Ultra smooth surface of diamonds, towards Å scale roughness for the (111) orientation

T N Tran Thi[1,2], S H Connell[1], T Mashego[3], B Fernandez[4] , D Mavunda[1, 5], J Härtwig[1,2]

1. University of Johannesburg, Johannesburg, South Africa
2. ESRF, Grenoble, France
3. iThemba LABS (Gauteng), South Africa
4. Institut Neel, CNRS, Grenoble, France
5. NECSA, South Africa

Introduction

Synthetic diamond of exceptional quality is required for many high technology applications. Two examples:

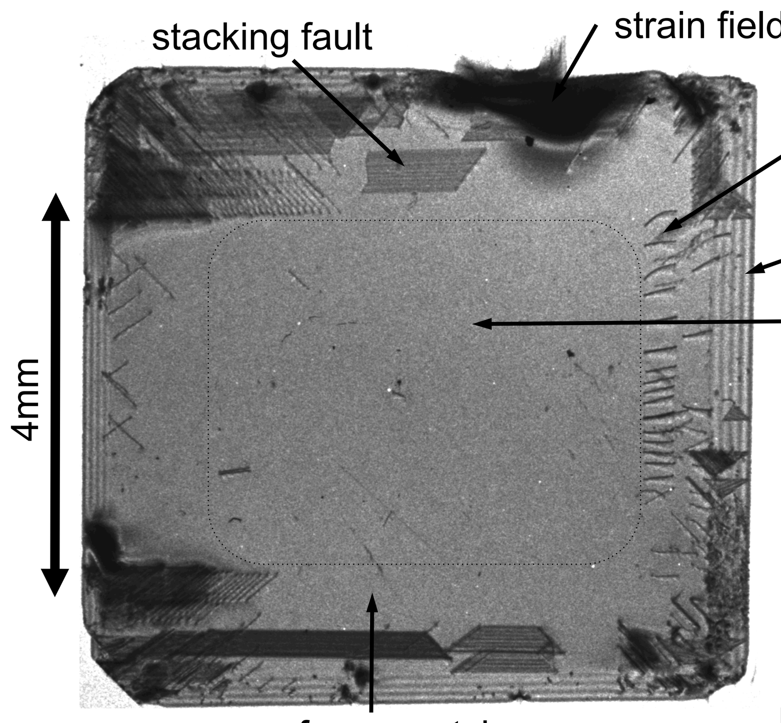
- δ-doped diamond as an electrical switch in FETs, expected to operate at high power and high frequencies;
- 111-oriented diamond plates that are needed at synchrotrons such as the ESRF to serve as beam splitter monochromators working in reflection geometry (Bragg case).

The surface is of special interest. It should be smooth, flat and defect free. For the diffraction application, the requirements is in addition a low miscut for the 111 surface.

Motivation: The various communities for the high tech applications of diamond are seeking a surface processing step to remove the scaife polishing damage (200nm depth), improve roughess (few A^0) and flatness (1m) and to also process the diamond 111 surface, the hardest known surface (~0.2^0 miscut)

Can have regions of sufficiently high Bulk Quality (White Beam Topograph)

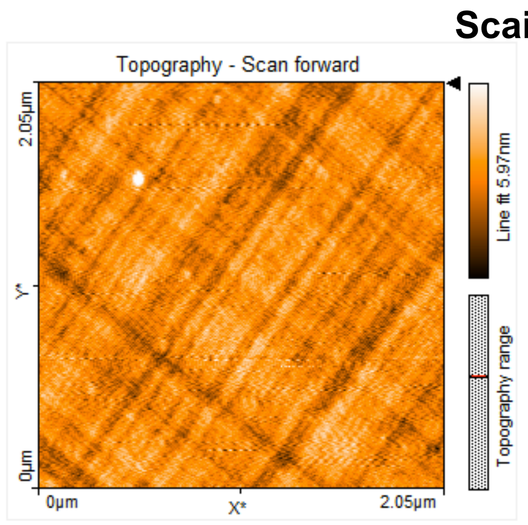
Pure synthetic HPHT IIa 100 plate (Element Six SA)



