



Desarrollo de Hardware y Software de un Sistema de Espectroscopia de Reflectancia Difusa (DRS) para Medir la Temperatura del Sustrato Semiconductor in-situ

Hardware and Software Development of a Diffuse Reflectance Spectroscopy (DRS) System to Measure Temperature of Semiconductor Substrates in-situ

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Resumen

La medición y el control de la temperatura del sustrato es una dificultad común para la técnica de epitaxia de haz molecular "Molecular Beam Epitaxy"(MBE), así como para otras técnicas de deposición de semiconductores. En este trabajo se presenta el desarrollo de un sistema de espectroscopía de reflectancia difusa (DRS) como método alternativo para la medición de la temperatura de materiales semiconductores en una cámara de ultra alto vacío (UHV). DRS es un método óptico para monitorear la temperatura de materiales semiconductores durante el proceso de crecimiento en una cámara de MBE. El sistema utilizado ha sido calibrado y probado para sustratos de tipo n-GaAs y de SI-GaAs. Se ha logrado una sensibilidad de 2 °C para el sistema. La precisión de la medida está limitada por la precisión con la que se puede determinar el borde de absorción de la señal de reflectancia difusa desde la muestra en función de la temperatura. Complementariamente, se presenta el desarrollo del hardware y software usados para la calibración del método de medición.

Palabras claves: Espectroscopia de reflectancia difusa, SI-GaAs, n-GaAs, medidas de temperatura

Abstract

Measurement and control of substrate temperature is a common difficulty for molecular beam epitaxy (MBE), as well as other semiconductor deposition techniques. In this work, the development of a diffuse reflectance spectroscopy (DRS) system is presented as an alternative method for measuring the temperature of semiconductor materials in an Ultra High Vacuum chamber. DRS is an optical method for monitoring the temperature of semiconductor materials during the growth process in an MBE chamber. The system used has been calibrated and tested to n-GaAs and SiGaAs substrates. A sensitivity of 2 °C for the system is achieved. Its accuracy is limited by the accuracy with which the absorption edge of the diffuse reflectance signal from the substrate can be determine as a function of temperature. The hardware, software development and system calibration are reported here.

Keywords: Diffuse reflectance spectroscopy, Si-GaAs, n-GaAs, temperature measurement

Introduction

One of the important parameter during the growth of semiconductor thin films in a vacuum chamber is the substrate temperature. It has a profound effect on the structure and composition of the epilayers and their interface with the substrate. The optical and electrical properties of the thin film layers also depend directly upon the substrate temperature. Since temperature is such a common physical quantity, its measurement is often treated casually in the growth systems. However, real substrate temperature is very difficult to measure in a high vacuum chamber. This is due to a poor thermal coupling between the heater, the substrate holder and the substrate. The two most common types of substrate temperature monitoring techniques in an Ultra High Vacuum (UHV) chamber are thermocouples and rarely infrared pyrometers. Thermocouple is an economical option, and relatively easy to use, however accurate temperature measurements using thermocouples require physical contact with the substrate. This is neither practical nor acceptable because the substrate would become contaminated. In addition, the loading/unloading of the substrate has to be as fast and as easy as possible. The thermocouple is usually installed in a thermal cavity behind the substrate close to the heater in a rotating sample holder, so that it is in radiative contact with the substrate holder filament. It is often assumed that the substrate temperature is equal to the temperature reading of thermocouple in such a design. The fallacy of this assumption will be clear by bearing in mind the thermal contact problem associated within an UHV-chamber. In a non-rotating sample holder, however, thermocouples may be in contact with the sample holder on the back of the substrate. It is presumed that the substrate temperature is the same as the temperature of the platform. However, the temperature reading may also deviate substantially from the real temperature of the substrate. In addition to the above-mentioned problem, there is another main difficulty with these designs; the thermocouple temperature reading can quickly reach the thermal equilibrium, while the real temperature of the sample has not. The temperature discrepancies between the real substrate temperature and the temperature reading using thermocouple are difficult to compensate by calibration of the thermocouples, because the temperature offset between the thermocouple and the substrate is not reproducible. This means the substrate temperature is very poorly represented by the thermocouples, both in the magnitude and in the temporal response. The most severe impact of deviation of temperature, reading by thermocouples from

real temperature, is on any investigation of semiconductor growth process, which depends on the substrate temperature.

Pyrometry is an optical technique, which can be used for measuring the sample temperature. It is relatively straightforward optical technique to use. There is no need for mechanical contact with the sample. A Pyrometer is usually placed outside the UHV-chamber to monitor the substrate temperature. However, semiconductor samples are not ideal black bodies. For example, the emissivity of n-type GaAs and Si-GaAs are about 0.60 and 0.40, respectively. The emissivity depends upon the wavelength and the temperature of the sample. This means that, keeping track of the emissivity change is essential in determining the real substrate temperature, using a pyrometer. This is a complicated task especially when the sample's surface characteristics (and hence, emissivity) are changing during growth process. As pyrometry is a passive optical technique, (i.e. one simply collects black body radiation emitted by sample), other sources of light in the growth chamber such as heater's filament, ion gauges etc., each may contribute to the detected light signal. This can lead to significant errors in the temperature reading even if the emissivity of the sample is well characterized. A particular problem with semiconductor substrates is that at low temperature the maximum black body radiation emitted by the substrates occurs in the far-infrared region of the spectrum, with relatively little light given off in the near infrared and visible regions. Semiconductor materials such as gallium arsenide, silicon and so on, are fairly transparent in the infrared region. Therefore, if one places a substrate in front of a heater and attempts to measure the temperature by a pyrometer, one will primarily collect emission from the heater's filament and not from the sample. This implies a restriction on the design of the pyrometer; it must collect light in the near infrared region where the sample is opaque. The emissions from the substrate are very low in this region until the sample reaches a threshold temperature. This implies a lower limit for the temperature measurements of semiconductor materials, using pyrometry technique.

