

SHOP NOTES

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Robust optical delivery system for measuring substrate temperature during molecular beam epitaxy

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The phenomenal growth in wireless communications and optoelectronic technology is making III-V semiconductor based devices an increasingly important component of the entire semiconductor market.¹ Unlike Si-based technology, where devices are fabricated primarily by ion implantation, III-V device structures must be fabricated one atomic plane at a time. One of the most powerful and flexible growth methods for III-V structures is molecular beam epitaxy (MBE). The ability of MBE to control the deposition of extremely thin films that is required for high-performance devices, as well as the capability to grow device layers with arbitrary compositions and doping profiles, is unequalled. However, even MBE as traditionally used suffers from poor day-to-day repeatability, and this is due, in part, to the lack of any means to accurately sense and control one basic process parameter, namely, the substrate temperature. This problem arises because the typical sensor used for temperature control is a thermocouple, which cannot be in good thermal contact with the substrate if one wants to produce high-quality, highly uniform material. The inaccuracies in substrate temperature, in turn, affect the overall progress within the MBE community at large. The reason for this is that the temperature profile used to produce a high-quality growth at one institution cannot be transferred to other institutions. The MBE community has tried to minimize these difficulties through the implementation of optical pyrometers for substrate temperature determination. Unfortunately, the accuracy of pyrometers is limited by stray light from the source ovens and from substrate heater filaments. In addition, pyrometer readings are affected by films deposited on the pyrometer viewport and by lack of knowledge of sample emissivity (which in many cases is changing during the growth of the structure). Finally, if one uses direct radiative heating of the substrate, the pyrometer becomes flooded with the infrared radiation of the heater filaments, making the technique even less accurate.² Other workers have suggested the use of

fundamental optical properties, such as the band gap of a semiconductor, and their temperature dependence as a vehicle for obtaining accurate and reproducible substrate temperatures.² One implementation of this idea is realized by using the broadband light emitted from the substrate heater filaments as the light source for performing an optical transmission measurement on the substrate.^{2,3} Using the substrate heater as a light source is very clever and has many advantages over thermocouple sensors and pyrometers. Namely, it determines the temperature without requiring physical contact with the substrate and this approach is not affected by films being deposited on the pyrometer viewport.³ In addition, this approach does not require internal modifications to the MBE machine to implement. However, there are two disadvantages with this approach. Since one cannot modulate the intensity of the heater light, the signal to noise ratio is affected by stray light. Also, if the heater power is shut off, no light source is available to measure the substrate temperature. Another implementation of *in situ* band gap determination is to bring white light into the pyrometer port and to measure the band-edge reflection spectroscopy using the back-reflected light coming from the pyrometer viewport.⁴ This method works well for bare substrates but suffers from optical interference effects, especially when the films are smooth and uniform in thickness (precisely what one wants to achieve with MBE). The most successful noncontact temperature measurement scheme was achieved by installing an optical pipe inside the MBE machine that allows an alternate light source to be brought to the back side of the substrate. This has been successfully implemented in MBE machines that have an in-line optical path to the back side of the substrate.⁵ Supplying an alternate light source that can be chopped prior to entering the substrate has proven to be the most precise substrate temperature measurement method available.⁶ However, in MBE machines where the sample is mounted on a manipulator that rotates about a perpendicular flange axis, no successful integration has been achieved. For the first time, a simple and robust solution has been found for

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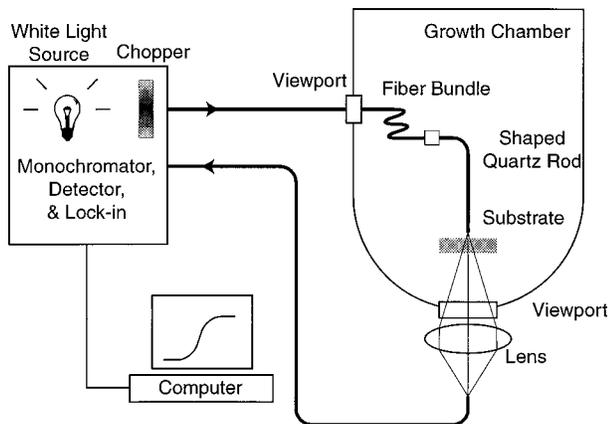


FIG. 1. Schematic diagram showing the entire optical delivery system used for transmission thermometry measurements. The system uses a 20 W white light source which is passed through a chopper. The light then moves through a fiber bundle to a viewport on the UHV growth chamber. Inside the vacuum chamber the light travels through a fiber bundle and then through the shaped quartz rod to the back side of the substrate. The transmitted light is collected through another viewport on the front side of the substrate and is then feed into the monochromator. After using lock-in detection, a computer digitizes the transmission spectra and calculates the substrate temperature.

integrating a noncontact transmission thermometry sample temperature measurement scheme into a Riber 32 MBE machine. The Riber 32 MBE machine falls into a class of MBE machines that utilizes a rotating manipulator for sample transfers, which makes this integration unusually complicated. The fundamental band gap of the substrate is measured from the transmission spectrum obtained after passing white light through the substrate. This approach allows the substrate temperature to be measured over a wide temperature range (e.g., 0–700 °C for GaAs) without making physical contact with the substrate. The temperature of the substrate is known within ± 2 °C and is updated approximately every second. We will discuss the advantages and disadvantages of using this optical technique for temperature determination in MBE.

