



# Influence of Pulse Energy/Duration, Wavelength and Incidence Angle on XUV/X-ray Damage Phenomena

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## XUV/X-ray laser damage to materials can be at least of two kinds

- 1) low fluence – multiple shots: single photon (nonthermal) damage
- 2) high fluence – single shot: thermal damage

D. A. G. Deacon: Optical coating damage and performance requirements in free electron laser, *Nucl. Instrum. Meth. Phys. Res. A250* (1-2), 283-288 (1986).

- 3) scientifically very interesting are intermediate irradiation conditions, i. e. few-shot near-threshold irradiation

# a-C sample preparation and characterization



**Preparation:** The a-C samples were (40-45)-nm a-C layers deposited on silicon substrates by GKSS/Incoatec (M. Störmer; Geesthacht, Germany; [www.incoatec.de/www.gkss.de](http://www.incoatec.de/www.gkss.de)).

The amorphous carbon films were produced on planar, well-polished silicon substrates in an ultrahigh-vacuum chamber by DC magnetron sputtering.

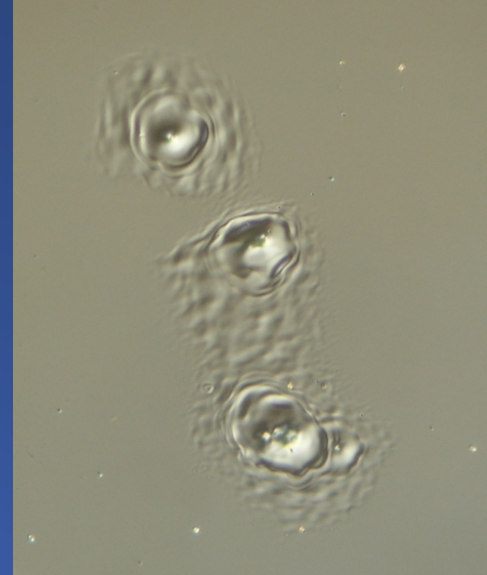
**Characterization:** The films were routinely characterized with unpolarized Cu-K $\alpha$  radiation using an X-ray reflectometer (Bruker AXS D8) equipped with a reflectometry stage and a primary Göbel mirror. The reflectometry curves were fitted using the Bruker AXS simulation software. Both film thickness and density were determined. Furthermore, the film properties were measured at relevant wavelengths in the XUV and soft X-ray range at the soft X-ray reflectometry beamline G1 (HASYLAB/DESY).

**(a)** S. Jacobi et al.: Characterization of amorphous carbon films as total-reflection mirrors for XUV free-electron lasers, *Proc. SPIE* **4782**, 113 (2002).

**(b)** B. Steeg et al.: Total reflection amorphous carbon mirrors for VUV Free Electron Laser, *Appl. Phys. Lett.* **84**, 657 (2004).

**(c)** M. Störmer et al.: Investigations of large x-ray optics for free electron lasers, *Proc. SPIE* **5533**, 58 (2004).

**Nanoscopy Lab** established in 2004 at IP-ASCR, Prague is equipped with



**A. Nomarski (DIC) microscope Olympus BX51** and

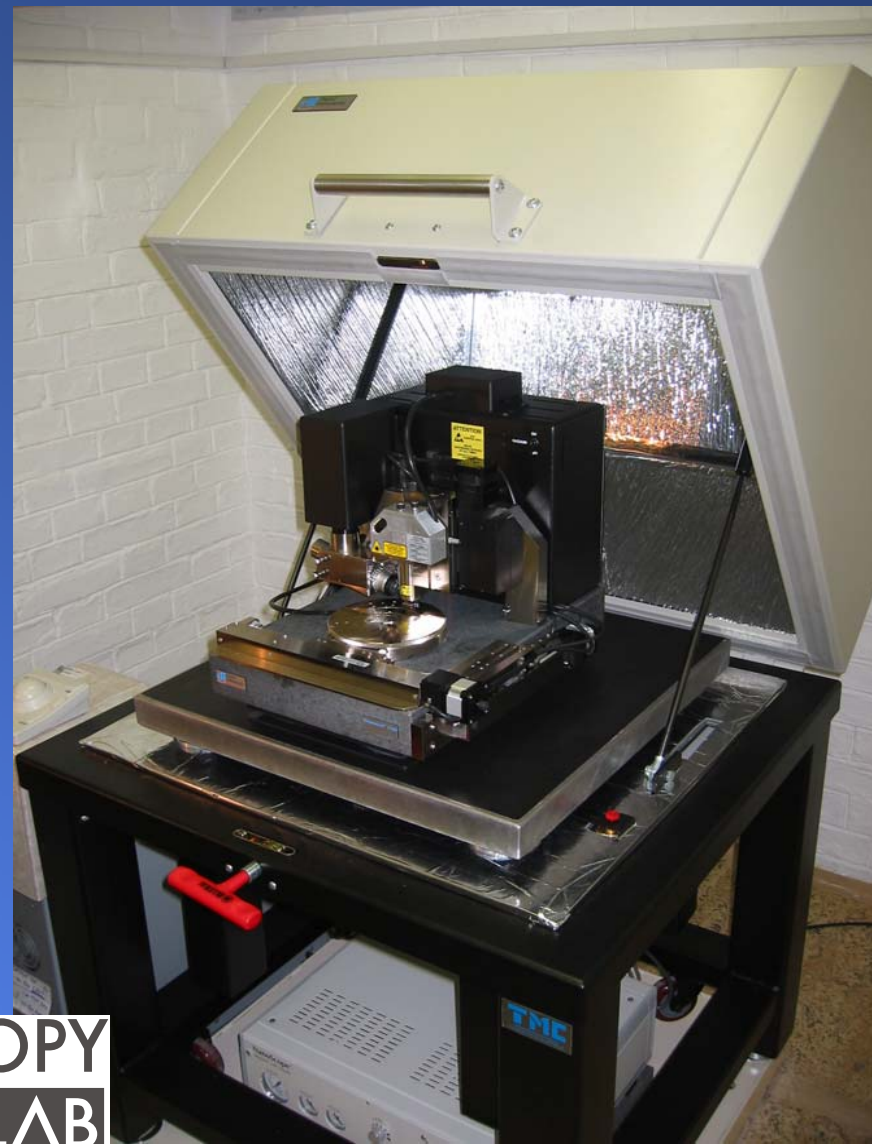
**B. VEECO NanoScope Dimension™3100 Scanning Probe Microscope**

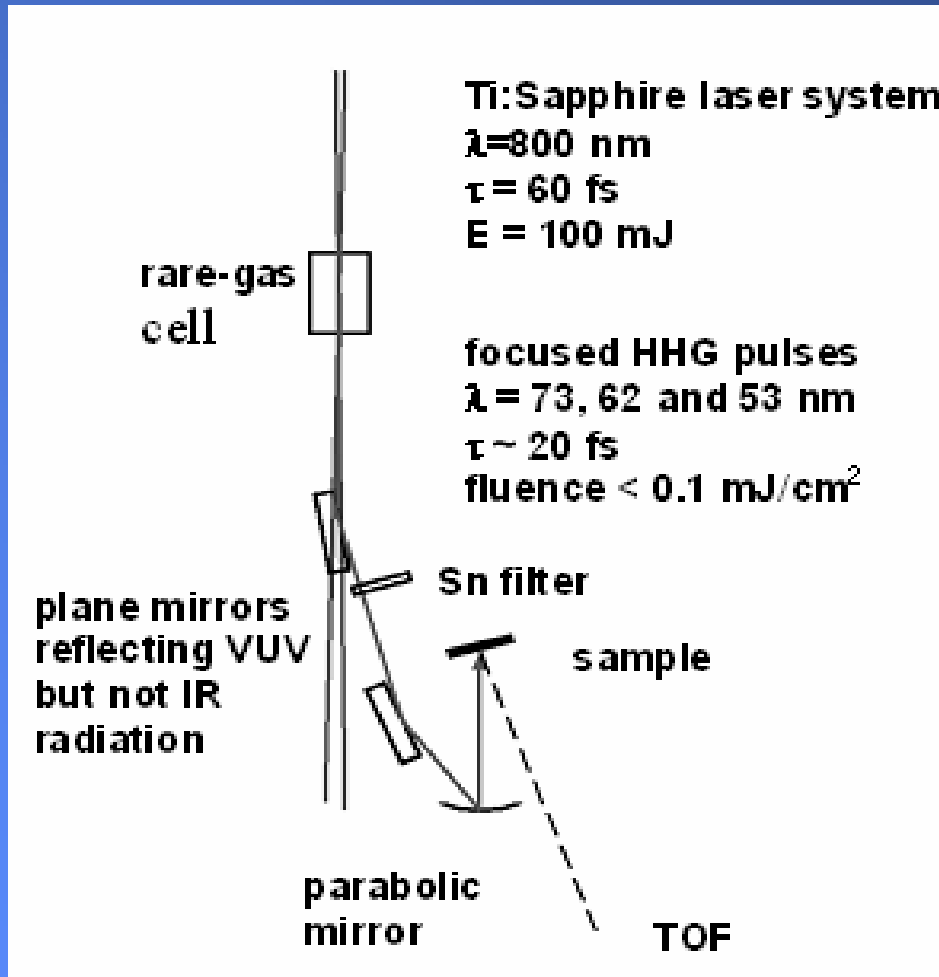
**The key components of the SPM instrument are as follows:**

- a) D3100 NanoScope Dimension™** performs all major AFM imaging techniques, of course including the tapping mode. Inspectable area is 120 mm x 100 mm; samples up to 200 mm diameter and 12 mm thick [large size optical elements; coatings on massive substrates; the large samples irradiated with TTF1 FEL].
- b) NanoScope IV Control Station** allows up to 10x faster topographical scanning and PhaseImaging in air with the tapping mode [TappingMode+™] than provided by other SPM controllers [including the NanoScope IIIa used in our previous studies].

c) **DAFMCL** [Dimension Closed-Loop SPM Microscope Head]  
SPM/AFM scanner with horizontal imaging area  $90\ \mu\text{m} \times 90\ \mu\text{m}$  nominal maximum and a vertical of  $5\ \mu\text{m}$  nominal maximum is used for a crater profiling resulting in an accurate determination of the ablation (etch) rate.