The lowest threshold temperature for the GaAs substrate for pyrometry temperature measurements is about 400°C (Wright et al. 1988). The need for an accurate, and non-invasive temperature measurement has led to the development of several innovative techniques such as; thermal expansion (Peuse et al. 1993), (Voorhees et al. 1991), ellipsometry (Conrad et al. 1993), infrared spectroscopy (Katzer et al. 1993), (Malalm et al. 1994), and band edge absorption (Hellman et al. 1987), (Weilmeier et al. 1991), (Johnson et al. 1993), (Guyer et al. 2000), (Balmer et al. 2003), (Schlereth et al. 2020).

Diffuse Reflectance Spectroscopy (DRS) is an optical method for measuring the temperature of a semiconductor substrate with a temperature dependent bandgap. It is based on optical absorption edge measurements. In this technique, a white light source illuminates the sample from outside the growth chamber. This allows chopping the light beam and consequently using the lock-in amplifier detection method. This reduces the effect of the background stray light from the other sources. The light beam is partially reflected from the front surface of the substrate (specular reflection), and partially transmitted through the substrate, where it is diffusely scattered (diffuse reflection). The reflection angle of specular reflection is equal to incident angle of light, while the diffuse signal is scattered over π steradians and contains information about the substrate bandgap. In this technique, an appropriate detector detects the diffuse light, which is located away from the specular reflection. The concept of Diffuse Reflectance Spectroscopy is shown in Fig. 1.

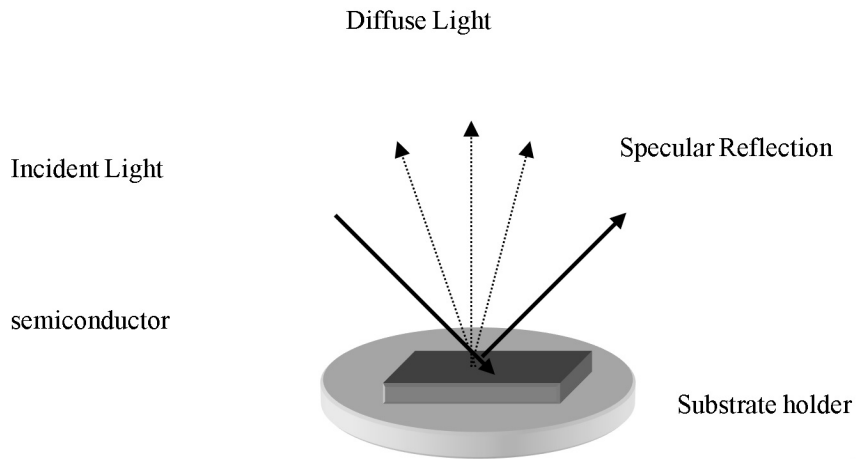


Fig. 1: Principal of the Diffuse Reflectance Spectroscopy (DRS).

A typical absorption edge is shown in Fig. 2, (Johnson et al. 1997), (Wang et al. 1997). It is important to note that the bandgap lies in the short wavelength region of the spectrum, where the optical signal is almost zero. Therefore, monitoring the change of wavelength position in the spectrum, corresponding to the energy bandgap would not provide an accurate and reproducible method to measure the temperature. This is because the absorption below the band edge depends on the quality of the sample and unintentional residual doping level. The residual absorptions below the band edge are intensified for diffuse light, which experiences multiple reflections inside the substrate. For accurate measurements of the real temperature, it is desirable to have a technique, which is as insensitive as possible to quality of the substrate. In other words, we need to choose a feature in the absorption edge spectrum, which is not dependent on the quality of sample.

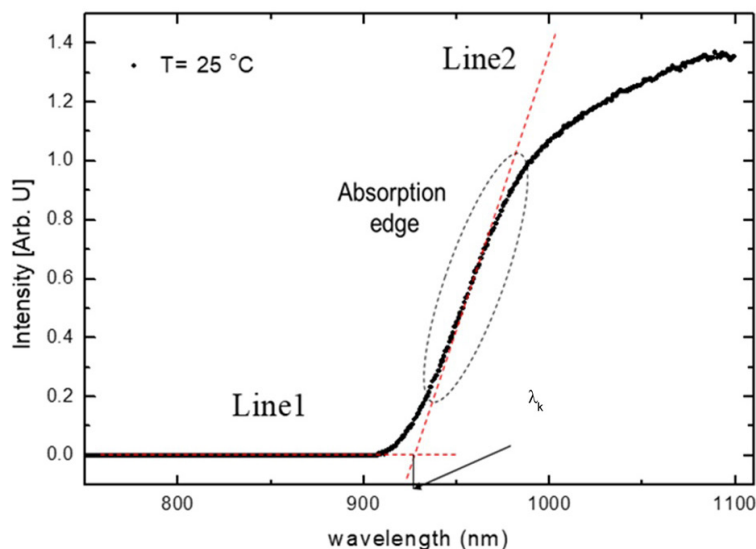


Fig. 2: Illustration of a typical absorption band edge and the asymptotic lines (Line 1 and Line 2), which is used to locate the Knee wavelength.

