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Homogeneous substrate heating using a CO_2 laser with feedback, rastering, and temperature monitoring

S. J. Barrington[a] and R. W. Eason
Optoelectronics Research Centre, University of Southampton, Southampton, SO17 1BJ, United Kingdom

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We report results from a highly versatile and scalable raster-scanned continuous wave CO_2 laser heater incorporating a dual wavelength photodiode-based temperature monitoring system with feedback control. The system is able to uniformly heat substrates inside a vacuum chamber, reduce the occurrence of substrate fracture and allow almost negligible heat loss to the deposition chamber and surroundings. The minimum achievable temperature difference across the substrate is, in practice, limited by the fastest achievable raster scan speed. © *2000 American Institute of Physics.* [S0034-6748(00)01311-3]

I. INTRODUCTION

Pulsed laser deposition (PLD), now an established method of thin film production, offers a rapid and cost effective technique for producing high quality epitaxial films.[1] In order to produce such films an appropriately orientated substrate is required which usually needs to be uniformly heated during the deposition process. Literature values for optimum substrate temperature most often fall within the temperature range of 600–800 °C although in some substrate–film combinations, the substrate may need to be heated in excess of 1200 °C.[2] The orientation and phase of the developing film can be highly dependent on the temperature of the substrate during deposition. Uneven temperature distributions across the substrate can lead to undesirable polycrystalline films or a mixture of amorphous and crystalline growth. Rapid heating coupled with poor thermal conductivity of the substrate can also cause severe thermal stress if the substrate is not heated homogeneously, often leading to substrate fracture. It is essential therefore that an even temperature profile of the substrate is achieved.

Tungsten heating elements, thermocoax and other resistive heating methods have been used with some success to achieve high temperature, homogeneous heating. These methods however, are extremely inefficient and result in unavoidable heating of the whole deposition chamber, causing severe outgassing from the chamber walls and any additional vacuum components. Efficient and uniform thermal contact between the heating element and the substrate also needs to be ensured which is not always practical at high temperatures even if a conductive paste (e.g., an indium–gallium alloy) is used. Resistive heating also implies a lengthy turnaround time between depositions before the chamber can cool enough to allow installation of a new substrate. This is a severe drawback when one considers that the typical deposition times required for the growth of a 4 μm film can be as short as 10 min.

CO_2 laser substrate heating in PLD has already been used with considerable success.[2–4] Such laser heating minimizes most of the problems associated with conductive heating and can heat a substrate with minimal heat loss to the chamber. A 40 W CO_2 laser is sufficient to heat a 1 cm^2 substrate up to 1200 °C, whereas a heating filament typically requires $\sim$1000 W to heat the substrate to the same temperature. To achieve an even heating profile across the substrate, beam homogenizers have been employed,[2,4] but these are often difficult to set up and align. Homogenizers, additionally, do not offer flexibility in the size or position of the substrate and, if used within the vacuum chamber, can quickly become coated with target material. This causes heating of the homogenizer due to increased absorbency and a corresponding loss in power delivered to the substrate.

II. HOMOGENEOUS HEATING

We report here a method of homogeneous heating using a continuous wave (cw) CO_2 laser and two scanning galvanometric mirrors (General Scanning Inc., Watertown, MA). We also describe a simple temperature monitoring system employed to determine the substrate temperature distribution. The apparatus is shown in detail in Fig. 1.

The mirrors are computer controlled to deflect the CO_2 laser along an appropriate two-dimensional (2D) array of points on the substrate. A laser diode pointer coincident with the CO_2 beam path is used to render the pattern tracked by the CO_2 beam visible to a charge coupled device (CCD) for ease of alignment. Figure 2 shows a CCD image of the laser pattern on the back of the substrate: a 6×4 array of points. By varying the dwell time of the laser on each point and the position and number of the points across the substrate, heating uniformity across the substrate can be achieved to a degree much higher than for nonscanned single beam techniques. Absolute uniformity is limited by the number of points that can be scanned across the substrate in a time fast enough to avoid temperature fluctuations due to the transient nature of the heat source on any one particular point.

Also seen in Fig. 2 is the alumina cradle that is used to hold the substrate and details of the nickel base. This design

[a]Electronic mail: s.barrington@physics.org

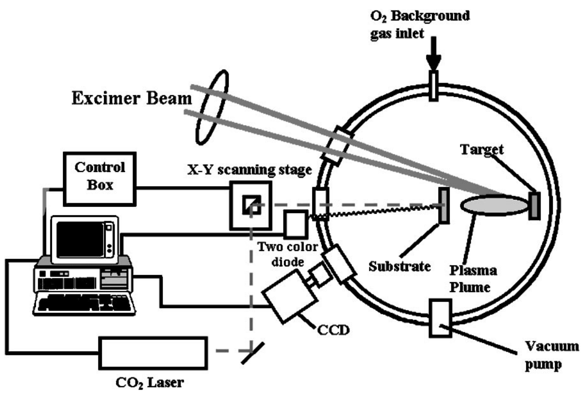

FIG. 1. Computer controlled raster-scanned laser heating apparatus.

ensures a good compromise between minimum heat loss, by conduction, to the surroundings, and flexibility of the mechanical construction.

