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WE GROW DIAMOND

Couches minces de diamant CVD pour applications mécaniques

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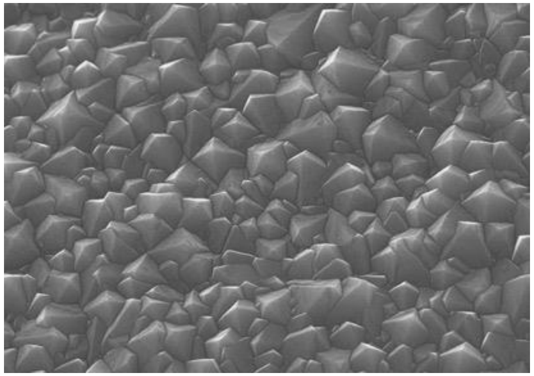
History

Early 90's	Late 90's	2005	2012
CSEM	CSEM / EPFL	Adamant technologies	NeoCoat

- NeoCoat relies on **25 years of innovation** and R&D in the field of CVD diamond
- All past developments and know-how of CSEM and Adamant in the field of diamond are now NeoCoat's proprietary know-how and assets.
- This experience includes **CVD reactor design, diamond growth and applications**
- NeoCoat's principal founders are involved in this history **since 2000**
- Since 2012, based on a new strategy focused **only on CVD diamond growth**

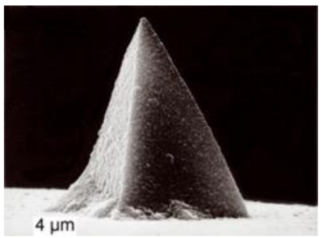
Expertise in diamond coating and CVD diamond solutions

2 ways to enter the market

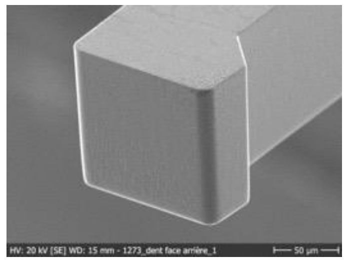


- **Diamond growth and coating services**
  - Diamond coating on various substrates and with different kind of films (microstructure, doping…)
  - Manufacturing of diamond parts
- **CVD reactors and processes**
  - CVD reactors based on various technologies and dedicated process
  - For markets on which NeoCoat does not aim to be active as job coater

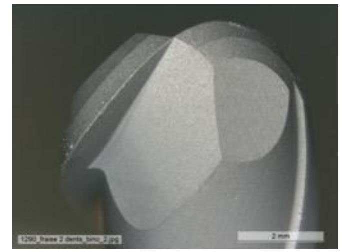
Our diamond coating offer



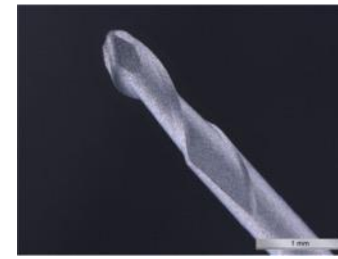
AFM probes

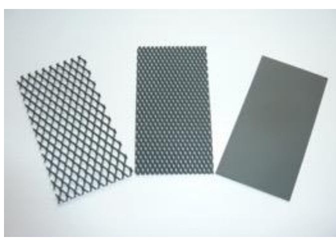


Micromechanical parts

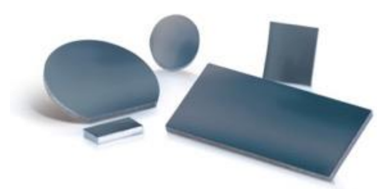


Tools

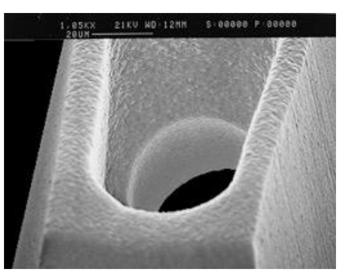




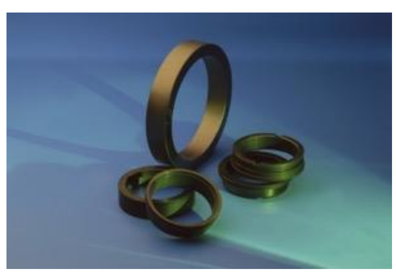
Boron-doped diamond electrodes



Thermal dissipation

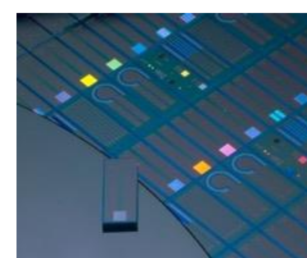


Coatings on complex shapes (dies, nozzles)



Diamond coated rotary seals

Single crystals



Diamond-based microsystems

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Current equipments

- 3 HFCVD reactors (up to ~0.5m2) -> MCD, NCD, BDD (doped)
- 1 MWCVD reactor -> single crystals, thick films, NCD
- Raman spectrometer and various other characterization tools

Our CVD Diamond Technologies

We rely on 3 CVD technologies to grow the right diamond for each application

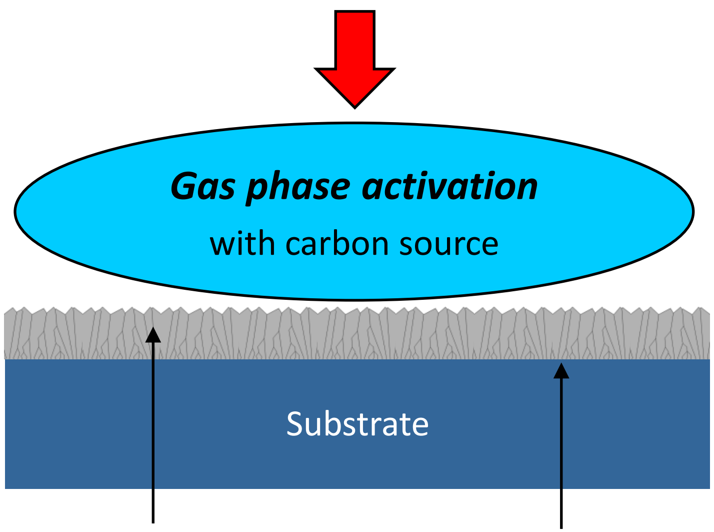
Diamond CVD principal

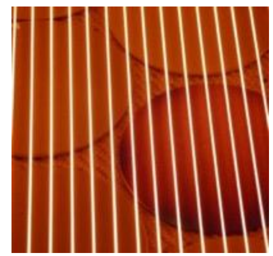
Energy supply

1/ **HF** (Hot Filament)