The diffuse reflectance spectrum has a sharp rise in the wavelength region at the onset of substrate transparency. In Fig. 2, two asymptotic lines (line 1 and line 2) are shown. Line 1 is the extrapolation of the background signal in the wavelength region shorter than the transparency wavelength region, and line 2 is the extrapolation of the steepest part of the spectrum. The intersection of these lines at the wavelength adjacent to the sharpest bend in the spectrum provides an optical signature of temperature dependency of the substrate. The wavelength, corresponding to these lines intersection “knee wavelength” is denoted by λ_k . The knee wavelength is related to the bandgap at 0 K, $E_g(0)$, by a simple relation.

$$E_g(0) = \frac{hc}{\lambda_k} \quad (1)$$

Where h and c are Planck’s constant and the speed of light, respectively. At temperatures higher than 0 K, the relation departs from this simple equation and no simple analytical expression can be used to relate the knee wavelength to the energy bandgap.

The objective of the current work was to produce a home-made DRS setup to measure and to monitor the substrate temperature during the growth of II-VI semiconductor epilayers on n-GaAs and SI-GaAs substrates. MBE growth of semiconductors is governed by the kinetic process rather than thermodynamic ones, therefore the substrate temperature, T_s , is a very important parameter in order to produce high quality samples. In the following, we present software and hardware development of a DRS system, including the calibration chamber.

Experimental method

Optical hardware development

An optical setup was built up, which contained a mini scanning digital monochromator (SDMC1-05) supplied by The Optometrics Group. It was equipped with an integral stepping motor. This configuration allowed the unit to be operated by a stepping motor controller. The monochromator has a 830 lines/mm-ruled grating, which is blazed at 1200 nm. The scanning range is between (750 –1700) nm. The diffuse light was led to the monochromator through an optical fiber bundle. The cross section of the optical fiber bundle was round with a diameter of 2 mm at the input end. The exit end was rectangular with a width of 0.6 mm in order to maximize the coupling with the entrance slit of the monochromator. A germanium photodiode detector follows the monochromator. The detector contains an internal amplifier. The pre-amplified signal from the detector was led to a lock-in amplifier, whose reference signal came from the mechanical chopper.

Calibration chamber development

In order to measure temperature of a semiconductor substrate in the growth chamber, there is a need for a predetermined calibration curve, which gives the substrate temperature, T_s , as a function of the “Knee Wavelength”, λ_k . Therefore, a small vacuum chamber was designed to be inserted in an isothermal calibration oven, see Fig. 3. The sample holder with an attached thermocouple to the back of the substrate holder was mounted in the vacuum chamber. It is important that entire calibration chamber to be heated homogeneously, therefore a series of heat shields were installed between the substrate and the optical ports to minimize any heat conduction to outside of the oven. Figure 3 shows the details of the calibration chamber. A series of temperature measurements were performed in the calibration chamber on an n-GaAs (001), and SI-GaAs (001) substrates. The respective calibration curves for these substrates were determined, see below in the results section.

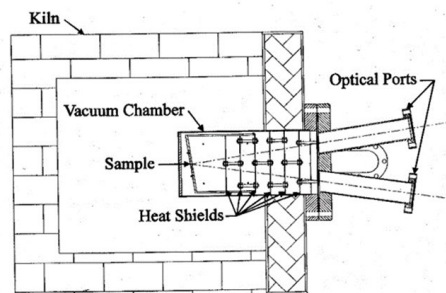
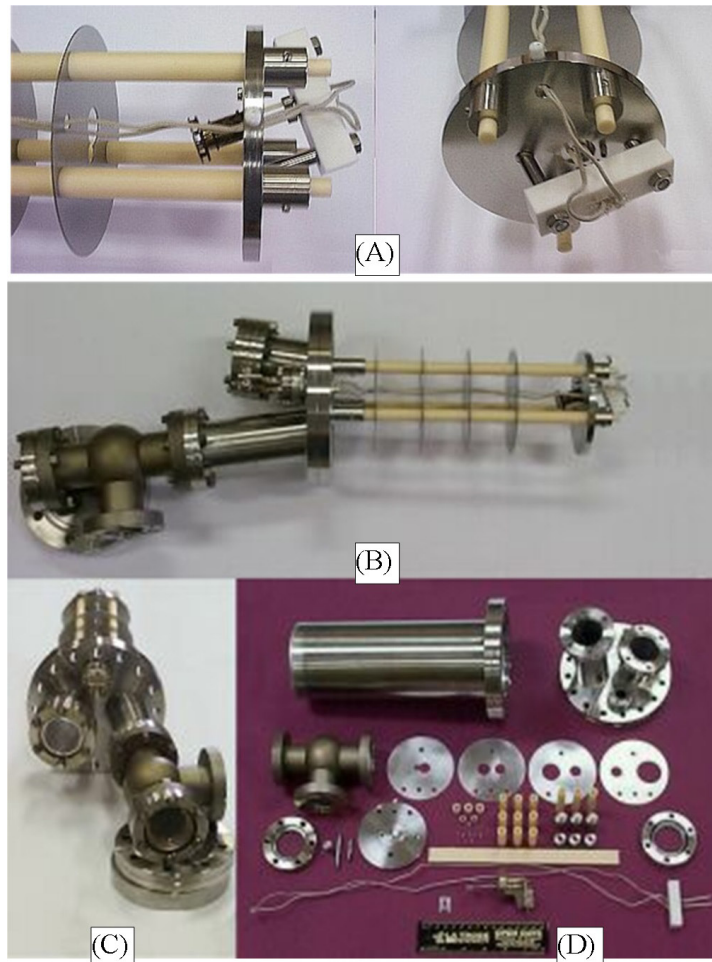


Fig. 3: Top panel (A): Sample holder and thermocouple assembly, panel (B): Sample holder stage and heat shields assembly, panel (C) Confluence of calibration chamber along with the optical viewports, attached to the calibration assembly (D): All parts and pieces of the calibration chamber, including the heat shield. A schematic sketch of calibration chamber and the oven is shown in the bottom panel.