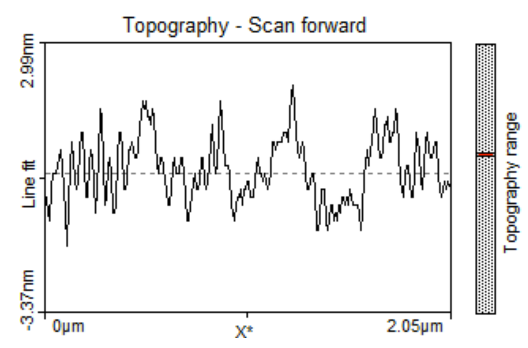
We present a summary of the different polishing techniques as well as their advantages and limits:

- Mechanical polishing with nano diamond powder.
- Oxygen electron cyclotron resonance (ECR) etching, oxygen radio frequency etching or microwave etching (ICP) in a O_2+SF_6+Ar gas mixture.
- Oxygen implantation, followed by annealing in vacuum at 950°C. "Lift-off" resulted from either a hydrogen plasma or an acid etch, and the final anneal in air at 500°C provided an additional soft isotropic etch.
- Hot metalling 1: The diamond is moved with low speeds over a surface of pure Fe at 1000^{o}C in vacuum for at least 3 hours
- Hot metalling 2: Mechanical diamond-grit-less sciafe polishing at high speeds, high temperatures and high loads

Scaife polishing with nano-diamond powder

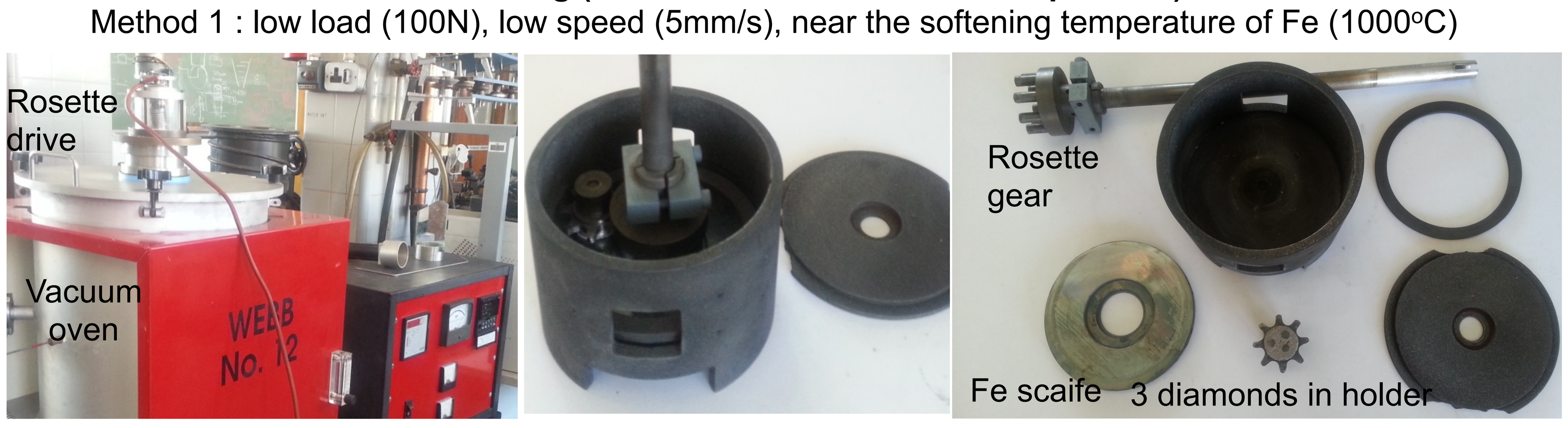


100 surface (<50nm diamond grit) using two polishing directions
→ 0.5 nm RSM of surface roughness with 4 μm^2 footprint
Believed that the depth of the subsurface damage is controlled by size of the diamond grit (including communition)



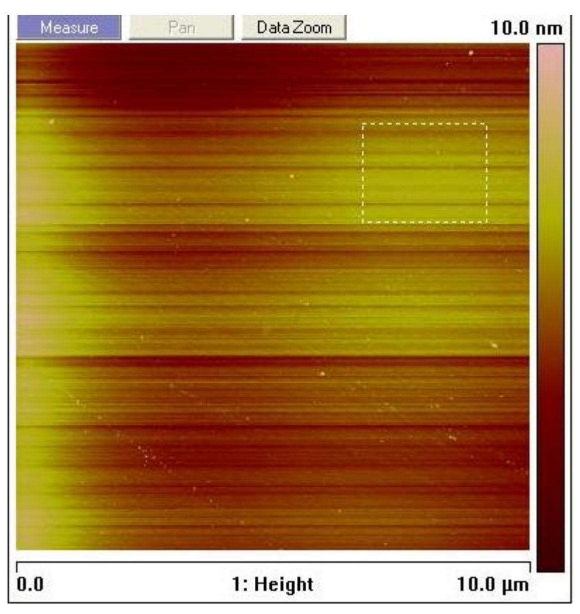
Hot-metalling (thermo-chemical-mechanical process)