2/ **MW** (Microwave Plasma)

3/ **MEPS** (Low temperature)





High temperature > 700°C

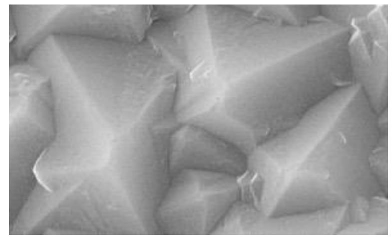
HF CVD
Large scale
Low growth rate

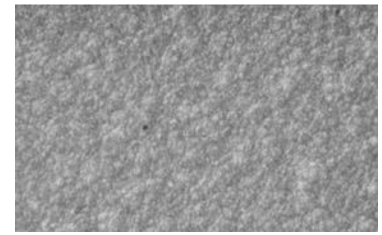
MW CVD
Small scale
High growth rate

Low temperature (100-500°C)

MEPS CVD
Large scale
Low growth rate
New substrates

Microcrystalline vs nanocrystalline

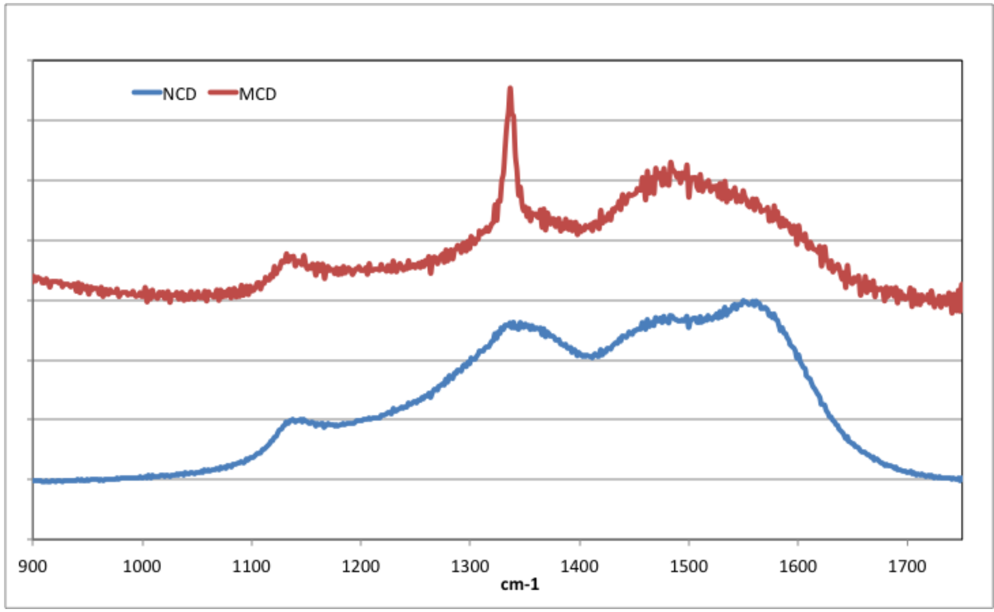




Properties	Microcrystalline	Nanocrystalline
Diamond purity (%)	>99%	>90%
Grain size	0.5 µm at 3µm	30-50 nm (independent on thickness)
Roughness (R_a)	80 nm at 3µm	10-30 nm (independent on thickness)
Hardness (GPa)	100-110	80-85
Biaxial Young Modulus (GPa)	1250	600-800
Residual stress on silicon substrate (MPa)	-300	-100
Thermal conductivity ($W/m^{-1}.K^{-1}$)	1500	10-20
Resistivity for 8000ppm BDD film (mΩ.cm)	3	>30

Diamond quality

Raman spectrometer



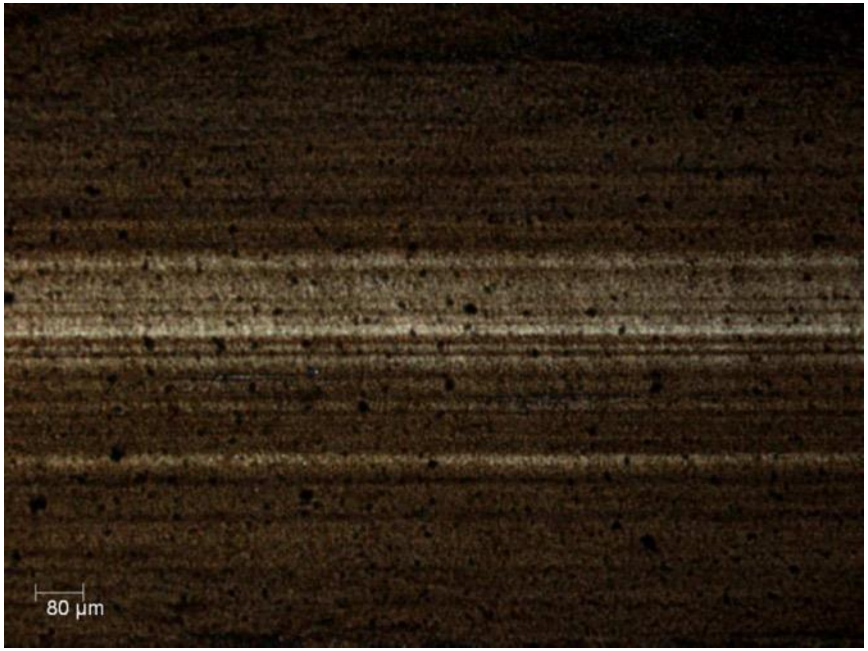
Typical Raman spectra for Microcrystalline and Nanocrystalline

