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Use of SiC band gap temperature dependence for absolute calibration of emissivity corrected pyrometers in III-nitride MOVPE

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Abstract

In this paper we will demonstrate a new method for temperature calibration by using the in-situ measured band-gap shift of SiC in conjunction with real-time emissivity corrected pyrometry. The complete procedure for temperature calibration and real-time wafer temperature measurement on transparent substrates will be presented.

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1. Introduction

Accurate monitoring of growth temperatures is essential during GaN metalorganic vapour-phase epitaxy (MOVPE) growth. The layer qualities, growth rates, abruptness of heterointerfaces and ternary alloy compositions are all dependent on growth temperature. Hence, in-situ measurement and control of the substrate temperature in nitride MOVPE is crucial for obtaining reproducible layer quality.

Thermocouples and pyrometers are the most widely used tools for measuring and controlling the process temperature. Thermocouples are often inaccurate because they make poor thermal contact with the sample. Due to susceptor and/or wafer rotation the thermocouple is far away from the growing layer. Therefore, the true wafer

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temperature has an offset to the process control temperature. This offset is not constant since changes in the susceptor setup or increasing reactor wall coatings and even different rotation gas flows alter the actual wafer surface temperature [1] and hence, this offset cannot be compensated for by calibration. All these indicated issues also hold for light-pipe sensors, which pyrometrically sense the temperature *indirectly* from underneath the susceptor setup. *Direct* pyrometry is also complicated due to the fact that optical interference occurs since the index of refraction of the substrate differs from the deposited film. Thus, the emissivity "changes".

Real-time emissivity corrected direct pyrometry [2] of the susceptor overcomes these problems. However, since optically transparent substrates such as sapphire or SiC are required for GaN MOVPE growth, the measured surface temperature represents the susceptor surface temperature and has to be corrected for the temperature difference between wafer and susceptor.

Presently, the most effective methods to calibrate real-time emissivity corrected pyrometers are procedures based on melting points or phase transitions [3]. To the latter one belongs the very convenient method based on the Si-Al-eutectic formation at $T = 577\,^{\circ}\mathrm{C}$. This phase transition of a thin Al layer on Si is accompanied by a sudden roughening of the surface, which leads to a dramatic loss in reflectance that can be seen with the naked eye, or more conveniently, can be detected by a reflectance sensor. However this method is rather time consuming and yields only one data point at 577 °C. The calibration accuracy can be influenced by Al-layer thickness. In addition in RF-heated MOVPE systems the melting temperature can be erroneous when the highly conductive Al-layer is heated by induction. It will be shown that melting/roughening temperature is then reached earlier. Hence this calibration method cannot be employed in RF-heated MOVPE systems.

In this paper we will demonstrate a new method for temperature calibration by using the in-situ measured temperature-dependent band-gap shift of SiC in conjunction with real-time emissivity corrected pyrometry. The complete procedure for temperature calibration and real-time wafer temperature measurement on transparent substrates will be presented.

This in turn will allow us to reach exactly the same run-by-run wafer temperature in any given MOVPE system. Especially for process transfer to other reactors, the accurate calibration of the pyrometer to the absolute temperature is then achieved. Thus, process temperature finally becomes directly comparable.

2. Experimental procedure

All experiments have been conducted in Aixtron horizontal MOVPE reactors at Forschungszentrum Jülich, FZJ (AIX 200/4 RF-S) and Ferdinand Braun Institute Berlin, FBH (AIX 200/4). Both MOVPE systems were equipped with low-strain UV transparent view ports for normal incidence optical access and with corresponding holes in the liner-tube.

The optical measurements at FZJ were performed with an EpiR M TT optical in-situ sensor having two separate measurement channels for emissivity corrected pyrometry and high accuracy reflectance measurements. Thus, two complementary physical quantities can be measured in real-time during the entire growth process: the true wafer temperature and the reflectance of the growing layer stack. The measurement is performed at 950 nm using a broad band detection, allowing detection of wafer temperatures above 450 °C [4].

