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Use of wafer temperature determination for the study of unintentional parameter influences for the MOVPE of III-nitrides

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In this paper we will first report on the use of real-time determination of wafer temperature for transparent substrates. With this method we will study the unintentional influence of growth parameter variations on the surface temperature. The effect on nitride growth optimization will be discussed.

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1 Introduction

The growth of III-nitrides with high crystalline perfection is a great challenge since there is a lack of commercially available native substrates on which homoepitaxial growth could be performed. Usually transparent substrates are employed such as sapphire or SiC which exhibit a lattice mismatch of up to 16% with respect to GaN. In order to deposit high quality material by MOVPE (metalorganic vapor phase epitaxy), GaN buffer growth strategies such as the two step procedure proposed by Amano et al. [1] are employed. First a nearly amorphous GaN layer about 25 nm thick is deposited at temperatures of around 500 °C. Then in a second step after annealing deposition is resumed at temperatures of about 1000 °C and above. Both growth as well as morphology development are strongly influenced by temperature. Furthermore the reactor pressure, the ammonia to trimethylgallium (TMGa) partial pressure ratio (V/III ratio) and the ambient – usually hydrogen or nitrogen – strongly influence growth and need to be optimized. The latter has been known to unintentionally alter the surface temperature in MOVPE [2, 3]. The knowledge and exact control of the substrate temperature is a necessity for parameter optimization but quite difficult to achieve. The methods commonly used are thermocouple measurements and pyrometry. Thermocouple measurements, however, are carried out far away from the growth front due to rotation of the substrates. Pyrometry is not exact due to changes in emissivity if materials of different refractive index are deposited on top of each other. Additionally if transparent substrates are used, the thermal radiance, cannot be measured directly by pyrometry. The pyrometer "sees" the radiation which originates from the underlying susceptor and passes through the substrate. The radiation is weakened by the substrate itself as well as by the scattering due to its unpolished back side. The demand can, however, be solved by employing an in situ optical sensor which incorporates a reflectometer together with a pyrometer allowing the correction of emissivity changes during growth [4]. It is then possible to simultaneously observe the morphology development and to separately determine the growth temperature. In addi-

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tion it becomes possible to see if the surface temperature unintentionally changes during the variation of other growth parameters. In this paper we will report on the measurement of the temperature for transparent substrates, which subsequently calls for calibration. It has been reported earlier [5] that the normal temperature calibration – the determination of the melting point or a eutectic point – cannot be carried out in all inductively heated reactors. Therefore a different calibration strategy will be described. In the second part of the paper we will deal with the unintentional influence of growth parameters on substrate temperature.

2 Experimental details

The temperature calibration experiments were conducted in two different horizontal Aixtron MOVPE reactors equipped with low strain UV transparent view ports for normal incidence optical access. In the first reactor, an AIX 200/4 RF-S for nitride growth, the substrates are heated inductively. The heating system is controlled by a light pipe sensor. The reactor is equipped with an EpiR M TT (LayTec) optical in-situ sensor which has two separate measurement channels for emissivity corrected pyrometry and high accuracy reflectance measurements, respectively. The true wafer temperature measurement is performed at 980 nm using a broad band detector, allowing detection of wafer temperatures above 450 °C. In the second reactor, an AIX 200/4 for conventional III–V growth, the substrates are heated by infrared radiation. Its heating system is controlled by a thermocouple measurement. This reactor is equipped with a LayTec EpiRAS-200 spectrometer that allows combined reflectance anisotropy spectroscopy (RAS) [6] and reflectance (R) measurements between 826 nm (1.5 eV) and 248 nm (5.0 eV). In the nitride reactor the calibration runs were carried out with substrate rotation and H_2 as the ambient using conventional two inch one side polished sapphire substrates as well as two inch, double side polished, 450 μm thick, un-doped 6H-SiC substrate. The temperature dependence of the SiC band gap was measured for the calibration. The temperature calibration in the reference reactor was conducted by means of phase transition measurements as described above with a *non* rotating substrate in order to exclude cooling effects due to the rotation gas flow. The measurement error due to the phase transition calibration is assumed to be ≤2 °C in this reactor. The temperature dependence of the SiC band gap was measured in addition [5].

The second set of experiments was only carried out in the temperature calibrated nitride reactor without introducing TMGa to the with GaN preconditioned reactor, however, using typical growth conditions. This horizontal reactor is equipped with a straight separation plate in the gas inlet which allows the separate injection of the TMGa and the ammonia. The standard inlet configuration was used in which the ammonia is injected closer to the substrate surface through the lower injection channel. A two inch one side polished α-sapphire wafer was placed in the reactor. At a total flow of 9 slm H_2 ambient the pressure was varied from 50–1000 mbar. Two different process control temperatures T_P were chosen, 1000 °C and 1100 °C. At a reactor pressure of 200 mbar and a total flow of 9 slm the ammonia content in the gas phase was varied from 0 to 3 slm and the nitrogen to hydrogen ambient gas ratio was varied from 0 to 100%. Finally at 200 mbar reactor the total flow in the reactor was varied from 5 to 14 slm. The last three experiments were carried out at 1100 °C process control temperature.