The instrument provides routinely a resolution requested for imaging the  $\sim 10\text{-nm}$  spaced LIPSS expected at the surfaces irradiated by XUV lasers.





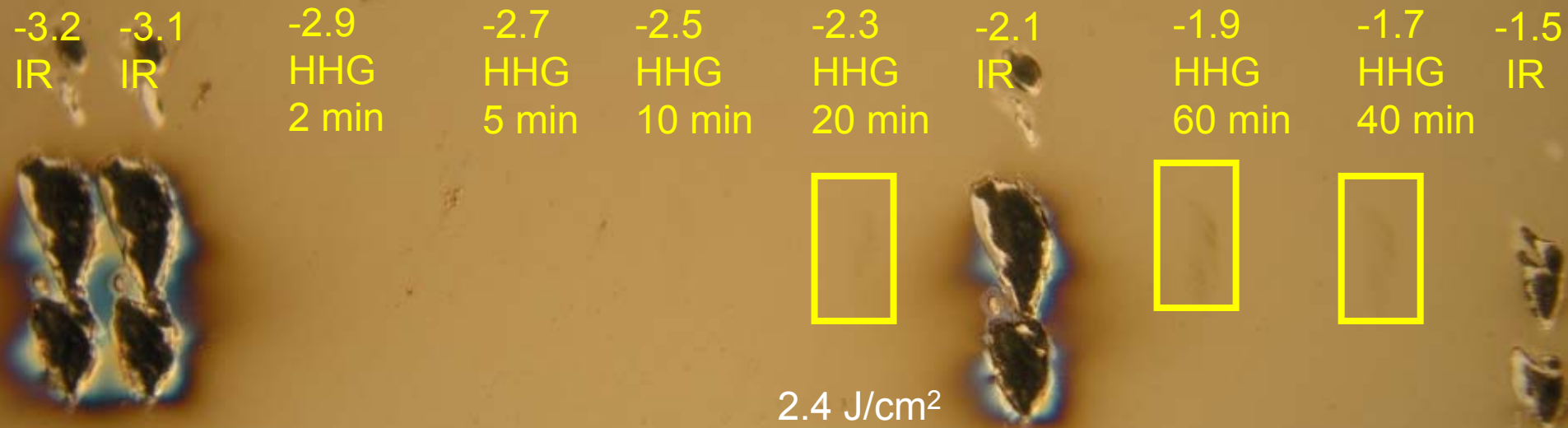
11th, 13th and 15th harmonics of LUCA beam operated at SPAM/DRECAM, CEA-Saclay - Hamed Merdji, Stéphane Guizard et al.

for more details see *Proc. SPIE* **5917**, 91-96 (2005)

**Scheme of the experiment with the beam of high-order harmonics.**



a-C/Si; SPAM/DRECAM, CEA-Saclay  
March 3 2005

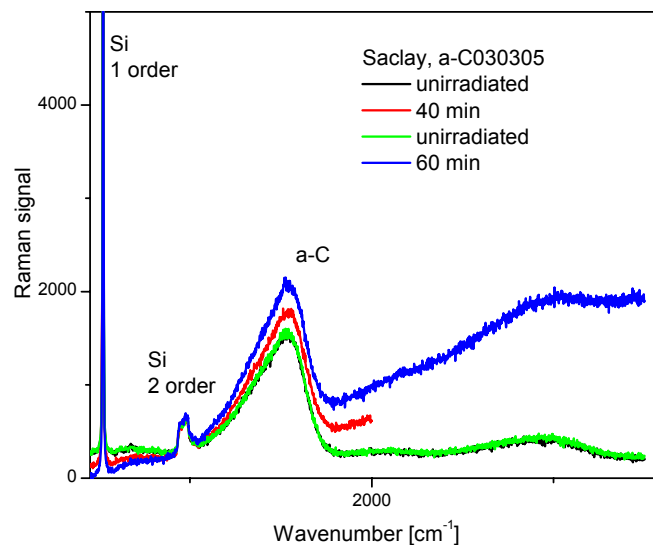


# a-C/Si irradiated by focused HH beam at SPAM-CEA in Saclay

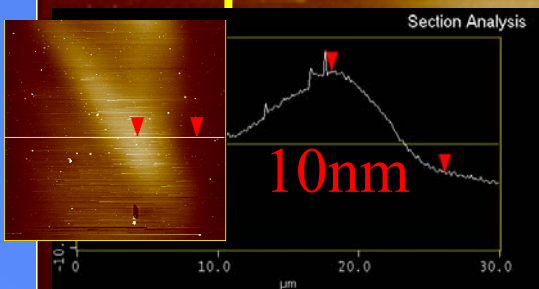
spot HHG1,  
40 min

spot HHG3,  
20 min

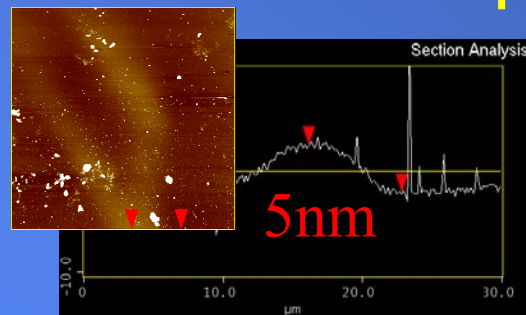
50  $\mu\text{m}$



fluence:  $\sim 0.1 \text{ mJ/cm}^2$   
reprate: 20 Hz



20  $\mu\text{m}$



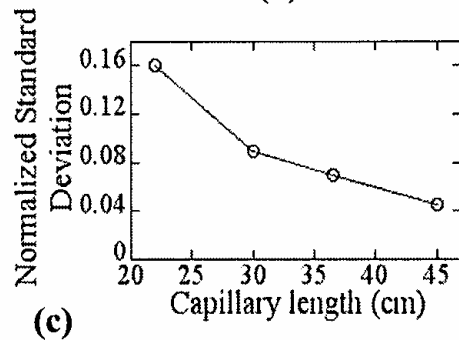
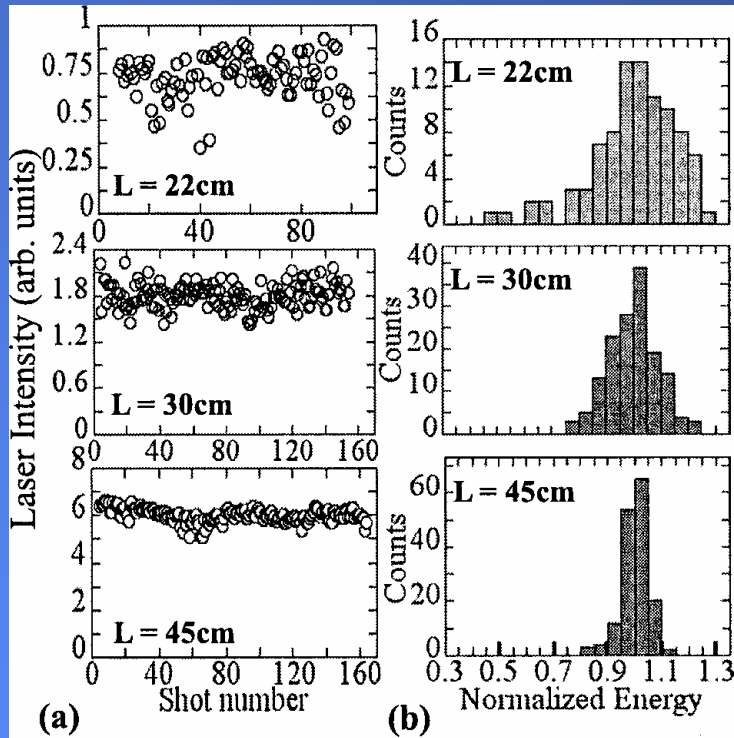
# Measurements of energy fluctuations of a saturated 46.9 nm Ar laser produced in Z-pinch capillary discharges

A. Ritucci,<sup>a)</sup> G. Tomassetti, A. Reale, and L. Reale

*Department of Physics, University of L'Aquila, gc LNGS of INFN, INFN, 67010 Coppito, L'Aquila, Italy*

