Light Hydraulic Effect in Laser Nanodiamond Synthesis, Optimizing Parameters for the Output Increase

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Abstract

Nanodiamonds firstly discovered in 1963 in detonation soot are highly efficient in various processes, - in electroplating, polymer compositions, polishing, lubricants and sintering [1]. However, the implementation of nanodiamonds in industry is still restricted due to their insufficient homogeneity mainly caused by non-controlled process of their fabrication by detonation of explosives in closed chambers. Light Hydraulic Effect (LHE) was applied for laser assisted synthesis of pure and uniform nanodiamonds. This method is based on the focusing laser radiation of high power density in liquid at some distance from specially prepared hydrocarbon target [2]. A few approaches of technology optimization were considered: using solid state Q-switch lasers and fiber lasers with laser pulses in the range from femto- to nanosecond pulse lengths, maximally suitable repetition rate and the delivered power density of 10^7 – 10^10 W/cm^2. The optimization of laser parameters of the existing laboratory process has a clear promise of raising the nanodiamond output by 2 orders of magnitude.

Reference

1. Ultrananocrystalline Diamond. Synthesis, Properties, and Applications, edited by O. Shenderova and D. Gruen, Published by William Andrew Publishing 2012

2. B. Zousman and O. Levinson; Chapter 5 in book Nanodiamond edited by Oliver Williams, RSC Nanoscience & Nanotechnology, London, 2014

Background. Nanodiamonds: Structure, Properties, Synthesis, Applications

Nanodiamonds (ND). Carbon nanoparticles with cubic diamond structure in the core and a surface shell of carbon-onion-like structure and various functional groups (Fig.1).

ND structure & properties. Diamond core: highest hardness (167 Gpa {111}) and wear resistance, highest thermal conductivity (2300 W/mK), high electrical resistivity (10^{13} Ωcm), low thermal expansion (1.0 × 10-6 K-1), wide band gap (5.47 eV {300 K}), high refractive index (2.417), low specific gravity (3.52), chemical and radiation resistance, high biocompatibility [wiki]; specific ND features: tiny size (< 100 nm, usually < 10 nm), large surface area (250-450 m²/g).

High and controllable chemical activity of ND surface enables desired interaction between ND and various materials and objects transferring them unique diamond properties.

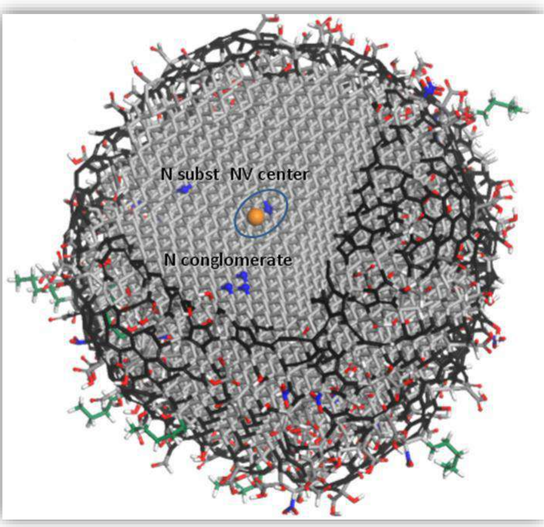
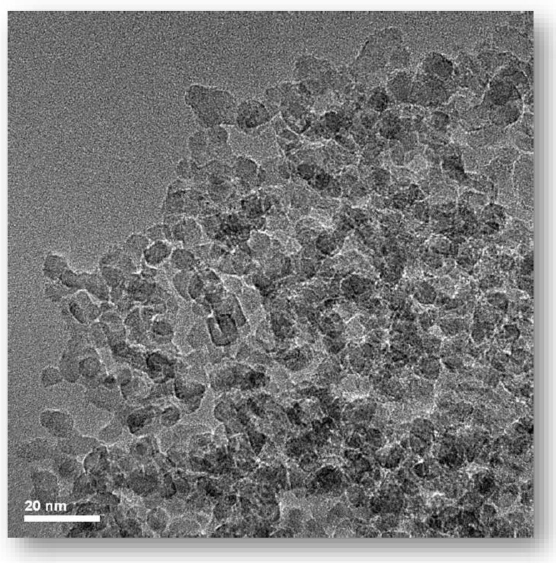


Fig. 1. ND structure:
***Gray**: cubic diamond lattice (sp3)*
***Black**: graphitic structure (sp2)*
***Red & green**: oxygen & hydrogen containing functional groups*
***Yellow**: N-V center*
The image of Dr. Vadim Mochalin, Drexel University.