Software development

The main task of the software interface is to scan the monochromator and record a list of the wavelengths and the corresponding signal values. This task requires making two different communications lines: the first with the monochromator using RS-232 protocol and the second with the data acquiring card using an ActiveX controller (OCX file) provided with the

card. The software interface, using visual basic code, was designed to make a RS-232 communication protocol between the computer and the microcontroller within the monochromator, which can obey and implement number of commands to control the stepper motor of the monochromator. The software was designed to make another communication with the data acquiring card which is installed in one of the PCI buses (32 – bit version) of the computer motherboard. A set of basic blocks functions were written and were used to the signals output from the lock-in amplifier and return raw data to the software interface.

The collected data were analyzed to directly obtain the knee wavelength to use it in a semiconductor wafer calibration process or to estimate the temperature if the calibration curve has already been determined. In this phase the software was given the ability to save all the calibration data in a special file format to make proper profiles for each semiconductor wafer as well as measurements reports and raw data.

The startup menu includes options to process a “**Measurement**”, “**Calibration**” or “**Analysis**” operation as shown in Fig. 4(A). The button “**Measurement**” opens the measurements form as shown in Fig. 4(B) where a temperature measurement operation can be carried out starting by assigning the serial communication port from a drop list contains all the RS – 232 serial ports exist in the computer and then configuring the serial port at the required settings for the monochromator controller. There are two options to acquire data, either from the data acquiring card “**Keithley card**” or from the look-in-amplifier. The “**Next**” button carries the configuration to the next step where the monochromator model is chosen from a drop list and the monochromator specifications will be loaded automatically from text file (“**MonoList**”) located in the program directory. The next step is to calibrate the monochromator calibration. After finishing the monochromator calibration successfully the procedure will move to measurement form where the user can move between scanning “**Configuration**” where the user can select the scanning wavelength range, steps and

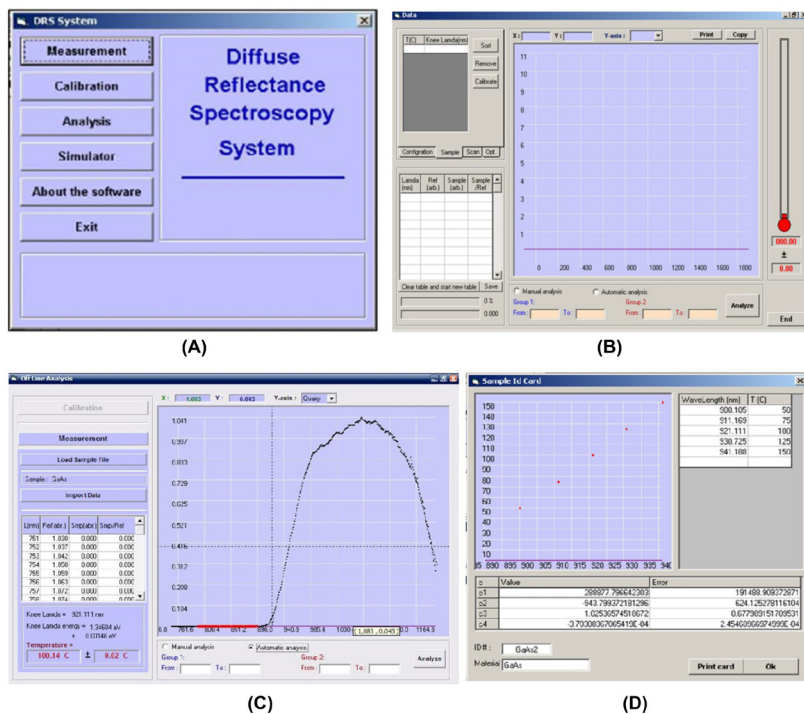


Fig. 4: various interface forms of the software

Selecting the data acquiring options, and **“Sample”** configuration to load a calibrated wafer profile, and proceeding the **“Scanning”** and when clicking on the headers of table, the user can select to scan for **“Ref.”** signal or for **“Sample”** signal, see Fig. 4(C). If the option **“Continuous”** is selected, then the program going to scan the sample several times as requested with the indicated time interval and an option to save the data in a selected directory. If the **“Calibration”** button is selected from the startup menu then the same form will appear as for **“Measurement”** the difference will be in the last step where instead of loading a calibrated wafer profile, a table of temperature and the corresponding knee wavelength will appear to gather data for the calibration, see Fig. 4(D). The saved data – either measurement or calibration data – can be analyzed separately by accessing to analysis form by choosing **“Analysis”** from the startup menu.

Results

Calibration Curve for n-type and SI-GaAs substrate

Figure 5(A) and (B) shows the normalized diffuse reflectance intensity at different range of substrate temperature for the n-type GaAs (001) and the SI GaAs (001), respectively. To minimize the effect of the wavelength dependency of the optical components such as lens, optical fiber bundle, and detector, the optical signals were normalized to the optical throughput of the system. The optical throughput was measured by removing the substrate and collecting the light reflected from a mirror.

The substrate temperatures, which were measured by thermocouples, are indicated on each curve in Fig. 5(A) and (B). It is shown that in the range above the bandgap (short wavelength including the visible part of the spectrum) the light from the lamp that is transmitted through the front surface of the substrate is absorbed. Thus, only the long wavelength light that is transmitted through the substrate is diffusely scattered, from the substrate back side, into the detection system.