F. Flora and L. Mezi

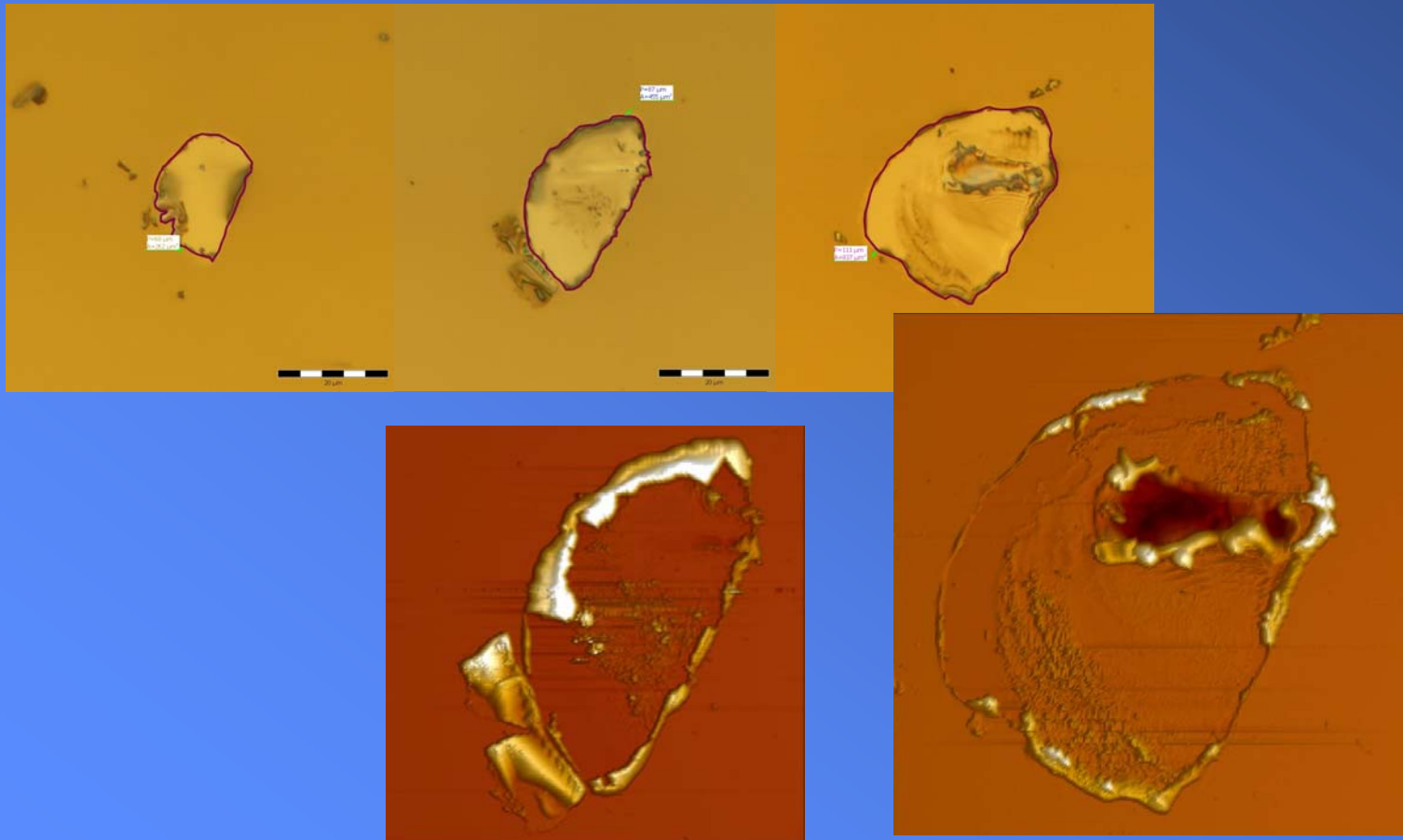
*ENEA Dipartimento, Innovazione, Divisione Fisica Applicata, CRE Frascati - C.P. 65, 00044, Italy*



a-C exposed to 1.7-ns pulses of 46.9-nm radiation

Single shot damage threshold was found to be around **1.1 J/cm<sup>2</sup>** using the Liu method