***Fig.2. HR TEM image of ND powder:** high aggregation*



For most applications ND surface treatment is needed to provide disaggregation and uniform ND distribution within chosen matrix

ND applications: additives to polymers, coatings and lubricants, fine polishing, adsorbents, catalysts, contrast agents in biomed research [1].

ND production. Currently ND with average size of 4-5 nm are produced by non-controlled and hazardous technology of detonation synthesis (TNT and RDX mixture). Non-constant quality and metal impurities restrict the possibility of ND implementation in industry and medicine. ND with average size of more than 30 nm are produced by High Pressure High Temperature (HPHT) method, the main lack – too wide PSD.

Laser ND synthesis: Novel technology of ND synthesis named Light Hydro-Dynamic Pulse (LHDP) has been developed and implemented in a laboratory scale ND production. Specially prepared hydrocarbon targets are treated with shock-wave caused by LHE in liquid (Fig.3)

Light Hydro-Dynamic Pulse Method

Technology. LHDP process includes:

- Forming multi-component targets containing carbon black and wax
- Laser treatments of targets in liquid (fig. 4)
- ND isolation: oxidation, flotation, washing, drying

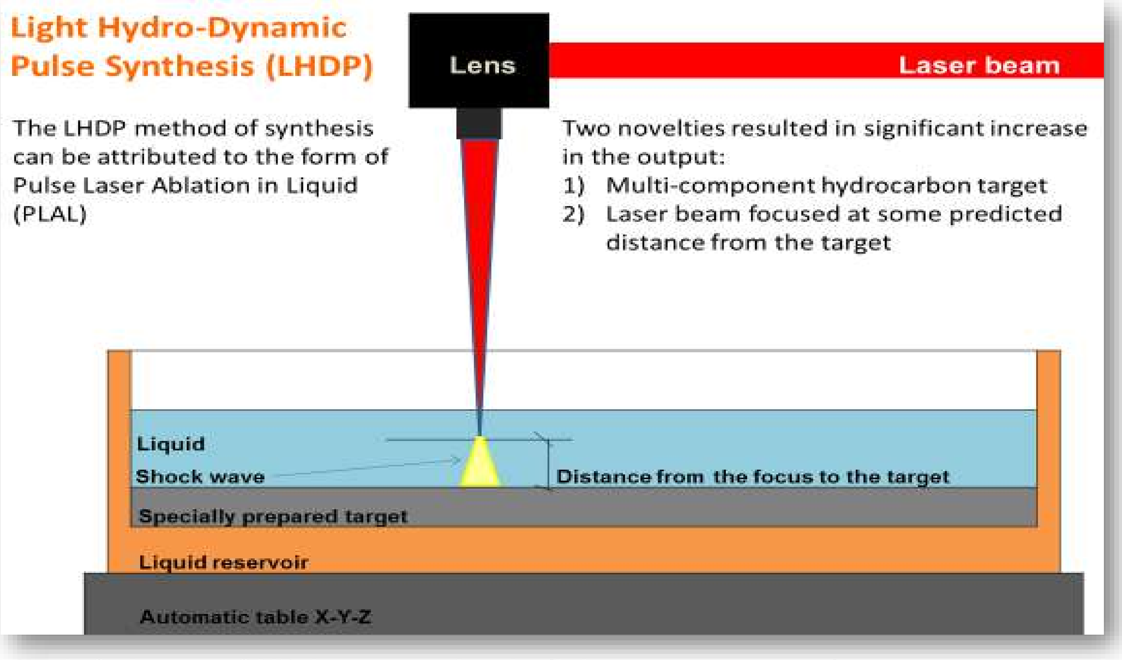


Fig.4. Scheme of ND laser synthesis (laboratory process)

Advantages:

- Controlled process providing constant ND quality
- Non-hazardous & environment-friendly process
- No security requirements and limitations
- High quality of ND (Table 1)

Trait	Analysis	DND	LND
Purity	Incombustible residue	0.4 – 8.0 wt.%	Non-detected (<0.02 wt.%)
Metals	SEM	Fe, Ti, Al, Ca	Non-detected
sp2	XPS	74.4 %	15.5 %
Size uniformity	PSD, TEM images	2-18 (50) nm; Av.: 4.5 nm	2-10 nm Av.: 4.5 nm

Table.1 Comparison of laser ND (LND) and detonation ND (DND) quality

Experiment, Results & Discussions

Experiment:

Laser Parameters

Laser type	Nd:YAG
Wave length	1.064 um
Pulse duration	30 ns
Maximal pulse energy	45 mJ
Frequency	150 Hz
Spot size (focusing optics)	30 um

Power density in spot was varied from 10^5 to 10^11 W/cm2.

