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Author(s):
J. C. Olson
M. O. Thompson
H. A. Davis
D. J. Rej
W. J. Waganaar
D. R. Tallant

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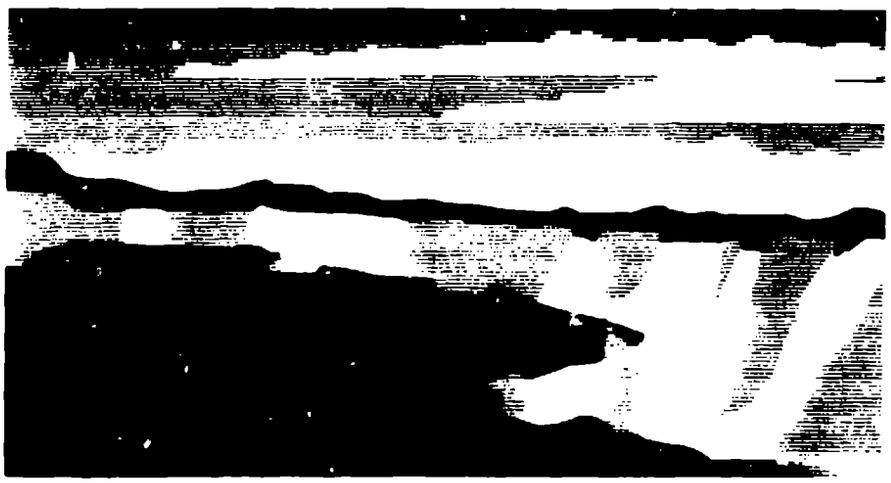

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SUBSTRATE HEATING MEASUREMENTS IN PULSED ION BEAM FILM DEPOSITION

J. C. Olson*, M. O. Thompson**, H. A. Davis*, D. J. Rej*, W. J. Waganaar*, and D. R. Tallant***

*Los Alamos National Laboratory, MS-E526, Los Alamos, NM 87544

**Materials Science and Engineering Department, Cornell University, Ithaca, NY 14853

***Sandia National Laboratories, P. O. Box 5800, Albuquerque, NM 87185-0343

ABSTRACT

Diamond-like Carbon(DLC) films have been deposited at Los Alamos National Laboratory by pulsed ion beam ablation of graphite targets. The targets were illuminated by an intense beam of hydrogen, carbon, and oxygen ions at a fluence of 15-45 J/cm^2. Ion energies were on the order of 350 keV, with beam current rising to 35 kA over a 400 ns ion current pulse.

Raman spectra of the deposited films indicate an increasing ratio of sp^3 to sp^2 bonding as the substrate is moved further away from the target and further off the target normal. Using a thin film platinum resistor at varying positions, we have measured the heating of the substrate surface due to the kinetic energy and heat of condensation of the ablated material. This information is used to determine if substrate heating is responsible for the lack of DLC in positions close to the target and near the target normal. Latest data and analysis will be presented.

INTRODUCTION

Pulsed ion beam deposition has the porential to become a low cost, high throughput (a few \$/m^2 at 50 m^2/hr for 1 μm coatings) film production process for flat panel displays, photovoltaic cells, and other applications. In this process, which is similar to pulsed laser deposition, an intense beam of ions (100-1000 keV, 10-100 kA, 0.1-1.0 μs) is directed toward a target, ablating some target material which condenses on a substrate as a thin film. An ongoing effort at Los Alamos National Laboratory is examining both science and technology issues related to pulsed ion beam deposition. The present work is concerned with heating of the substrate by the hot, dense ablation plume from a graphite target and the effect of this heating on the properties of the deposited material. Substrate heating by the ablated material was examined using thin film metal thermometers.

EXPERIMENTAL SETUP

A schematic of the experimental configuration is shown in Figure 1. The Anaconda generator at Los Alamos produced the intense ion beams used in these experiments. The machine is coupled to a focusing magnetically insulated diode, out of which a 400 keV, 30 kA, 700 ns ion beam is extracted. Ablation targets are placed 35 cm from the anode, at the beam focus, where a peak beam energy density of approximately 30($\pm$15) J/cm^2 is

