Seeded Growth of Nanocrystalline CVD Diamond Films with Nanodiamonds

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High quality nanocrystalline CVD diamond films are grown for a variety of applications including MEMS, NEMS resonators, coatings, silicon-on-insulator wafers, optical pellicle beamsplitters, and particle detectors. These films can be continuous at thicknesses of as little as 30 nm, have exceptional mechanical and thermal properties, and have surface roughness varying from 3 nm (RMS) at 60 nm thickness to ~ 20 nm at 1 micron thickness. The seeding procedure is a vital step in fabrication of these smooth, particle- and pinhole-free high quality nanocrystalline diamond films, with many of the properties depending on the seed density. This work is an effort to determine and understand the critical aspects of the seeding process in order to achieve higher nucleation density and a uniform, monolayer distribution of seeds.

The standard seeding technique we use involves the ultrasonic treatment of a silicon substrate in an ethanol-based nanodiamond suspension derived from explosively formed nanodiamonds. Currently we are able to produce high quality diamond films with a seed density greater than 10^{12} cm^{-2}. Based on empirical data, we believe that the thin film surface roughness is a function of nucleation density, seed distribution uniformity, and film growth conditions.

We have examined the effects of the concentration in the nanodiamond seeding solution and the seeding procedure, including treatment time, use of ultrasonic excitation, and rinse and drying procedures. The growth conditions remained constant in this experiment: nanocrystalline films were deposited on 100 mm Si/SiO_2 wafers using microwave plasma CVD. The presence of the SiO_2 layer was necessary in order to allow a brief dip in HF to highlight pinholes. For consistent initial conditions, all substrates underwent the SC-I cleaning procedure followed by megasonic rinsing and spin drying. Subsequently, a CVD diamond film was grown to 300 nm thickness, which had previously been determined to be optimal for these tests.

Increasing nanodiamond concentration by a factor of two resulted in pinhole free films and visually appeared to be of a very good quality. Optical microscope analysis (Nomarsky Interference Contrast) revealed a surface non-uniformity in a shape of some large (hundreds of micrometers) areas with differing thickness. It appears that the increase in nanodiamond concentration produces an uneven deposition of a multiple layers of seeds, which ultimately increases film roughness. This imperfection becomes unnoticeable once films reach ~600 nm.

Based on this outcome, we were compelled to focus our research on the parameters of ultrasonic treatment in order to understand the peculiarities of nucleation mechanism. The samples in the first series were seeded by means of ultrasonic treatment for various duration of time. For the second series the absence of ultrasonic treatment was the key, though the seeding solution was ultrasonically agitated before its use. Samples were dipped or soaked in the seeding solution for a various times. The SEM analysis of seeded samples, as well as 300 nm thick films was performed to determine the nature of the surface properties.

Nanodiamond particles tend to agglomerate in solution and are easily attached to the treated sample. The role of ultrasonic preparation of the solution is to lower the agglomeration in solution as well as on the surface of the sample. The concentration of the seeding solution is an important factor. Ultrasonic treatment of the wafer in the seeding solution for a sufficient amount of time allows deposition of an evenly distributed layer of seeds on the surface of the substrate and suppresses the uneven build up of seeds and the resulting non-uniformity in film thickness. Together with the optimized seeding solution, proper ultrasonic treatment is a critical step for the fabrication of very smooth and pinhole free nanocrystalline diamond films.