III. TEMPERATURE MONITORING

Temperature measurement using a thermocouple is impractical as this will inevitably induce heat sinking in the substrate and thus promote temperature inhomogeneity. Temperature is instead measured using a two-color (silicon and germanium) photodiode (Hamamatsu part K1713-03). Within this single package an infrared-transmitting silicon photodiode is mounted over an infrared-detecting germanium photodiode. This requires very little space and alignment and also requires no external beam splitters. The temperature of the substrate can be calculated from the ratio of the intensity observed by the two diodes within their respective spectral regions. A practical solution for doing this is to assume the emissivity of the substrate is similar for the two observing photodiode wave bands (i.e., assume a graybody) and that the spectral response curve of the two diodes can be

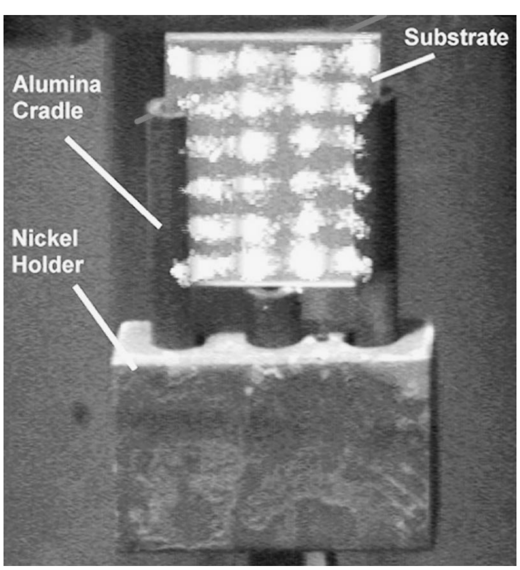

FIG. 2. Raster scan pattern across the substrate using an alignment diode laser to render the path visible. The alumina cradle and nickel holder are also indicated.

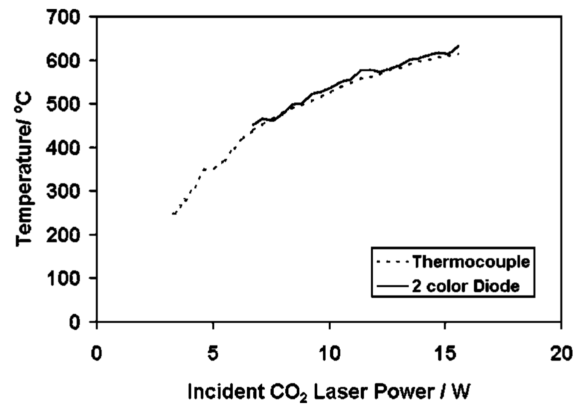

FIG. 3. Temperature measured with the calibrated two-color diode and thermocouple against incident CO_2 laser power.

approximated to a central effective wavelength. The equations for temperature determination can then be simplified to[5]

$$T \sim \frac{B}{(\ln Q) - A} + C,$$

where Q is the ratio of the two diode outputs.

A, B and C are constants to be determined by calibrating ratio sets $(Q_1, Q_2, \ldots, Q_n)$ against known temperatures $(T_1, T_2, \ldots, T_n)$ and evaluated through a least squares method. Since this method should be independent of the emissivity, the calibration only needs to be done for one material. It should be noted however that metals are not graybodies so some deviation from this curve may be seen should a metal substrate be used. Coating the back of the substrate in black soot can solve problems arising with non-graybodies or substrates semitransparent to CO_2 laser radiation (10.6 μm). The temperature data from the diode can be sent via the computer back to the laser power control to stabilize the temperature and automate a constant, gradual heating process, thereby reducing chances of substrate fracture. Figure 3 shows a graph of the temperature calculated from the two-color diode with a thermocouple reading for comparison against incident power. In this case the thermocouple was firmly heat sunk to the front surface of the substrate (heating is from the back) in a way designed to minimize temperature inhomogeneity. This process, however, renders the substrate unsuitable for deposition purposes.

