Исследование механизма спекания детонационных наноалмазов при высоком давлении

Ф.М. Шахов[1], С.В. Кидалов[1], А.Я. Вуль[1], В.Т.Лебедев[2], Д.Н.Орлова[2], Ю.С.Грушко[2] А.Н. Озерин[3]

[1] Физико-технический институт им. А.Ф. Иоффе, Санкт-Петербург,

[2] Петербургский институт ядерной физики им. Б.П. Константинова, Гатчина,

[3] Институт синтетических полимерных материалов им. С.Н. Ениколопова, Москва

Fedor.Shakhov@mail.ioffe.ru

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Fedor Shakhov

Outline

1. Detonation Nanodiamonds (ND).

2. High pressure high temperature sintering of ND.

3. X-Ray diffraction of sintered ND.

4. Small Angle Neutron Scattering (SANS) of sintered ND.

5. Conclusions.

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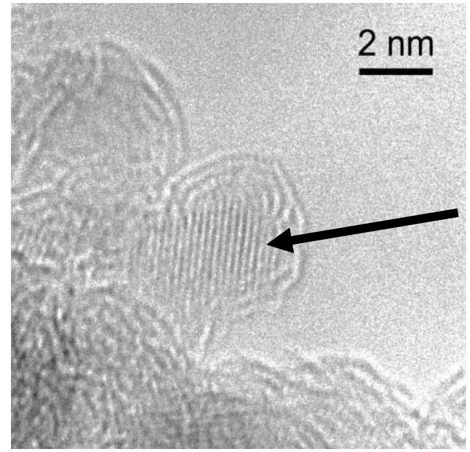
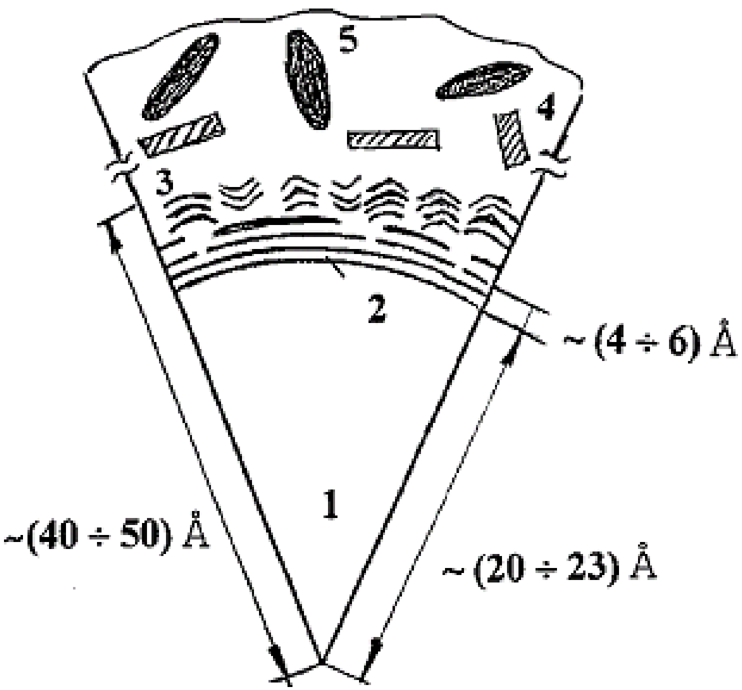
Nanodiamonds

Synthesis conditions:
combustion of a mixture (trinitrotoluene+hexogen) at P>18 GPa and T>2700 ^{0}C



