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FET proactive 7: Molecular
Scale Devices and Systems

Project N° 245122

DIAMANT
Diamond based atomic nanotechnologies
Small-Medium Collaborative Project

DELIVERY D2.3
Control of surface roughness

A delivery of Work package 2
Engineering atomic and artificial molecular and nanophotonic devices in diamond

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Partner name IMEC, E6

Version n° 1

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PU	Public	
PP	Restricted to other programme participants (including the Commission Services)	
RE	Restricted to a group specified by the Consortium (including the Commission services)	
CO	Confidential, only for members of the Consortium (including the Commission services)	CO
TN	Technical Note, only for members of the Consortium	

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01.08.2011			Consortium comments
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Person month for execution of the deliverable: 2 IMEC, E6: 2

Abstract:
The task is oriented to the technology, characterisation of the surface roughness of mono crystalline diamond on sub-nm technologies used are diamond etching and polishing with sub-nm resolution and resulting surface roughness control

DESCRIPTION

The aim of this deliverable was to prepare ultra-flat single crystal diamond surfaces with Rms < 1 nm using material produced at E6 and at IMEC. The material at E6 is prepared by a proprietary PE CVD technology. The single crystal diamond at IMEC is prepared using a commercial reactor SEKI-ASTeX PDS 17, shown on the following figure. Differences are in the complementarity of strategies for obtaining low Rms films. While at IMEC strategies was derived towards the optimisation of growth conditions to reduce Rms during the growth E6 work concentrated on polishing.

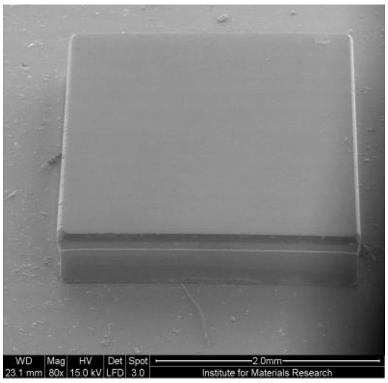


Fig. 1 *Schematic photo (a- left) MW – PE CVD apparatus used at IMEC for diamond growth and epitaxial diamond (b- right) grown at IMEC on commercial E6 HPHT substrate.*

To obtain low defect high quality diamond with low surface roughness both the PE-CVD process, including pre-treatment of the substrate surface before PE CVD epitaxial growth, the polishing and post-processing has to be optimised to high level.

At IMEC the effort was devoted to the control of the diamond substrate surface before the growth as well as the conditions for obtaining low defect density CVD diamond. The optimisation of the HPHT surface quality consisted of the selection of suitable substrates with low dislocation density and mono-growth sector substrates as well as (Ar/O2) plasma treatment before the deposition, allowing to prepare low defect density HPHT surface that could be further used for the epitaxy in PDS 17 system. Several growth condition regions have been mapped to obtain high quality crystals, by changing the CH4/H2 gas ratio, the substrate temperature, arrangement of the substrate holder as well as MW power, gas pressure etc. The following figure shows a step-flow growth of CVD diamond with large step bunching. The high quality i.e.low defect density of diamond is important not only for the value of the decoherence times, or other luminescence centres but also for the final surface quality and depth of the polishing damage that has to be removed.

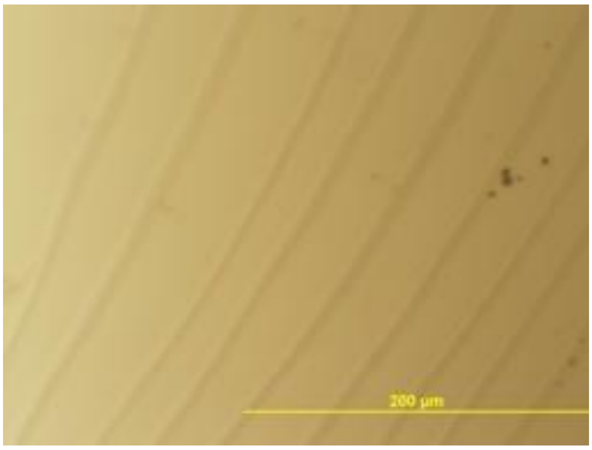


Fig. 2. *Macro-step bunching for 500 micron thick as -grown CVD diamond prepared at IMEC.*

By comparison of figure 1b and 2 it is evident that the growth conditions for samples from Fig 1b (8% CH4 in H2), Figure 2a 2% (CH4in H2) have a detrimental influence on the morphology of the final layer which effects the selection of right polishing method. While the surface in Fig.1 b is nearly atomically flat after the growth without any polishing (though with a waviness on larger area), the surface in Fig.2 shows macroscopic features. The production of nearly atomic flat surfaces was the main success of the IMEC group during the first 12 month of the project. For the final surface it is not only its microscopic flatness but also the surface waviness which might imply that CVD grown samples have to be polished and post treated to obtain ultra flat surfaces, depending on the final application. At both patterns, significant work has been carried out to reach these requirements.