fluence increases from left to the right



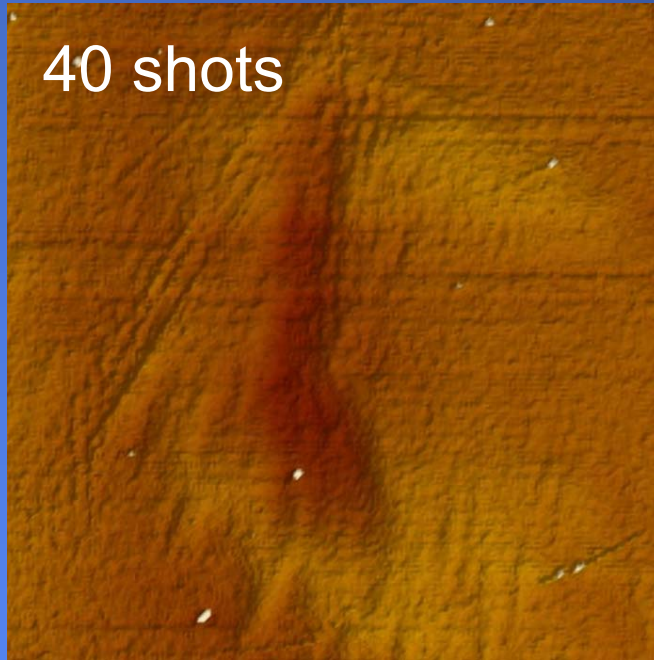
30x30  $\mu\text{m}^2$

5 nm NANOSCOPY



IP-ASCR-Prague

40 shots



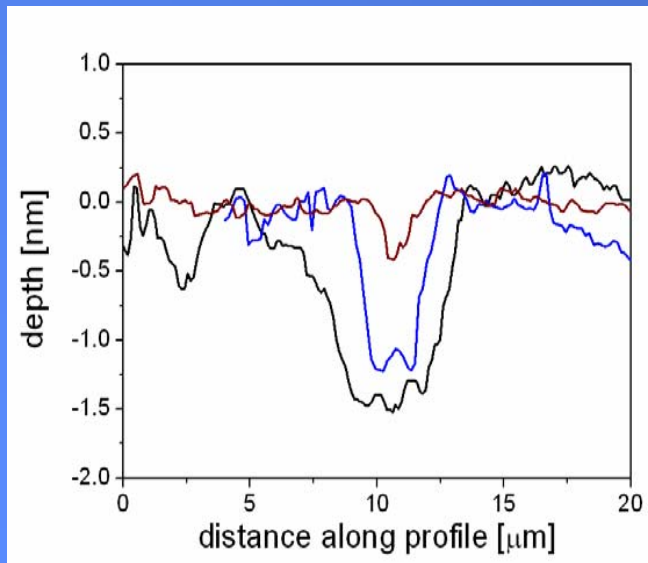
20 shots



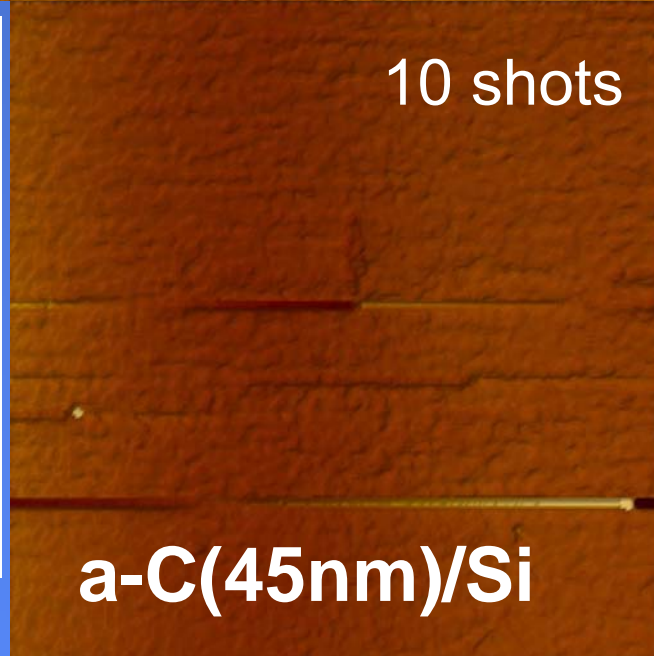
46.9 nm

1.7 ns

0.5 J/cm<sup>2</sup>

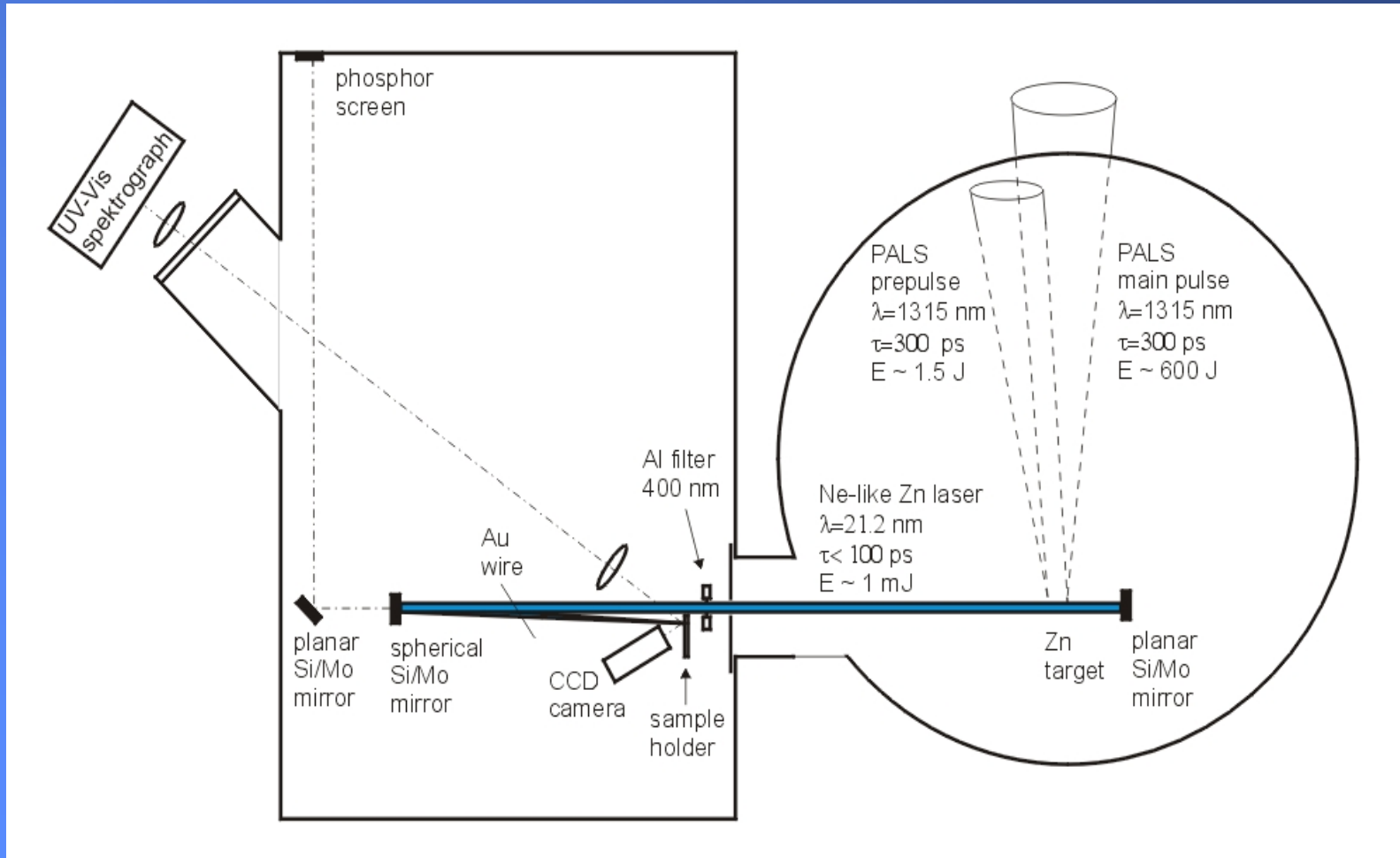


10 shots

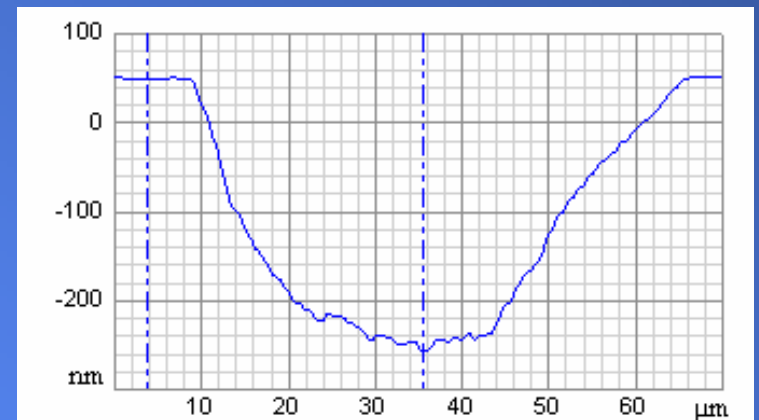
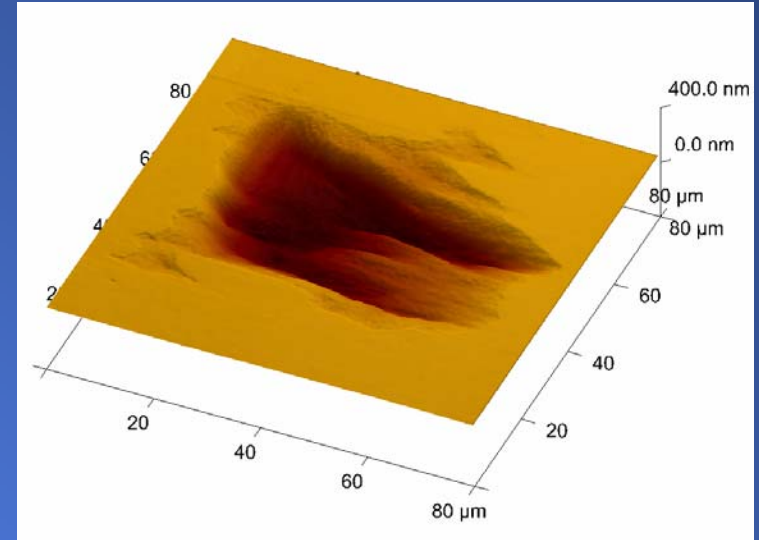
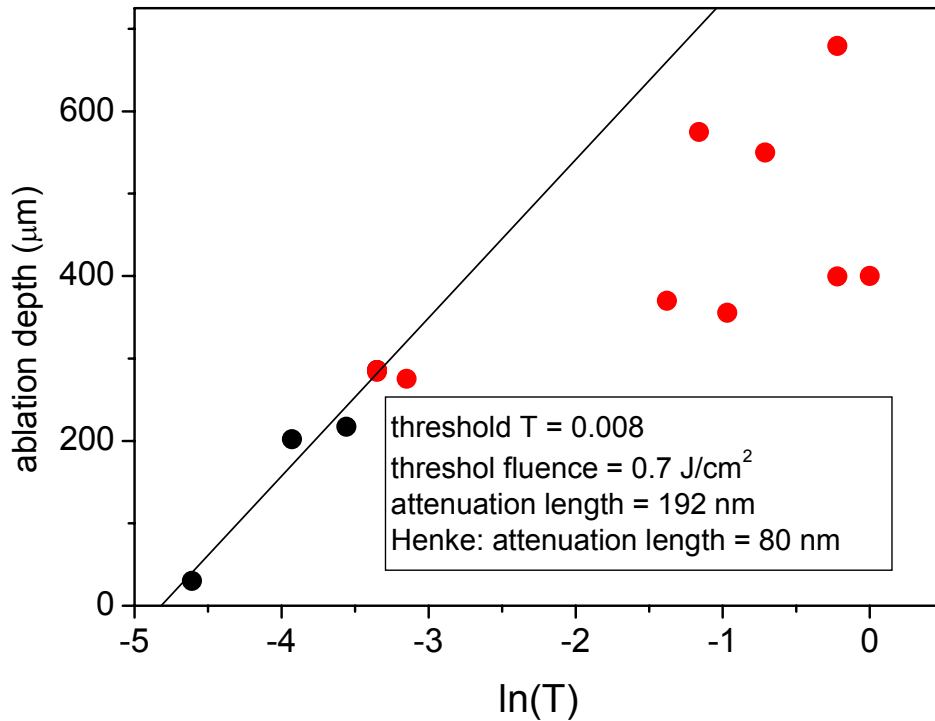