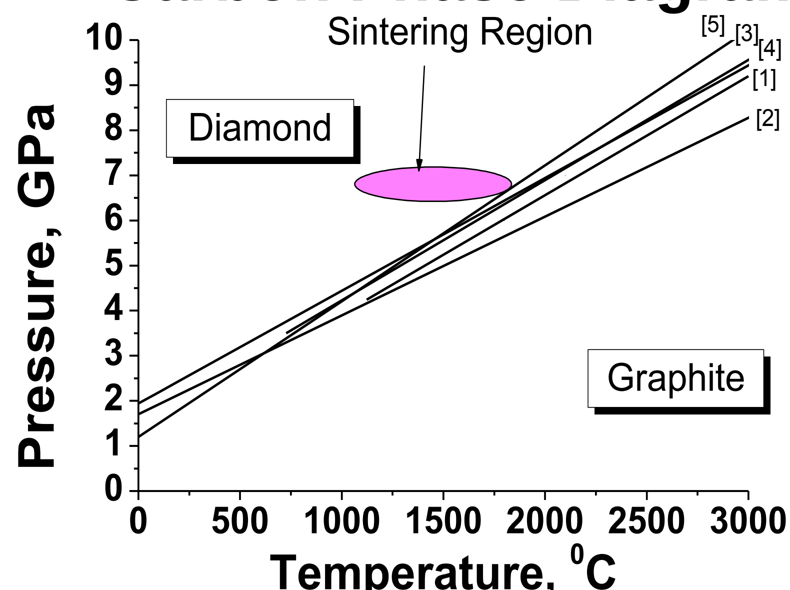
HRTEM image

Diamond core



Structure model of detonation carbon:
1 – diamond core, 2 - “onion-like” shell,
3 - “sets” of graphite-like aromatic clusters,
4 - graphite particles, 5 - metal oxides.

Carbon Phase Diagram



Vul’ A.Ya. et all. 1998

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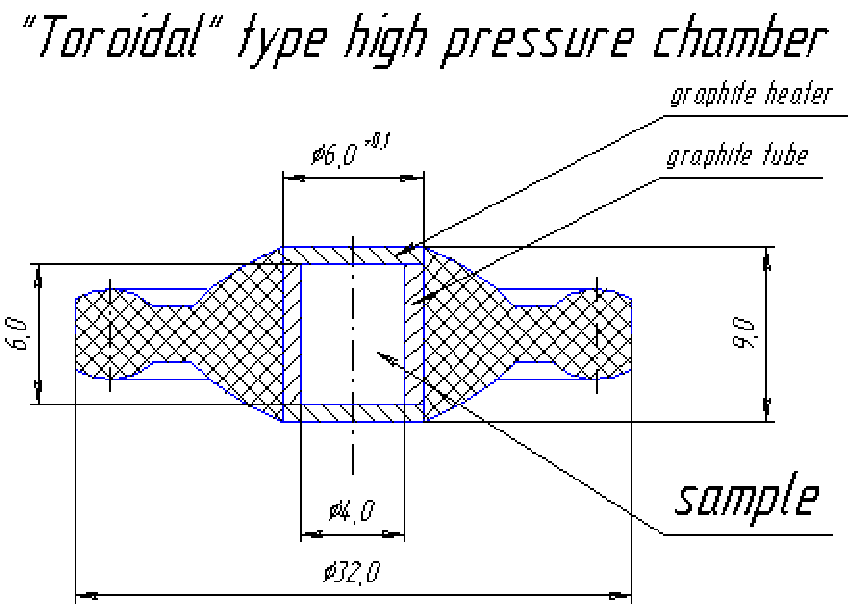
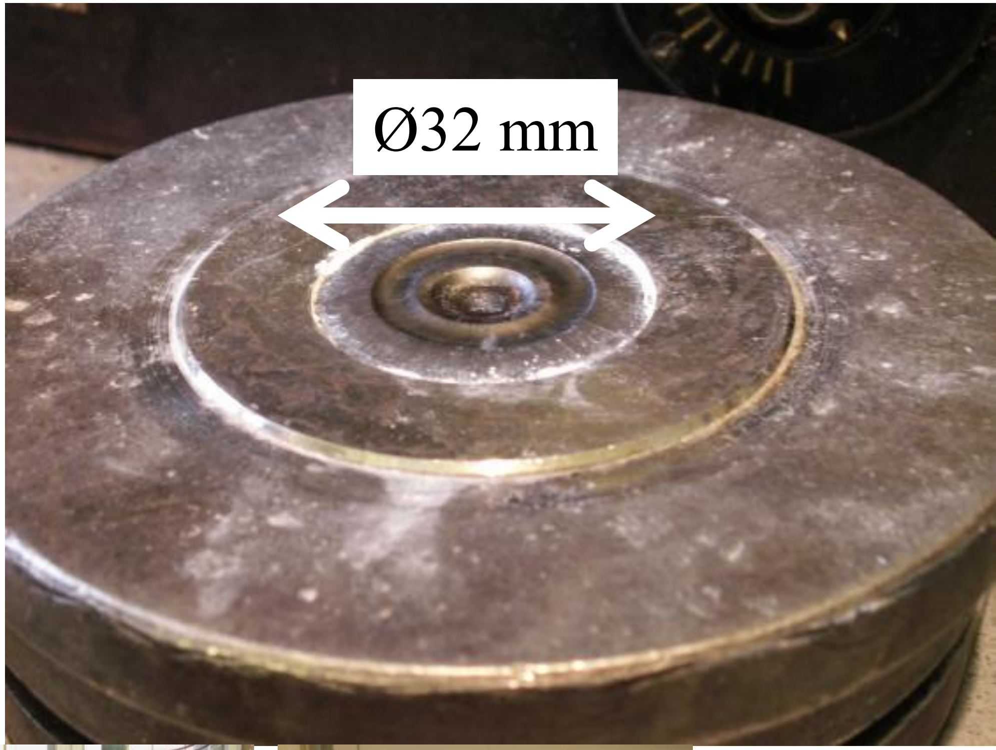
“Toroid” High Pressure Chamber