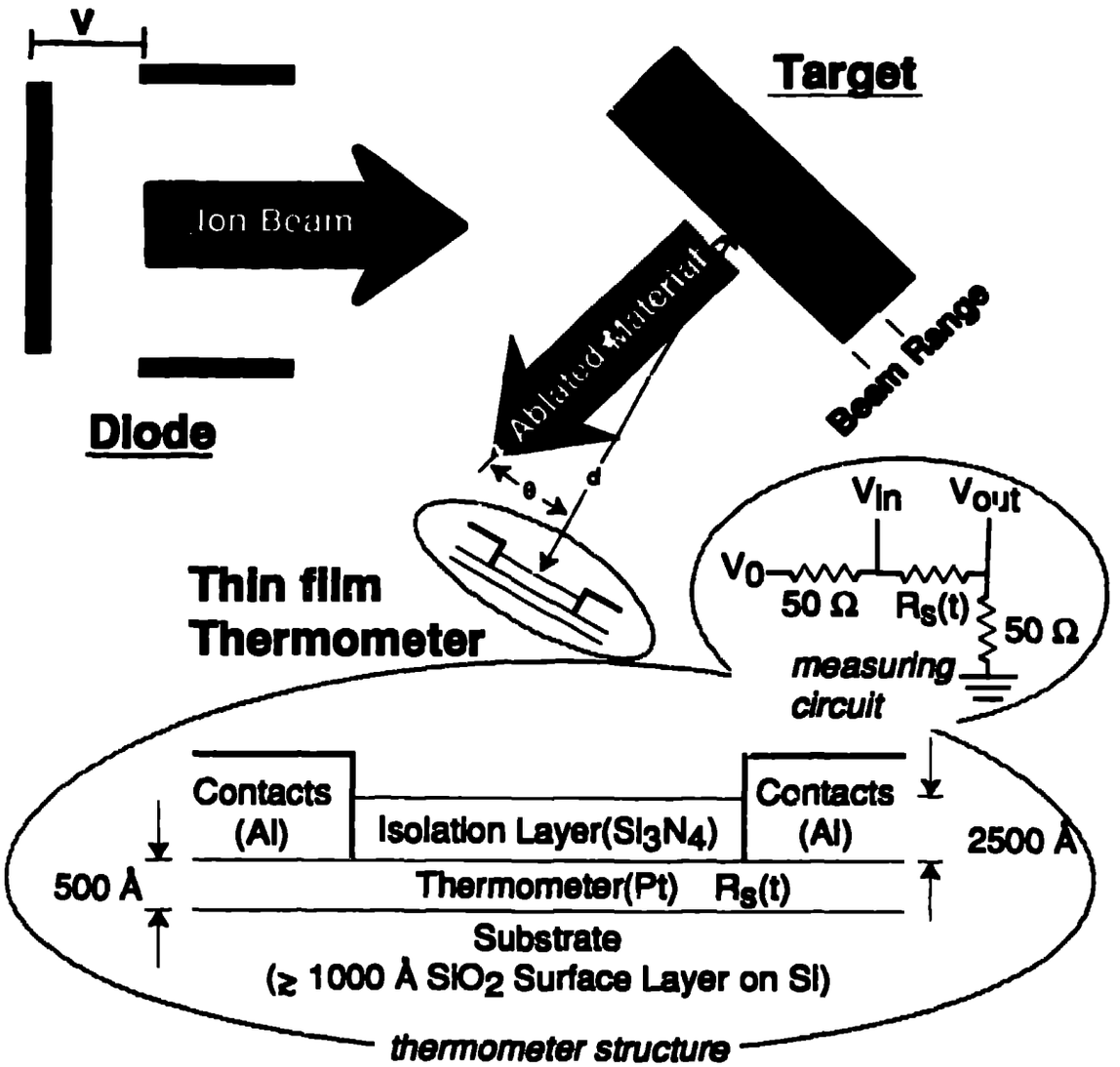

FIG. 1. Schematic of the experimental setup and the thin film thermometer.

delivered [1,2]. A single machine pulse typically ablates 10 mg of carbon from a POCO graphite target.

Substrate heating for various target-substrate separations and orientations was examined by the use of thin film metal thermometers. This technique, which relies on the temperature dependent resistance of a thin metal film to provide heating information with nanosecond time resolution, has previously been used to examine ablation in pulsed laser deposition experiments and melt dynamics in pulsed laser induced melting experiments [3–7]. Our experiments varied the target-thermometer separation, d, from 10 to 20 cm and varied the thermometer position relative to the target normal from θ=0 to θ=30°.

Thermometer construction and a circuit description are shown in Figure 1. The relatively thick (2500 Å) Si_3N_4 isolation layer served two purposes: to diminish capacitive copling between the thermometer and beam created plasmas; and, more importantly, to prevent plasmas from shorting out the resistance of the thermometer. Details about the fabrication and calibration of this type of thermometer may be found elsewhere [3]. Thermometers in our experiments had room temperature resistances around 150 Ω and temperature coefficients of resistance ($\gamma = 1/R\ dR/dT$) of 0.0032 K^{-1}.

