How do I disperse diamond powders in solution?

In many applications, having a well-dispersed solution of nanodiamonds in either water or a solvent is critical to achieve desirable results. Aggregates of nanodiamonds (and, indeed, most nanomaterials) do not perform as well as well-dispersed particles in many applications. Starting with diamond powder allows for precise control over the concentration of diamonds in solution, but suspending the diamonds in solution can be tricky without the proper instrumentation.

It is important to understand that the 'dispersibility' (the extent to which diamonds can be dispersed without aggregation in a solvent) is a complex issue that depends on multiple factors, some of which are inter-related with each other, such as: (1) nanodiamond surface chemistry, (2) particle size, (4) pH, and (5) solvent type. For consideration on these topics, see ***My nanodiamond particles are unstable in solution, what should I do?*** or ***What solvents can be used with nanodiamonds?*** For the purposes of this discussion, it will be assumed that you are working with a solvent in which the particular diamonds are well dispersed, and you are starting with diamond in powdered form.

Methods of Dispersing Diamond Powders

There are several common laboratory techniques for suspending nanodiamond powders in solution: (1) ultrasonic dispersion with an ultrasonic bath, (2) ultrasonic dispersion with a cavitation ultrasonic horn, and (3) vortex mixing. A schematic of each approach is shown below.

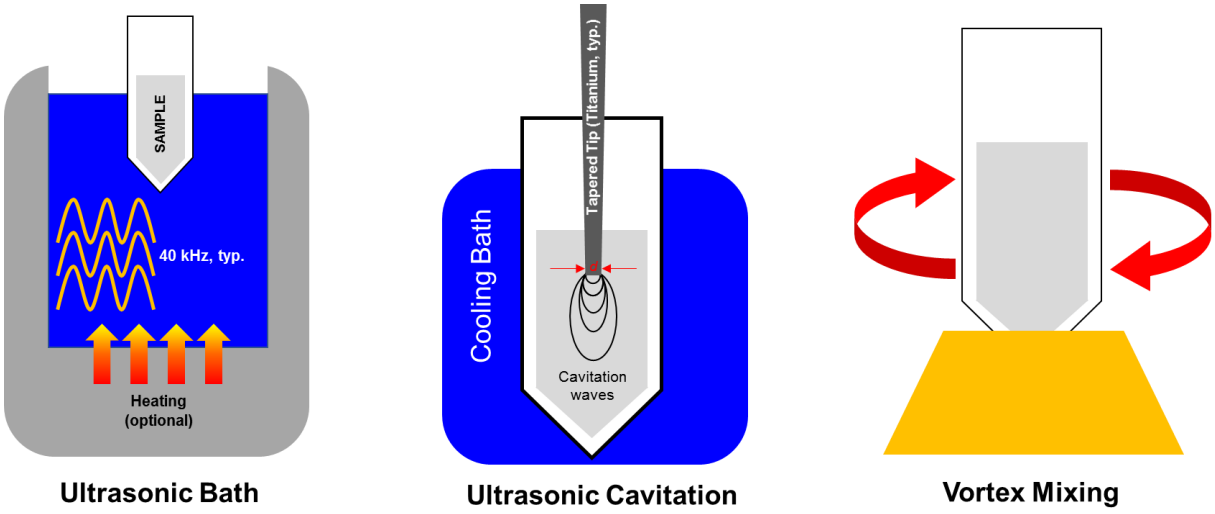

Figure 1. *Schematics of each dispersion technique. With an **ultrasonic bath**, a sample contained within a vessel is dipped into a bath in which ultrasonic waves are passed through, and ultrasonic cavitation occurs with appropriate conditions. Heating can be applied in many laboratory ultrasonic baths. With horn-based **ultrasonic cavitation**, a metallic (typically titanium) probe with tip diameter 'd' is placed within the sample containing liquid, and ultrasonic waves generate cavitation near the probe tip. This method provides very high-power densities. **Vortex mixing** is primarily used to re-constitute particles after centrifugation, or to facilitate homegenous mixing of two or more components quickly. It can be used to facilitate solution of diamond powder to a small extent, but it does not provide any means of disaggregation if aggregation does occur, which is typical when starting with nanodiamond powder.*

