# OPTICAL MONITORING OF TECHNOLOGICAL PROCESSES FOR FABRICATION OF THIN-FILM NANOSTRUCTURES

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## *ABSTRACT*

Thisworkillustratesapplicationofthe uniquefiber-optic instrumentationforin situmonitoringofseveral technologicalprocessescommonlyusedinfabricationof semiconducting thin-film nanostructures. This instrumentation is basedonprinciplesoflow-coherenttandeminterferometry, whichdetermineshighsensitivityandprecision in measuring basic technological parameters, such as thickness of forming layers, temperature and bending of the substrate.The probing wavelength $\lambda = 1.55$ μm allows carrying out the measurements on majority of substrates for semiconductor technology: Si, SOI, GaAs, InP, GaP, $Al_2O_3$, diamond, $ZrO_2$:Y. Monitoring of such processes as MOVPE, MBEandplasmaetchingin various set-ups was realized. The absolute resolution achieved in these experiments was limited only by calibration accuracy and corresponds to $1^{\circ}$Cat sensitivity of $0.01^{\circ}$C. The accuracy limit in estimating the thickness of layers during their growth is 2 nm.

**Key words:** in situ characterization, optical monitoring, thing films, plasma etching, MBE, MOVPE

## *INTRODUCTION*

Modern technologies for manufacturing of micro- and nanostructures and devices demandabsolute reproducibility at each stage. Besides, efforts are made to improve the production output within a single process. Therefore, presence of reliable instrumentation for in situ control of the technologically important parameters [1] is mandatory. For most processes (including film deposition, annealing or etching of the finished thin film structures), fundamental parameters are the substrate temperature and the rate of growth or etching. Up to now, the substrate temperature is routinely controlled by thermocouples or pyrometers.The accuracy of these methods is fairly low. For the first class, the lack is absence of a good, reproducible thermal contact, especially at reduced pressure. In the second case, uncertainty of irradiation sources and significant variation

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in optical properties of the objects themselves during experiment are disadvantages. Thus, accurate determination of both temperature and rate of the epitaxial growth of semiconductors in the processes of molecular-beam epitaxy (MBE),metalorganic vapor phase epitaxy(MOVPE) or plasma etching (PE) represents a very important and still challenging problem.

### *OPERATING PRINCIPLE*

The proposed technique is based on the principle of low-coherent tandem interferometry (*Fig. 1*) [2, 3]. The instrumentation consists of a low-coherent source of light and two interferometers (delay lines) optically coupled via an optical fiber.

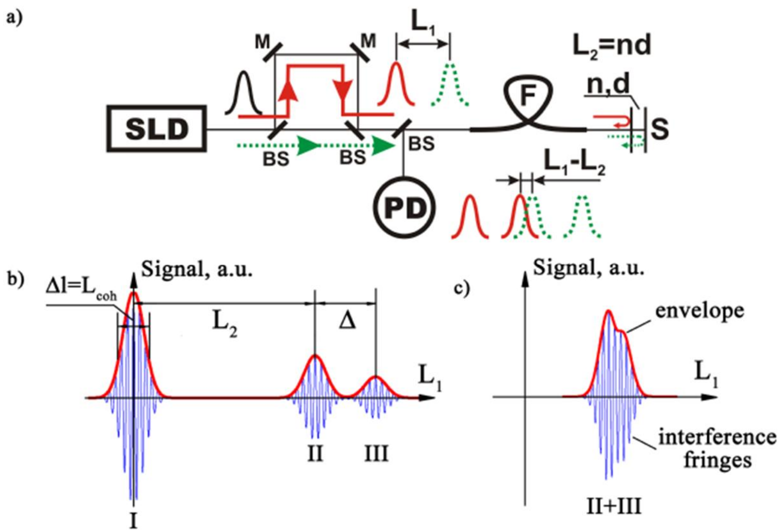


**Fig. 1**– Optical set up of the apparatus. SLD – a low coherent light source, PD – photodiode, F – optical fiber, BS – beam splitter, M – mirror, S – sample (wafer under test)

First delay line (reference) is placed in the measurement unit. Second delay line is essentially the wafer under testing and operates similarly to a Fabri-Perho interferometer. Two waves reflected from the front and rear surfaces of the wafer have a difference in the optical paths, which is equal to the doubled optical thickness of the wafer $L_2 = 2nD$. Hereinafter, $n$ and $D$ are the refraction index and the geometrical thickness of the wafer, correspondingly. Interference at the device output occurs if I) $L_1$ or $L_2 < L_{coh}$ and II) $\Delta L = |L_2-L_1| < L_{coh}$. Here, $L_1$ and $L_2$ are differences of optical path lengths (delays) of the fist and second interferometers, respectively, and $L_{coh}$is a coherence length of the light source.