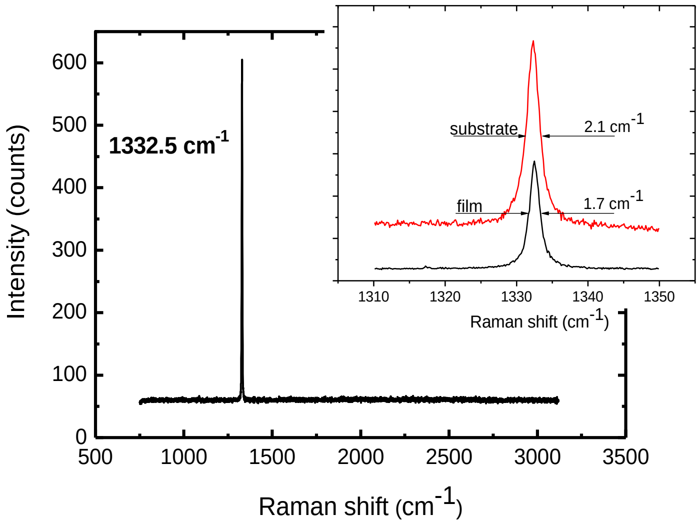


Fig 3 *Raman spectra of the film from Figure 1b., prepared at IMEC.*

The deposited layer shows a very narrow FWHM Raman line of 1.7 cm-1 , confirming high quality of the deposited epitaxial diamond. FWHM of the Raman line of the I b substrate is actually larger than the resulting CVD epitaxial layer.

The group at IMEC has investigated defects such as strain induced birefringence arising from bundles of dislocations lying almost parallel to the [001] growth axis. Some of these specific birefringence patterns consist of four or eight bright petals, depending of the observation conditions. The following figure shows Raman imaging investigation of such defects. Further on dislocations can were found to nucleate at the epitaxial interface. In each case, dislocation nucleation can be caused by particulate (carbon) contamination. High compressive strain was evidenced in the vicinity of such carbon particles, which was found to decrease in magnitude as the film grew in thickness. Therefore optimisation of the surface preparation as well as the growth conditions is important for obtaining optimal surfaces.

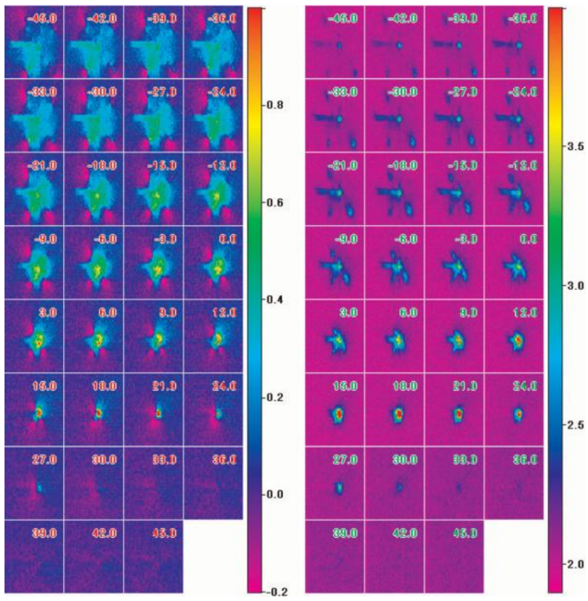


Fig. 4*Stack of frequency left) and FWHM Raman line width(right) from the sample surface to the deeper part in the substrate. The film thickness was probed in steps of 3 mm. The value given in each image corresponds to the position of the piezoelectric device along the z-axis. The ''□45 mm'' coordinate corresponds to the sample surface, and the interface is approximately located at the ''21 mm'' coordinate. The frequency images (left) are given as a shift of the diamond line from the expected position for unstrained diamond.*

Subsequently, IMEC has been working on post processing of the PE CVD layers. At first layers were polishing using a new technique developed at IMEC, which gives extreme flatness. This technique comprises first polishing using high rotation speed skyfe (with a holder adapting the surface movement) followed by a chemical polishing, This two step polishing achieves in first stage roughness about 1 micron and in the second stage record roughness about 0.1 nm. This is unique result compared to the current state of the art. In addition to that, we have used a plasma polishing to achieved still better nearly atomically flat surfaces. After the chemical polishing

