Journal of Crystal Growth 413 (2015) 42–45

Contents lists available at ScienceDirect

Journal of Crystal Growth

journal homepage: www.elsevier.com/locate/jcrysgro

Real-time measurement of substrate temperature in molecular beam epitaxy using low-coherence tandem interferometry

D.V. Yurasov [a,b,*], A.Yu. Luk'yanov [a], P.V. Volkov [a], A.V. Goryunov [a], A.D. Tertyshnik [a], M.N. Drozdov [a], A.V. Novikov [a,b]

[a] Institute for Physics of Microstructures, Russian Academy of Sciences, 603950, GSP–105, Nizhny Novgorod, Russia
[b] Lobachevsky State University of Nizhny Novgorod, 603950, 23 Prospekt Gagarina, Nizhny Novgorod, Russia

ARTICLE INFO

Article history:
Received 13 October 2014
Received in revised form
8 December 2014
Accepted 9 December 2014
Communicated by E. Calleja
Available online 17 December 2014

Keywords:
A1. Impurities
A3. Molecular beam epitaxy
B2. Semiconducting silicon
Low-coherence interferometry
Real-time temperature measurements

ABSTRACT

It was demonstrated that the low-coherence interferometry technique can be successfully applied to real-time substrate temperature evaluation during molecular beam epitaxy in a wide range down to room temperature. The proposed technique was used for formation of silicon layers delta-doped by antimony. Due to shortening of the growth interruptions needed for temperature switching the low-coherence interferometry technique allows improving the crystal quality of the grown samples and reducing the material and time consumption. These advantages become extremely beneficial with lowering of the growth temperatures.

1. Introduction

The temperature control during epitaxial growth is crucial because the properties of nanostructures often depend strongly on the temperature at which they were fabricated [1,2]. The modern trend of miniaturization of microelectronic components has also one specific consequence among the others – the tendency of lowering of the structures fabrication temperature. The latter is caused by the demands to limit the intermixing of materials in the heterostructures, smearing of the doping profiles, out-diffusion of dopants, etc. This makes use of moderate and low temperatures to be more and more important in the epitaxial growth processes [3–8]. In epitaxy, the calibrated thermocouples and pyrometers are usually employed substrate temperature measurements. They have some merits and demerits. The pyrometers have an advantage of real-time measurements but the temperatures they can detect are limited from below 400–500 °C (due to rapid decrease of the emissive power that is proportional to the fourth power of temperature). The thermocouples, after careful calibration (by eutectic and melting points of certain materials, see [9] for example), can measure temperature in a rather wide range but they have a grave disadvantage of time lag. As a result, the thermocouples can be used for the correct measurements of temperature only under steady-state conditions. So if the growth run requires the different layers to be grown at different temperatures one will be forced to interrupt the growth process and wait for the thermostabilization in order to define the temperature exactly. This shortcoming of thermocouples becomes extremely important at low temperatures, i.e. in the range where pyrometers are also ineffective. In some cases the time necessary for thermocouple stabilization can be several times longer than the time that is really required to cool or heat the sample to the desired value. Such time delays (from a few to tens of minutes) are often unwanted not only because of the increased material and power consumption but also due to the sample surface contamination. The latter may deteriorate the crystal quality of the grown structures and worsen their optical and electrical characteristics [1].

Some other non-invasive techniques of temperature measurements such as band-edge thermometry [10], fluoroptic thermometry [11], diffuse reflectance spectroscopy [12] or ellipsometry [13] were reported, but they are also not free from some drawbacks or technological difficulties in application. For example, the band-edge thermometry is difficult to use for the formation of heterostructures. The latter is connected with the fact that the absorption spectra of the deposited layers may sufficiently differ from the substrate absorption spectrum. Besides, the doping level may also influence the substrate absorption spectrum that requires additional calibration [14]. The interference effects additionally complicate the correct interpretation of the data obtained by the band-edge thermometry method [15]. The

* Corresponding author. Tel.: +7 831 4385037; fax: +7 831 4 609111.
E-mail address: Inquisitor@ipm.sci-nnov.ru (D.V. Yurasov).

http://dx.doi.org/10.1016/j.jcrysgro.2014.12.008
0022-0248/

more detailed review of the thermometry methods and their advantages and disadvantages can be found elsewhere [16]. So the real-time determination of the substrate temperature in epitaxy is challenging at low temperatures. In this article the method of low-coherence tandem interferometry is proposed in order to overcome this problem.