a-C(45nm)/Si

# focusing scheme of Ne-like Zn soft x-ray laser

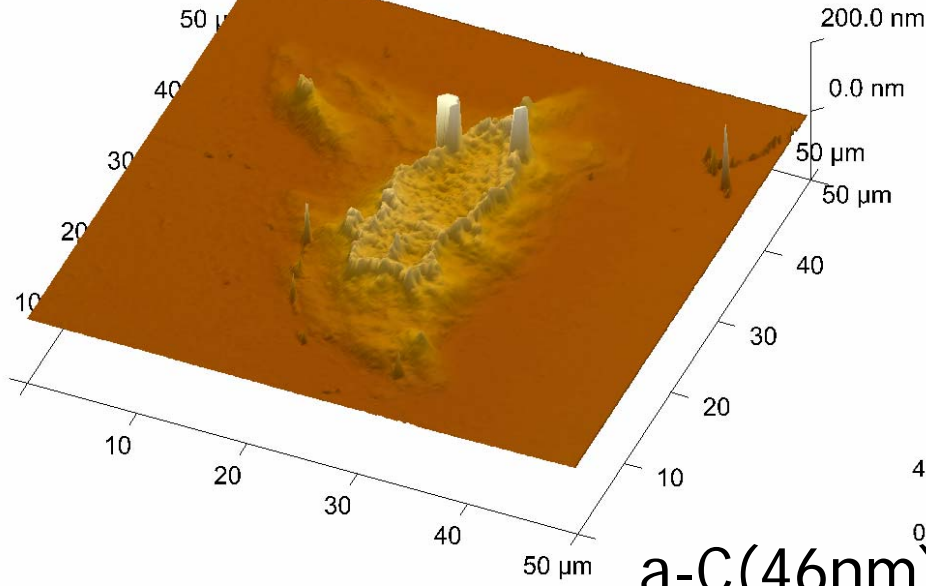


# PMMA ablation induced by 80-ps pulses of 21.2-nm radiation

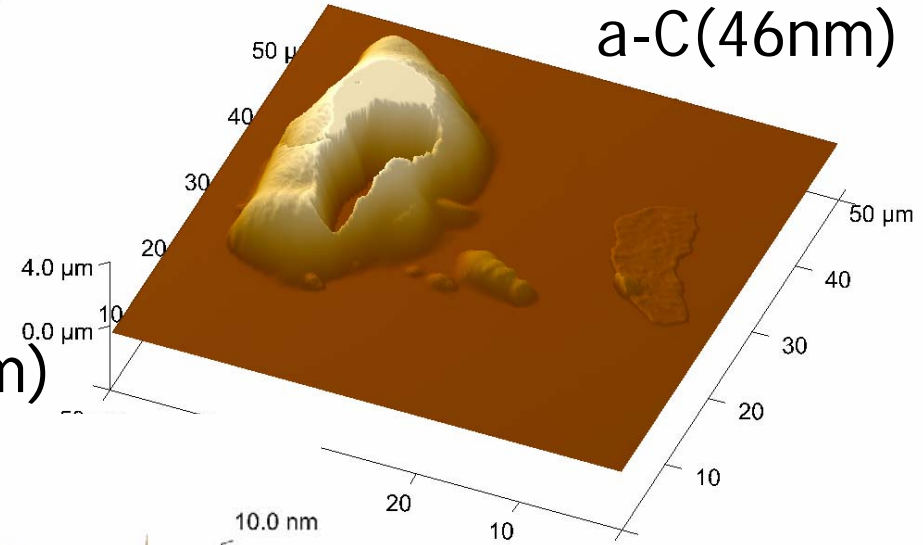


Al filter:  
 $T=10\%$

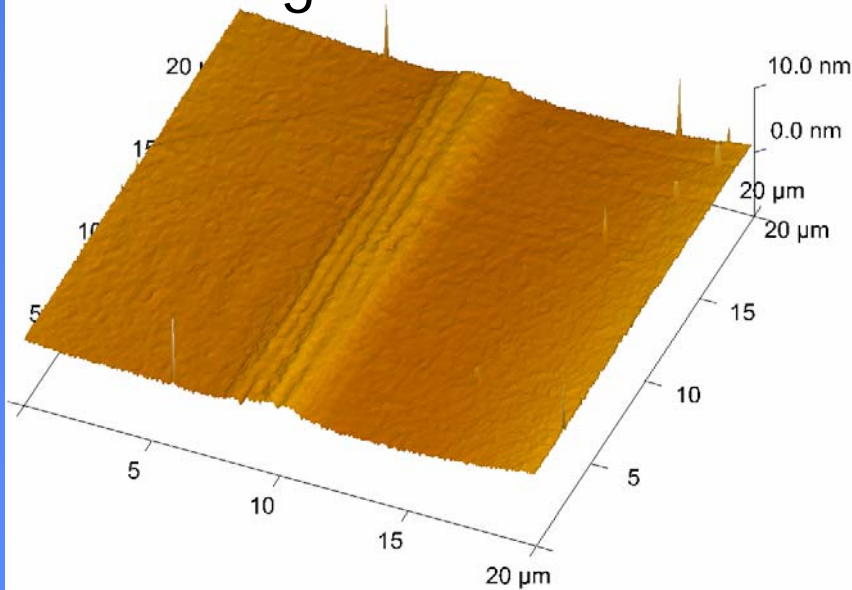
a-C(890nm)



a-C(46nm)



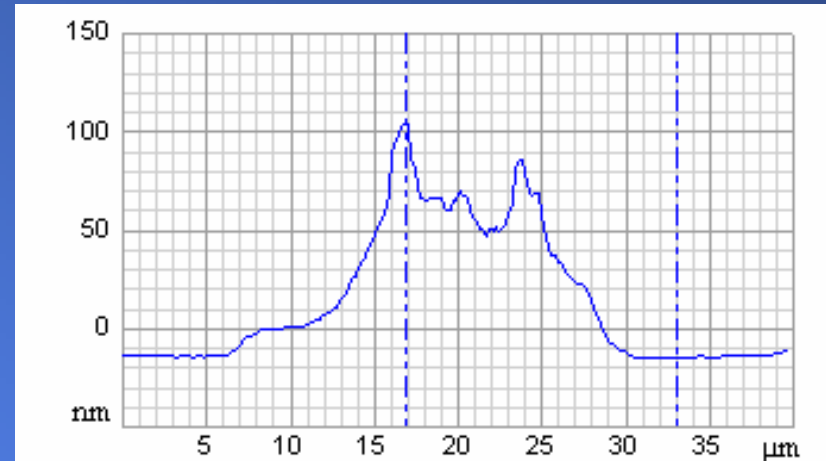
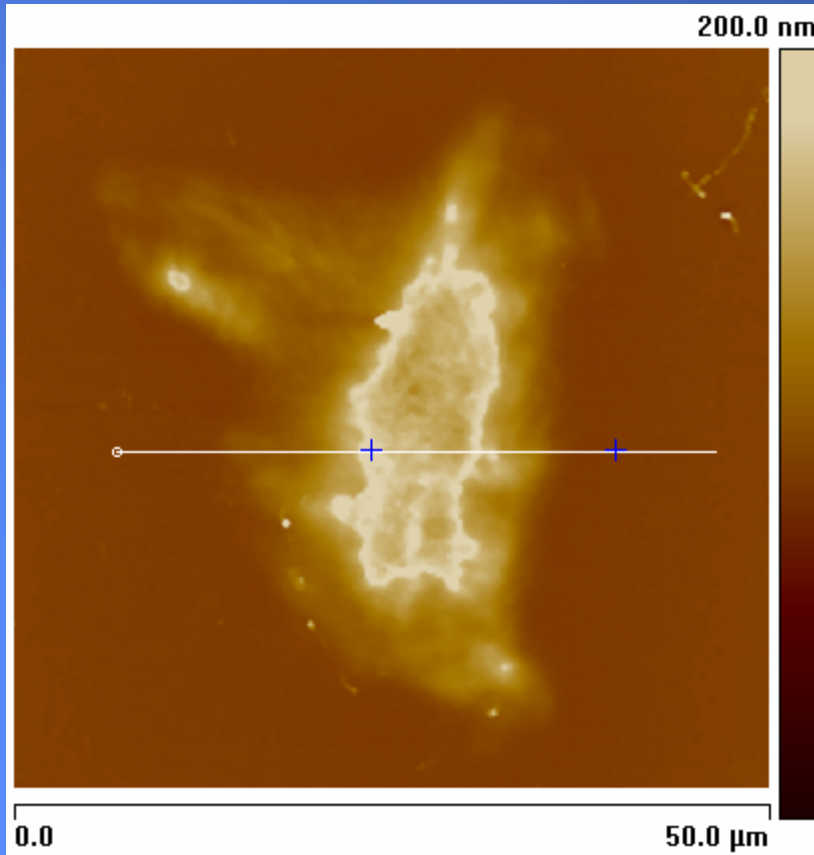
a-C(46nm)  
 $5^\circ$