Method 1 : low load (100N), low speed (5mm/s), near the softening temperature of Fe (1000^{o}C)

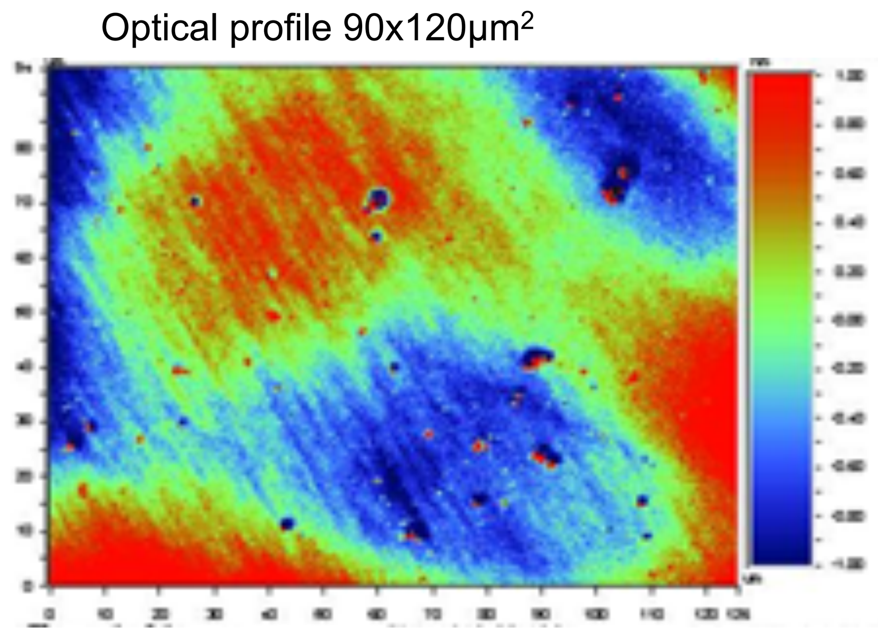


Specifications
Diamond gritless process.
Rosette motion of work-piece.
Low load, low speed, heated pure Fe scaife.
Vacuum oven at 1000^{0}C for ~ 3 hours.
Material removal via C-diffusion into Fe.
Removal rate: ~3μm/hour.

AFM measurement
10μm^2 footprint
→ 0.5 nm RSM of surface roughness



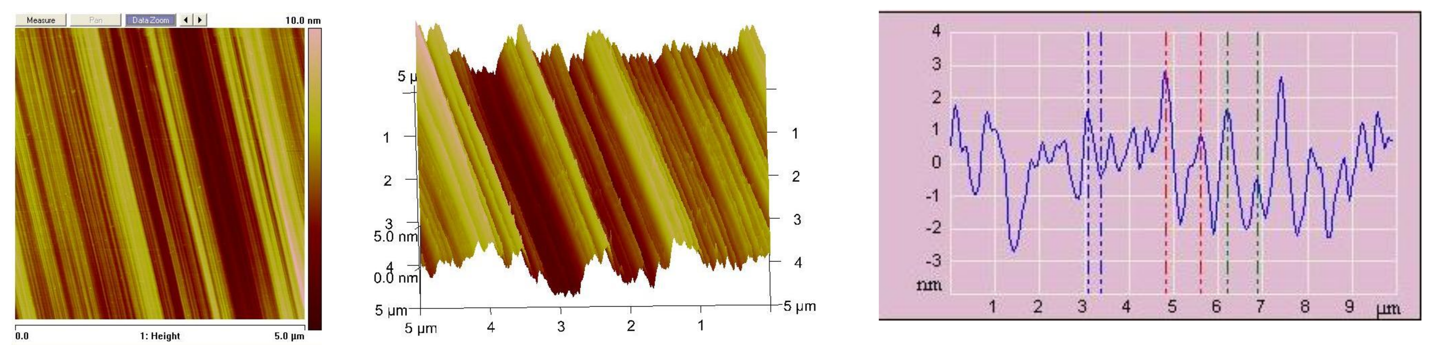
Optical profile 90x120μm^2



Parallel polishing lines at Armstrong scale.
May be related to scaife preparation (smoothness).
Surface flatness at the larger mm scale as wave periods.
Defects remain on the surface.