The spectrum of the diffusely back-scattered light was obtained by scanning the wavelength through the wavelength region below and above the substrate bandgap. The knee wavelength shifts to longer wavelength as the temperature increases. The reduction of the diffuse reflectance signal at longer wavelengths, corresponding to high temperatures, is due to an increase in the residual sub-edge absorption, which intensifies at higher temperatures. The signal at the short-wavelength region, where the substrate is opaque, is caused by residual diffuse scattering from the front side of the substrate and stray light scattered in the vacuum chamber. This background signal can be subtracted in order to locate the position of knee wavelength in the spectra.

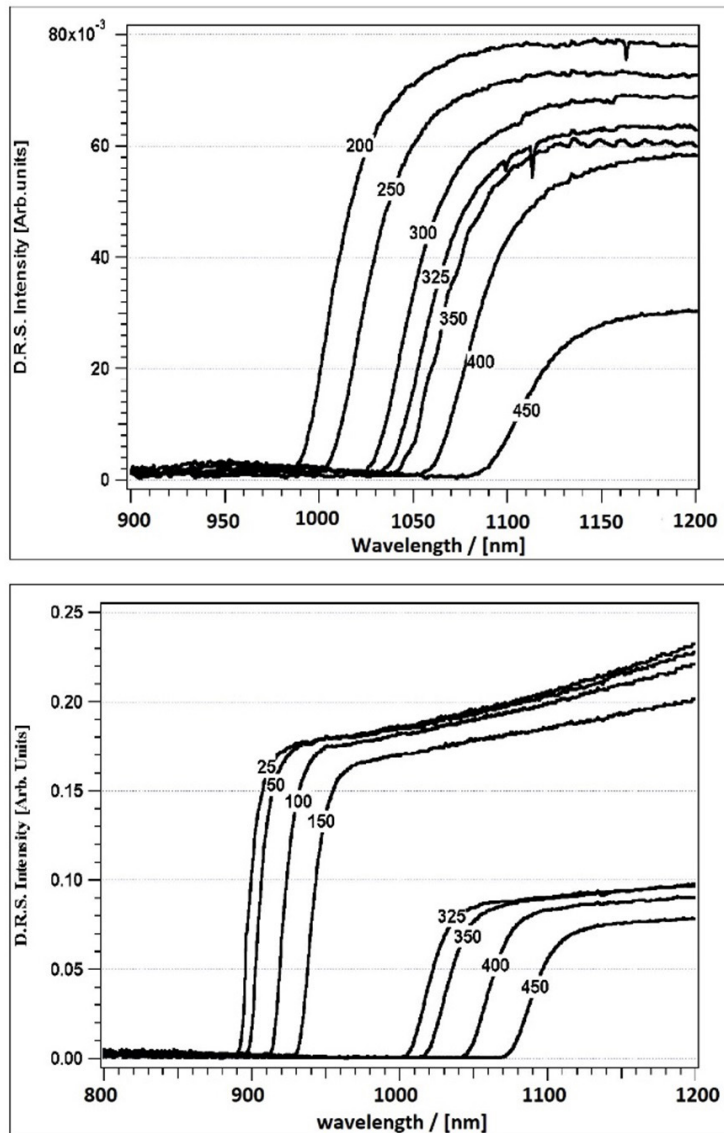


Fig. 5: Normalized absorption edge signal of the n-type GaAs substrate (A), and the SI GaAs substrate (B).

The normalized diffuse reflectance spectra for a SI GaAs (001) and an n-type GaAs (001) are compared in Fig. 6. Three curves for the substrate temperatures at 200 °C, 400 °C, and 450 °C are shown for each sample. The shift of the Knee wavelength due to increasing temperature is larger for n-type GaAs. The amplitude of the DRS signal is also less for n-type GaAs due to large number of electron in conduction band, which causes intra-band absorption of the diffuse light.

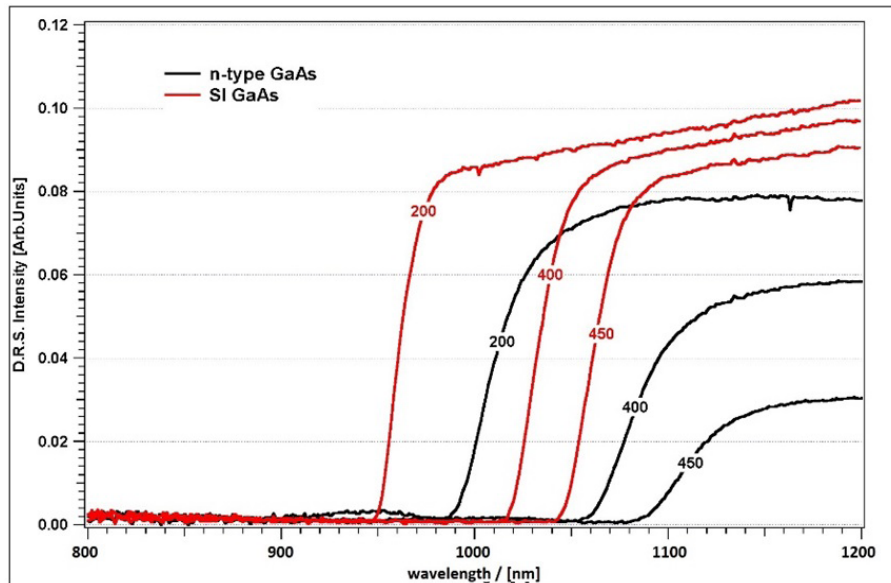


Fig. 6: Comparison of DRS signal from the n-type GaAs and the SI GaAs.

The system was calibrated against a thermocouple attached to the substrate, over the temperature range of 25°C to 550°C, inside the calibration chamber. The calibration was carried out for n-type and SI GaAs wafers. The thermocouple was restrained by a Ta-strip, spot-welded to the sample stage, so that the thermocouple junction was in permanent contact to substrate during the calibration. The temperature was determined from the position of the knee wavelength in the spectra. The procedure is described in (Pearsall et al. 1995). The data basically were used to fit a linear regression to the base-line reflectance below the knee wavelength and a separate linear regression was fitted near the region of maximum slope. The intersection of these lines determines the knee wavelength. These values were used to calibrate the position of the knees against the thermocouple temperature attached to the substrate.

