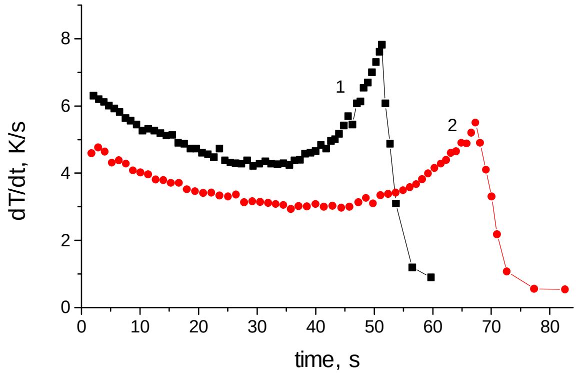

Figure 7. Temporal evolution of heating rate in plasma etching of polymer film on the silicon crystal. Sharp decrease in dT/dt at t ≈ 50 s (1) and t ≈ 70 s (2) is due to the end of exothermic surface reaction. Conditions and designations as in Fig.6.

5 CONCLUSION

Laser interferometric thermometry of transparent plates is under development for monitoring the wafers temperature in plasma processing and to research heat phenomena in plasma surface interaction. Using laser thermometry it has been possible to perform a first regular study of mechanisms and kinetics of heat transfer from plasma to surface.

REFERENCES

[1] A.N.Magunov, Laser thermometry of solids in plasmas, *Instruments and Experimental Techniques*, **43**(2) (2000).
[2] R.A.Bond, S.Dzioba, and H.M.Naguib, Temperature measurements of glass substrates during plasma etching, *J.Vac.Sci.Technol*. **18**(2) (1981) 335-338.
[3] V.M.Donnelly, D.E.Ibbotson, and C.-P.Chang, Interferometric thermometry measurements of silicon wafer temperatures during plasma processing, *J.Vac.Sci.Technol*. **10A** (1992) 1060-1064.
[4] A.N.Magunov and E.V.Mudrov, Silicon wafer temperature measurements with laser interferometry in a plasma chemical reactor, *High Temperature* **30**(2) (1992) 300-305.
[5] M.C.M. van de Sanden, A.D.Bailey III, J.A.Gregus, and R.A.Gottsho, Scaling of Si and GaAs trench etch rates with aspect ratio, feature width and substrate temperature, in: M.C.M. van de Sanden (ed.), *Abstracts of the 12th European Sect. Conf. on the Atomic and Molecular Physics of Ionized Gases* (Noordwijkerhout, The Netherlands, 1994), ECA. Vol.18E, p.396-397.
[6] R.Kurosaki, J.Kikuchi, Ya.Kobayashi, et al., In-situ Si wafer temperature measuring using pulse modulated infrared laser interferometric thermometry for CVD film deposition, *Proc.SPIE,* Vol.2635, 1995, p.224-233.
[7] A.N.Magunov, Scanning calorimeter for measurements in a nonequilibrium low-temperature plasma, *Plasma Physics Reports* **23**(11) (1997) 940-948.
[8] A.N.Magunov, Heat-transfer instabilities in the interaction of a nonequilibrium plasma with a solid surface, *Plasma Physics Reports* **25**(8) (1999) 646-651.
[9] C.Foissac, P.Supiot, O.Dessaux and P.Goudmand, Comparison of Pyrex plate and spectroscopic temperature measurements for gas temperature monitoring in a nitrogen afterglow, *Plasma Sources Sci.Technol*. **8**(4) (1999) 603-610.
[10] A.N.Magunov, Laser interferometric thermometry of semiconductors and dielectrics, *Instruments and Experimental Techniques*, **41**(3) (1998) 6-18.
[11] A.N.Magunov and S.V.Merkulov, Amplitude and shape of the Fabry-Perot optical resonances in laser interferometric thermometry of semiconductors and dielectrics, *Technical Physics*, **70**(2) (2000).
[12] O.V.Lukin and A.N.Magunov, Determination of the instant of etching completion by a calorimetric method, *Russian Microelectronics*, **27**(6) (1998) 378-385.

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