Sintering conditions:

P = 5 - 7 GPa

T = 1000 - 2300 C (AC current)

t = 11 – 25 sec   *V* = ~ 0.1 ccm

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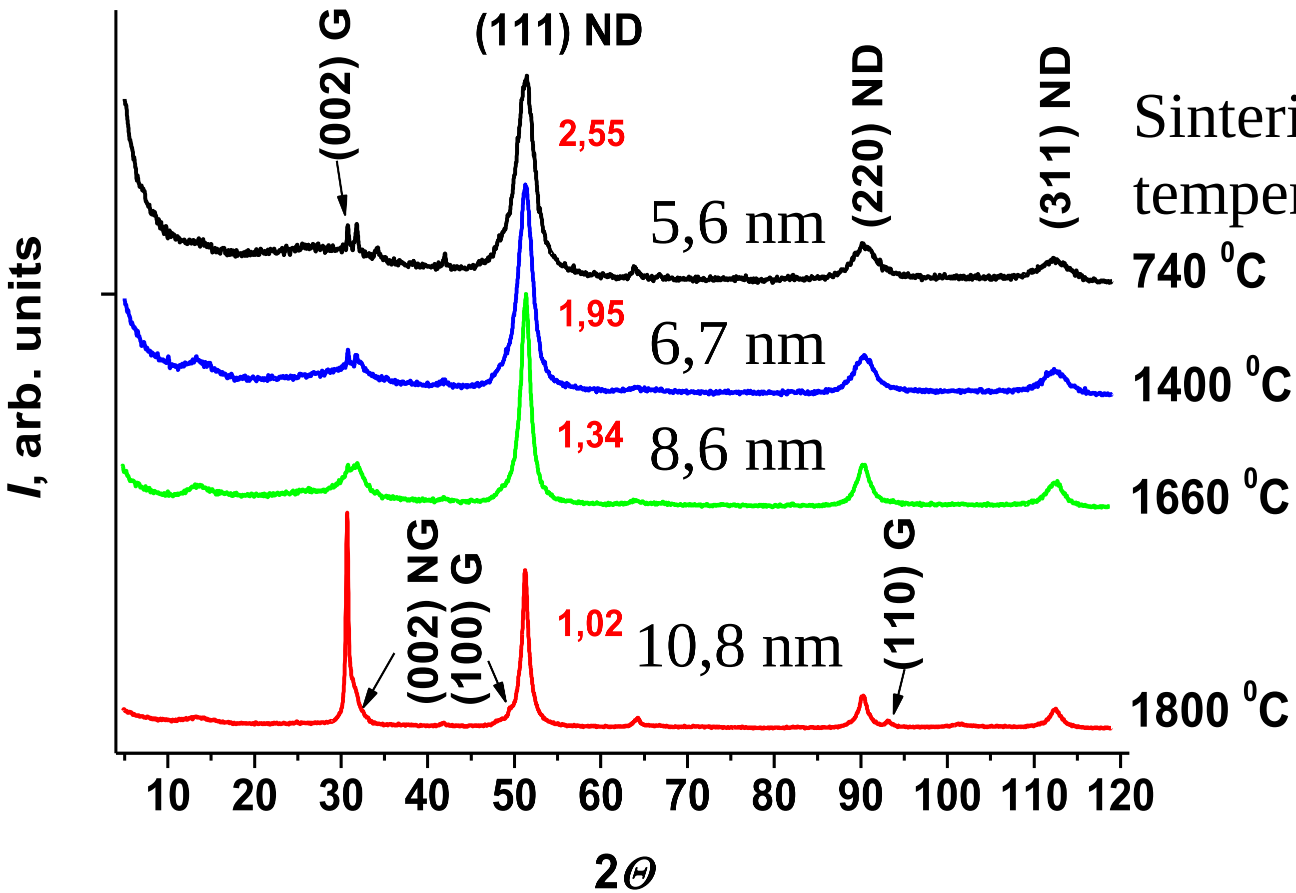
X-ray Diffraction Patterns of Sintered Nanodiamonds

Sintering conditions:
6 GPa, 11 s.