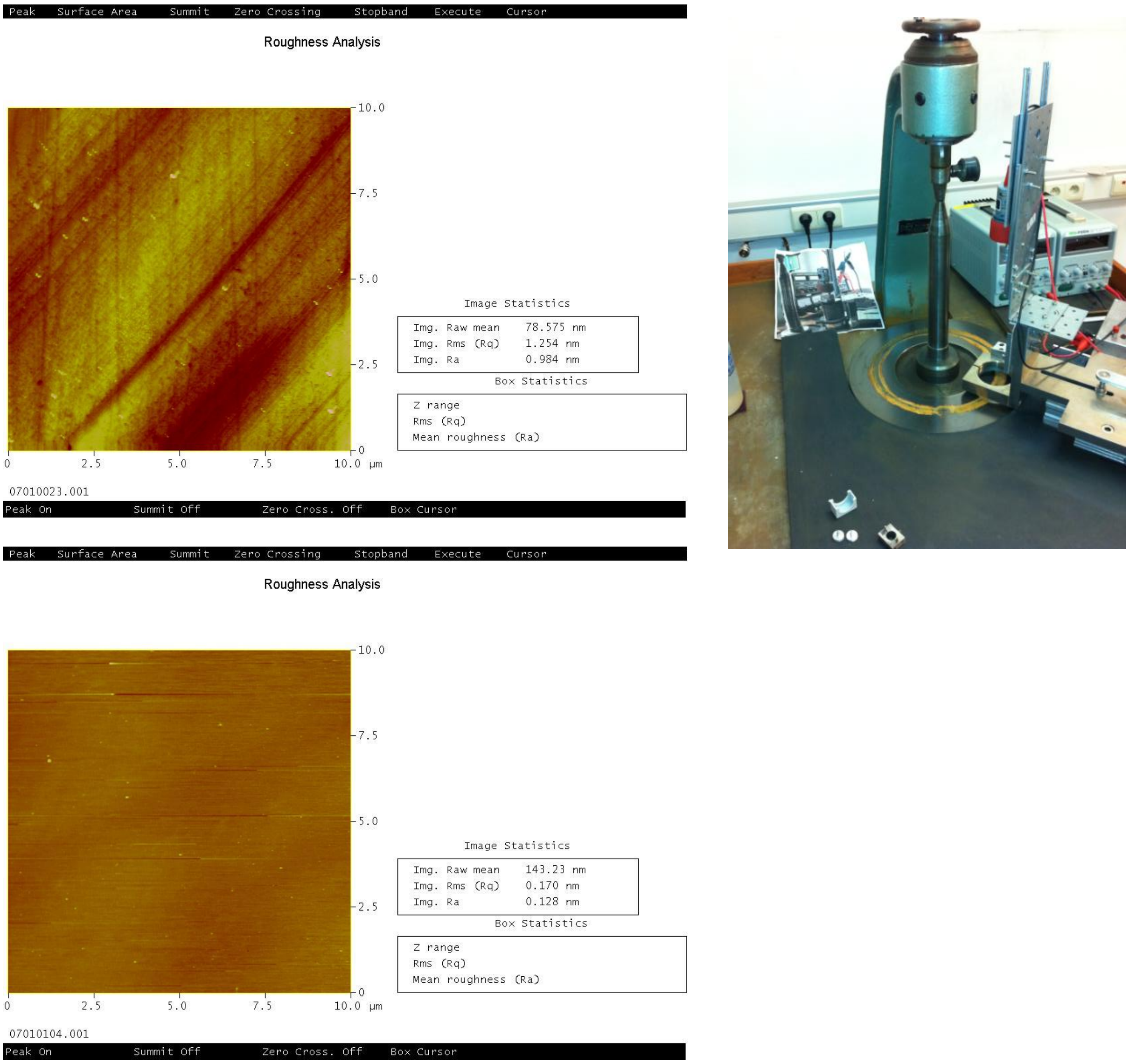
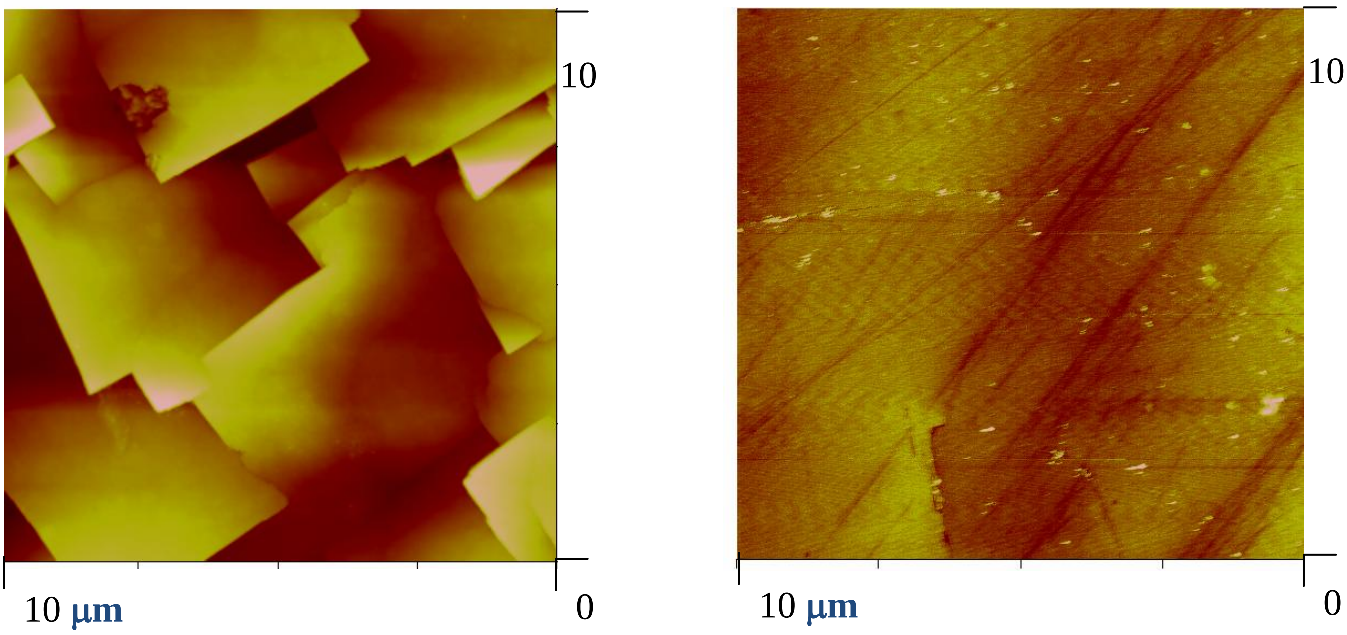