Mechanical properties

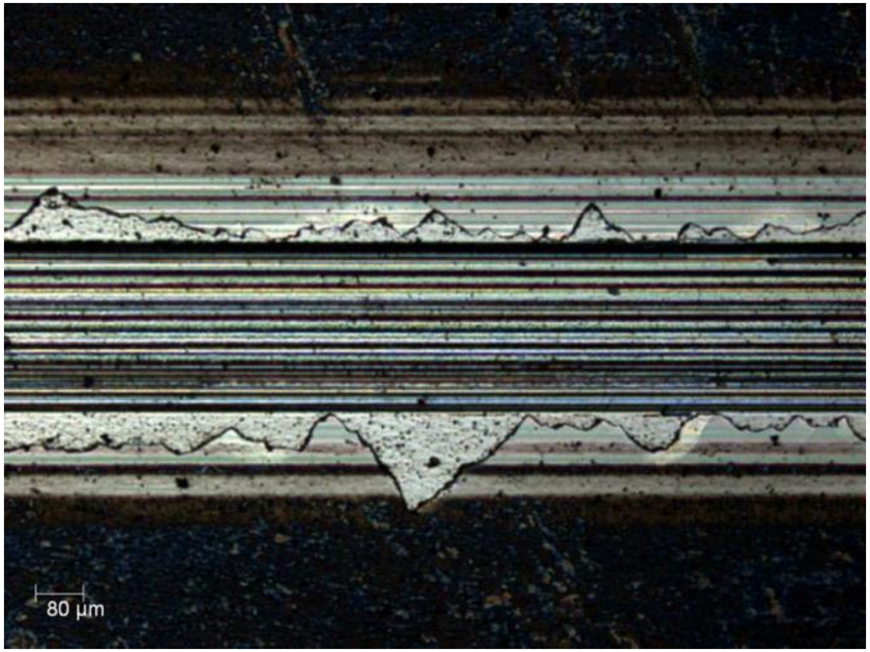
Sample	Coating	RMS	Purity	Hardness (GPa)	Young Modulus (GPa)	Intrinsic Stress (MPa)
A	Poly	120	97%	106	1250	+100
B	Nano	33	94%	-	930	+100
C	Nano	25	90%	86	810	+200
D	Nano	15	80%	81	780	+600
E	Nano	14	75%	74	620	-
F	Nano	25				
G	Poly	5	-	-	-	+100

From L. Vandenbulcke, M.I. De Barros / Surface and Coatings Technology 146 –147 (2001) 417–424

Adhesion tests : pin on disk



Good adhesion



Delamination

Adhesion

- **Interface**
  - Cleaning
  - Removing catalytic elements
  - Seeding, early-growth conditions

- **Substrate roughness**
  - Mechanical anchoring effect

- **Residual stresses**
  - Thermal stresses = deposition temperature
  - Intrinsic stresses = grain size

Material issues

- **Fe, Co, Ni**
  - Fast diffusion of carbon
  - Catalytic effect on methane to form soots

- **Non-forming carbides**
  - Weak adhesion

- **High-temperature behaviour**
  - Phase change
  - Mechanical properties

Material issues

H																	He
Li	Be											B	C	N	O	F	Ne
Na	Mg											Al	Si	P	S	Cl	Ar
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	La *	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
Fr	Ra	Ac **															
		*	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	
		**	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lw	

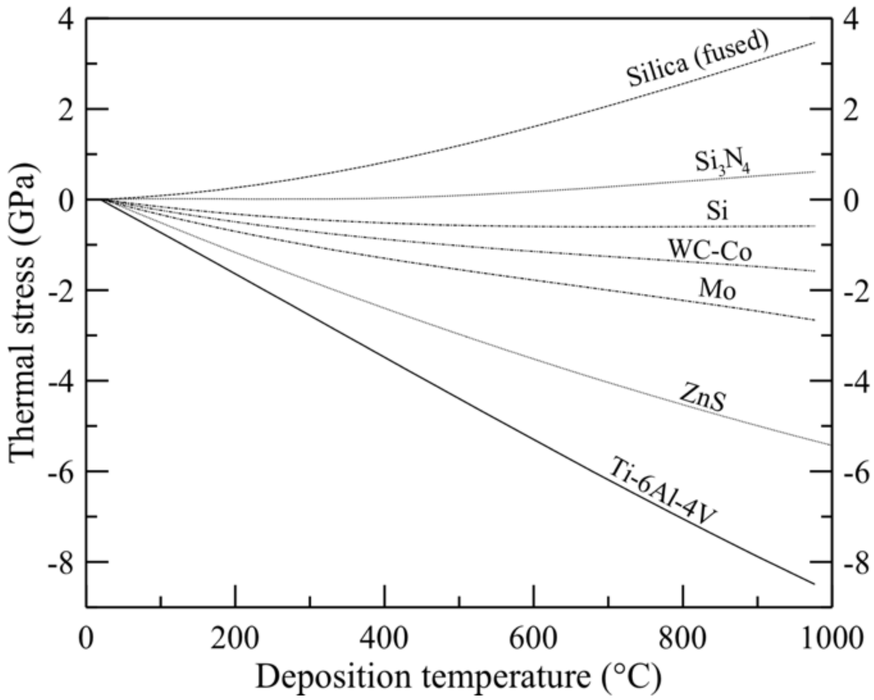
dopants

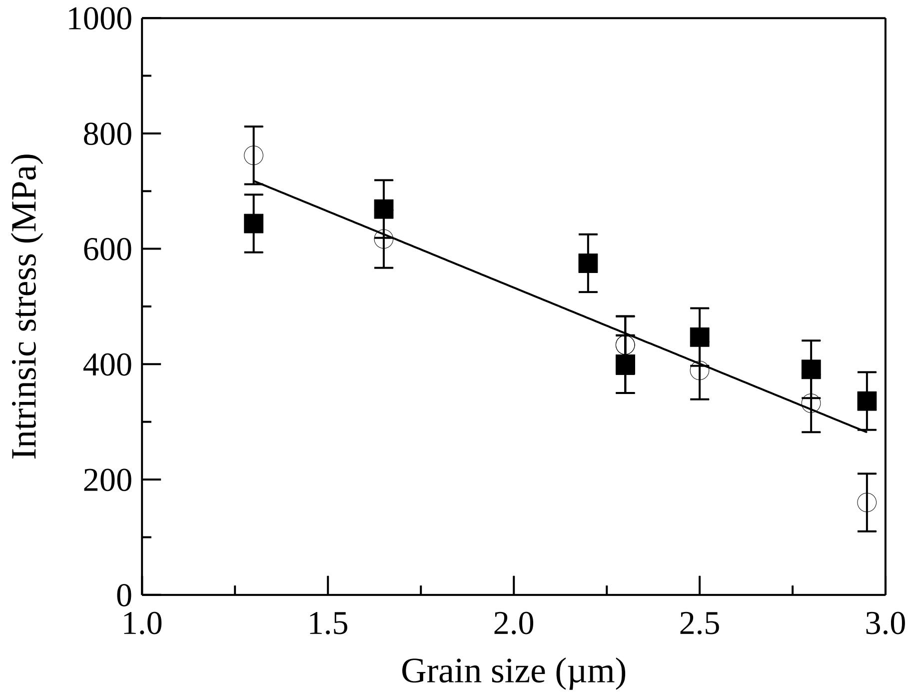
as elements = Schottky contacts, as carbids = well adherent ohmic contacts