Co, K_a = 1,789 Å

Selyakov-Sherrer equation:

$$W(2\theta) = \frac{\lambda}{D\cdot\cos(\theta)}$$

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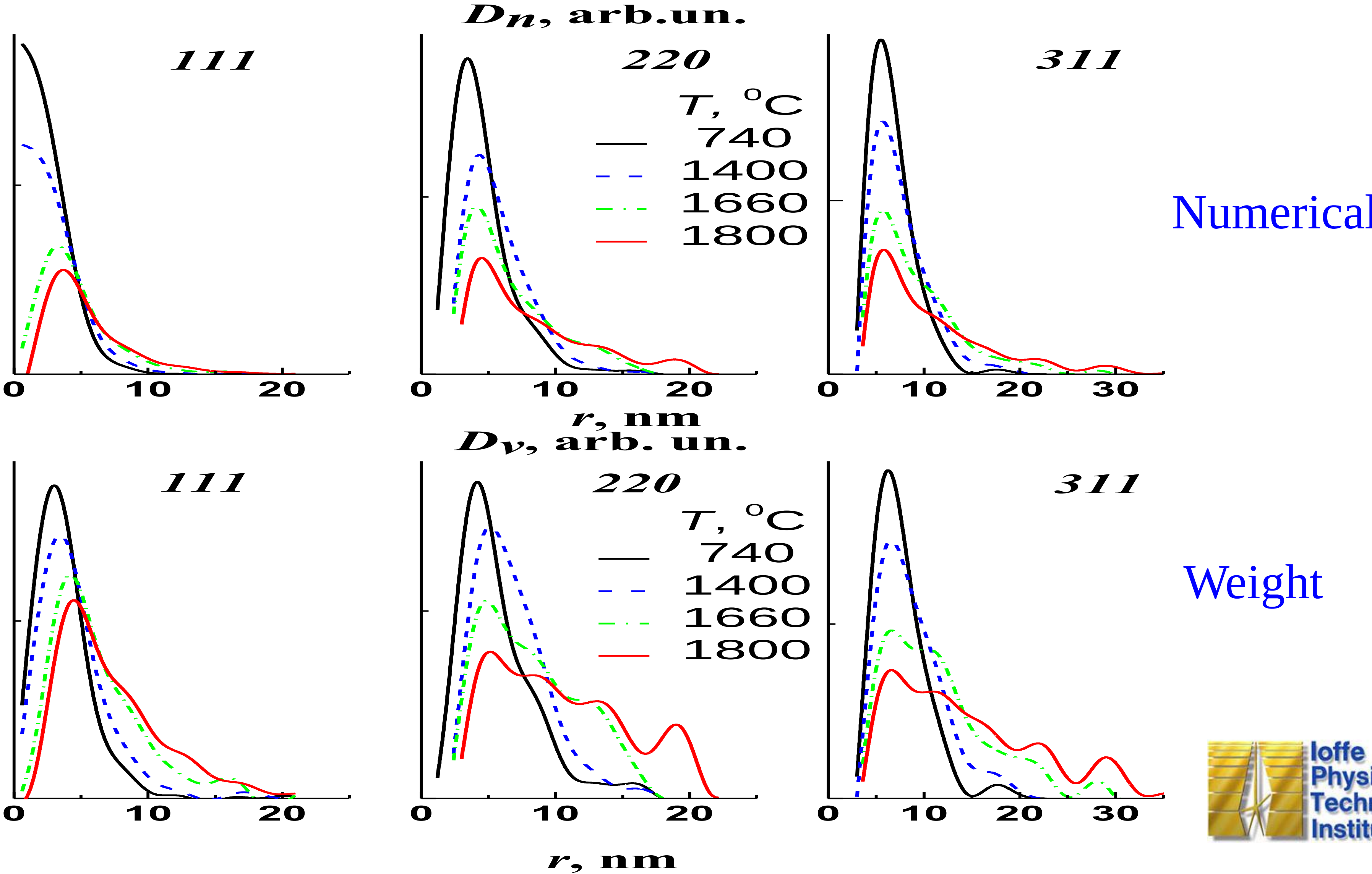
Crystallite Size Distribution Derived From X-Ray Diffraction Spectra by Fourier Transformation