Temperature measurement was accomplished by measuring the time-dependent thermometer resistance, $R_s(t)$. This was done by applying a pulsed bias, V_0, of 10 V to the measuring circuit. The voltages above and below(with respect to ground potential) the thin film thermometer layer, $V_{in}(t)$ and $V_{out}(t)$, respectively, were monitored using HP54111D digitizing oscilloscopes. $R_s(t)$ was then calculated from

$$R_s(t) = \frac{100}{\frac{V_0}{V_{in}(t)-V_{out}(t)} - 1}. \tag{1}$$

The quantity $V_{in}(t) - V_{out}(t)$ should remain equal to V_0 and provided a measure of the noise. Using (1), the time dependent thermometer temperature was computed from

$$T(t) = \frac{1}{\gamma}\frac{R_s(t) - R_s(0)}{R_s(0)}. \tag{2}$$

Heating power on the substrate was found by deconvolving the solution to the heat equation

$$T(t) = \int_0^t \frac{P(t')}{\sqrt{\pi \rho c_p \kappa (t - t')}} dt' \tag{3}$$

where ρ, c_p and κ are the density, heat capacity, and thermal conductivity of the thermometer. For the time scales of interest in our experiment, it is sufficient to use ρ, c_p and κ for the bulk material, namely silicon. From the form of (3) it is clear that the experimentally measured $T(t)$ can be applied to other substrate materials simply by scaling by the appropriate ρ, c_p and κ.

RESULTS

Figure 2 shows the substrate temperature rise during carbon deposition at three different positions: d =15 cm, $\theta = 0°$; d =20 cm, $\theta = 0°$; and d =20 cm, $\theta = 30°$. Figure 3 shows the heating power due to the ablation plume at the same three positions, and Figure 4 shows the estimated temperature rise of a glass substrate at those positions. We also took data with the conditions d =10 cm, $\theta = 0°$.

Expansion of the plume is clearly evident in the power data. Substrate heating decreased with increasing angle and with increasing target substrate separation, from 70 kW/cm^2 for approximately 10 μs at d =10 cm, $\theta = 0°$ to 5 kW/cm^2 for approximately 20 μs at d =20 cm, $\theta = 30°$. Arrival time of the leading edge of the ablation plume was used to compute a plume expansion velocity. The measured value was 1 cm/μs on the target normal, decreasing to 0.4 cm/μs at $\theta = 30°$. These values are necessarily crude, due to low statistics and large shot to shot irreproducibilities in the pulsed ion beam, but never the less are in reasonable agreement with the plume expansion velocity of 2 cm/μs measured by visible light framing pictures. A better measure of the plume energy content can be made by integrating the power to find energy, and dividing the energy by the number of atoms deposited. 25 nm of film at a density of 1.7($\pm$0.3) g/cm^2 is deposited in a typical pulse [8], and the energy found by integrating the power curve for either position on the normal is 0.7 J/cm^2, giving a plume energy of 19 eV/particle. This calculation assumes that all the heat absorbed by the substrate is contributed by particles which condense on the surface, and is therefore likely to be an overestimate of the plume temperature.

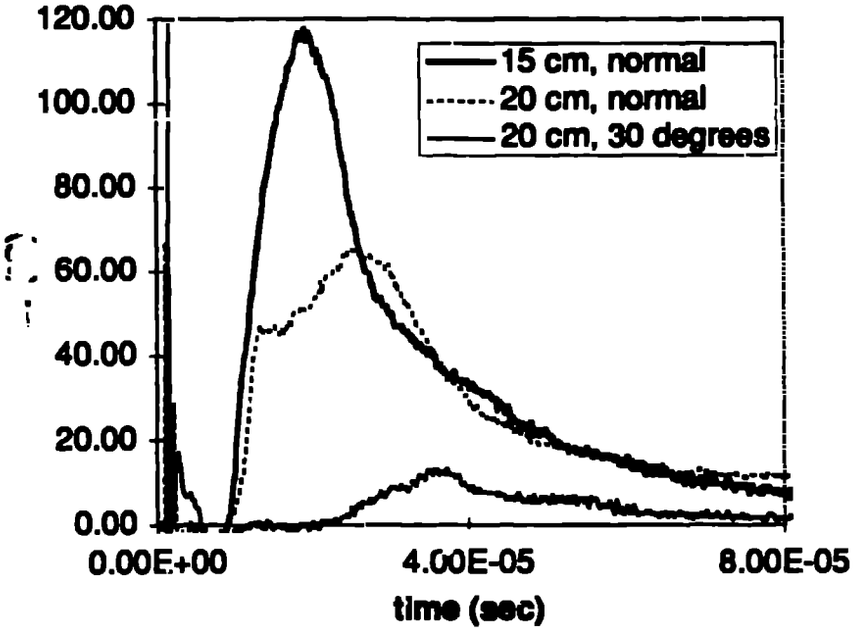

FIG. 2. Temperature rise as measured by thin film thermometers located: d =15 cm, θ = 0° (thick solid line); d =20 cm. θ = 0° (thin dashed line); and d =20 cm, θ = 30°(thin solid line).