as elements = Schottky contacts, above 600 °C graphitization and solid solution

as elements = ohmic contacts, bad adherence, no reaction at elevated temperature

Residual stresses

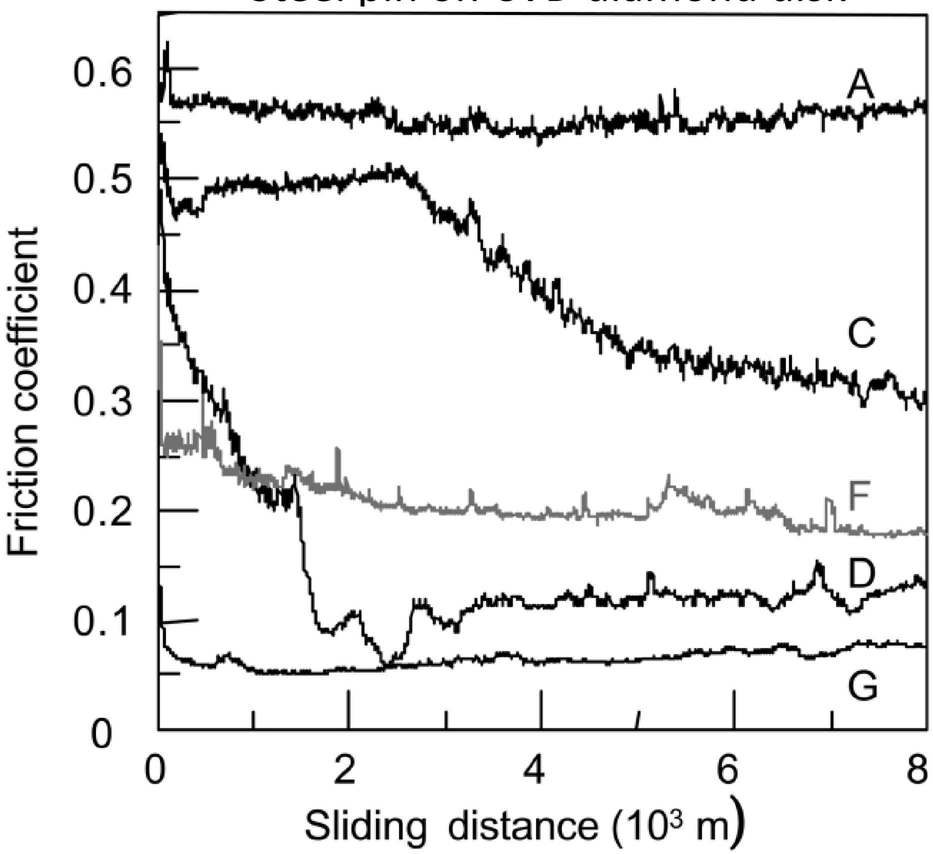


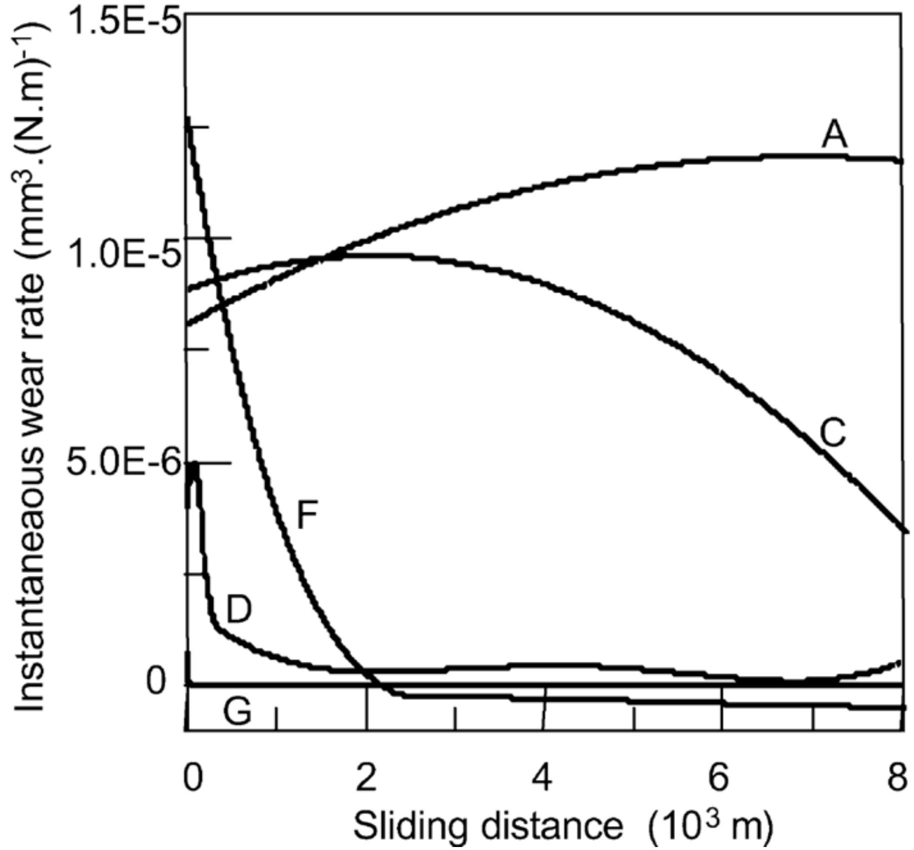


From D. Rats at al, JOURNAL OF APPLIED PHYSICS, 78 (1995), 4994-5001

Sliding tests

Steel pin on CVD diamond disk





From M.I. De Barros et al. /Wear 249 (2001) 68–78

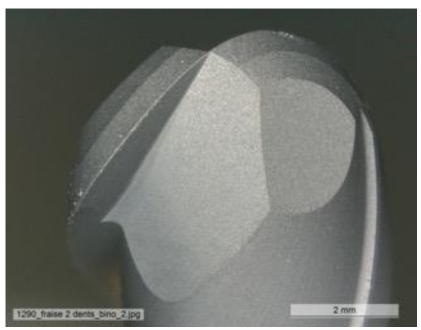
Diamond coatings on tools

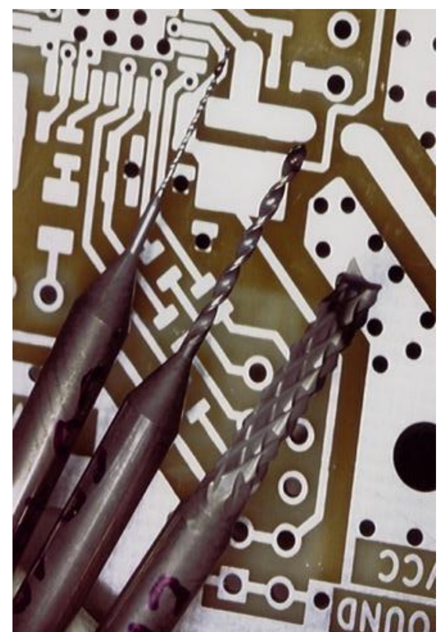
Specific substrate pretreatments to avoid interaction with cobalt