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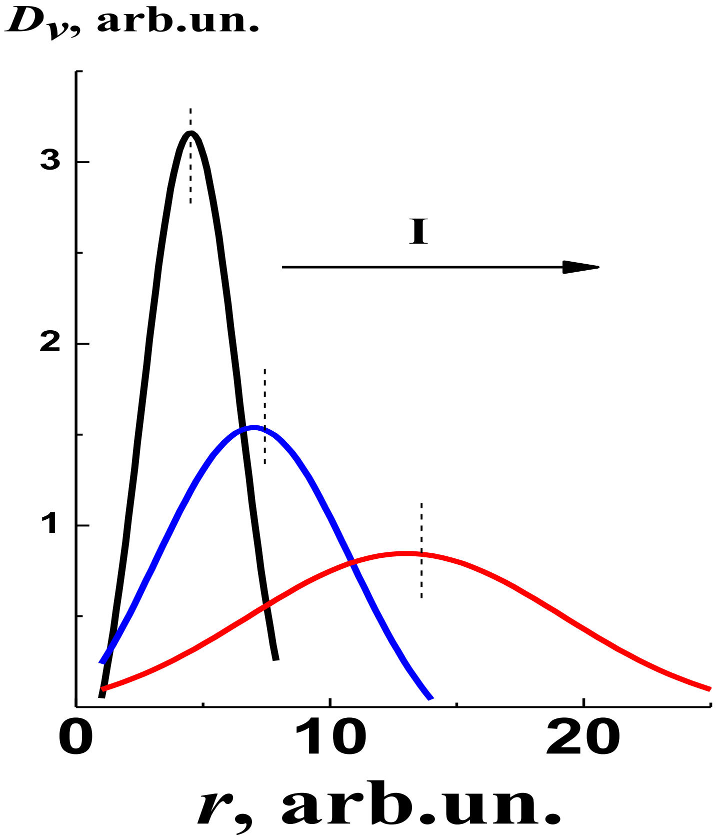
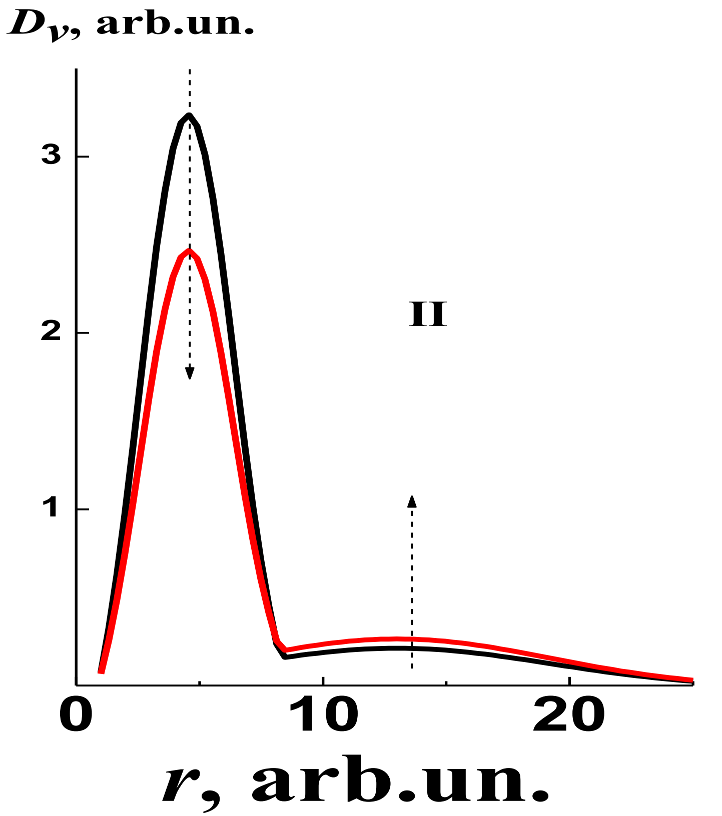
Particle Size Distribution at Different Growth Mechanisms



«Continuous recrystallization»



«Oriented attachment»