At FBH the in-situ measurements were performed with a LayTec EpiRAS-200 spectrometer that allows combined reflectance anisotropy spectroscopy (RAS) [5] and reflectance (R) measurements between 826 nm (1.5 eV) and 248 nm (5.0 eV).

A notable difference in the epitaxy equipment is the heating system. While the FBH reactor is heated via irradiation of the susceptor with IR lamps the FZJ system is inductively heated via a RF-coil situated underneath the susceptor setup. Temperature control is achieved by a thermocouple (FBH) or a light pipe sensor (FZJ) as described above.

The calibration runs at FZJ were performed under standard GaN epitaxy conditions (rotating sample) under H2 ambient. Using gas foil rotation the samples' rotation frequencies varied between 1 and 2 Hz. The temperature calibration at FBH was conducted by means of phase transition measurements as described above with a *non*-rotating susceptor in order to exclude cooling effects due to rotation gas flow. The measurement error due to the phase transition calibration is assumed to be less than ±2 K. For the absolute temperature calibration based on the determination of the eutectic or melting-point at FZJ 150 nm thin layers of Ag and Au on both sapphire and Si substrates were used in order to achieve data points in different temperature regimes close to GaN epitaxy conditions. The melting point for Ag and Au on sapphire wafers is 962 and 1063 °C, respectively. The Phase transition of Ag and Au on Si occurs at 830 and 370 °C, respectively [6]. These samples have been carefully and stepwise heated up to their respective melting or phase transition points.

The temperature-dependent SiC band gap shift measurement were carried out using two inch, double side polished, 450 μm thick, 6H-SiC substrate, supplied by NovaSiC. In this set of experiments reflectance spectra with a resolution of 0.01 eV were recorded for a range of true temperatures at FBH and nominal "light pipe" temperatures at FZJ.

3. Results and discussion

3.1. Principle of the temperature measurement technique

The pyrometry measurement is based on the detection of the incandescence from the wafer according to the Planck equation and Kirchhoff's law [7,8].

Using the Wien approximation [for $\lambda T < \lambda_{\max} T$] the inferred temperature from the surface is determined as

$$T_{\text{surface}} = \frac{1}{T_\lambda} + \frac{\lambda}{c_2}\ln(\varepsilon_\lambda) \tag{1}$$

with T_{surface} as the effective surface temperature, λ is the detection wavelength, T_λ as the spectral radiance temperature and ε_λ as the spectral emissivity [9].

From Eq. (1) it is obvious that successful pyrometry requires accurate knowledge of the spectral directional emissivity of the target. Since the emissivity of the target depends upon such factors as the incident light angle, material, temperature, surface roughness, doping level, dopant used, etc., it is not always appropriate to assume that the emissivity is a known quantity. Even the roughness of the unpolished side of a wafer can alter the emissivity [10]. Additionally, the target emissivity can change during growth as the epitaxial structure is deposited. This changing emissivity effect will produce an error when using a pyrometer to measure substrate temperature. These issues are overcome with emissivity corrected pyrometry. Simply stated, this in-situ measurement technique requires the combined functionality of a conventional pyrometer and a reflectometer. The reflectance of the substrate is measured at the same wavelength at which the pyrometer measures the thermal radiance. By using the equation

$$E = 1 - R, \tag{2}$$

the spectral directional emissivity (E) of the substrate surface can be calculated from the measured spectral directional reflectivity (R). Now, it is apparent why transparent substrates cannot be measured pyrometrically. At the detection wavelength of 950 nm the optical radiation from the susceptor passes through the transparent substrate and the pyrometer cannot distinguish between substrate radiation and susceptor radiation. The second problem is that the substrate emissivity at our detection wavelength is negligible yielding no usable blackbody radiance. In order to determine the temperature difference between susceptor and wafer we followed two approaches.