no filter

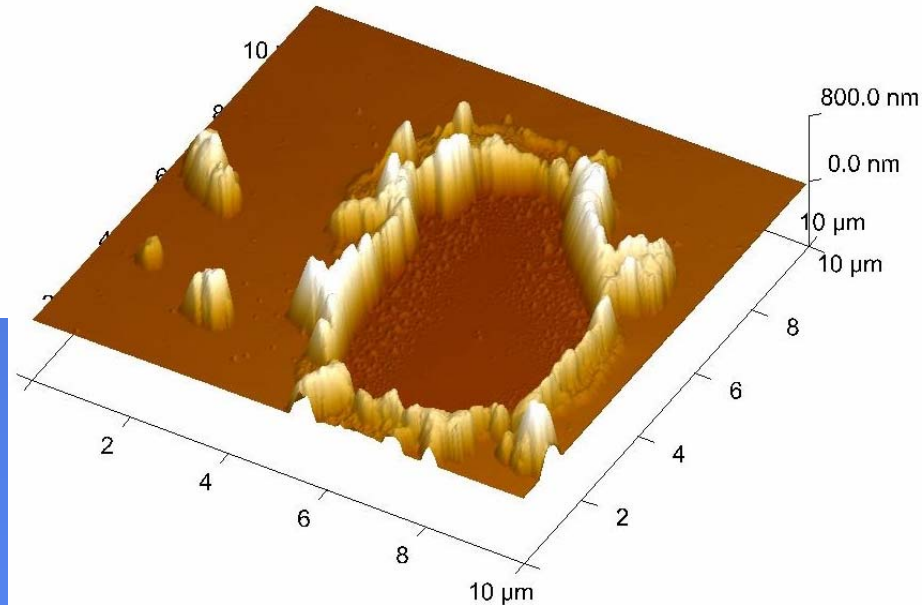
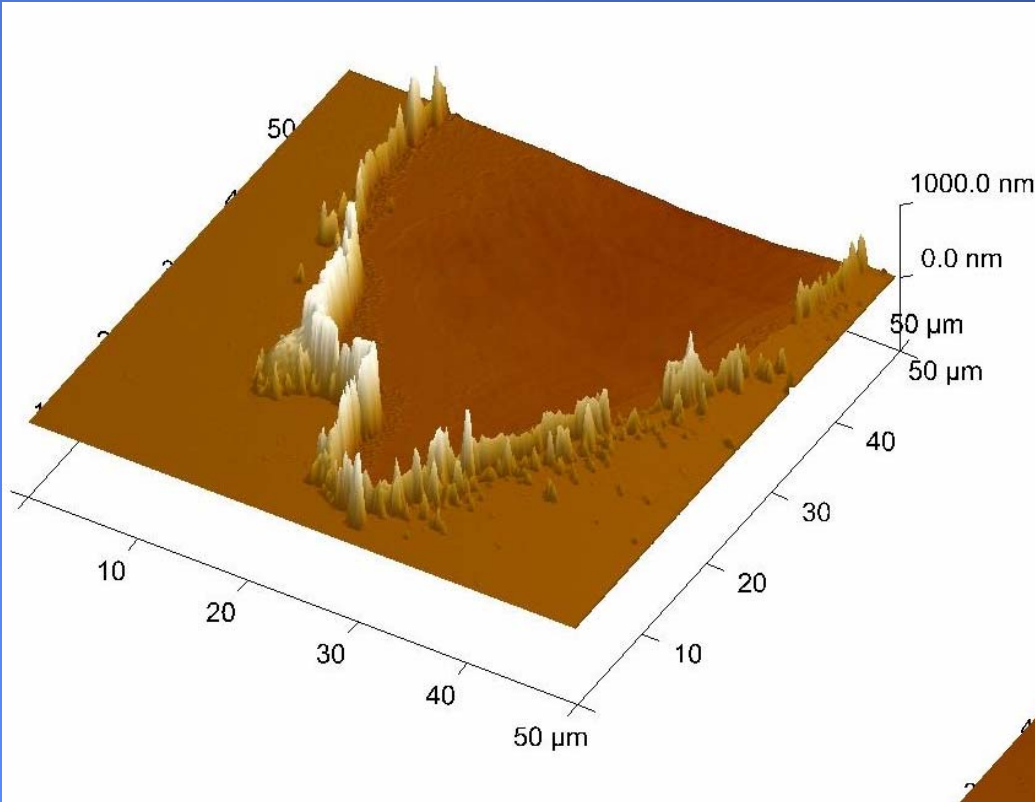


a-C thick layer irradiated by 80-ps pulses of 21.2-nm radiation



a-C(890nm)

# 21.2-nm plasma-based laser induced damage to 100-nm a-Si layer deposited on fused silica substrate by Ch. Hecquet



crystallization of a-Si layer irradiated by keV synchrotron radiation was reported 15 yrs ago

**F. Sato, K. Goto, J. Chikawa:** Solid-Phase Epitaxy with X-Ray Irradiation to Grow Dislocation-Free Silicon Films at Low Temperatures, *Jpn. J. Appl. Phys. Part 2* **30**(2A), L205 (1991)

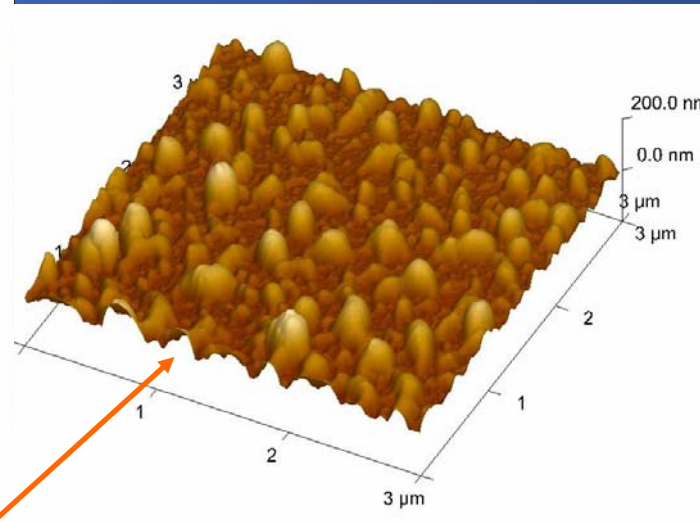
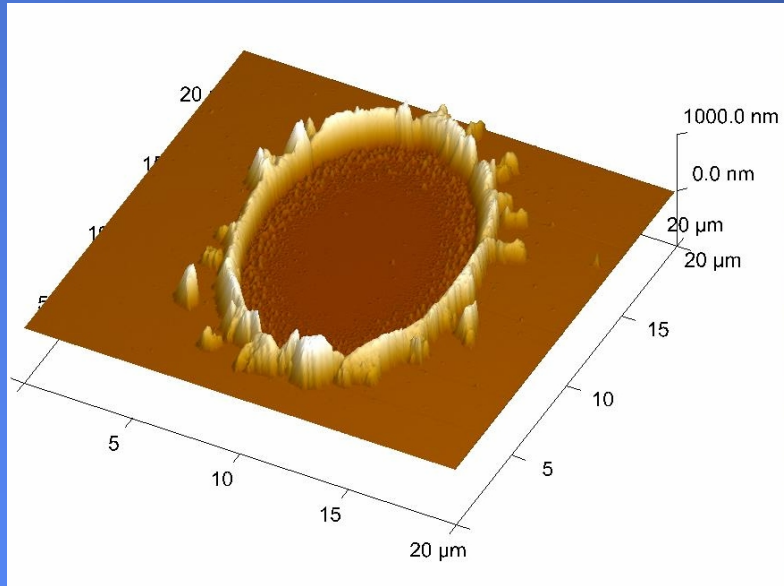
Radiation recrystallization of a-Si was explained by assuming that vacancy-interstitial pairs are formed with X-ray irradiation by Auger processes.

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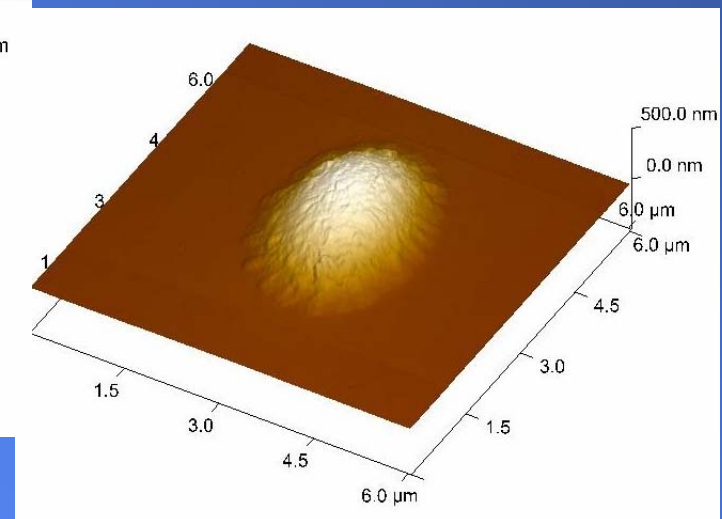
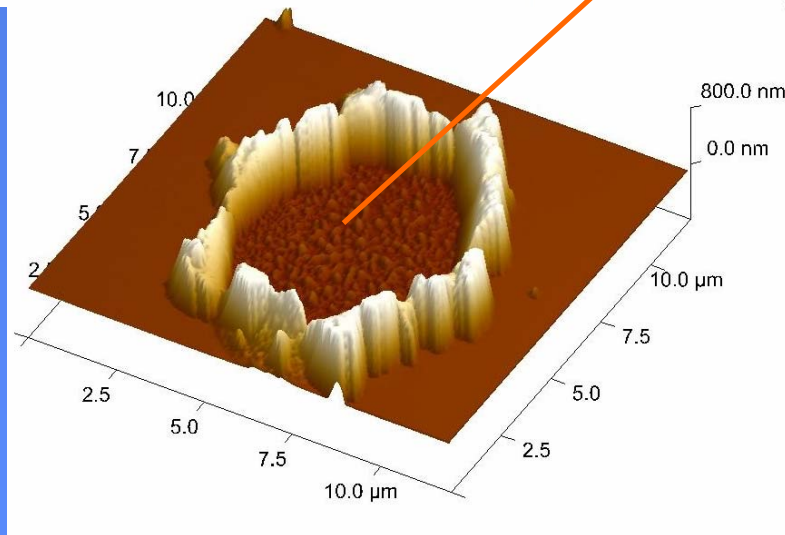
**T. Matsumura, H. Katayama-Yoshida, N. Orita:** Vacancy-Interstitial Pair-Formation Mechanism of X-Ray-Irradiation-Induced Crystallization in Amorphous Silicon Studied by ab initio Molecular Simulation, *Mat. Res. Soc. Symp. Proc.* **377**, 275 (1995)

The bistable dangling bonds were found to be responsible for the nonthermal vacancy-interstitial-pair-formation resulting in a reduction of crystallization  $E_a$ .