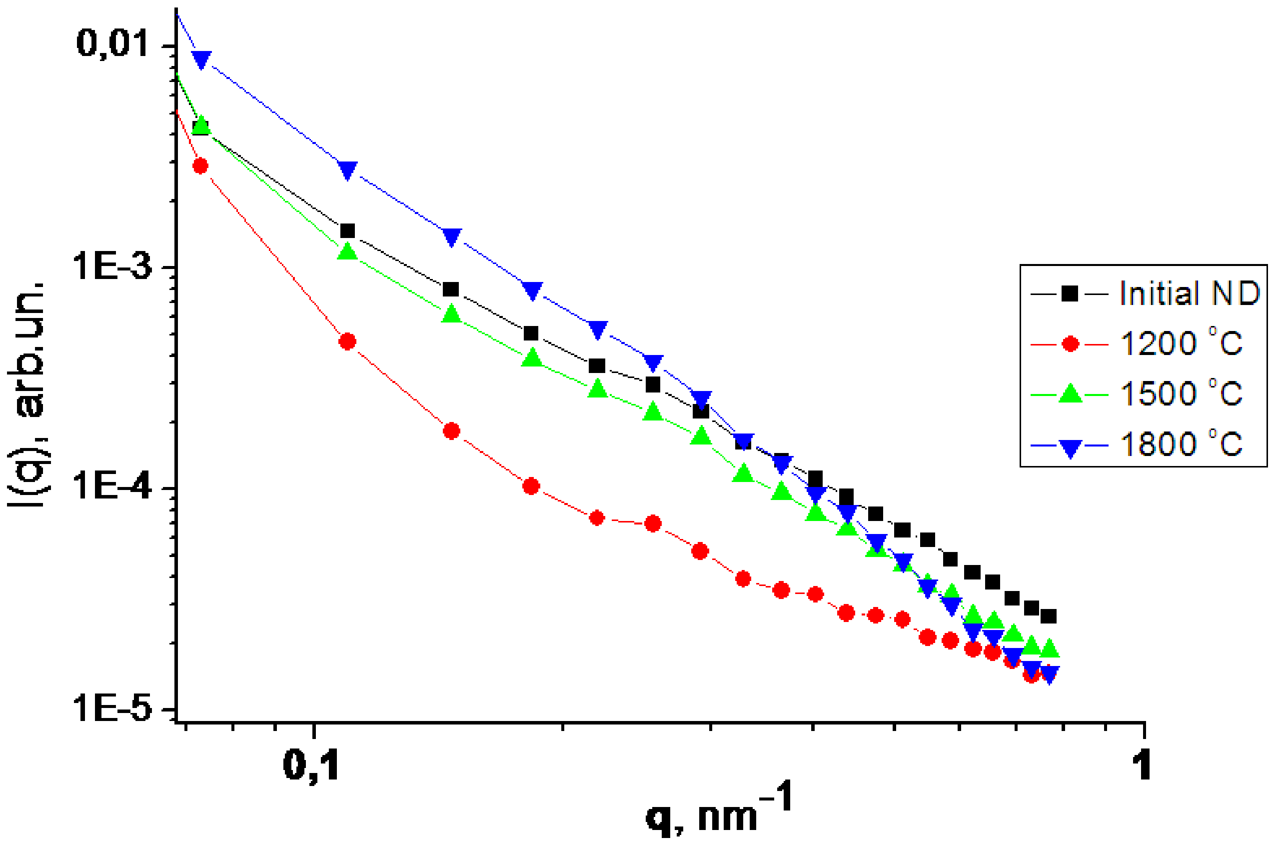
Small Angle Neutron Scattering (SANS) of Sintered Nanodiamonds

Diffractometer «Membrana-2»