Fig.5 *from the top: HPHT diamond substrate rough-polished til < 1 nm, chemical polishing till record 0.1 n. On the tight side the detail for the chemical polishing setup used.*

CVD poly-diamond

CVD poly-diamond

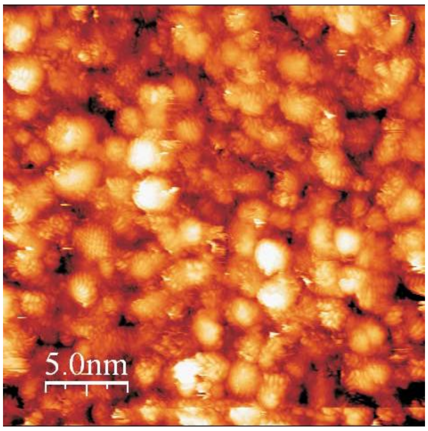


roughness (RMS): 50 nm

roughness(RMS): 0.8 nm

Fig6. *IMEC has also developed successful polishing of thin CVD diamond films on the Si substrate, allowing to reduce surface roughness below 1 nm, without removing the film of thicknessof about 8micrn thick*

Diamond have been further treated Ar plasma and then by H plasma and fully atomically flat surfaces have been achieved. The picture shows also the equipment used.



(b)

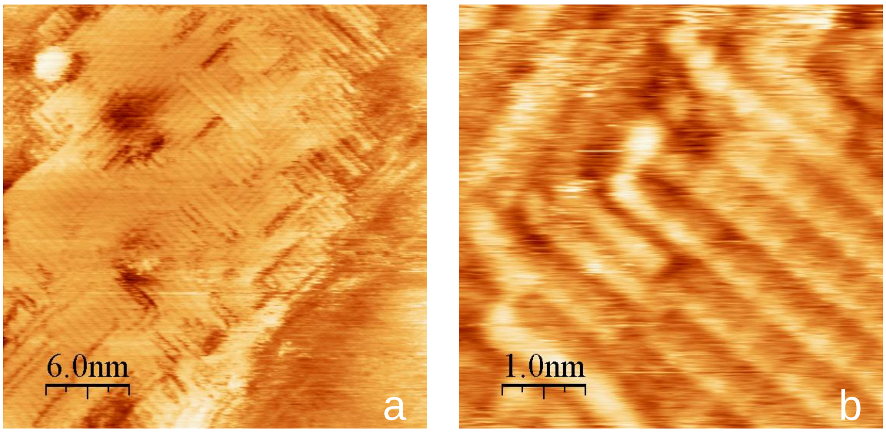


Fig. 6AFM pictures of Ar plasma treated (a) and hydrogen plasma treated (b) CVD diamond surface. The hydrogen treatment led to atomically flat CVD diamond surfaces on the area of several mm square

Polishing developments at Element 6.