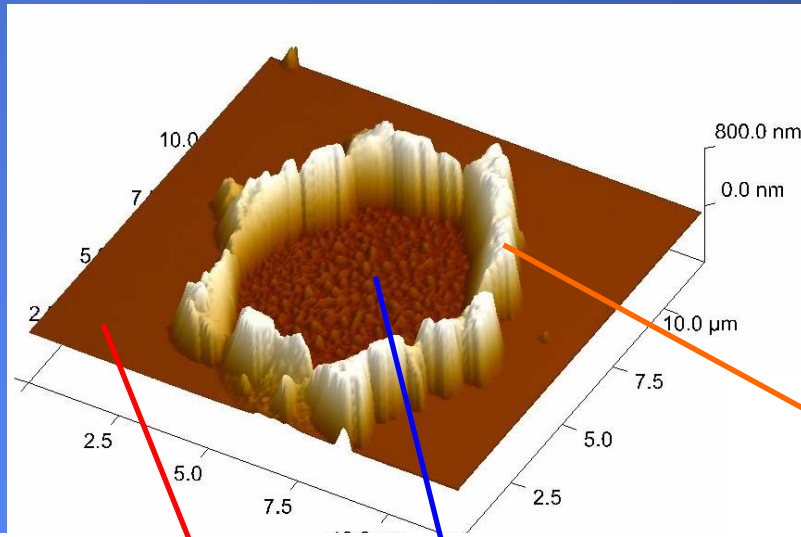
# FLASH-induced damage to 100-nm a-Si layer deposited on fused silica substrate by Ch. Hecquet



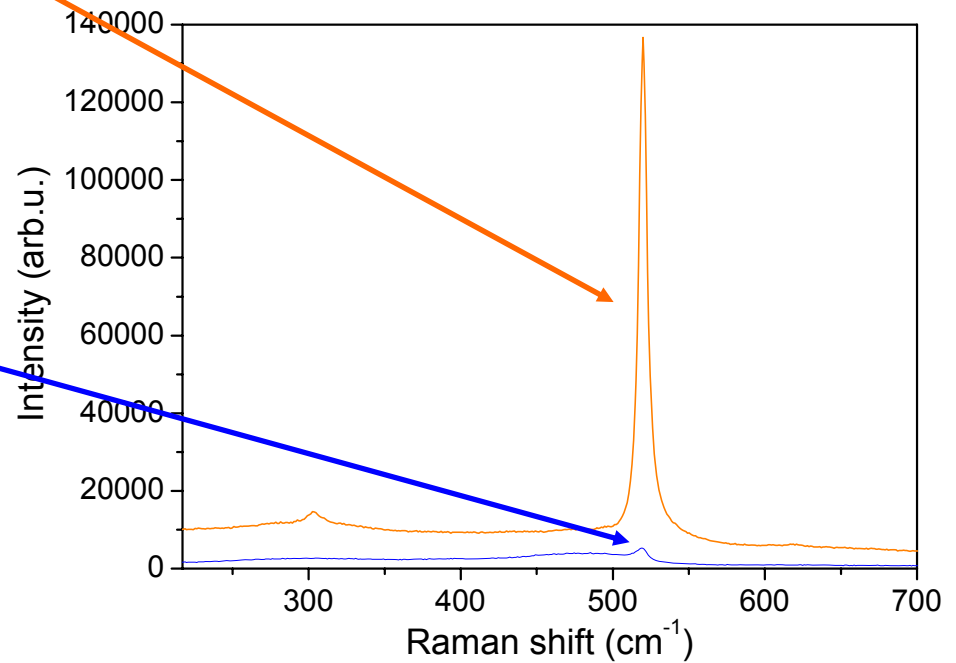
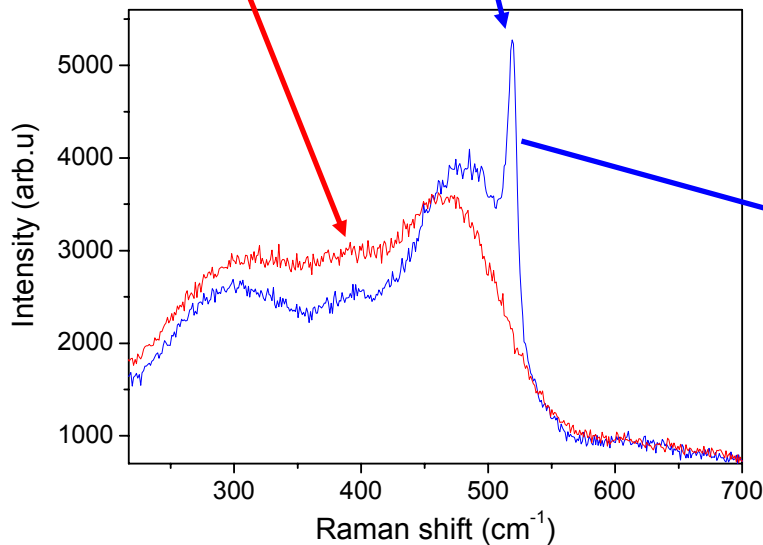
21.7 nm  
10 fs



# FLASH-induced damage to 100-nm a-Si layer deposited on fused silica substrate by Ch. Hecquet

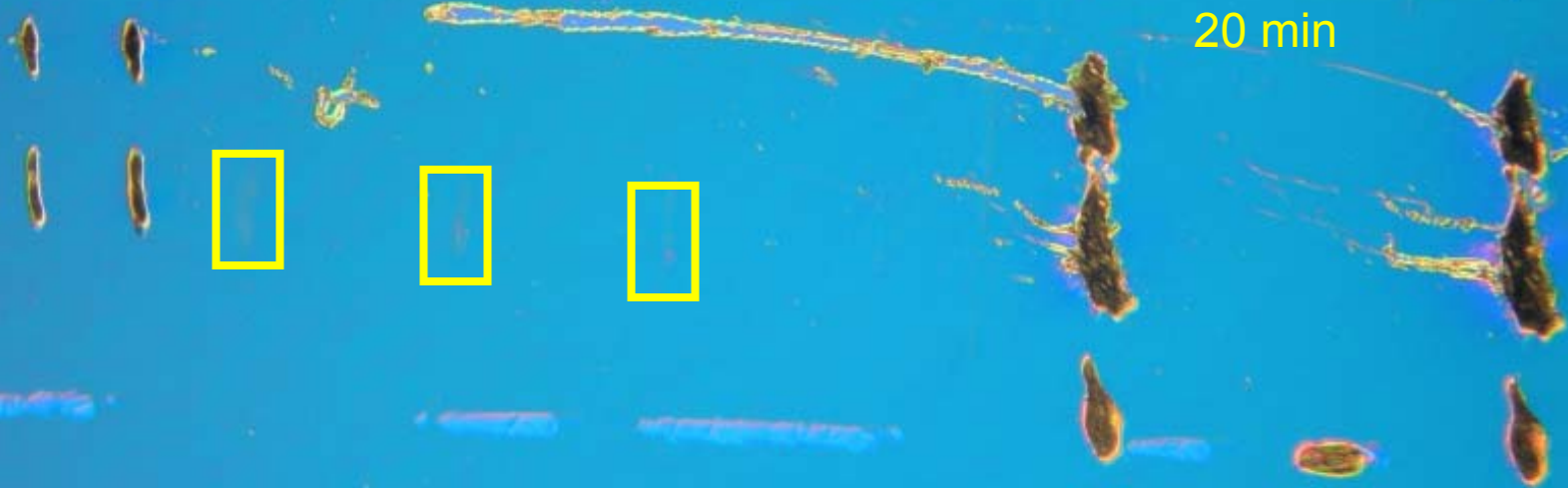


micro-Raman spectra  
785 nm, 2-micron spot



500-nm PMMA/Si; Silson, UK  
SPAM/DRECAM, CEA-Saclay  
March 2 2005

7.5 IR    7.4 IR    7.3 HHG 20 min    7.1 HHG 10 min    6.9 HHG 5 min    6.7 HHG 2 min    6.5 IR    6.3 filtered IR no HHG 20 min    6.1 IR