*Fig.1b* shows the signal at the device output during tuning the length of the first delay line. Peak #I at $L_1 = 0$ corresponds to the first condition. It is determined by the autocorrelation function of the light source. Peak #II at $L_1 = L_2$ corresponds to the second condition. This peak is determined by the cross-correlation function of reference delay line and the wafer. Peak #III appears in presence of an additional reflecting surface located near either surface of wafer (*e.g.*, unruffled surface of the susceptor). If the gap between this additional surface and the surface of wafer Δ meets the conditions $\Delta < L_{coh}$, then peaks #I and #II merge giving one peak with distorted shape (*Fig.1c*). Similar distortions arise when additional layer with different refraction index is formed on the wafer surface. By taking this into account, absolute optical thickness of the wafer can be measured as a distance between positions of the envelope maximums of the Peaks #I and #II (measurements "along the envelope"). Similarly, value of the gap between wafer and susceptor is derived from the distance between Peaks #II and #III.

Position (phase) of interference fringes inside the Peak #2 is more sensitive to changes of the optical thickness and less sensitive to the form of the envelope curve. Therefore, recording of the phase shift of the interference fringes (measurements "along the phase") is preferable for the measurements of minor relative changes in optical thickness.

Thus, we are now able to monitor both growth (etching) process and the temperature of objects using the same experimental set-up. In the latter case, dependence of optical thickness $d$ on the temperature $T$ is used. It may be written as follows: $d = n(T)D(T) = d_0(T_0)(1+f(T))$. Here, $d_0(T_0)$ is optical thickness measured at the known temperature $T_0$, and $f(T)$ is a calibration curve.

For the InPsubstrates:

$$f_g(T) = 5\times10^{-8}T^2 + 8.1\times10^{-5}T - 8.6\times10^{-3} \text{ at } T_0 = 100\,^\circ C \quad (1)$$

$$f_{ph}(T) = 2.7\times10^{-8}T^2 + 5.9\times10^{-5}T - 6.2\times10^{-3} \text{ at } T_0 = 100\,^\circ C \quad (2)$$

Here and after $f_g(T)$ is the calibration curve for the phase velocity of the probe light and $f_{ph}(T)$is the calibration curve for the group velocity of the probe light.

For the GaPsubstrates:

$$f_g(T) = 3.6\times10^{-8}T^2 + 4.9\times10^{-5}T - 5.2\times10^{-3} \text{ at } T_0 = 100\,^\circ C \quad (3)$$

$$f_{ph}(T) = 1.8\times10^{-8}T^2 + 4.8\times10^{-5}T - 4.9\times10^{-3} \text{ at } T_0 = 100\,^\circ C \quad (4)$$

For the GaAssubstrates:

$$f_g(T) = 4.5\times10^{-8}T^2 + 9\times10^{-5}T - 9.4\times10^{-3} \text{ at } T_0 = 100\,^\circ C \quad (5)$$

$$f_{ph}(T)=3.1\times10^{-8}T^2+7\times10^{-5}T-7.3\times10^{-3}\text{ at } T_0=100\,^{\circ}\mathrm{C} \quad (6)$$

For the diamondsubstrates:

$$f_g(T)=3.9\times10^{-8}T^2+9.1\times10^{-7}T-3.9\times10^{-4}\text{ at } T_0=100\,^{\circ}\mathrm{C} \quad (7)$$

$$f_{ph}(T)=2\times10^{-8}T^2+3.9\times10^{-6}T-5.9\times10^{-3}\text{ at } T_0=100\,^{\circ}\mathrm{C} \quad (8)$$

For the $ZrO_2$:Ysubstrates:

$$f_g(T)=1.8\times10^{-8}T^2+6.4\times10^{-6}T-8.3\times10^{-4}\text{ at } T_0=100\,^{\circ}\mathrm{C} \quad (9)$$

$$f_{ph}(T)=5\times10^{-9}T^2+9.8\times10^{-6}T-1\times10^{-3}\text{ at } T_0=100\,^{\circ}\mathrm{C} \quad (10)$$

For the sapphire substratesвдиапазонетемператур400 – 1400 K:

$$f_g(T)=4.327\times10^{-9}T^2+1.451\times10^{-5}T-1.494\times10^{-3}\text{ at } T_0=100\,^{\circ}\mathrm{C} \quad (11)$$

$$f_{ph}(T)=3.364\times10^{-9}T^2+1.543\times10^{-5}T-1.577\times10^{-3}\text{ at } T_0=100\,^{\circ}\mathrm{C} \quad (12)$$