3.2. Calibration using phase transition/melting point

In order to calibrate the pyrometer for $\Delta T_{\text{wafer,suceptor}}$ experiments with different phase

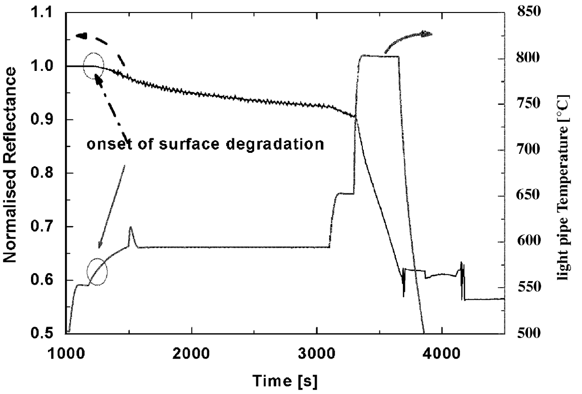

Fig. 1. Reflectance and temperature transient of calibration run based on melting point determination of a thin Ag layer on sapphire. The unexpected and untimely slow degradation of the Ag-layer at a temperature of 570 °C is highlighted.

transitions and melting points, as mentioned above, were used.

Fig. 1 displays the reflectance transient for the melting point experiment Ag on sapphire as well as the respective "lightpipe" temperature, which yields a higher temperature than the actual surface bears. The sudden degradation of the Ag-layer surface is expected at 962 °C. However, surface roughening occurs already at lower "lightpipe" temperatures of about 570 °C. Keeping temperature constant at 590 °C the reflectance slowly drops. With increasing temperature this effect becomes more evident and at around 730 °C an abrupt change in the reflectance is observed. Apparently there is a huge difference between the nominal melting point and the measured actual value.

We attribute this untimely slow degradation of the surface to inductive heating of the metallic film due to the RF-heating of the susceptor material. However, this method of absolute temperature calibration is not applicable for *our* RF-heated system and another method for absolute temperature calibration is necessary.

3.3. Band gap temperature dependence

The band gap of many IV and III–V materials varies strongly with temperature. Therefore, spectral reflectometry holds possibilities for utilizing this shift to reference the substrate temperature during MOVPE growth [11–13]. The band gap can be determined from the radiation (spectrally analysed in the EpiR M TT) reflected from the susceptor and transmitted through the substrate. Since double side polished SiC substrates were used the detected reflectance provides a larger signal level. The first set of experiments is conducted at FBH, where the IR heating system allows the absolute temperature calibration by phase transition measurements as described above. Thus, the detected temperature is the true surface temperature.

Fig. 2 displays the inferred reflectance spectra from 2.0 to 4.0 eV for an unintentionally doped SiC wafer for a range of temperatures. The spectra are normalized and have been referenced to the room temperature spectrum. Thus, the difference in reflectance due to the different transmission/absorption is emphasized. These spectra display a sharp falling edge which corresponds to the onset of absorption in the layer. This onset is correlated to the band gap. With increasing substrate temperature the position of the falling edge moves to lower energies as the band gap decreases. Using a suitable fit function (straight lines in Fig. 2) the position of the absorption onset versus the absolute temperature was determined as seen in Fig. 3.

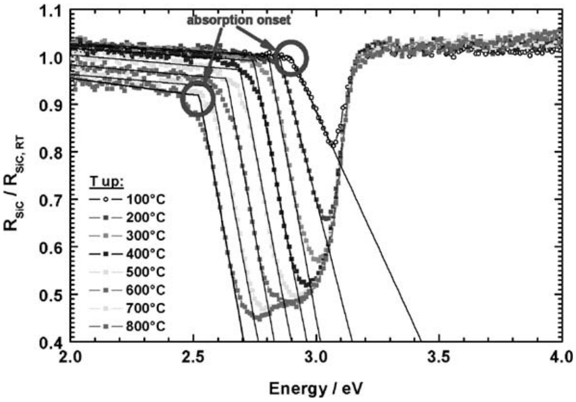

Fig. 2. Normalised reflectance spectra referenced to the room temperature spectrum of an undoped double side polished SiC wafer for a range of absolute temperatures. The straight lines represent the fit function used to determine the onset of the absorption.