The calibration curve for a 350 μm thick n-doped (10^{18}) GaAs substrate is shown in Fig 7. The knee wavelengths were extracted from DRS signals, using the method that has been explained above. The data points were fitted by a 4th degree polynomial, which is given in equation below.

$$T_S(\lambda_k) = -16115 + 41.948\lambda_k - 3.6716 \times 10^{-2} \lambda_k^2 + 1.12 \times 10^{-5} \lambda_k^3 \quad (2)$$

Where λ_k is the knee wavelength in nm and T_S is the substrate temperature in °C. The knee wavelength depends on many factors other than temperature, such as doping level, substrate thickness. Any calibration measurements with a high level of accuracy and precision have to account for these parameters. An empirical method has to be employed to characterize the temperature as a function of sample thickness and doping level. Different types of substrate will result in different coefficients of the 4th degree polynomial fit. The corresponding calibration curve for SI-GaAs substrate is not shown here for simplicity.

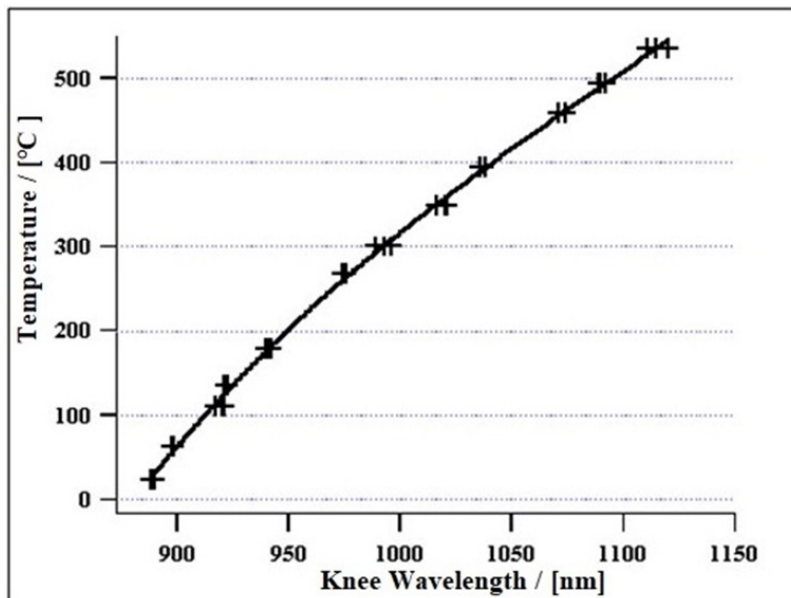


Fig. 7: Temperature calibration curve for n-type GaAs substrate.

To investigate the sensitivity and precision of the system, temperatures of the substrates were also measured for two successive temperatures with differences in the range 1 to 5 °C. Figure 8 shows the temperature dependence of DRS spectra for an n-GaAs substrate at two different temperatures $T_1=100^\circ\text{C}$ and $T_2=105^\circ\text{C}$. The knee wavelengths at $\lambda_{k1} = (951.7\pm 0.1)$ nm for T_1 and $\lambda_{k2} = (954.5\pm 0.1)$ nm for T_2 can unmistakably be resolved. The inset (Fig. 8) shows the DRS spectra of SI-GaAs substrate with temperature difference of 1 °C, the knee wavelengths are barely resolved. Whereas, they were clearly distinguished by the software for two successive temperatures with a difference of 2 °C. Therefore, the error in temperature measurements by the system was estimated to be ± 2 .

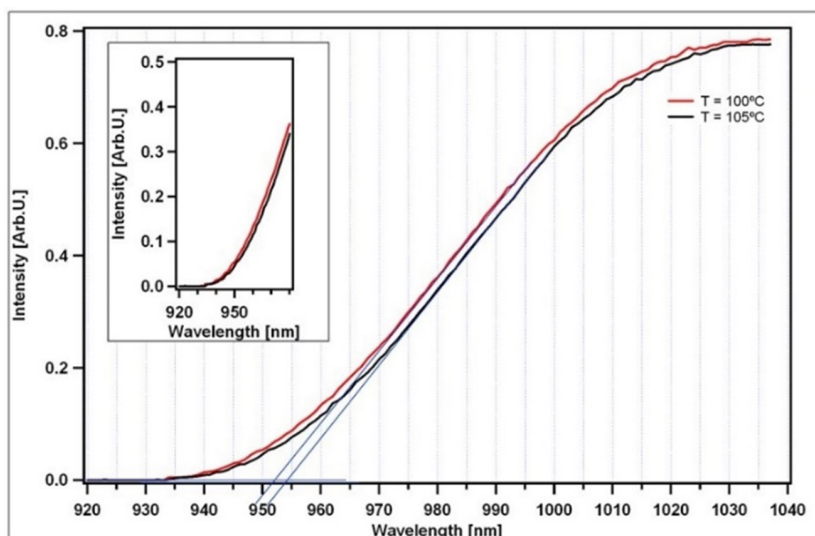


Fig. 8: Temperature dependence of DRS spectra for n-GaAs substrate at two different temperatures $T_1=100^\circ\text{C}$ and $T_2=105^\circ\text{C}$, inset: temperature dependence of DRS spectra for SI-GaAs substrate at two different temperatures $T_1=200^\circ\text{C}$ and $T_2=201^\circ\text{C}$

In addition to the precision performance of the system, repeatability of the system was also studied. Temperature of the substrates were measured at different constant temperatures various times. The results (not shown here for simplicity) indicated of a high repeatability performance of the system.