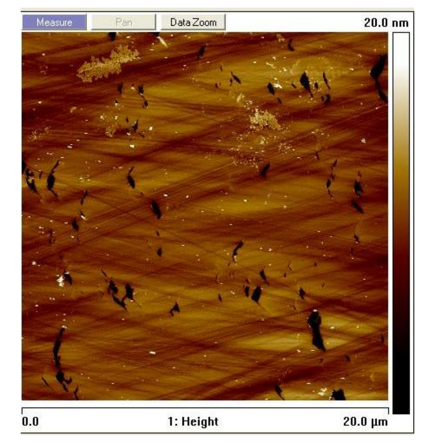
What are the problems related to Surface Quality ?

AFM for a scaife polished surface typical of the HPHT diamond on the left



- parallel polishing lines of a few nm width (r_{rms} 4 nm)
- packed polishing lines (1 mm period) superimposed on
- parallel polishing waves (200 mm period) (R_{rms} 40 nm)

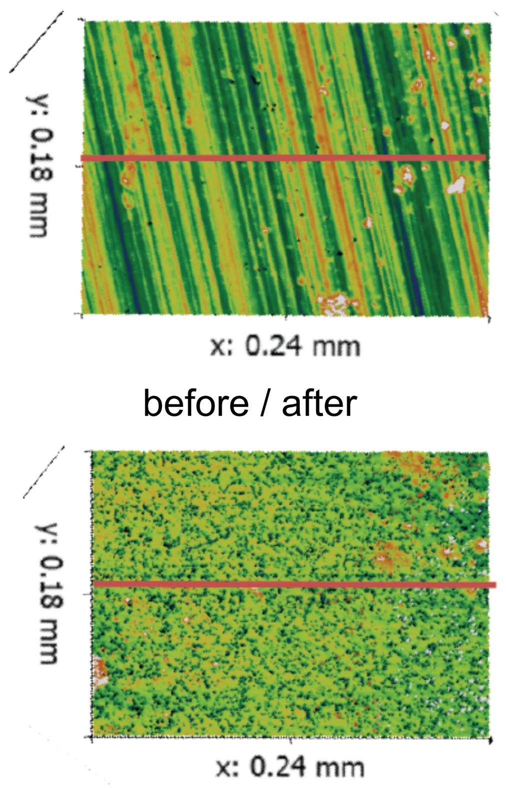
In the case of CVD, there are also holes from emerging bundles of dislocations



issues
- Roughness
- Flatness
- (near sub-) surface damage
- 111 no scaife polishing for low miscut 111 surface

ECR and ICP results

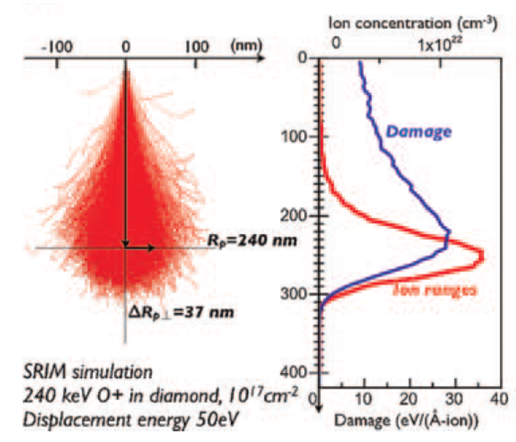
Method	Plasma	Etch rate (μm.h^{-1})	Results
ECR	Oxygen	~ 6	Fast etch rate but increased surface roughness
ECR	Argon	~ 12	Fast etch rate but preferential etching along damage pits and bulk defects
ICP	Argon-chlorine	~ 4	Slower etch rate but decreased surface roughness in well polished sample
ECR	Hydrogen	~.5	Very slow but not efficient