For the Si substrates в диапазоне температур300 – 800 K:

$$f_g(T)=3.469\times10^{-8}T^2+6.6327\times10^{-5}T-6.674\times10^{-3}\text{ at } T_0=100\,^{\circ}\mathrm{C} \quad (13)$$

$$f_{ph}(T)=2.117\times10^{-8}T^2+5.445\times10^{-5}T-5.669\times10^{-3}\text{ at } T_0=100\,^{\circ}\mathrm{C} \quad (14)$$

## *EXPERIMENTS*

Experiments were done in several types of reactors.GrowthofGaN based structures by MOVPEtechnique was performed in a home-made apparatus with a vertical-type reactor [4].Plasmachemicaletchingofsilicon-on-insulator (SOI) structures was carried out in aPlasmalab 80Plus reactor (Oxford Instruments). Thesereactorswereequippedwithaopticalwindow positioned in front of the measured sample, which provides normal incidence of probing light on the sample. GaAsstructures were grownusing MOVPEtechnique inEpiquipVP502-RPwith a horizontal-type reactor. Here, the inputofoptical irradiation was implemented through the reactor wall perpendicular to the sample. Finally, growthofSi/Ge structures was carried out by means ofMBEusing aRIBER station. Inthiscase, theprobinglightwasshedon sample at the angle of 45°. Toprovidetheinputandoutputoftheprobinglightinthe same optical fiber, a mirror was installed into the reactor.

### *RESULTS AND DISCUSSION*

*Fig. 2* presents “finger print” of growth of a GaN film on sapphire wafer.

In the inset to Fig. 2a, formation of the seeding GaN layer with thickness of 100 nm at 550 $^{o}$C is monitored for the first time (thickness variations “along the phase” are shown).

In *Fig. 3*, some results comparing the temperature of GaAs substrate in Epiquip reactor measured by a thermocouple and our instrumentation are given.

*Fig. 4* displays therecordofplasmachemicaletchingofthethree-layered SOI structure (a 500 µm thickn-Sisubstrate, a 1 µm thick $SiO_2$l ayer, and 1.5 µmthickp-Si layer) usingareactive$BCl_3$ – Armixture.

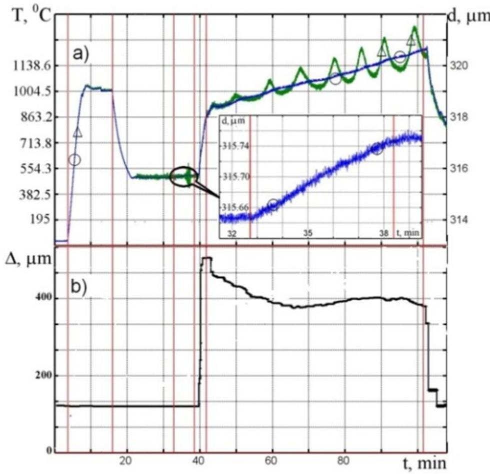


**Fig. 2** – Growthof a GaN film on sapphire wafer.a) Changes inopticalthicknessalong envelop (green line) and along phase (blueline). b) Changes inthick-nessof gap between susceptor plane and rear surface of wafer

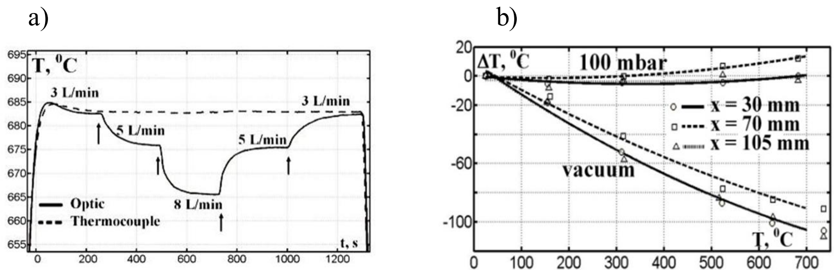


**Fig. 3** – Comparison of the GaAs substrate temperature.a) Changes ina substrate tem-perature due to changes flow of a carrier gas ($H_2$) were recorded by optics and not rec-orded by thermocouple. b) Difference in temperature measured by a thermocouple and our instrumentation (ΔT) on pressure in the chamber, as well as on the camera point (X = 0 is inlet of the chamber)