There are primarily two custom components that made the incorporation of the optical thermometry system possible. First, a shaped quartz rod that guides the light from a fiber bundle to the back side of the substrate (see Fig. 1). The quartz rod has a diameter of 2 mm and the ends are flame polished so that they are optically flat. One end of the quartz rod is positioned 1–2 mm from the substrate. Due to the high temperature stability properties of quartz, there are no contamination issues to consider (even though the quartz rests only millimeters away from the hot sample and heater filaments). If a stainless steel coated fiber pipe were used this close to the heater, severe outgassing and subsequent contamination problems would occur. The quartz rod has a 90° bend with a radius of curvature of 25.4 mm and an overall length of 200 mm, so that the other end of the rod directly faces the conflat flange holding the sample manipulator. The second critical component which made the integration successful is an ultrahigh vacuum (UHV) compatible optical

fiber bundle, which is attached to one end of the quartz rod and directs the light to an optical fiber port on the manipulator flange. The individual fibers are made of borosilicate and are not coated. The fiber bundle is encased in a stainless steel monocoil, and between the fiber bundle and the stainless steel is a layer of a fiberglass material called “natural Silverflex.” This material is used to avoid fiber breakage and is UHV compatible, unlike the conventional polymer materials used to coat fibers (the fiber bundle was supplied by Fiberguide Industries). The fiber-fiber feedthrough is simply a standard glass viewport with a special mount which rigidly supports the fiber flat against the viewport (the fiber-fiber UHV conflat feedthrough was supplied by CI Systems). The sample and the quartz rod assembly rotate as a rigid body $\sim 180^\circ$ between the sample’s introduction position and the sample’s growth position about a line perpendicular to the manipulator flange and approximately centered on the manipulator flange. A 60 cm long fiber bundle connects the quartz rod to the manipulator flange which are separated by about 20 cm. The extra 40 cm length of fiber allows the optical system to easily accommodate the 180° twist without breaking. In addition, the fiber bundle is mounted approximately 10 cm from the center of the manipulator flange and thus only needs to flex $\pm 5^\circ$ during the $\pm 90^\circ$ swing of the sample from its transfer to its growth position. The quartz rod is *only* fixed at the point where it attaches to the fiber bundle, otherwise it simply rests against the manipulator components, providing a strain free mount that allows independent thermal expansion and contraction. The entire assembly is UHV capable, and our system reaches a base pressure of 3×10^{-11} Torr when cooled with liquid nitrogen.

The quartz rod and fiber bundle assembly are the only components inside the vacuum chamber. On our manipulator, the optical delivery components are relatively simple to install and to replace. The MBE machine is a custom modified commercial system (model 32 from Riber). The primary internal modifications were moving the thermocouple sensor from the center of the 3 in. heater to a new position approximately 5 mm away, and also making a 3 mm diam hole through a polycrystalline boron nitride (PBN) plate that sits between the heater filaments and the substrate. As stated above, the quartz rod simply rests against the edge of the PBN plate and is only held to a rigid mount at the other end. This allows the quartz rod and manipulator assembly to flex without putting tension on one another. Our entire chamber has been cycled from room temperature to 200–250 °C many times without any damage occurring to the quartz rod or the fiber bundle. In addition, we rotate the manipulator several times per day from the sample transfer position to the sample growth position, and this motion has never caused the quartz rod or fiber bundle to fail.

In order to upgrade an existing MBE system to utilize the optical delivery system described here, one key requirement is having a pyrometer viewport in front of the sample while in the growth position (ideally, directly in front of the sample). A second requirement is to modify the sample

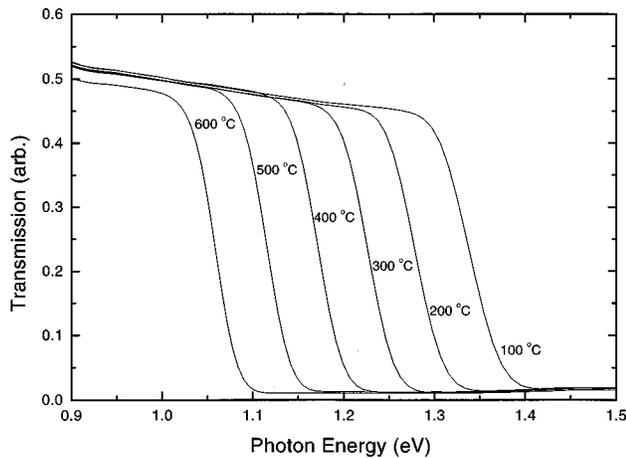


Fig. 2. Six transmission curves are shown as a function photon energy. Each curve was taken after the substrate temperature remained unchanged for more than 30 min, while being heated with a constant current power supply. The sharp drop in the transmission spectra occurs once the photon energy is large enough to cause band-to-band transitions.

heater and manipulator to make space for the quartz rod and fiber bundle.