Nanocrystalline diamond for high precision machining of composites, PCB…

Microcrystalline diamond for graphite machining

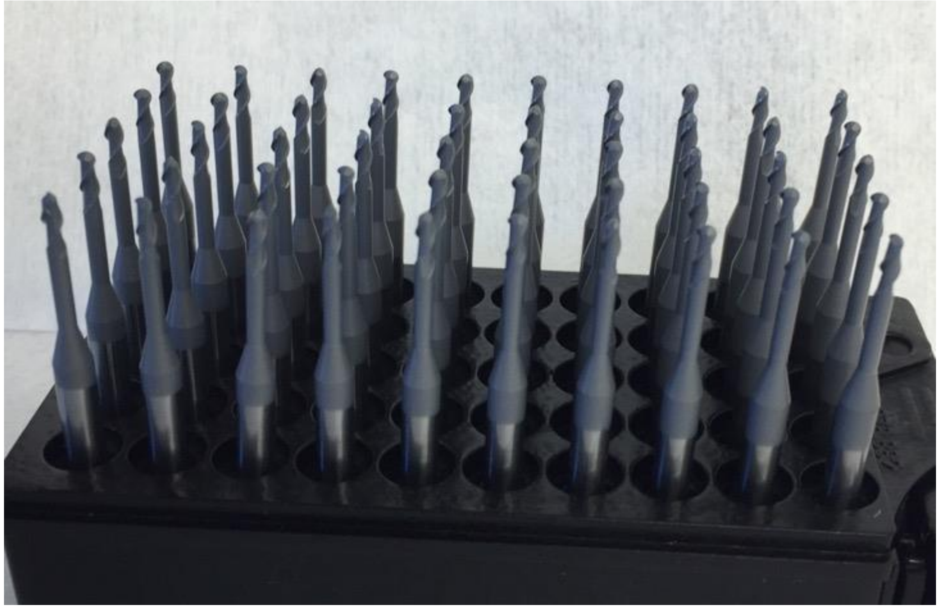
Coatings on small dental precision tools



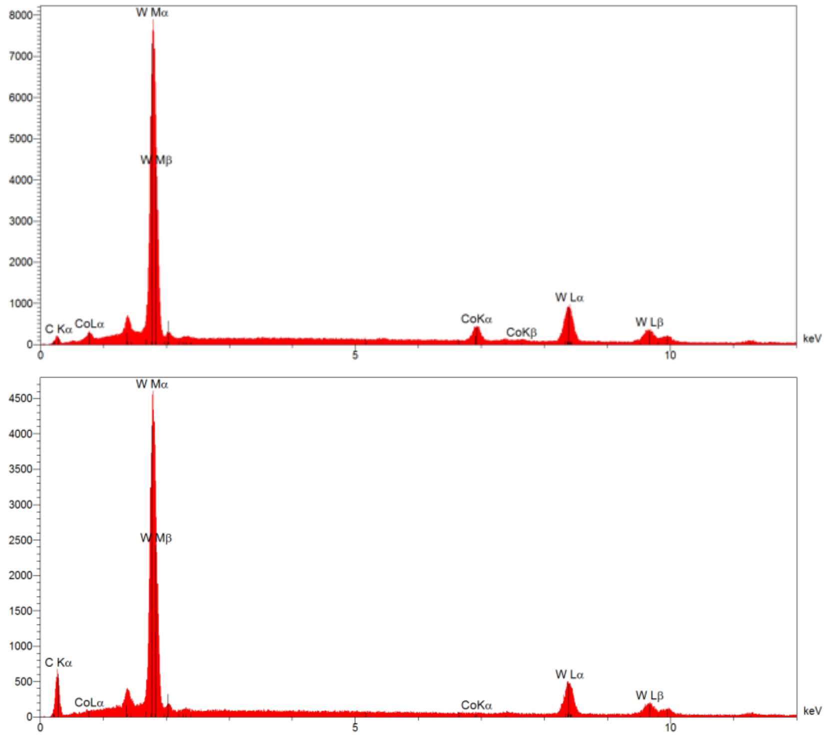


Cobalt etching

Objectives: Remove contaminants (oil…) and cobalt-free surface



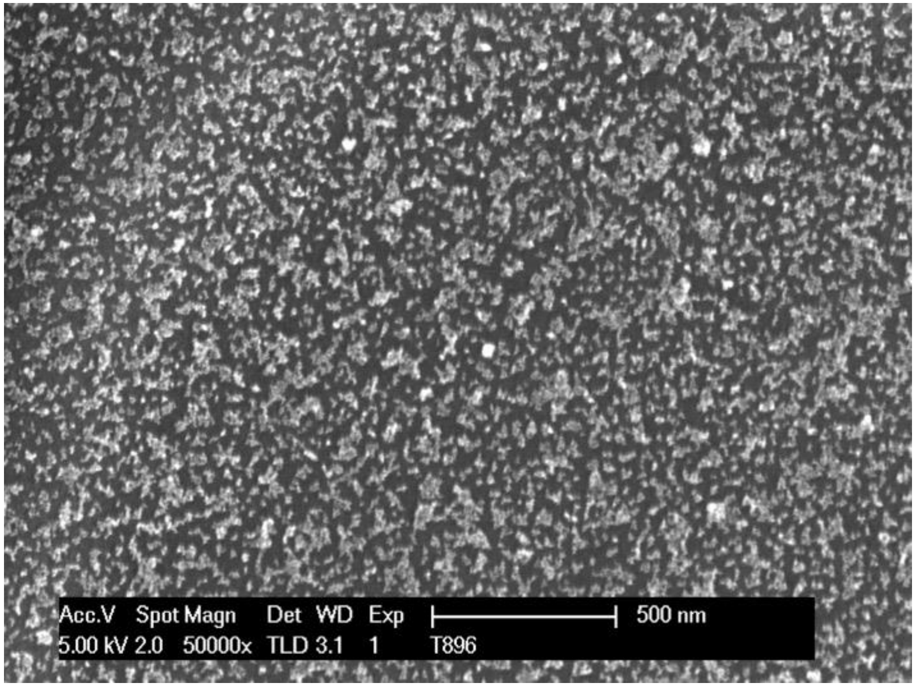
Typical image of tools after cobalt etching