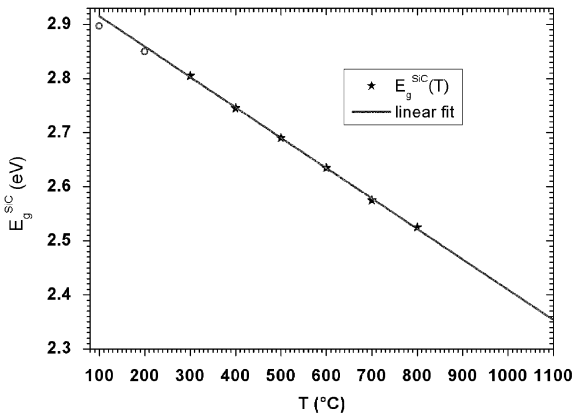

Fig. 3. The resulting correlation between absorption onset and absolute temperature as found at Ferdinand-Braun-Institut.

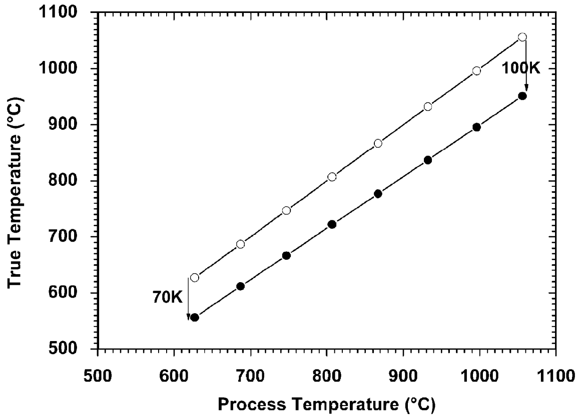

Fig. 4. Referencing the temperature at FZJ, empty circles, to the absolute temperature of FBH, full circles. The true surface temperature of the SiC wafer at a process control temperature $T1 = 620\,°C$ is at $550\,°C$ and for $T1 = 1050\,°C$ it is $950\,°C$.

Now, a gauging normal is established since the correlation of the band gap shift (or more correctly "absorption onset") with the absolute temperature is determined. Using this reference the reactor at FZJ can be calibrated. For that purpose the SiC measurement were repeated in the FZJ reactor in a similar way and the absorption onset was determined. Fig. 4 shows the comparison of the two resulting band gap plots versus the temperature. The resulting process temperature correction for the reactor at FZJ is displayed. The true surface temperature of the SiC wafer at a process control temperature $T1 = 620\,°C$ is at $550\,°C$ and for $T1 = 1050\,°C$ it is $950\,°C$. Assuming that sapphire wafers of equal thickness qualitatively show the same thermal behaviour our calibration method can also be employed to determine the surface temperature of sapphire wafers during growth. Once calibrated in this manner our reactor measures the correct wafer temperature for all growth runs on SiC and sapphire, provided that the wafer is not bent or its surface is roughened. Now this calibration method can be applied each time the reactor geometry or sensor position is changed.

4. Conclusions and outlook

In the foregoing discussion, it was shown that direct temperature measurement is a must though it is challenging for transparent substrates since calibration for the temperature offset susceptor/wafer is necessary. When using RF heated systems great care must be exercised when using a calibration based on melting point or phase transition measurements. A new method for temperature calibration by using the in-situ measured band-gap shift of SiC in conjunction with real-time emissivity corrected pyrometry for transparent substrates has been demonstrated. Direct transfer of process temperature to any reactor now becomes possible.

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