Comparison thermocouples and DRS system temperature measurements

In order to compare the performance of the DRS system temperature measurements to the conventional thermocouples temperature readings, and the response of the DRS system had been tested attached to an Ultra High vacuum system, as shown in Fig. 9.

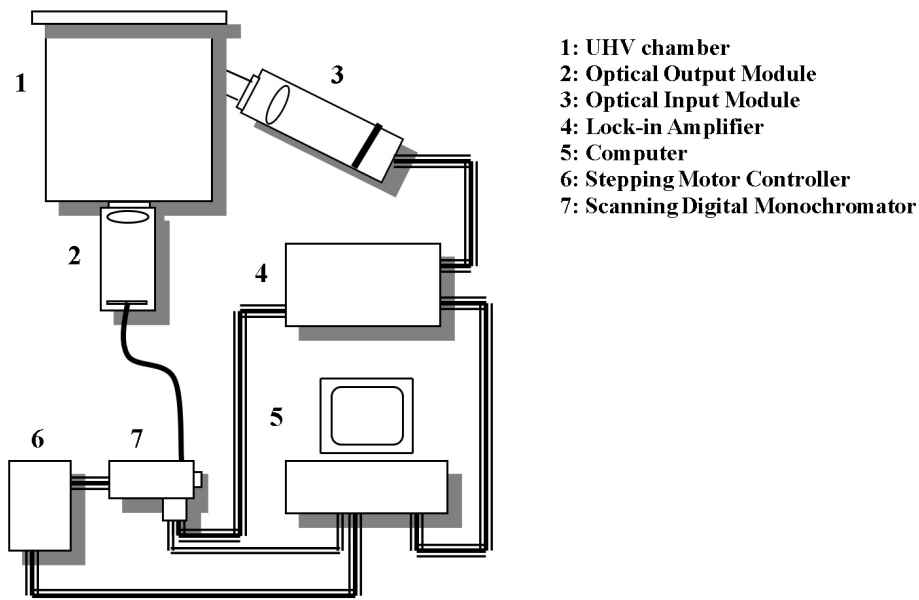


Fig. 9: Schematic diagram of Diffuse Reflectance Spectroscopy set up attached to the UHV chamber

The substrate temperature ramped up from 25°C to 450°C in different steps. The ramping rate was fixed at 0.35°C/s on the thermocouple controller. The substrate temperature was measured by the DRS system and simultaneously was monitored by a thermocouple. Fig. 10 illustrates the substrate temperature measurement performed by thermocouple in actual MBE chamber (solid line) and DRS system (markers) attached to the MBE chamber. The plot shows a temperature deviation about between 70°C (at low temperature) and 170°C (at high temperature). The temperature difference depends on the thermal conductivity between the substrate and substrate holder, which is mediated by indium or gallium indium soldering. These temperature offsets depend directly upon the thermal coupling between the substrate and the substrate heater.

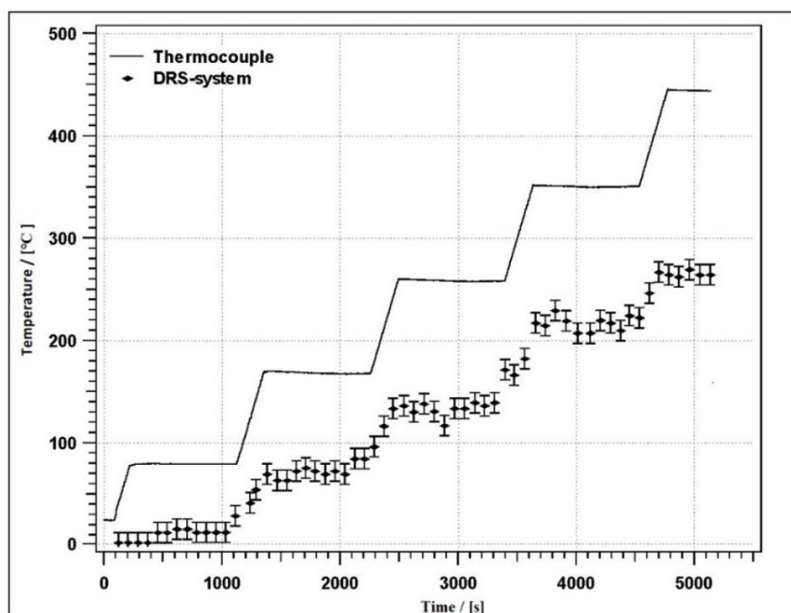


Fig.10: The substrate temperature monitored by thermocouples and the DRS system.

Discussion

In the following, the limitations of DRS technique to measure the semiconductor materials is presented. First of all, it should be emphasized that the calibration curve which is presented above would not be universal for all substrates, as it has been stated above. It depends on the thickness, doping level, and doping types (n or p) of the substrate. The system was designed and meant to be used to measure the substrate temperature during MBE growth of II-VI semiconductor epilayers as accurate as possible. This is only possible if the calibration curve has already been obtained for that particular substrate. In case of changing the substrate of different thickness or different doping type, the calibration process needs to be repeated for the new substrate.