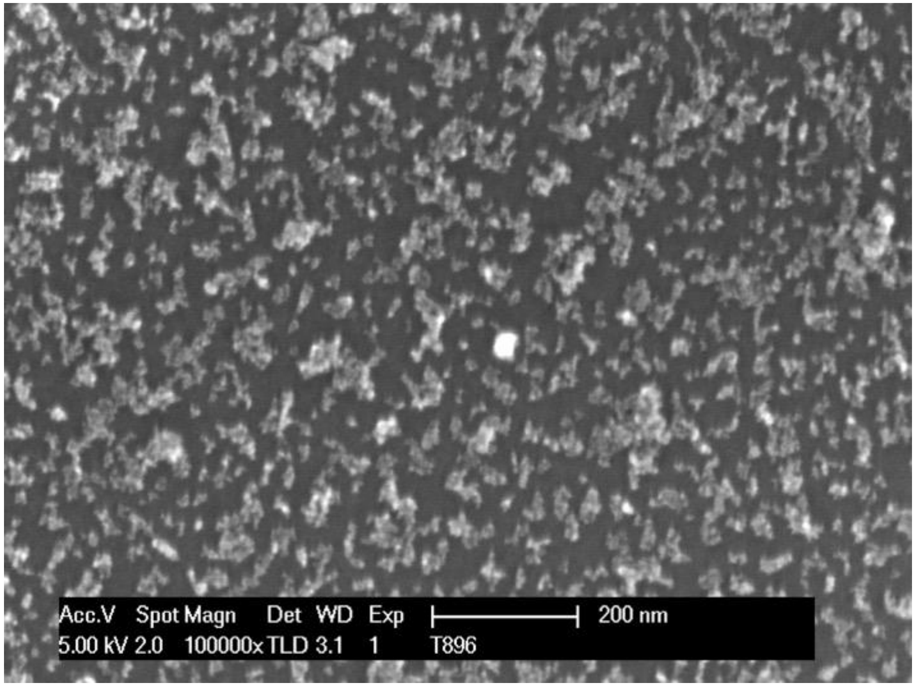


XRF spectra before and after etching

Nanoseeding

Objectives: High density of diamond nuclei (seeds)





SEM images of carbide surface after seeding

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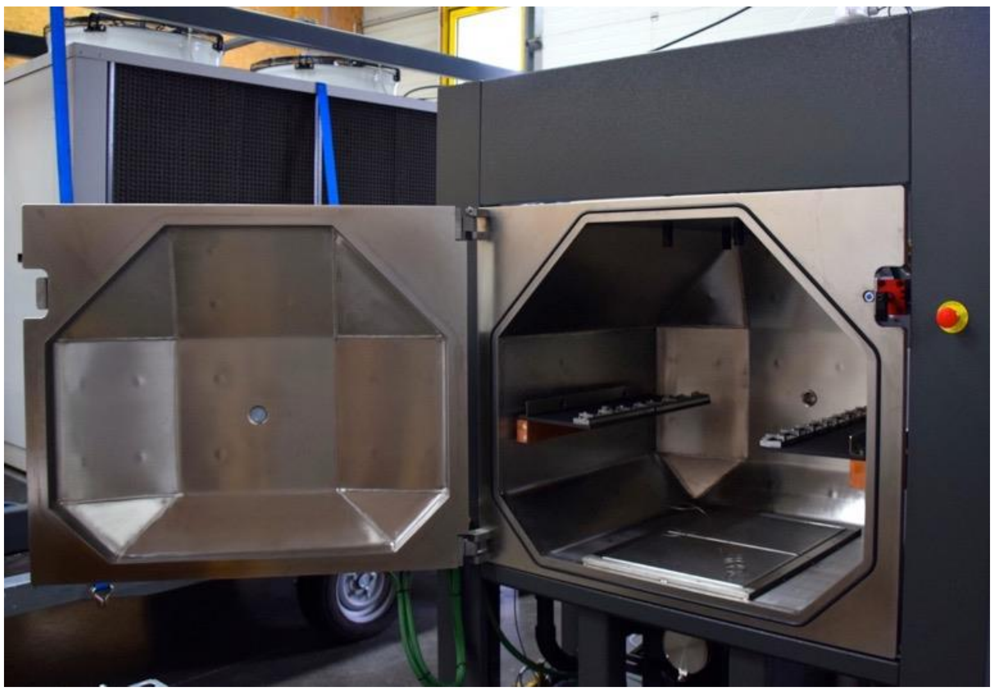
Wetbench

All chemical pretreatment (cleaning, cobalt etching and nanoseeding) could be performed in wetbench