ND were weighted & analyzed with XRD (Fig. 5)

Results and discussions

1. Minimal power density for ND formation is 10^6 W/cm2. Only 40 % of the powder is diamond, the rest: amorphous C
2. 10^7 W/cm2 power density provides 100 % ND crystallinity with particles average size 4.5 nm.
3. Increase in the power density from 10^7 to 10^10 W/cm2 enhanced the output from 0.9 to 2.0 g/hour not affecting ND size & crystallinity.
4. The power density of 10^11 W/cm2 resulted in the appearance of bigger ND particles (20-30 nm in average, ~15 %) in the regular ND powder with average particles size of 4-5 nm.

Optimization of the target content resulted in the increase in the productivity from 2.0 to 3.0 g/hour.

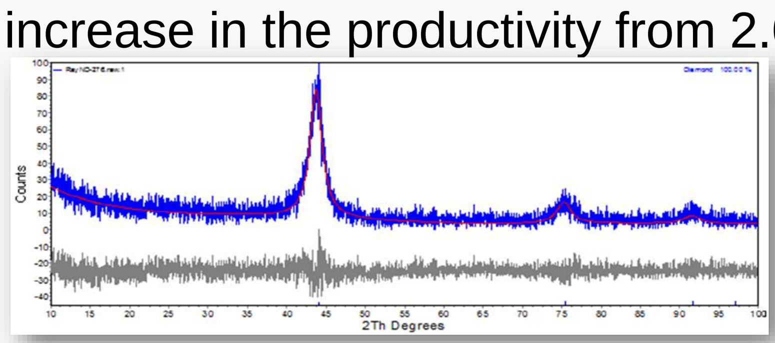


Fig.5. XRD spectrum indicates diamond cubic lattice and average size of 4.5 nm (formula Scherrer)

From Lab to Fab / Conclusion

Actual task. For industrial Production of Advanced Nano-Diamond Additives (PANDA) in accordance to the costs and market analysis, the ND output should be increased from 3 to 600 g per hour. Thus, laser beam parameters should be optimized.

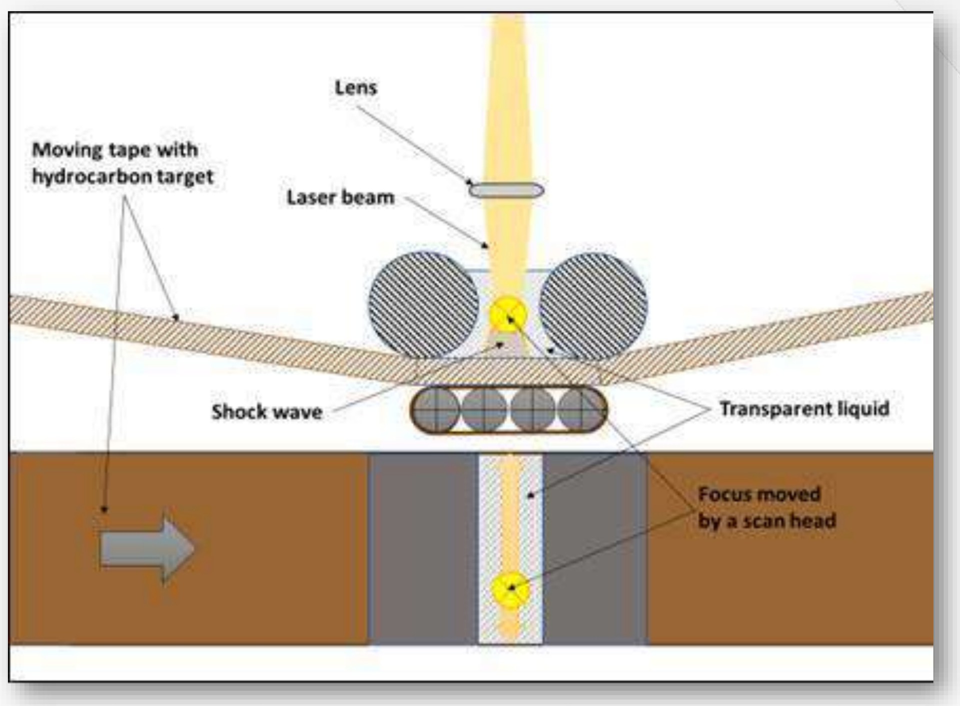


Fig.6.Scheme of ND laser synthesis (industrial process)

Industrial laser system has been designed: high-repetition rate Q-switch laser with the disc amplifier suitable for an immediate implementation. The beam is moved by galvanometric-based beam deflector (scan head) in X-direction (15 cm), while the target is moving with a step of 80 um along the Y-axis continuously. Z position of the target is controlled by the target delivery system.

Repetition rate: 30 kHz

Speed of the target in Y direction: 8.6 cm/min.

Calculated productivity: 600 g/hour

Advantages: continuous target motion & low cost

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