2. Experimental setup and measurement principle

The equipment for the substrate temperature evaluation consists of a low-coherence radiation source (superluminescent diode, SLD) and two interferometers (delay lines) optically coupled article an optical fiber (Fig. 1). The first delay line L_1 (reference) placed in the measurement unit (MU) is based on the scheme of Michelson interferometer. The second delay line $L_2 = 2nd\sqrt{1-\sin^2\varphi/n^2}$ is formed by two beams, reflected from the front and rear surfaces of the wafer under test (S). Hereinafter, n and d are the refraction index and the geometrical thickness of the wafer, φ - an angle of light incidence, L_1 and L_2 are the differences of the optical path lengths (delays) of the first and second interferometers, respectively. Due to the arrangement of the material sources in the Riber SIVA-21 MBE system used (the electron beam evaporators for Si and Ge are situated directly below the sample holder, that is typical for SiGe MBE machines) it is impossible to maintain the normal incidence of the laser beam on the sample surface which is the optimal condition for the measurements. Therefore the laser source and the detector have been placed outside the growth chamber, close to the inclined viewport, so the light angle was approximately 40 degrees. Additional mirror (M_{add}) mounted inside the growth chamber reflects the probe beams back to the wafer and to the optical fiber. The special protective screen is mounted close to the mirror in order to cover it from the material flux and prevent its coating. Interference at photo detector (PD) occurs if (I) L_1 or $L_2 < L_{coh}$ and (II) $\Delta L = |L_2 - L_1| < L_{coh}$. L_{coh} is a coherence length of the light source. Interference peak 1 (Fig. 1) at $L_1 = 0$ corresponds to the condition (I). It is determined by the autocorrelation function of the light source. Interference peak 2 at $L_1 = L_2$ corresponds to the condition (II). This peak is determined by the cross-correlation function of the reference delay line and the wafer. Absolute optical thickness of the wafer can be measured as a distance between the positions of the envelope maximums of the peak 1 and the peak 2. The temperature value can be obtained from the temperature dependence of the sum of wafer expansion and the wafer refraction index. Usually, the second term of this sum is greater than the first one by a factor of 10 [17]. The dependence of the optical thickness D on the temperature T can be written as follows:

$$D = n(T) \times d(T) = D_0(T_0) \times (1 + f(T)). \quad (1)$$

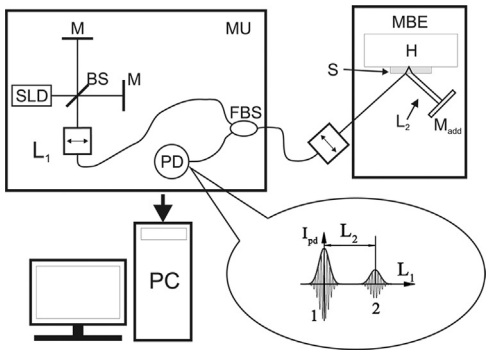

Fig. 1. The scheme of the experimental setup. MU- main unit, SLD – superluminescent diode, BS- beam splitter, M – mirror, FBS – fiber beam splitter, PD- photo diode, MBE – growth chamber, S- sample, H – heater, M_{add} – additional mirror, PC – personal computer with ADC and special software.

Here $D_0(T_0)$ is an optical thickness measured at a known temperature T_0, $f(T)$ is a calibration curve. This $f(T)$ curve is determined both by the wafer material and by the characteristics of the light source. The technique of $f(T)$ evaluation is presented in [18]. The ultimate accuracy of the proposed technique may reach the value of 0.1 °C. The main limiting factors are the inaccuracy of the measurements of the shoulder length difference in the reference interferometer and the inaccuracy of the temperature determination during the calibration curve evaluation. The difference between the optical parameters of the deposited layer and the substrate may also introduce some uncertainty, but in most cases the thickness of the grown layer is much smaller than the substrate thickness so such an uncertainty is very small and may be neglected.

Under existing conditions the accuracy was mainly determined by the inexactness of the initial temperature setting and can be estimated as ± 2 °C.

The measurement technique [19] and the results of its employment for the monitoring of the technological parameters in MOVPE and plasma-chemical treatment processes are described in more detail in Refs. 20–22. Recently, the applicability of the proposed technique to the molecular beam epitaxy (MBE) was examined [23]. In the current article the low-coherence tandem interferometry method of temperature evaluation was applied for the first time for real MBE growth process. All experiments were made using a solid source MBE system Riber SIVA-21 designed for the growth of Si-based structures. The MBE system was equipped by two e-guns for Si and Ge evaporation, effusion cells for donor (Sb) and acceptor (B) impurities. A W-Re thermocouple and an infrared pyrometer IMPAC IS 12 specially developed for silicon served as the standard technical instrumentation for the temperature measurements.

3. Results and discussion.

As was shown above the proposed approach for the temperature measurement does not require any fitting parameters. One only needs to know the calibration curve for the substrate material $f(T)$ (1). This curve for a Si wafer and used SLD with the central wavelength of 1545 nm and the bandwidth of 60 nm was measured separately, prior to all other experiments using the method presented in [18].

The data obtained using the interferometer were at first compared with the values measured by the IR pyrometer. The measurements were carried out as follows. After loading of a double-side polished Si(001) substrate to the growth chamber the starting temperature point (room temperature) was set. Then the standard thermal annealing at 800 °C was performed and the sample was cooled down to the first fixed value $T_{tc} = 50$ °C. The corresponding value of T_{int} was recorded. After that the sample was slowly heated up to the next fixed value given by the thermocouple (T_{tc}=const) and kept up at this temperature for several minutes for the thermal stabilization. After stabilization period the values given by the interferometer (T_{int}) and by the pyrometer (if possible) (T_{pyr}) were measured. Then the sample was heated again up to the next point (next fixed T_{tc}) and the procedure repeated. The whole data set (which represents in fact two calibration curves) obtained as was described above is presented in Fig. 2.

One can see that the values given by the interferometer and by the pyrometer agree quite well with each other in the whole temperature range where both methods are valid (450-600 °C). The temperatures below 450 °C cannot be accurately measured using the pyrometer (because it's close to the lower limit of pyrometers working range), while the temperatures above 600 °C are difficult to measure using the interferometer due to