

Diamond CVD, NanoBOND/DEBOND System, and Furnace Characterization

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Primary CNF Tools Used: MOS Clean Bench & Tanks, MRL E2 CMOS Wet Oxide, MRL B3 LPCVD LTO, Woollam RC2 Spectroscopic Ellipsometer, Filmetrics F50, Bruker DektakXT Profilometer, Zeiss Gemini SEM, Zeiss Ultra SEM, Keyence VHX-7100 Digital Microscope, NanoBOND 200, NanoDEBOND 200

Abstract:

Summer project goals included work on the Nordtech funded Seki Diamond chemical vapor deposition tool and the Nordtech funded OnNano NanoBOND 200 and NanoDEBOND 200 system. The goal with the Seki Diamond tool was to characterize and perform a hardware assessment of the tool. The work completed on that project was the development of a nanodiamond seed crystal coating process, essential for serving as a nucleation point for diamond chemical vapor deposition. Project time was limited when the tool was shut down for repair. This caused a pivot to the NanoBOND 200 and NanoDEBOND 200 system. The task was to perform a hardware assessment of the system and develop processes for operation, with the ultimate goal being a complete operating manual.

Summary of Research:

Nanodiamond Seed Crystal Process

The Seki Diamond tool functions by using a microwave plasma, 99% Hydrogen and 1% methane. In the plasma is a diamond seed crystal which the methane will contact, depositing its carbon atom onto the diamond lattice structure. The remaining hydrogen in the methane molecule will join the existing hydrogen plasma cloud. This reaction builds a layer of diamond, atom at a time on top of a diamond seed crystal. The reaction will only grow diamond on diamond. For this purpose, it is essential to the process that we have proper diamond seed crystals for diamond chemical vapor deposition.

The Seki Diamond tool uses a molybdenum puck as its base for seed crystals. Seed crystals can either be monocrystalline, one large diamond with a singular lattice direction, or polycrystalline, many smaller diamonds with various lattice directions. To initially

characterize the diamond growth rate, polycrystalline seeds were preferred for their much lower cost. The seed crystals are roughly 20nm in diameter, requiring a scanning electron microscope for imaging. This factor meant the original molybdenum puck would have a surface too rough to observe any seed crystal deposition. Instead, two-inch silicon wafers were preferred for their availability and smooth surface, even though they will never be used in the Seki Diamond tool. A ScienceDirect article recommended starting with a 50% dimethyl sulfoxide (DMSO) diamond solution to 50% methanol. A two-inch wafer was placed in this 50/50 solution and moved into an ultrasonic bath for 10 minutes, after the bath wafers were rinsed with more methanol and dried with nitrogen [1]. When this recipe was observed under the Zeiss Gemini SEM560 scanning electron microscope, many of the diamonds had collected into water spots and were not evenly dispersed across the wafer. To solve this the process was modified to see if a lower concentration, different cleaning method, and different sonication times, improved results. The ideal recipe found was a 25%/75% DMSO to methanol solution, sonicated for 15 minutes, and cleaned with isopropanol (Figure 1).

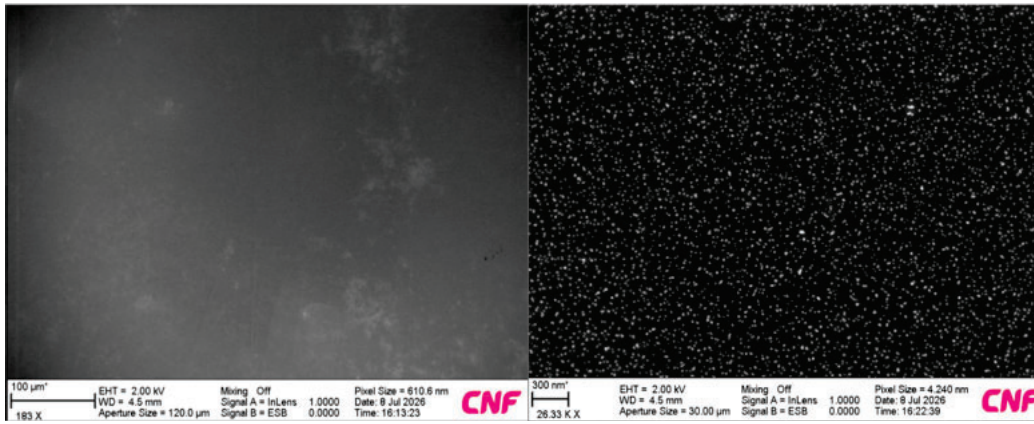


Figure 1: Nanodiamond Seed Crystals. 15 minutes, 25% DMSO, cleaned with IPA.

NanoBOND 200 and NanoDEBOND 200

The hardware assessment for both the NanoBOND 200 and NanoDEBOND 200 involved testing, experimentation, and troubleshooting. The NanoBOND 200 and NanoDEBOND 200 system functions by coating a carrier wafer with a bonding polymer and bonding to either another target wafer or wafer piece. Theoretically the carrier is thicker and stronger than the target, adding structural support as the target undergoes process steps. When the carrier has served its purpose, the bonded stack is then slid apart in the NanoDEBOND 200 tool. Initially much time was devoted to developing a spincoating process for MicroResist, mr-glue-xp bonding polymer. The given process yielded streaking and uneven polymer surfaces. Through trial and error, a two-step spinning recipe was created for the three different carrier wafer sizes, four inch, six inch, and eight inch.

Our operating process had to be adjusted for the original setup of the tool. The NanoBOND 200 has two vacuum chucks, one for the carrier and one for the target, however neither chuck has an included aligning mechanism. To resolve this, a wafer aligner for each chuck was modeled and is being machined. The NanoDEBOND also required other adjustments. One being that during the debond process the carrier is supposed to be held in place on the carrier vacuum chuck not only by vacuum, but by a physical lip impeding the direction of sliding. This lip was measured to be 600 microns thick, 75 microns thicker than a standard four-inch wafer. This led to bonded stacks on four-inch carrier wafers breaking as they hit the lip when debonded. A fix to machine down this lip is on the way. Another critical flaw with the debonder was the lack of any refined wafer removal mechanism. Originally the debonded carrier and target wafers were removed with tweezers or by hand. Since they are oriented vertically and at temperatures over 100

degrees Celsius this is suboptimal. An effective solution was using a vacuum wand to remove the wafers. After the hardware assessment and subsequent solutions, a full manual of operation was made, including all operation steps for spin coating, bonding, and debonding, as well as general information useful for future users.

Furnace Characterization

Additionally, during transition time between projects time was allocated for work on the E2 Wet Oxide furnace and the B3 LPCVD furnace. The goal for the E2 wet oxide was to create a Deal-Grove equation for oxide thickness. This was done using nine trial runs of varying time and temperature. A model was made that can predict thickness with 80% accuracy, which needs more refining before further use. Work on the LPCVD furnace used three runs of 35 wafer batches to measure uniformity across wafer position in the tube. This successfully identified variation is presented in the front of the tube.

References:

- [1] Shenderova, O. A., & ScienceDirect. (2010, March). Seeding slurries based on detonation nanodiamond in DMSO. *Diamond and Related Materials*, Volume 19(2-3), 260-270. <https://www.sciencedirect.com/science/article/pii/S0925963509002830>