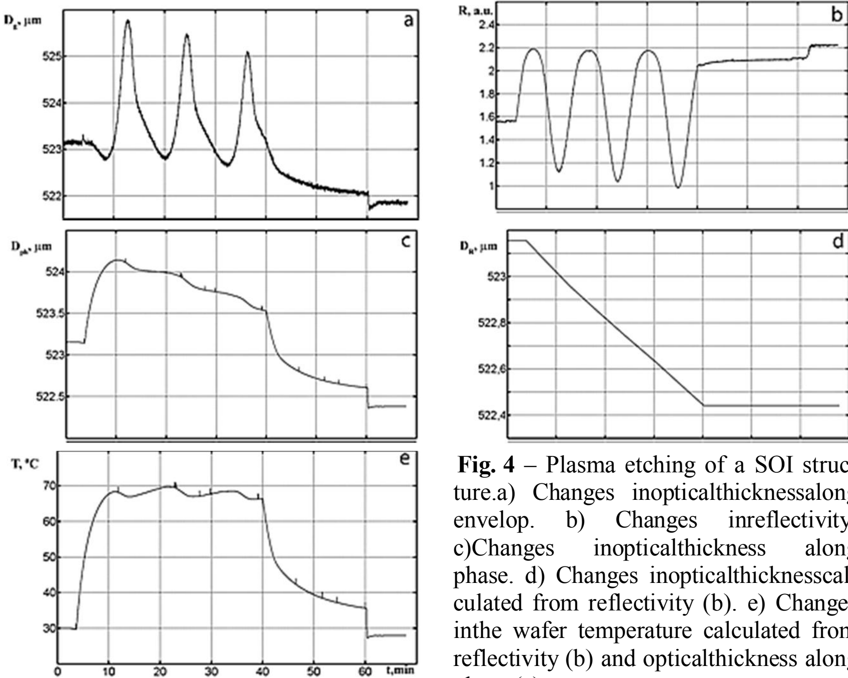


**Fig. 4** – Plasma etching of a SOI structure.a) Changes inopticalthicknessalong envelop. b) Changes inreflectivity. c)Changes inopticalthickness along phase. d) Changes inopticalthicknesscalculated from reflectivity (b). e) Changes inthe wafer temperature calculated from reflectivity (b) and opticalthickness along phase (c)

Asseenfrom Figure 4a, estimationofthicknesschangesalong the envelope seems difficult, since the observed oscillations have very complicated shape. However, ingeneralthepictureis similar to that observed when reflectivity factor (Fig. 4b) is registered: $I \sim \cos(kh+\varphi)$. Here $\varphi$ isanadditionalphaseshiftthatappearsduetolightinterferenceinunderlyinglayers, $k$is a wave number, and$h$is an optical thickness of the film.

In Figure 4a and 4c (changes “along phase”), the temperature induced jump of thickness during around 3600 seconds is seen, which is associated with rapid cooling of sample by incoming cold air from the room. As expected, reflectivity factor (Fig. 4b) is weakly affected by the temperature and depends exclusively on the layer thickness. In Figure 4d the changes in the layerthickness derived from variations of the reflectivity factor are plotted, whereas Figure 5e shows the changes in the substrate temperature that was calculated as difference between 4c and 4d. Atthat, the calibration data taken from Ref. [5] were used forconvertingofchangesinopticalthicknesstothetemperature.

### *CONCLUSIONS*

By using the novel principles of the optical thickness registration for transparent wafers, we introduced high-accuracy instrumentation for in situ

optical monitoring of the real substrate temperature (the accuracy is equal to ± 1°C) and bending (the accuracy is limited by the roughness of the susceptor surface, here it is equal to ± 3 μm), as well as the thickness (the accuracy is equal to ± 2 nm) of the growing (etching) layers during MOVPE, MBEand plasma etching processes.

Serious disagreement (10 – 100 °C) between the readings of the thermocouple and the temperature measured by the proposed optical method was observed in all cases. This disagreement rises with increasing substrate temperature and gas flow, as well as with decreasing pressure in the MOVPE reactor. InMBEconditions, thediscrepancy between the temperature values measured by a pyrometer and by our instrumentation was revealed.Disagreement rises with decreasing substrate temperature and with presence of hot parts inside the reactor. Inplasmaetchingconditions, thetemperaturemayriseupto 200 °Cdepending on the conditions of discharge maintenance and on presence/absence of the stage cooling.

It was shown that changes of overall optical thickness of the structures due to changing temperature can be distinguished from variations in physical thickness of the layer. To do this, the changes of amplitude of zero interference peak caused by an alteration of the interference conditions inside of the layer with increasing (decreasing) thickness should be accounted for. As result, simultaneous determination of both temperature and thickness of the treated layers becomes possible.

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