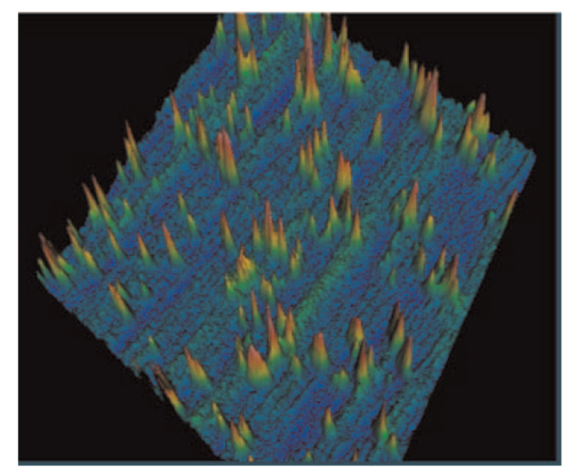
Roughness improves. Achieve: ~2nm
→ not enough for our application

But also problem seen by topography:
Topographs of volume dominated by "damaged" surface

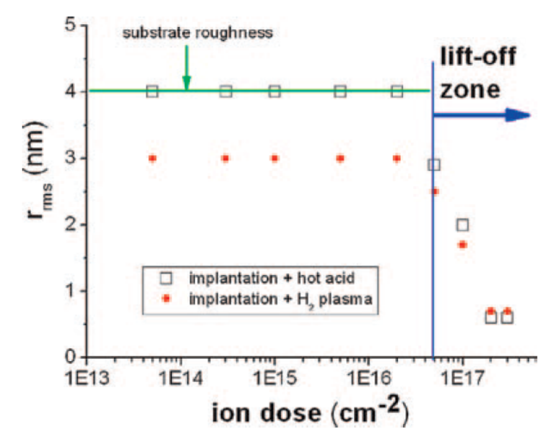
Implantation and lift-off process



O$^+$ implantation,
5 MeV, 10^{17} cm^{-2}
Creates buried damaged layer
Annealing in vacuo at 950ºC.

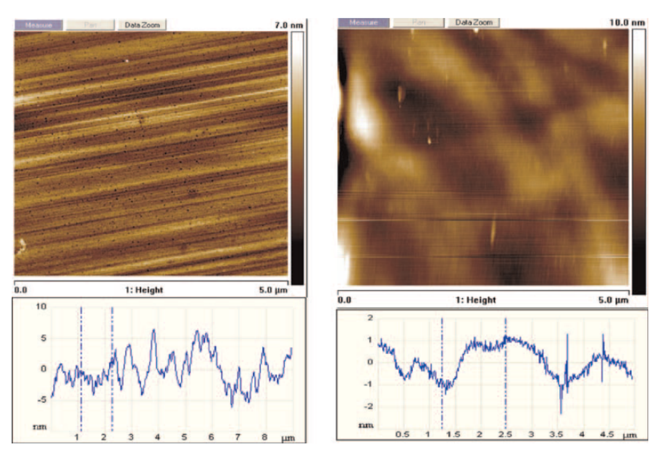


Optical profile after H plasma / hot-acid lift-off 50 x 50 mm^2



After final annealing / etch to to smooth the new surface and remove residual implantation damage