λ = 0,3 nm

$\Delta\lambda/\lambda$ = 0.25

$\mathbf{q} = (4\pi/\lambda)\sin(\theta/2) = 0.03 - 0.8\ \mathrm{nm}^{-1}$



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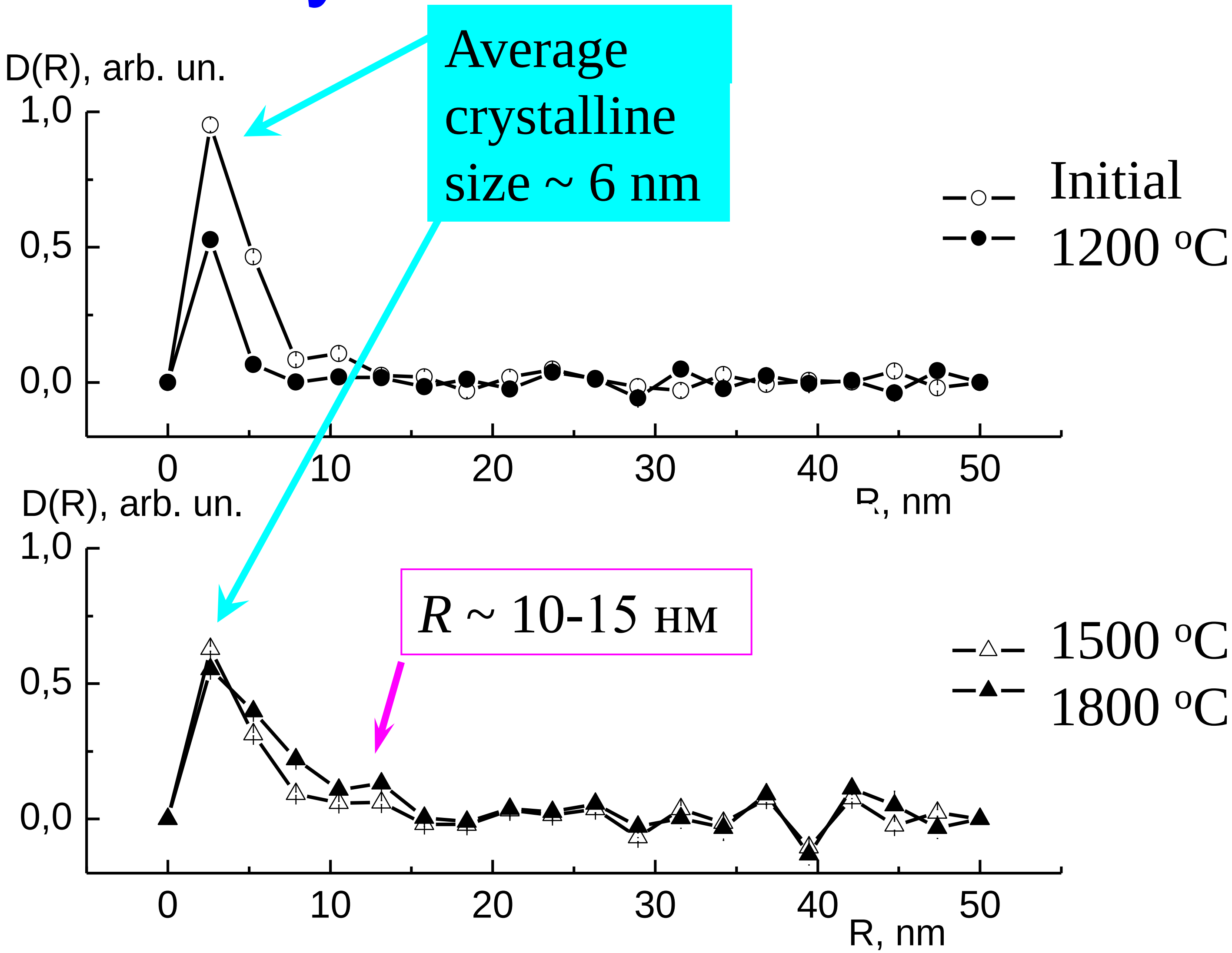
Crystallite Size Distribution Derived From Small Angle Neutron Scattering by Fourier Transformation



