Laser Reflectance Interferometry for In-Situ Growth Monitoring and Characterization of Polydiamond, Nanodiamond, graphene, CNTs, and Nitride Semiconductors

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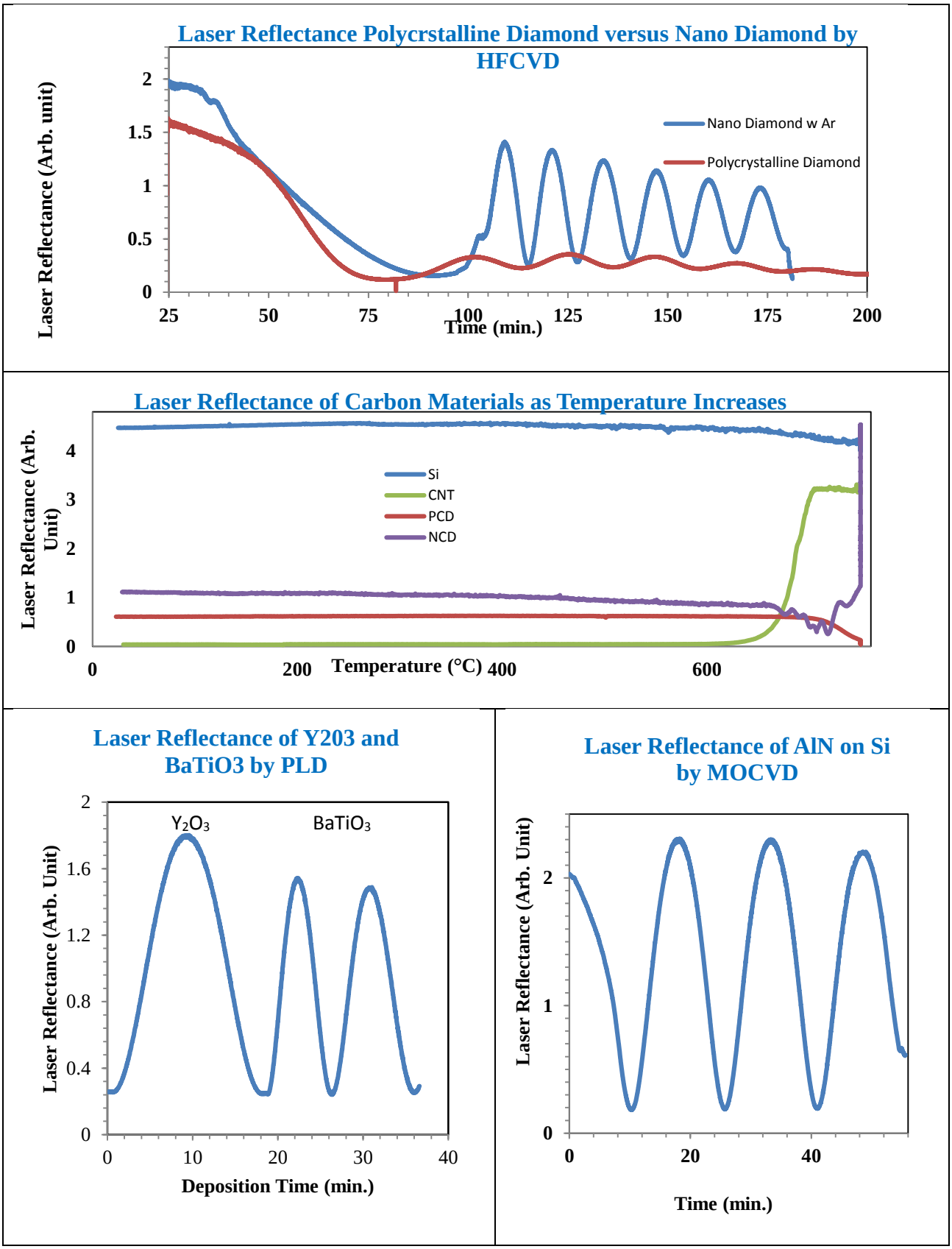
Laser reflectance interferometry tool (LRI) is developed for in-situ measurement of the growth characteristics of carbon based thin films materials. LRI tool integrated with hot-filament CVD (HFCVD) was used to grow films of diamond, nanodiamond, graphene, and thermal-CVD was used for carbon nano tubes (CNTs). LRI allows the in-situ measurement of the growth rate and the surface roughness of the samples as they were grown. This process provides real time information into the growth of films and can quickly illustrate growth features. The in-situ measurements allow for quick determination of the effectiveness of initial diamond seeding of the films. By knowing the wavelength of the laser and by knowing the refractive index of the diamond film, growth rate and film thickness can be determined.

Using LRI integrated HFCVD; growth parameters of poly and nanodiamond films were correlated such as seeding process and optimization, CH_4 concentration, negative biasing, filament temperature, and Ar/H_2 ratio on nanodiamond growth. LRI results clearly indicate that seeding procedure strongly affects initial growth stages of diamond film through early start of oscillations. As the film starts to grow the laser reflectance decreases, until nucleation layer is continuous on the substrate. After that laser reflectance starts to increase and oscillations can be measured. Since the time from peak to peak is used to measure the growth rate of the sample, LRI can be used to determine how growth parameters affect the growth rate and surface morphology of the deposited sample. Filament temperature had the greatest effect on the growth rate of diamond samples. Increasing CH_4/H_2 flow decreased time to nucleation, but had little effect on the growth rate once the film had nucleated. Increasing CH_4 concentration increased the growth rate. SEM measurements were conducted to confirm the in-situ film thickness measurements using LRI.

LRI is also used for characterization of combustion of carbon materials. The materials tested were CNTs, polycrystalline diamond, and nanodiamond films heated in air. Each phase of carbon form (polydiamond, nanodiamond, CNTs) has it its own characteristic behavior. The characteristic onset combustion temperature strongly depends on the form of the carbon (sp^3 vs. sp^2). The LRI for polydiamond has constant reflectance until it decreases at 700°C. Raman spectroscopy showed this was due to the destruction of the sp^2 bond but that the diamond (sp^3) counterpart remained intact. CNTs and nanodiamond both showed constant laser reflection until total destruction of the carbon films. CNTs were completely and combusted at 660°C, nanodiamond at 740°C, indicated by a strong change in reflectivity. These results will be presented in the light of laser reflectivity monitoring tool integrated with Blue Wave HFCVD or other CVD or PVD techniques used for monitoring growth and characterization of carbon and related optical thin film and coating materials.

Figures:

Below we highlight important results related to this research work. The additional supporting results will be presented in the conference.


Laser Reflectance Polycrstalline Diamond versus Nano Diamond by HFCVD
Laser Reflectance (Arb. unit)
Time (min.)
Nano Diamond w Ar
Polycrystalline Diamond
Laser Reflectance of Carbon Materials as Temperature Increases
Laser Reflectance (Arb. Unit)
Temperature (°C)
Si
CNT
PCD
NCD
Laser Reflectance of Y203 and BaTiO3 by PLD
Laser Reflectance (Arb. Unit)
Deposition Time (min.)
Y2O3
BaTiO3
Laser Reflectance of AlN on Si by MOCVD
Laser Reflectance (Arb. Unit)
Time (min.)