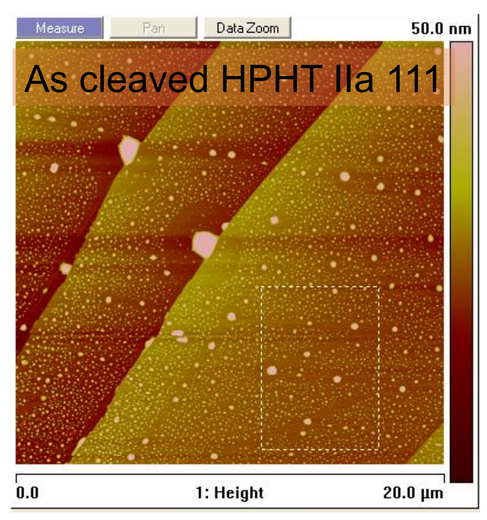
→ 0.6 nm RSM of surface roughness

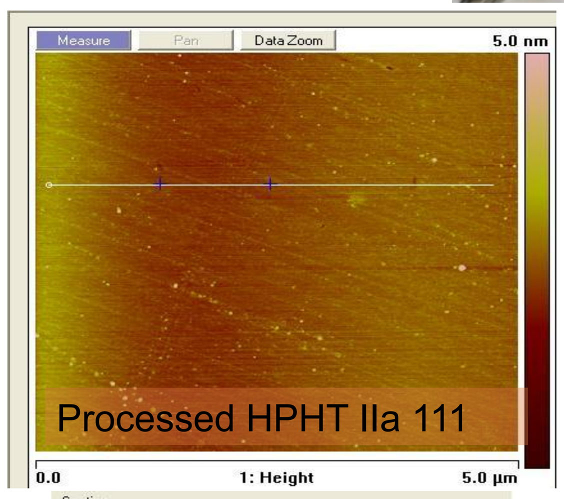


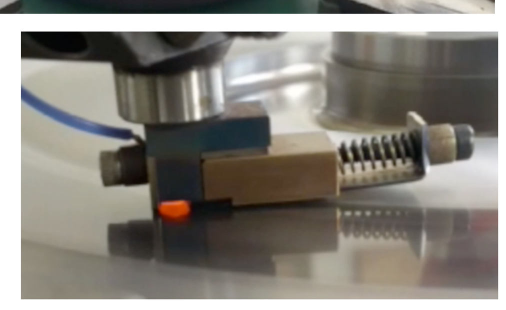
Hot-metalling (thermo-chemical-mechanical process)

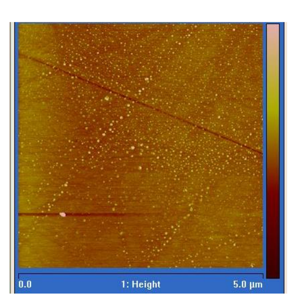
Method 2 : high load (20000N), high speed (300m/s), pre-heated scaife, no diamond grit

Specifications
Diamond gritless process.
Planetary motion of scaife.
High load, high speed ore-heated cast-Fe scaife.
Scaife temperature 450^{0}C for ~ 0.5 hours.
Final diamond temperature via friction.
Material removal via C-diffusion into Fe.
Removal rate: ~ 5μm/hour.

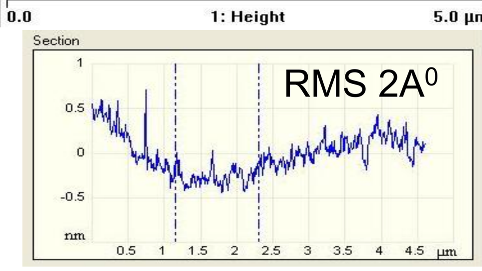






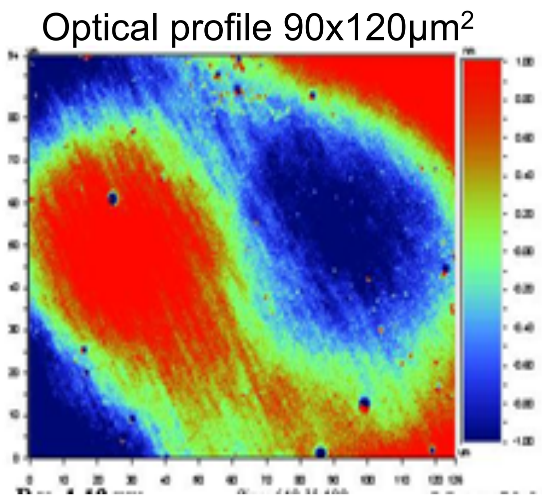


RMS 6nm
Terraces



Surface areas of 5x5um^2 are very smooth with RMS ~ 2A^0 at many different local areas. Only feint parallel polishing lines remain visible. Surface undulations remain.

Optical profile 90x120μm^2



Conclusions

1. The thermo-chemical-mechanical techniques appear very promising.
2. This method can address the 111 diamond surface
3. The best results include a surface with a roughness less than 2Å (rms) level over 10x10μm^2 areas as measured by AFM and optical profiling, for the (111) surface orientation.
4. The polished diamonds are still to be checked by X-ray diffraction synchrotron topography at beamline BM05, at the ESRF, France. This check for extended strain fields originating from the surface and extending into the bulk will indicate if any surface defects have been introduced from the polishing process.
5. Future experiments will optimise the systems for performing this process.

References

[1] T N. Tran Thi et al, Phys Stat Solidi A 208 (2011) 2057-2061
[2] J. R. Hird and J. E. Field, Proc. R. Soc. Lond. A 2004 460, 3547-3568