Volume

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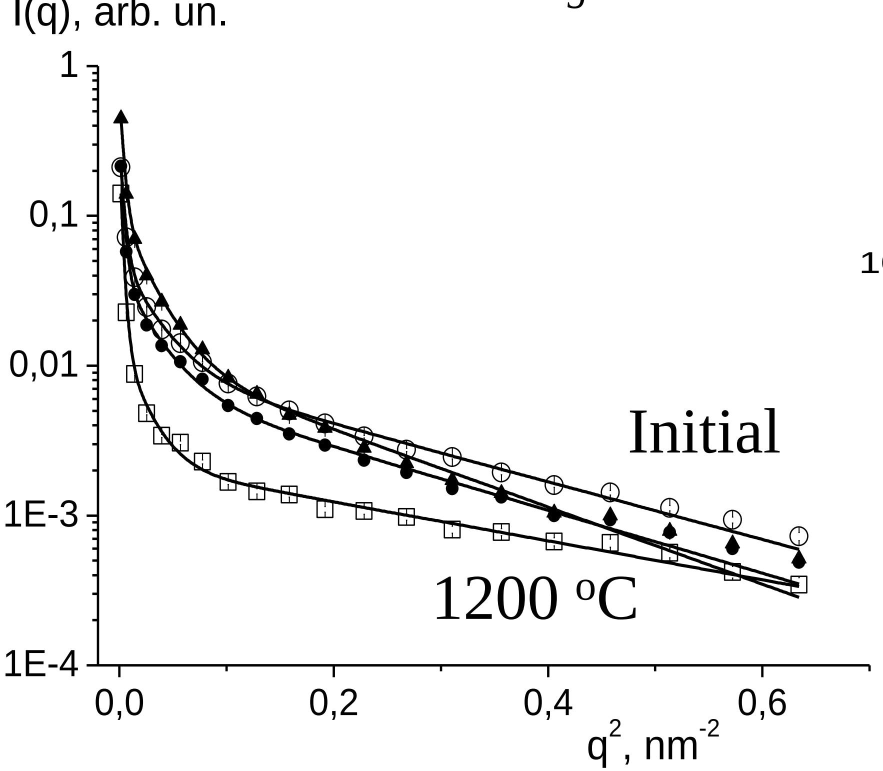
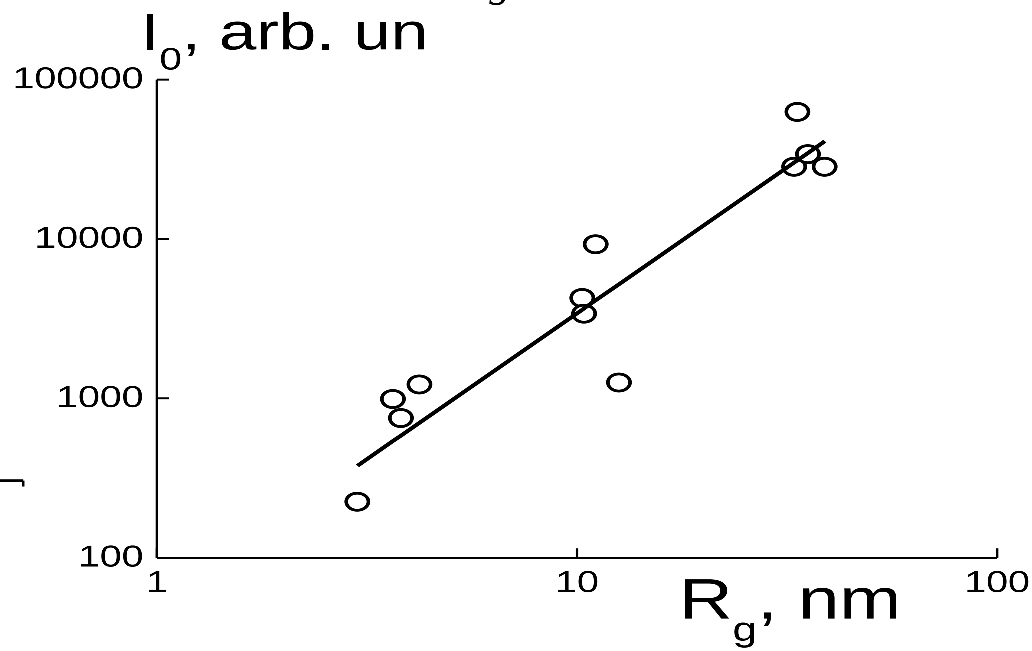
Guinier's Law – Particles Shape

$$I(\mathbf{q}) = I_{01}\exp[-(\mathbf{q}R_{g1})^2/3] + I_{02}\exp[-(\mathbf{q}R_{g2})^2/3] + I_{03}\exp[-(\mathbf{q}R_{g3})^2/3]$$



$I_0 \sim R_g^D;\ D = 1.8$



	Temperature, °C		
nm	1200	1500	1800
R_{g1}	3,00	3,81	4,22
R_{g2}	12,6	10,4	11,1
R_{g3}	38,9	35,5	33,5

Large Fractions Are Aggregates of Small Particles with Chain Structures

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Conclusions

Initial ND – 5 nm

Sintered ND – up to 30 nm

Thermal conductivity – 5-50 W/(m*K)

Sintering Mechanism:

X-Ray → Oriented Attachment

SANS → Smooth Particles with Sharp Interface and Chain Structure

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Grants

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