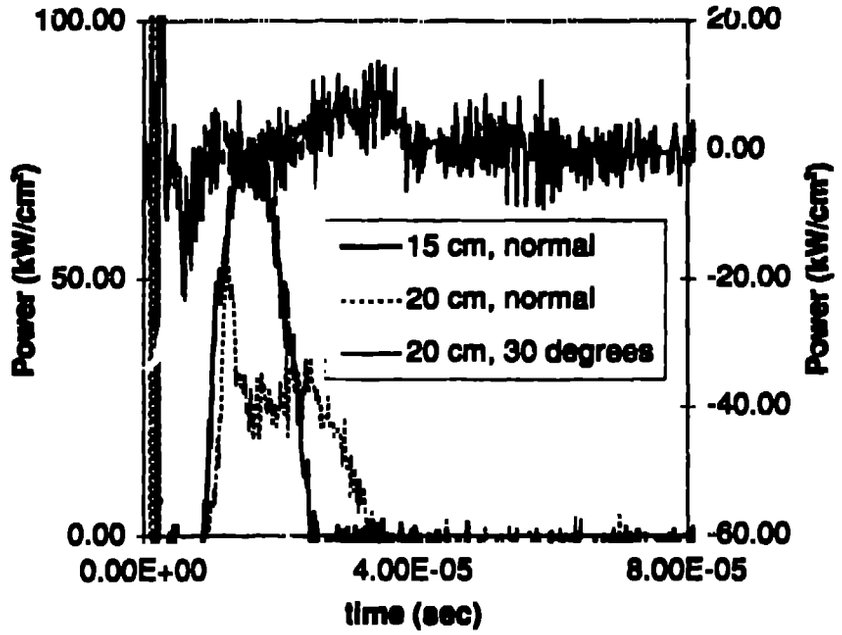

FIG. 3. Calculated heating power due to the ablation plume at positions: d =15 cm, θ = 0° (thick solid line - left vertical axis); d =20 cm, θ = 0° (thin dashed line - left vertical axis); and d =20 cm, θ = 0° (thin solid line - right vertical axis).

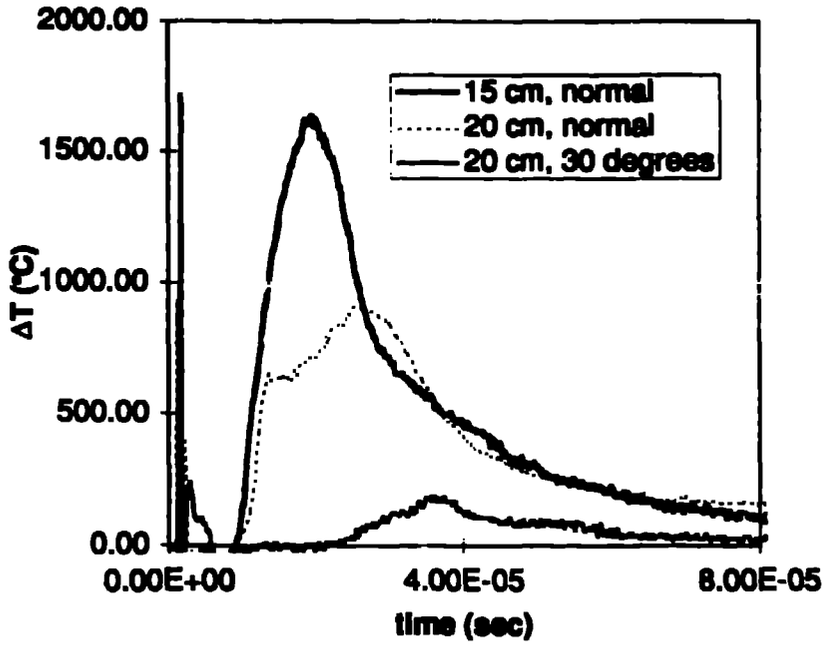

FIG. 4. Calculated surface temperature rise for glass substrates located: d =15 cm, θ = 0° (thick solid line); d =20 cm, θ = 0° (thin dashed line); and d =20 cm, θ = 30°(thin solid line).

DISCUSSION

The Raman spectra of films deposited at d =22.5 cm, θ = 0° and d =15 cm, θ = 50° showed diamond like character, but the film obtained at d =15 cm, θ = 0° was glassy [8]. This appears to be due to the high substrate temperature reached during deposition at this position, which promotes formation of thermodynamically stable sp^2 bonds. Plume energies appear well-suited to the formation of diamond-like carbon [9], and deposition onto a more thermally conductive substrate should allow the production of high quality DLC films at high rates even on the target normal at low target-substrate separations.

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