Multi-tank wetbench with automated handling

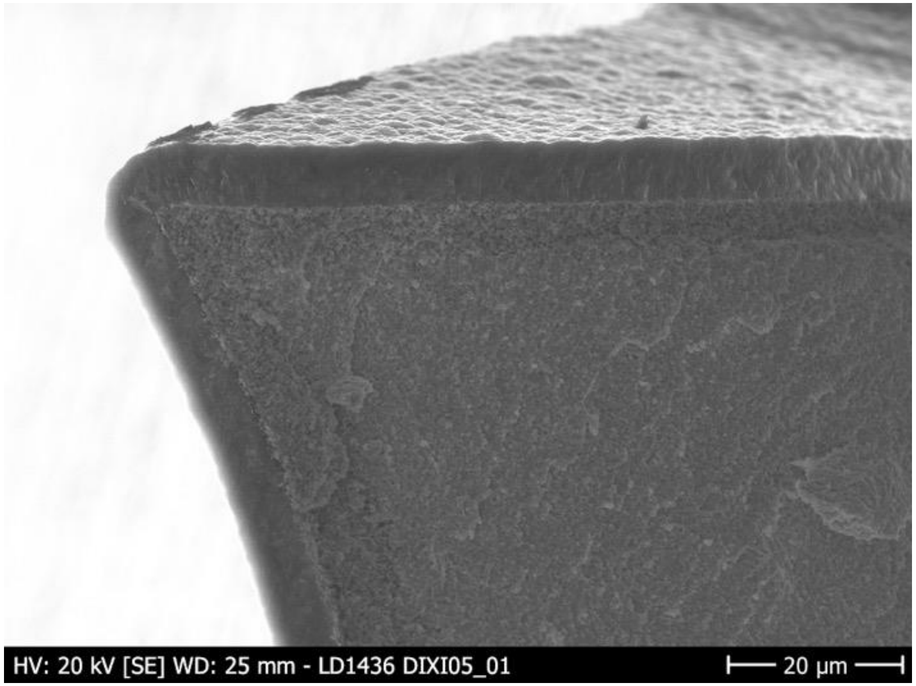
CVDiam® HF60 : loading



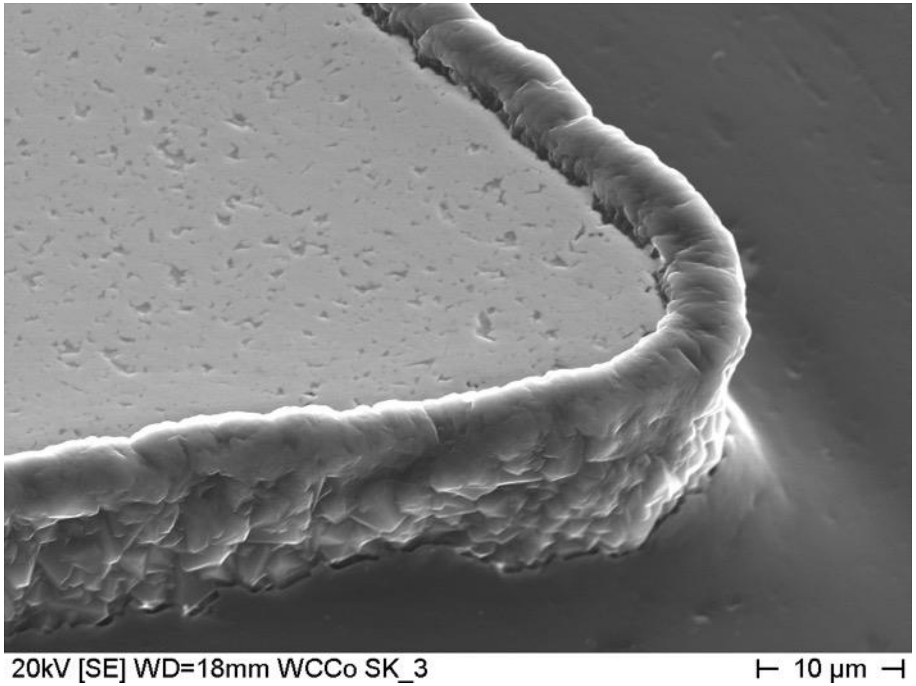
Easy loading thanks to large front door

Graphite box to adjust filament-tool spacing

Diamond coated tools (cross-section)



Nanocristalline : Endmill edge



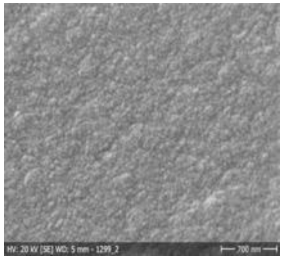
Microcristalline : Insert edge

Diamond coated tools: Comparison of performances

- Drill ø8mm
- Carbon-fiber–reinforced polymer (CRFP)
- Parts of Boeing 777
- 10µm nanocristalline diamond film

Coating	Run-Out Radial (µm)	Number of holes
No coating	3	10
Supplier A	600	250
Supplier B	100	325
Supplier C	50	600
NeoCoat	10	550

Micromechanical parts

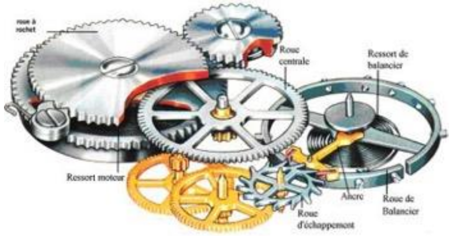


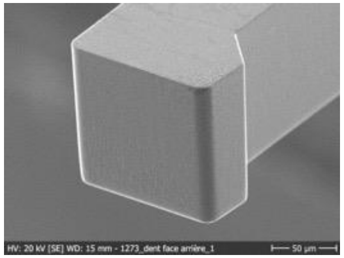
Nanocrystalline diamond for improved tribological properties