	Ultrasonic Bath	Ultrasonic Cavitation	Vortex Mixing
Typical Specifications	-40 kHz operating frequency (typical) -Maximum power: ~100 watts/gallon (26 watts/liter) -1 L to 20 L bath volumes	- 25-30 kHz operating frequency (typical) - Maximum power: 500 W - >10 kW - 250 uL (sample) to 20 L/h (batch processing) with flow cells	- ~100 to ~3000 rpm operation - Pressure activated by sample vessel
Advantages	-No sample contamination -Cooling (or heating) provided by bath -Simultaneous treatment of multiple sample -Cost ($100-$3k range typical)	- Excellent dispersion and disaggregation (high power density) - Rapid treatment times (seconds to minutes) - Small treatment volumes possible (uL), and flow cells can allow for large process treatments - Small footprint	- Simple to use - No external contamination of sample - Compatible with biological materials (gentle) - Inexpensive ($100 to $500 typical) - Very small footprint
Disadvantages	-Poor disaggregation, lower power density -Non-uniform sample treatment (tricky sample positioning in bath) -Long treatment times (minutes to hours) -Attenuation of ultrasonic waves through sample vessel wallsPotentially large footprint	- High cost (>$4 k typical) - External cooling necessary, sample can become overheated quickly - Sample contamination from ultrasonic tip over time - Tips require replacement over time, and cost on the order of several hundred dollars	- Does not provide a means of disaggregation - Diamonds can stick to sample vessel side walls in some instances
Recommendation	***Not recommended*** for obtaining well dispersed, homogenous nanodiamond suspensions. They can be used to help reconstitute materials after centrifugtation (esp. when used in conjuction with vortex mixing).	***Highly recommended*** for achieving optimal disaggregation and dispersion of nanodiamond powder into solution.	***Recommended*** as a general use tool when working with nanodiamond. Vortex mixing provides a simple means of reconstituting centrifuged material, and it is necessary when working with biological materials.

Tech-Note: Dispersing Diamond Powders

Typical Procedure for Dispersing Diamond Powder Using Ultrasonic Cavitation Probe

The following is a general use procedure that was written to prepare a 10 mg/mL (1% w/v) 100 nm carboxylated HPHT diamond solution. It can be adopted to other diamond powders and sample holding vessels; however, some procedural development may be needed depending on the specific material. The procedure was written using a Cole Parmer 750 W ultrasonic processor with a 1/8" probe.

1. Weigh an amount of 50 mg of nanodiamond powder, and add to a 15 mL conical polypropylene centrifuge tube.
2. Add up to approximately 5 mL of deionized water to the centrifuge tube.
3. Set the amplitude of the ultrasonic processor to 22%.
4. Fill a separate 15 mL conical centrifuge tube to at least 5 mL of volume with deionized water, and immerse the ultrasonic horn into the water. Briefly (~5 seconds) turn on the ultrasonic processor to rinse any material that may have been on the surface of the probe.
5. Remove the probe from the deionized water, then briefly pulse the ultrasonic horn (~0.5 s) to remove residual water from the probe.
6. To a separate container, add cold water (ideally, ice-chilled water), and then place the centrifuge tube containing the diamond and water suspension into the cold water bath.
7. Submerge the ultrasonic probe into the sample vial. Make sure the probe is not touching the side walls of the centrifuge tube, or, if it is, make sure the tube walls can be freely moved away from the probe tip. Do not force the probe tip to the bottom of the centrifuge tube.
8. Turn on the ultrasonic processor and let it run for 3 minutes (*this time can be made much shorter in reality, in this case, less than 1 minute would be sufficient*). Make sure powder does not settle on the bottom of the centrifuge tube, and move the tube up and down to facilitate removal of the material from the bottom of the vial if necessary.
9. Preparation of 10 mg/mL (50 mg, 5 mL) 100 nm solution is complete.

Useful Links

Cole-Parmer Ultrasonic Processors
Replacement Ultrasonic Probe Tapered Microtips
Ultrasonic Processor Supplier (Lab and Industrial Scale) (Hielscher)
Vortex Mixer Supplier
Ultrasonic Baths/Cleaners (Fisher Scientific)