3 Results and discussion

3.1 Determination of true surface temperature

The pyrometry measurement is based on the detection of the incandescence from the wafer [7, 8]. Successful pyrometry requires the accurate knowledge of the emissivity of the substrate. As soon as growth is carried out, the emissivity changes. By recording the reflectance at the same wavelength as the pyrometer wavelength – in our case at 980 nm – the emissivity can be determined and used to correct the pyrometry output. For transparent substrates, the temperature difference between susceptor and substrate surface needs to be taken into account. To this end the strong band gap dependence of a semiconductor on temperature can be employed. In reflectometry a sudden decrease in reflectance is observed when the energy approaches the band gap. This offers the possibility of correlating the absorption onset energy to

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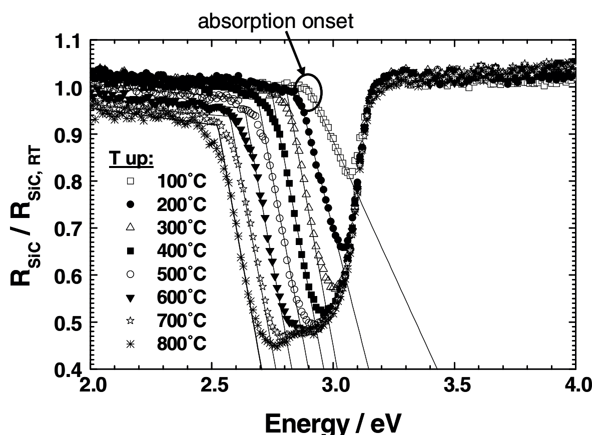

Fig. 1 Normalized reflectance spectra of an un-doped SiC wafer for a range of temperatures. The straight lines represent the fit function used to determine the onset of the absorption.

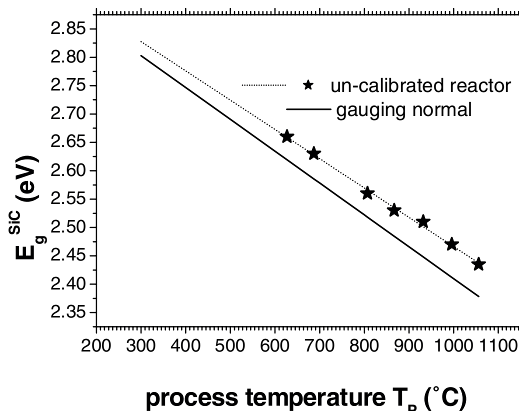

Fig. 2 Correlation between absorption onset and absolute temperature: full line gauging normal; dotted line is a fit for the measurements in the un-calibrated reactor.

the temperature of the wafer surface [9–11]. Therefore the reflectance spectra of a double side polished un-doped (transparent) SiC substrate were recorded as a function of temperature [5]. First the measurements were carried out in a reactor in which the exact wafer temperature was calibrated by means of a melting point measurement with an error below 2 °C. The reflectance spectra were normalized to a room temperature spectrum in order to enhance changes in the reflectance. The onset of absorption is clearly seen by a sharp falling edge in the normalized reflectance. 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. 1) the position of the absorption onset versus the absolute temperature was determined. The respective band gap dependence on temperature is presented in Fig. 2 as a full line for the reference reactor. Now, a gauging normal is established since the correlation of the absorption onset with the absolute temperature is determined. Then the measurement was repeated for the un-calibrated reactor. Using the gauging normal the reactor can now be calibrated with an accuracy in the absolute temperature within 3 °C. Assuming that sapphire wafers of equal thickness qualitatively show the same thermal behavior the calibration method can also be employed to determine the surface temperature of sapphire wafers during growth. Once calibrated in this manner the emissivity corrected pyrometer measures the correct wafer temperature for all growth runs on transparent substrates such as SiC and sapphire, provided that the wafer does not exhibit curvature or its surface is roughened.