By nature of its extreme hardness and other mechanical properties processing diamond to give flat low damage surfaces is notoriously difficult. While many workers in the field have reported low flatness few have reported details on low damage. During mechanical processing sub-surface damage can be created (eg. vacancies, interstitials and dislocations) in the near surface region. In terms of future devices targeted by Diamant, shallow implantation into the near surface region, <100 nm from the surface, of material is likely to be critical. Hence any residual processing damage could lead to defects with unpaired spin, shortening the decoherence times, in addition to adding other luminescence centres which could cloud the desired luminescence or alternatively lead to spectral instability of the desired centre.

E6 polishing recipes have produced {100} single crystal (SC) diamond surfaces with a surface roughness <1 nm.

Whilst deliberately minimized, these surfaces are likely to contain some level of sub-surface crystal damage. It has been shown that this can be mitigated through a subsequent reactive ion etch step, which is capable of maintaining the smooth surface morphological properties, whilst at the same time physically removing the top layer of damaged diamond[1] (Figure 7).

[1]I. Friel, et al., Diamond Relat. Mater. (2009), doi:10.1016/j.diamond.2009.01.013

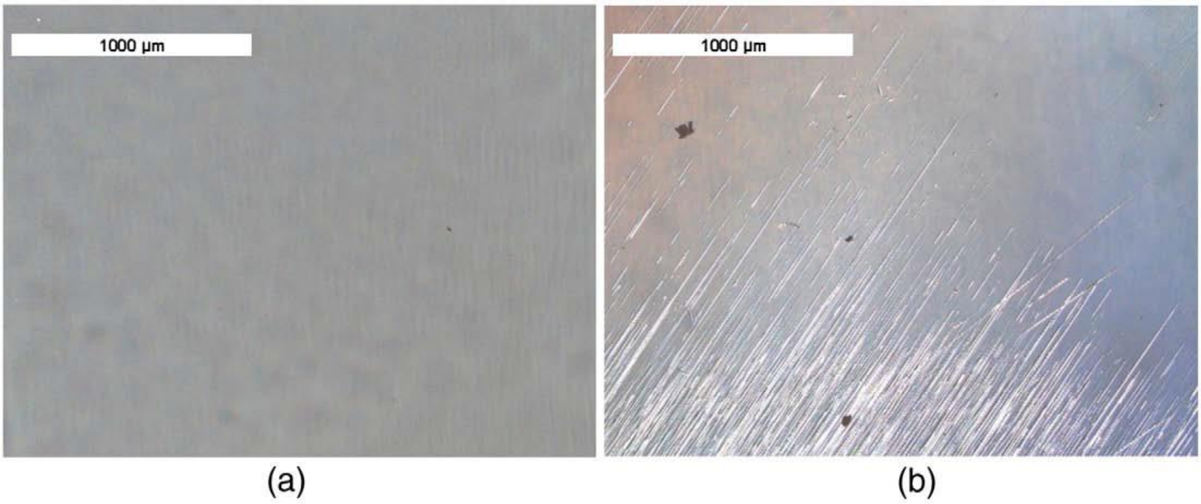


Fig 7 *DIC micrograph comparison of mechanically polished diamond surfaces following (a) Ar/Cl ICP etching and (b) O_2 plasma etching (image from Ref 1).*

RESOURCSES USED TO EXECUTE THE DELIVERABLES:
IMEC 2 PM, E6 2 PM

RELATION TO OTHER DELLIVERBLES/TASKS:
D2.1 Ultra-high-purity bulk 12C single-crystal CVD diamond
D2.2 Characterization of ultra-high-purity single-crystal samples
D2.4 AFM diamond probes
D2.5 Spectroscopy of new defects
D2.6 High yield production of defects
D2.7 Coherence time for shallow defects

CONLUSIONS:
The diamond growth was optimised at IMEC to prepare high quality CVD diamond films. By selection fop the growth conditions it has been possible to prepare nearly atomically flat CVD diamond surfaces even for thick epitaxial layers with Rms below 1 nm. However this layers show certain waviness.

At E6 already developed technology was further optimised to produce high quality and ultrahigh purity CVD diamond samples. E6 has demonstrates polishing with surface roughness below 1 nm and low defect density.