To monitor the relative temperature distribution across the substrate we have used a commercial silicon CCD camera (Pulnix 2010) and 10× macrozoom lens. At the temperatures routinely used in PLD this is sensitive enough to detect sufficient radiation from the substrate to give an accurate picture of the relative temperature profile with a maximum resolution of 640×480 pixels. The information from the CCD can also be fed back to the scanner via the computer to alter the temperature profile. By extending the dwell time of the laser on a particular array point, which is perhaps cooler than its neighbors, the relative temperature distribution can be controlled to maximize its homogeneity. This is particularly useful to offset the otherwise inevitable increased heating in the center of the substrate. Once set up it becomes trivial to adjust the scanning equipment via the computer to

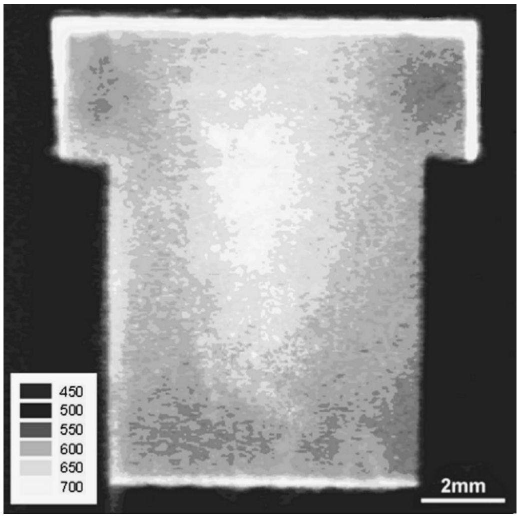

FIG. 4. Thermal image of the heated substrate without a feedback system, showing elevated heating in the center area.

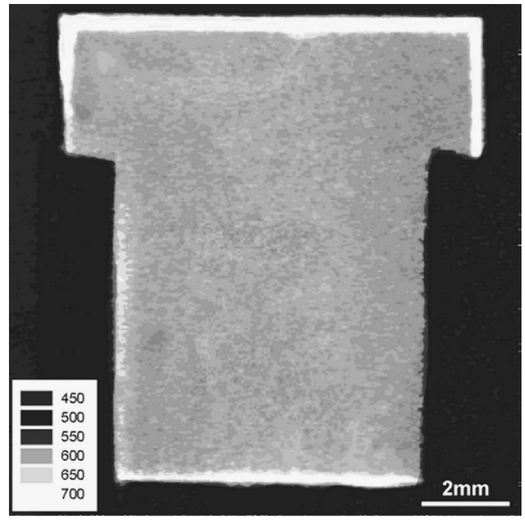

FIG. 5. Thermal image of the heated substrate with a feedback system enabled, showing a more constant heat distribution across the substrate.

allow for different sized or positioned substrates without any tricky realignment problems. Currently our system allows movement, expansion and contraction of the raster scan during heating and while in vacuum, permitting final adjustment and optimization if necessary.

IV. RESULTS

Figure 4 shows a typical temperature profile, obtained from a CCD camera, of a heated $Y_3Al_5O_{12}$ (YAG) substrate. This was recorded with no feedback control, making the dwell time of the laser at each point on the surface equal. The increased intensity seen at the edge of the substrate is not indicative of an elevated temperature as indicated. The high intensity seen is due to the light from within the substrate that is only able to escape at the edge due to total internal reflection at the face. The CCD camera is viewing the substrate at a slight angle, causing the top edge to appear brightest.

Ignoring such edge effects, Fig. 4 shows temperature variations between 590 and 690 °C over the substrate, which has an area of 1 cm^2.

Figure 5 shows the temperature distribution across the same substrate but with the feedback loop enabled. The beneficial effect this has on temperature homogenization can be clearly seen. In Fig. 5 the temperature variations across the same substrate of between 600 and 650 °C have been recorded.

V. DISCUSSION

We have found that for our requirements an array of 6×4 points produces a heating effect that is adequately uniform across the substrate. We can think of no reason why this technology cannot be scaled up to much larger dimensions or to higher precision. Larger dimensions would imply either a large increase in the array size of the raster scan or an increase in the beam size, which can be easily implemented by placing an appropriate lens in front of the scanning unit. Higher accuracy in heating homogeneity would imply having a larger array size, but this is then limited by the speed the scanning mirrors can complete one cycle of the raster scan. Ideally the mirrors should not take longer to complete one cycle than it takes for any particular point in the scan to noticeably change temperature due to the transitory nature of incident radiation.

In further work we will investigate more sophisticated methods of raster scanning to minimize cycle time. Heating of rotating or moving substrates, which are employed to improve thickness uniformity, would also be possible. Additionally, controlled heat gradients can be applied to the substrate (given a substrate able to withstand such thermal stress), causing different doping concentrations of certain elements whose inclusion in the film is heat dependent, as has been reported before for titanium doping in sapphire.[6]

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