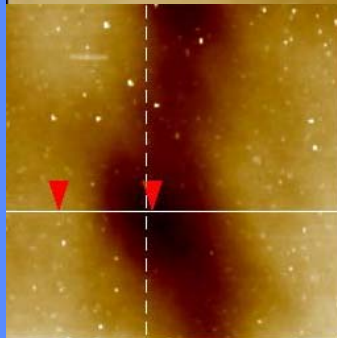


400 μm

# PMMA irradiated by the HH beam

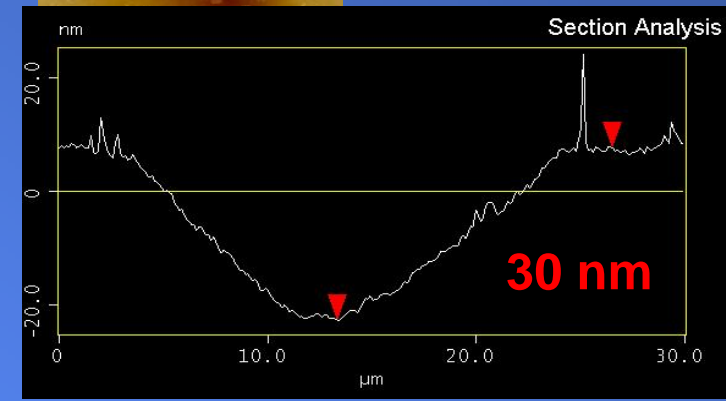
60 minutes

40  $\mu\text{m}$  x 40  $\mu\text{m}$



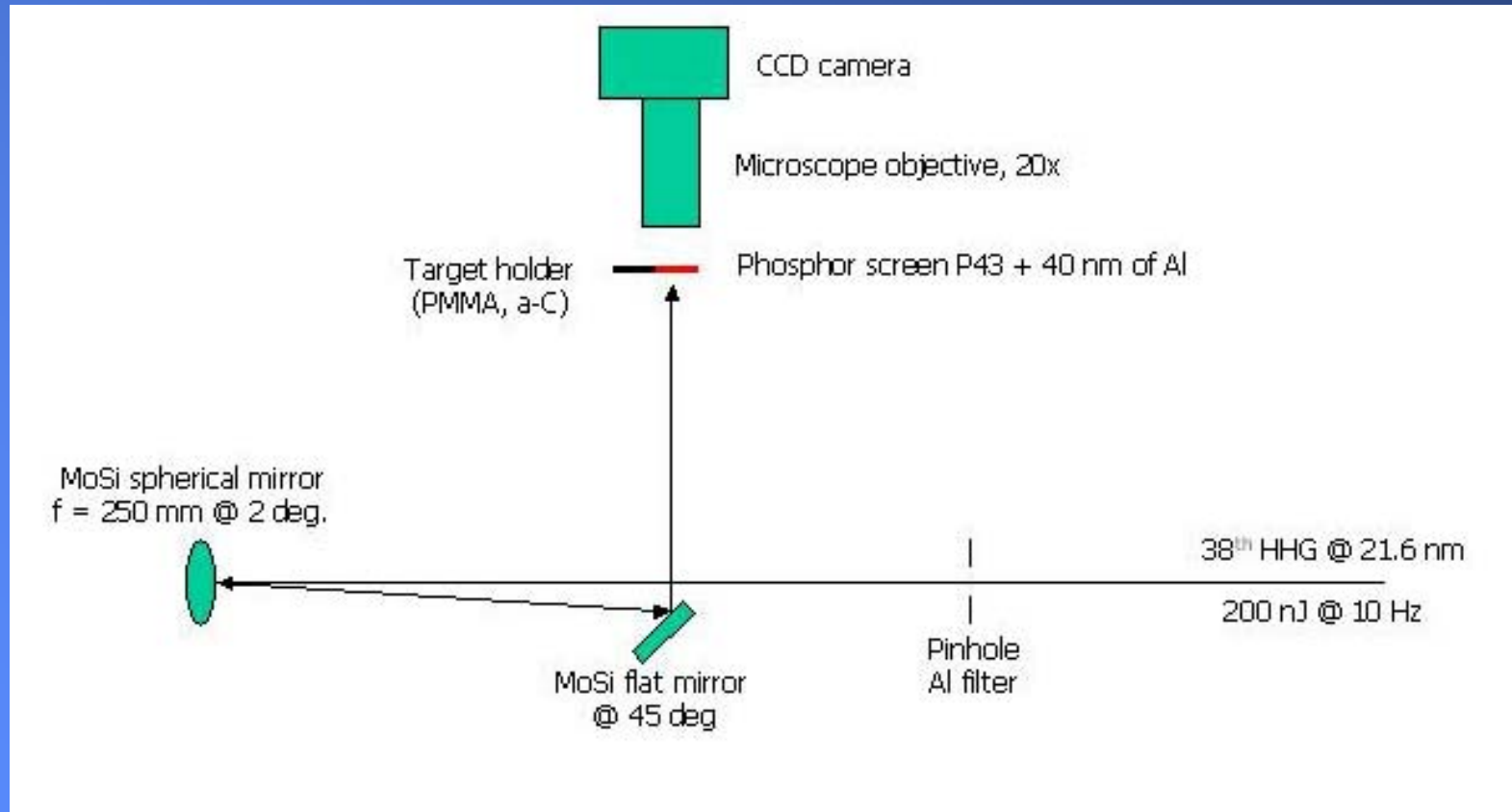
30 minutes

30  $\mu\text{m}$  x 30  $\mu\text{m}$



# Joint experiment on radiation damage by HHG at 21 nm: Spt 2006

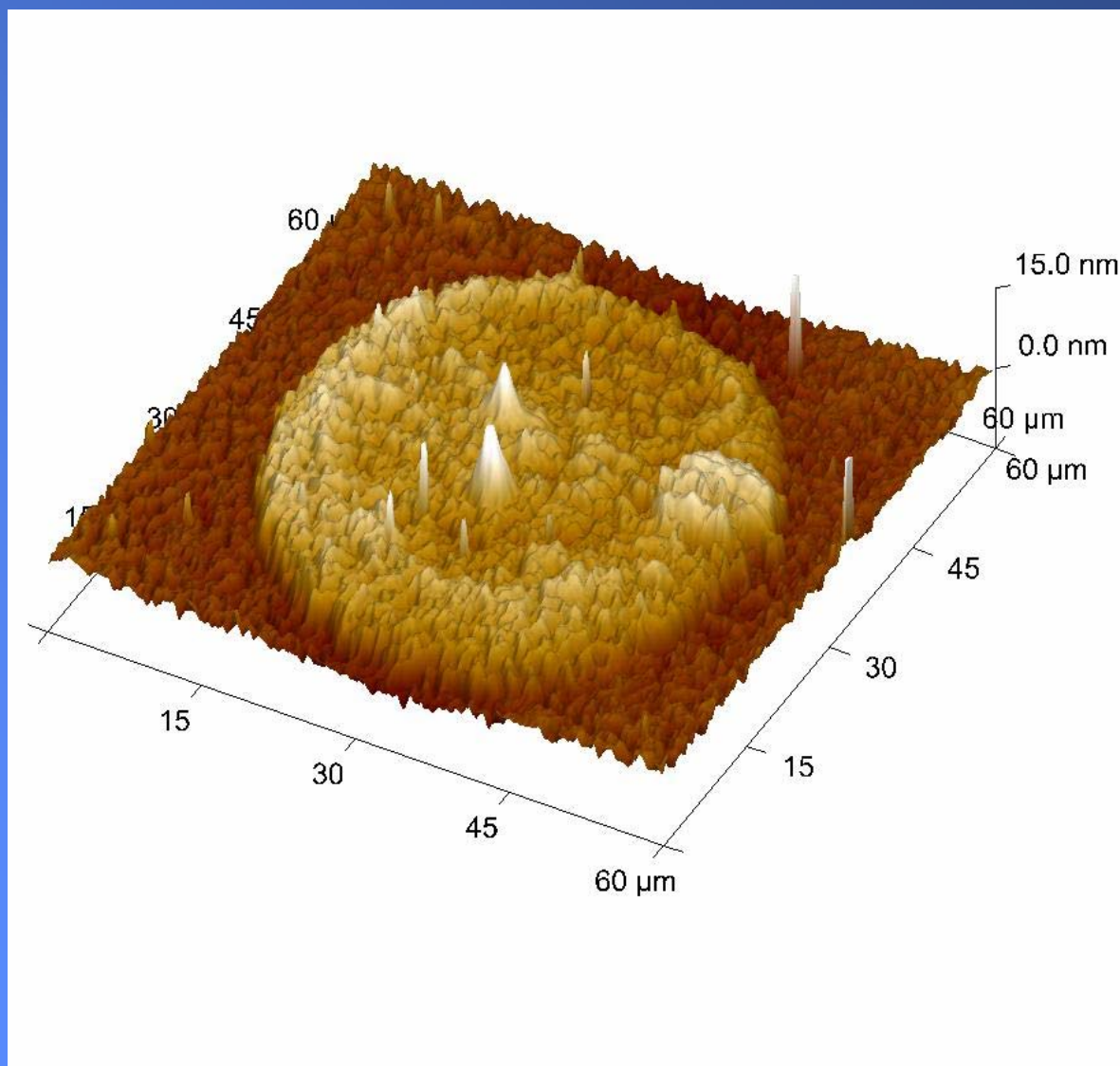
*Collaboration of DXRL (T. Mocek), NSL (L. Juha) and KAIST (C. H. Nam)*



- single-photon damage; around threshold behavior
- to be compared with results from PALS, FLASH and SLIC



# soft X-ray harmonics: PMMA expansion



# conclusion



Raman spectroscopy and AFM analysis showed re-crystallisation in a-Si layers irradiated with focused coherent short-wavelength beams, i.e., Ne-like Zn laser and FLASH.

Single-shot threshold value of  $1.1 \text{ J/cm}^2$  was found plotting the damaged areas determined by means of AFM. Investigating consequences of the multiple-shot exposure it has been found that an accumulation of 10, 20 and 40 shots at a fluence of  $0.5 \text{ J/cm}^2$ , i.e. well below the single-shot damage threshold, causes irreversible changes of a-C thin layer which can be registered by both AFM and DIC microscopy. In the center of the damaged area, AFM shows a-C removal to a maximum depth of 0.3, 1.2 and 1.5 nm for 10-, 20- and 40-shot exposure, respectively.

Ablation threshold of PMMA irradiated by 80-ps pulses of 21.2-nm radiation seems to be at least two orders of magnitude higher than that of PMMA exposed to 32-nm radiation in 25-fs pulses.

Short-wavelength high-order harmonics cause PMMA expansion while XUV ones initiate just materials erosion.

**Prague, 16-20 April 2007**



**SPIE European Congress on Optics and  
Optoelectronics**

**Damage to VUV, EUV&X-ray Optics  
(COO106)**

**at the moment, 27 abstracts submitted**

<http://spie.org/Conferences/Calls/07/eec/conferences/index.cfm?fuseaction=COO106>