3.2 Influence of growth parameters on temperature

3.2.1 Influence of pressure

There have been some reports [12, 13] that the quality of (GaIn)N layers can be greatly improved if the reactor pressure is increased. Before starting such an investigation it has to be verified that the substrate surface temperature does not change during the increase of reactor pressure. In this study the influence of the reactor pressure on the substrate temperature was investigated. In order to omit any influence of the height of our rotation disc with respect to the inductive field, the rotation was stopped. At a process control temperature T_P of 1100 °C and at 1000 °C the reactor pressure was varied. The influence is presented in Fig. 3. For both process control temperatures the surface temperature increases strongly with pressure by about 30 °C until 400 mbar is reached and then stays nearly constant above this pressure. By increasing the pressure in the reactor the heat is transferred more efficiently from the susceptor to the gas phase. A cooling effect on the process control temperature is to be expected. Indeed the heating power is increased in the reactor to keep the process control constant (not shown here). The surface temperature,

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however, does not stay constant. The heating power increase is higher than necessary to keep the surface temperature constant. Obviously the heat transfer towards the substrate is stronger than towards the pyrometer situated quite far away from the substrate. One reason may be the reflection of radiation back from the parasitic deposition of the reactor ceiling and walls towards the substrate. A heat transfer from the gas phase back to the substrate could also be possible. The observed temperature difference of 37 °C would surely influence growth strongly since the growth regime would be shifted more clearly from the diffusion controlled into the thermodynamically controlled regime [14].

3.2.2 Influence of total flow

The gas velocity is an important parameter with which the layer uniformity can be tuned in horizontal reactors [15]. Especially for Al-containing nitrides it is important to reduce the residence time in the reactor in order to avoid a strong depletion of the nutrients before the susceptor. Therefore the influence of total flow for both hydrogen and nitrogen ambient on temperature was investigated at 200 mbar reactor pressure and 1100 °C process control temperature. For both ambients only a weak linear decrease in surface temperature was found of 7 °C and 4 °C for H_2 and N_2 ambient, respectively, when the total flow in the reactor was increased from 5 slm to 14 slm. It would be expected that the increasing total flow rate has a cooling effect in the reactor since heat transport away from the substrate increases with the flow rates and would be more efficient for hydrogen as for nitrogen gas due to the sevenfold higher thermal conductivity of hydrogen. In this case the process control temperature does not "notice" the slight temperature change in the reactor. The observed temperature variation, however, is small and will not have a big influence on growth.

3.2.3 Influence of ammonia content

Next we studied the possible influence of NH_3 content in both hydrogen and nitrogen ambient. The thermal conductivity of ammonia is between that of hydrogen and nitrogen. If nitrogen is used as the ambient the temperature *increases* with the increase of ammonia content by 20 °C. If hydrogen is used, a slight tendency for temperature *decrease* is observed. Figure 4 shows this comparison for the two different ambients and the influence of ammonia on the surface temperature. If the nitrogen ambient is exchanged in part by ammonia the average thermal conductivity of the ambient is increased so that a cooling effect in the reactor is to be expected. If hydrogen ambient is partially exchanged for ammonia the average thermal conductivity of the gas decreases and a warming effect in the reactor can be expected. These effects are indeed noticed by the process control pyrometer. The heating system reacts

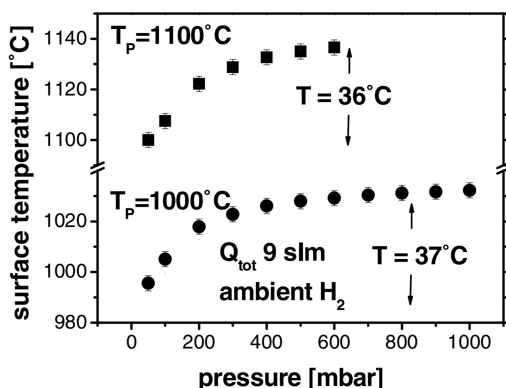

Fig. 3 Influence of reactor pressure on surface temperature at 9 slm total flow, hydrogen ambient and 1100 °C and 1000 °C process control temperature T_P.

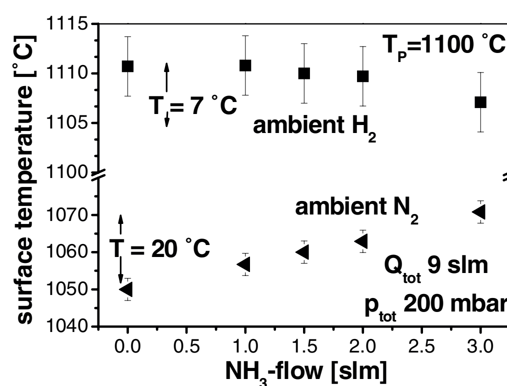

Fig. 4 Influence of ammonia content at 9 slm total flow, 1100 °C process control temperature T_P for both nitrogen and hydrogen ambient.