Outside the vacuum chamber we use commercially available hardware and software (model NTM1 from CI Systems) systems for performing the optical transmission measurements, shown schematically in Fig. 1. This system uses chopped white light from a 20 W lamp in order to separate our light source from stray light due to the heater, ion gauges, and cells. This light is directed to the back side of the substrate using the fiber bundle and shaped quartz rod discussed earlier. The light which is transmitted through the wafer is collected through a viewport on the growth surface side of the sample. We want to note that the optical throughput of the interface components is less than 2.5%. The primary loss of light collection is due to beam divergence from the quartz rod to the viewport. A simple lens system collects the light and focuses it onto a fiber bundle, after which the light is piped into a fast-scan grating monochromator with a silicon-germanium detector and a lock-in amplifier. A computer reads the transmission spectra and then calculates the fundamental band gap using either a first or second derivative technique. The temperature of the substrate is determined by comparing the band gap obtained from the above measurement with one obtained from a database. The entire process of collecting and digitizing the light intensities over a wavelength range from 600 to 1600 nm and extracting a temperature is about once per second. We now use the substrate temperature obtained with this system as a feedback for setting the amount of current delivered to the heater filaments.

As a demonstration of the systems performance, transmission spectra as a function of photon energy were acquired using a semi-insulating GaAs(001) 2 in. indium free mounted substrate and are shown in Fig. 2. A fresh "epiready" wafer was used, the oxide was removed and about 1 μm of GaAs was grown at 580 $^{\circ}\text{C}$ using a growth rate of 1 $\mu\text{m}/\text{h}$ determined using reflection high-energy elec-

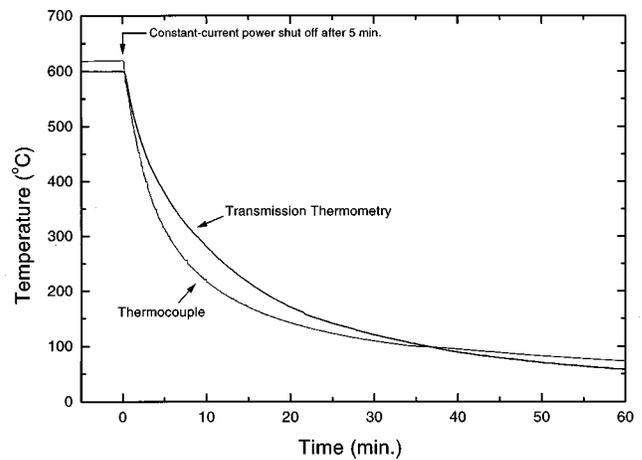


Fig. 3. Thermocouple temperature and calculated transmission thermometry temperature shown as a function of time. A constant current power supply maintained a wafer temperature of 600 $^{\circ}\text{C}$ (the thermocouple read 615 $^{\circ}\text{C}$) for 5 min after which the current was set abruptly to zero. The data show that the dynamic response is very different for the thermocouple and the substrate.

tron diffraction (RHEED) oscillations. The sample was cooled to room temperature and then subsequently heated to 100 $^{\circ}\text{C}$ using a constant current power supply. The current was manually adjusted until the temperature value was 100 ± 2 $^{\circ}\text{C}$ and did not change for 30 min. At the end of this period, a transmission spectrum was recorded. This procedure was repeated for 100 $^{\circ}\text{C}$ increments up to 600 $^{\circ}\text{C}$. The sharp drop in the transmission curves is due to a sharp increase in the absorption of light due to band-to-band transitions. The fundamental band gap decreases in an approximately linear manner as the temperature is increased from 0 to 700 $^{\circ}\text{C}$.

A significant advantage of supplying our own light to the back of the substrate is that once the substrate heater is turned off, we can still accurately measure the substrate temperature. Thus, we can accurately determine the cooling rate. We were surprised to find that the thermocouple temperature dropped much faster than the temperature of the substrate, as shown in Fig. 3. After holding the substrate at 600 $^{\circ}\text{C}$ for 5 min (the thermocouple reads 15 $^{\circ}\text{C}$ higher), the power to the substrate heater was set to zero. The thermocouple reached 250 $^{\circ}\text{C}$ in about 7 min, whereas the substrate required about 12 min. Often a heterostructure requires that growths of different materials be carried out at different temperatures. Using the thermocouple as a gauge of the response time for the substrate to change temperatures may not be accurate. If a particular layer has a narrow temperature window for high-quality deposition, then starting the growth too soon could lead to a poorly defined structure.

The key advantage of incorporating the optical transmission temperature measurement system into our MBE chamber is fast and accurate temperature determinations which are derived from a fundamental optical property. Thus, our knowledge of the temperature is not subject to variations due to different substrate mounting strategies or to even which MBE machine is used to perform the measurement.

One drawback for our system is that we can only measure the substrate temperature at the center of the substrate. We would need to install multiple quartz rods across the back side of the wafer to measure the temperature at other points on the wafer. It is known that a temperature gradient can occur between the center of the wafer and the edge of the wafer. With our measurement system we cannot determine this gradient. Another disadvantage of this optical detection system is that it does require that one install components inside the MBE machine.

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