Thicknesses: from hundreds of nm to tens of µm

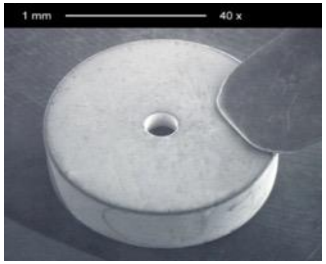
Coatings mainly on silicon and carbide

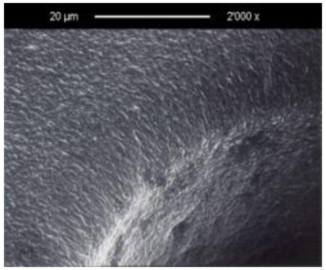
Watch parts



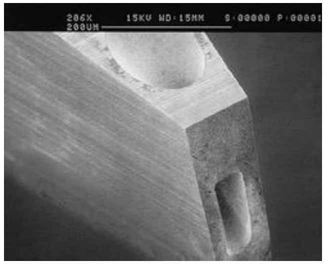


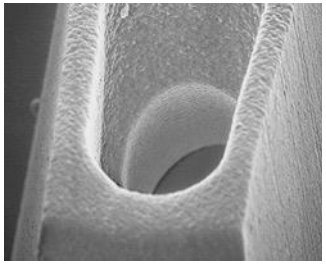
Water jet nozzles





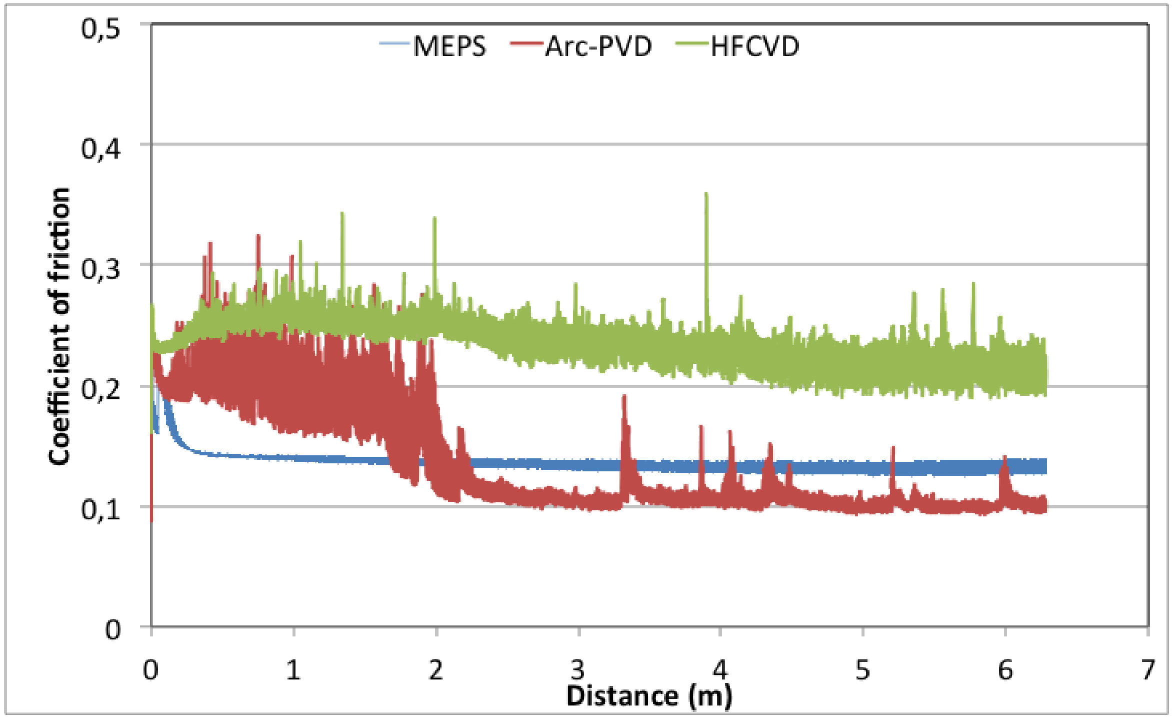
Microstructured tools …





Tribological properties

- Nanotribometer (CSM Instruments)
- Sapphire ball (2mm in diameter)
- F = 10mN
- v = 5mm/s



From H.-A. Mehedi, J. Achard, D. Rats et al, Diamond & Related Materials 47 (2014) 58–65

Tribological properties

Sample	Roughness (nm)	Friction Coefficient	Ball Wear Rate (10^{-14} $m^3.N^{-1}.m^{-1}$)
MEPS	5	0.12	0,3
HFCVD	20	0.22	20
Arc-PVD	<5	0.10	3

From H.-A. Mehedi, J. Achard, D. Rats et al, Diamond & Related Materials 47 (2014) 58–65

Rotary seals

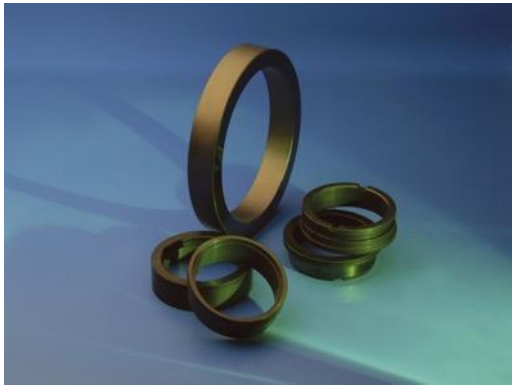
Diamond coating on SiC rotary seals

Microcrystalline or nanocrystalline coating with optional post-coating polishing

Coating thicknesses in the range of 2 to 10 µm

For use in high-performance pumps (oil industry, water with suspended solid)

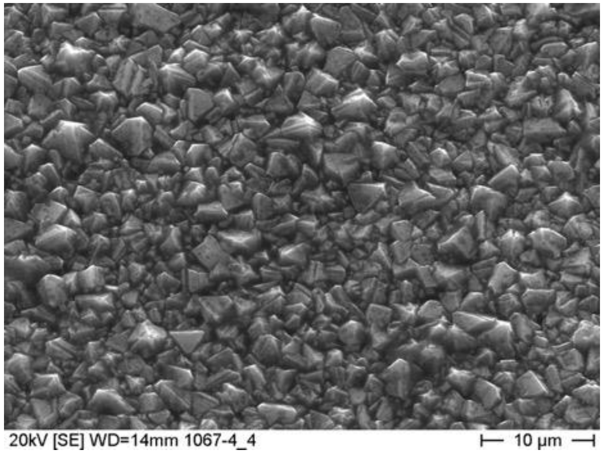
Increase of lifetime with a factor 10 to 100



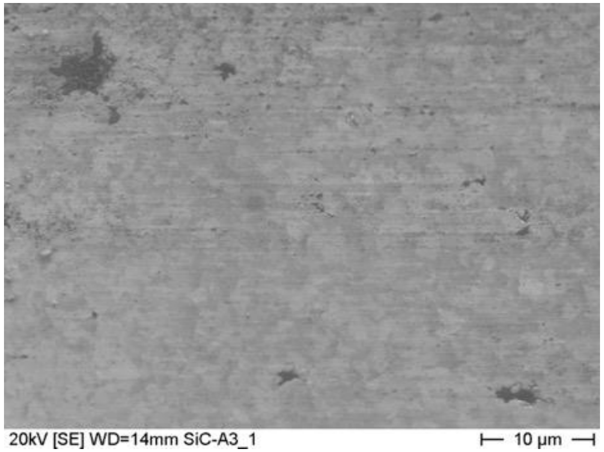
Rotary seals

Increase of lifetime with a factor of 10 to 100

Friction coefficient (diamond on diamond) < 0.1



Diamond coating « as-grown »
Microcrystalline structure



Surface polishing after long-term use
No delamination observed

Conclusion

- CVD Diamond is an exceptional material, but a strong know-how and expertise are needed to successfully coat it
- Mechanical properties depends on diamond microstructure (grain boundaries)
- Tribological properties depends on surface roughness
- Several mechanical applications are available:
  - Cutting tools
  - Micromechanical parts
  - Seals

Thank you for your attention

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Innovative CVD
Diamond Solutions