The second shortcoming of the technique as a temperature measurement technique will be presented during the actual growth process. We should bear in mind that the growth of epilayers with a different refractive index than that of the substrate, cause thin film interference in the epilayers upon exposure to the light beam. These interference fringes can affect the substrate temperature measurements by the DRS system. The growth of epilayers with smaller bandgap than that of the substrate absorbs the light at wavelengths where the substrate is transparent. This may limit the dynamic range of DRS temperature measurements. Thin film interference can be induced in any epilayers (with smaller or bigger bandgap than the substrate bandgap). Even though, these interference fringes can be used to measure the growth rate (Li et al. 1997) and index of refraction of the epilayers, but the interference effect will produce small changes in the shape of the diffuse reflectance signals in vicinity of the Knee wavelength. Consequently, this will cause that the temperatures obtained from the DRS spectra seem to oscillate during the growth. These shifts in the temperature reading of the substrate are obviously undesirable.

In relation to the current study, an epilayer of ZnSe (wide bandgap semiconductor) has been grown on a GaAs (narrow bandgap semiconductor). The temperature measurements during the growth produced thin film intensity oscillation in the spectrum of the light transmitted through the epilayer. The index of refraction of the wide bandgap ZnSe epilayer is lower than that of the substrate. Therefore, the light as it travels through will be reflected at the interface, which will then interfere with the incident wave causing thin film interference in the epilayer.

Figure 11 (top panel) shows the normalized diffuse reflectance signal for a n-GaAs substrate with a ZnSe epilayer of thickness 1.6 μm on top. The fringes, which caused by interferences can be seen. These fringes will play havoc with the position of the absorption edge. The calibration curve obtained based on these data is also shown in Fig. 11 (bottom panel). The effect of the fringes in the diffuse reflectance signal is illustrated by the fluctuations in the calibration curve, which are marked with arrows in Fig. 11 (bottom panel). These features are not presented in the calibration curve of a bare GaAs substrate, as shown above. To overcome this problem a series of measurements for ZnSe epilayers with a various thickness is necessary. A more comprehensive theoretical algorithm is needed to eliminate the effect of fringes on the temperature measurements during the growth, using DRS system. Therefore, the system can only be used in a passive mode to monitor substrate temperature before the growth of the epilayers. An active, closed-loop control of substrate temperature reading during the growth of multilayer heterostructures can provide a better quality of the structures. Any further improvement and development of the technique should be focused on these issues.

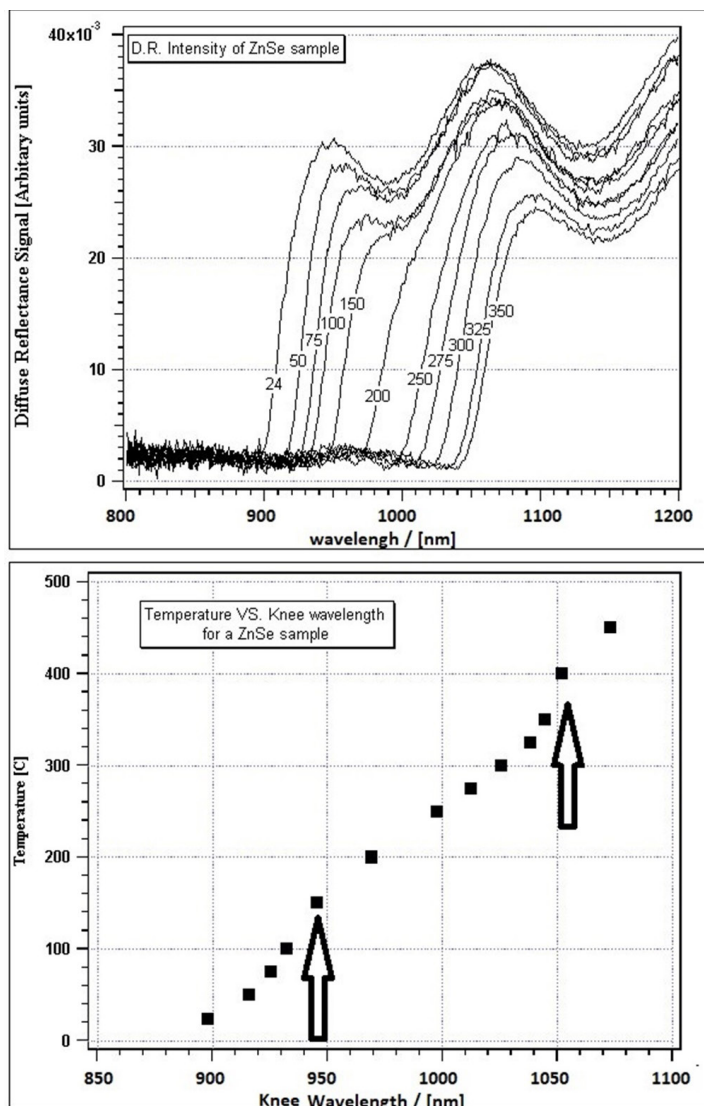


Fig.11: The substrate temperature monitored by thermocouples and the DRS system.

Conclusion

An optical temperature measurement device for use in measuring the temperature of semiconductor wafers, in an MBE chamber was designed and constructed. The temperature is inferred from the position of absorption edge of the diffuse reflected light of the back of the substrates. The position of the absorption edge of semiconductor materials, like their band gap, depends on temperature. The accuracy of the system is dependent on the accuracy with which the absorption edge of the semiconductor can be determined. A relative sensitivity of about 2°C estimated for temperature measurement. This sensitivity is independent of temperature in the range of 25°C to 550°C. The system was calibrated and tested against a thermocouple attached directly to a SI-GaAs and n-type GaAs substrates in a calibration chamber, and consequently it has been used to monitor substrates temperature in an UHV chamber of II-VI MBE growth system. The system is proved to be superior to the conventional thermocouples which are usually located at the back of the sample holder. It is also superior to pyrometry in that its sensitivity is independent of temperature, as it is not either sensitive to the changes in semiconductor materials emissivity during the MBE growth.

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