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with an increase in heating power with increasing ammonia content for the nitrogen ambient and a decrease for the hydrogen ambient. The substrate surface temperature, however, does not stay constant. Obviously again the heat transfer influence towards and through the substrate (detected sensitively by the true temperature measurement system) is stronger than towards the process control pyrometer situated quite far away from the substrate. Further investigations are necessary to clarify the exact origin of the temperature variation.

3.2.4 Influence of nitrogen/hydrogen ambient ratio

Finally the ambient composition was varied from pure hydrogen to pure nitrogen. The thermal conductivity of hydrogen is seven times as high as that of nitrogen. The experiment is of interest for In-containing compounds since their growth is preferentially performed in nitrogen ambient. Figure 5 presents the recorded influence of the nitrogen content on the substrate surface temperature. All in all a strong decrease of surface temperature of 50 °C is observed as the hydrogen ambient content decreases and the ambient gradually changes to nitrogen. The influence of nitrogen is at first small. As soon as the nitrogen content in the gas phase surpasses 1/3 of the total gas flow, the decrease in surface temperature becomes large. The temperature drop is even so large for the pure nitrogen ambient that a smooth layer growth is not to be expected. This may be the explanation for reports on the inferior morphology of GaN layers grown in nitrogen carrier gas compared to that of hydrogen carrier gas [16, 17]. The thermal conductivity of the gas phase again apparently plays an important role and seems to be correlated to the observed decrease in substrate temperature. The heating power used for keeping the process control temperature constant is also include in Fig. 5. As soon as the thermal conductivity of the ambient decreases the light pipe measurement used for the process control "sees" a higher temperature and therefore decreases the heating power for the inductive heating system. The reduction in heating power is too much since the surface temperature decreases. Even if the heating power is kept constant at a value used to obtain a process control temperature of 1100 °C, the surface temperature as well as the process control temperature increase when the nitrogen content of the gas phase increases and therefore the thermal conductivity decreases. This observation is presented in Fig. 6. After the gas takes up heat – the amount is related to its thermal conductivity –, the heat is transferred back to the substrate surface which is not detected by the light pipe measurement. It is therefore not sufficient to keep the heating power of the system constant during the parameter variations in order to have a constant surface temperature. The real-time determination and control of the substrate temperature is necessary if the influence of a parameter is to be studied.

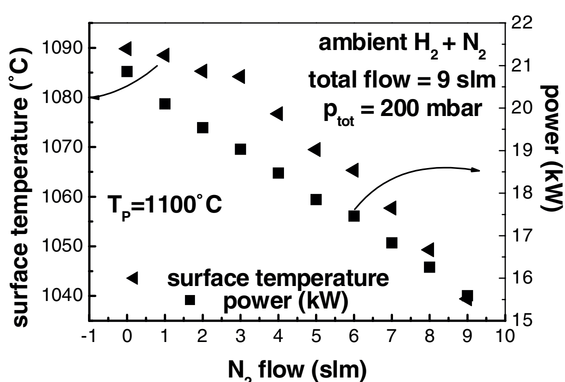

Fig. 5 Influence of nitrogen content on surface temperature at 9 slm total flow, hydrogen ambient and 1100 °C process control temperature. The heating power for the different ambient mixtures is also presented.

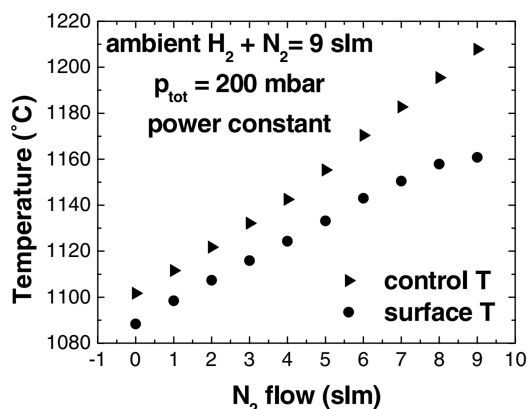

Fig. 6 Influence of nitrogen content on surface and process control temperature at 9 slm total flow, hydrogen ambient and constant heating power.

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4 Summary and conclusions

It was demonstrated that by using reflectance measurements together with emissivity corrected pyrometry absolute substrate surface temperatures could be determined in the MOVPE reactor on transparent substrates. With this substrate surface temperature determination method the influence of reactor pressure, total flow, ammonia content in the gas phase as well as the nitrogen to hydrogen ambient ratio on temperature were investigated. An unintentional influence of growth parameters on surface temperature was observed. A correlation was found between the ability of the gas phase to transfer heat and the change of surface temperature at otherwise constant process temperature. The higher the heat transfer ability is, the higher the surface temperature. The exact origin of the observation is not yet quite clear. Further investigations such as modelling experiments are to be carried out to help understand the unintentional variation of the surface temperature by parameter variations.

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