

PERIODIC STRUCTURES FORMATION ON BERYLLIUM, CARBON, TUNGSTEN MIXED FILMS BY TW LASER IRRADIATION

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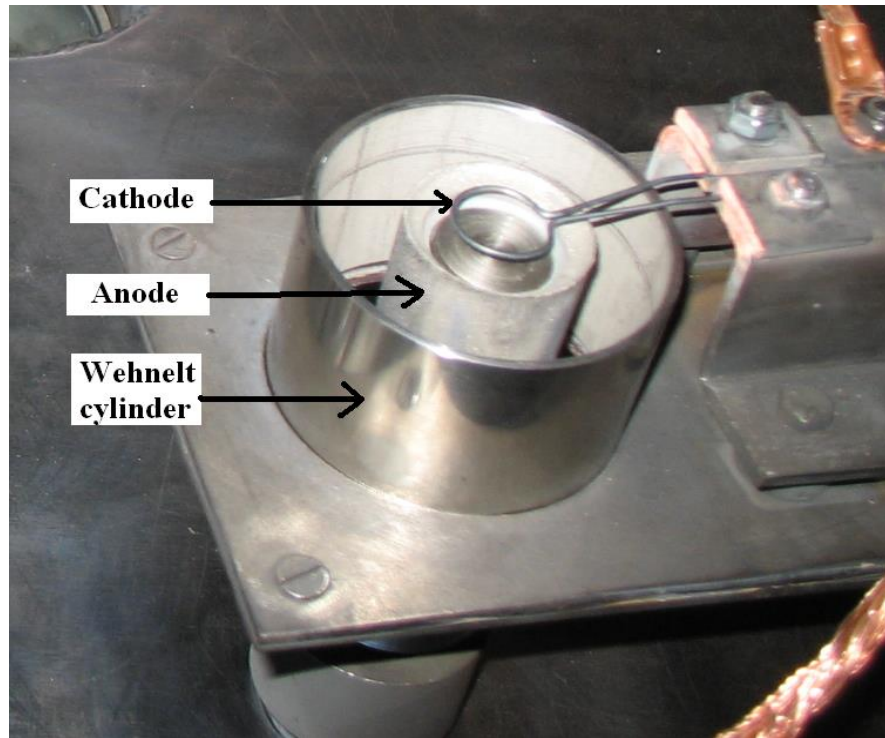
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Outline

- Experiments: Film preparation and laser irradiation
 - Devices and setup
 - Direct laser-matter interaction
 - Indirect laser-matter interaction
- Results and discussions
 - Morphology
 - Structural changes
- Conclusions

Experiments: Sample Preparation - TVA Method-



**TVA evaporator and plasma running in
pure Be vapors**

TVA Systems



Licensed laboratory to work with beryllium and beryllium containing composites



- Vacuum deposition systems:**
- stainless steel chambers,
 - glass, quartz and germanium windows;
 - volumes; 250 l, 1000 l
 - base pressure; $6 \cdot 10^{-7}$ torr
 - mechanical pump (60; 250 m³/h)
 - buster pump (200; 500 m³/h),
 - diffusion pump (3000; 8000 l/s)

Laser Facilities - Magurele

ELI-NP

High power laser system, -
- Power 2 x 10PW

- Gamma beam, high intensity, up to 20MeV, produced by Compton scattering of a laser beam on a 700 MeV electron beam produced by a warm LINAC

CETAL

High power laser system
- Power 1 PW,
- Pulse width 100 fs
- Wavelength 800 nm
- Rep. Rate 0.1 Hz (1PW)

TEWALAS

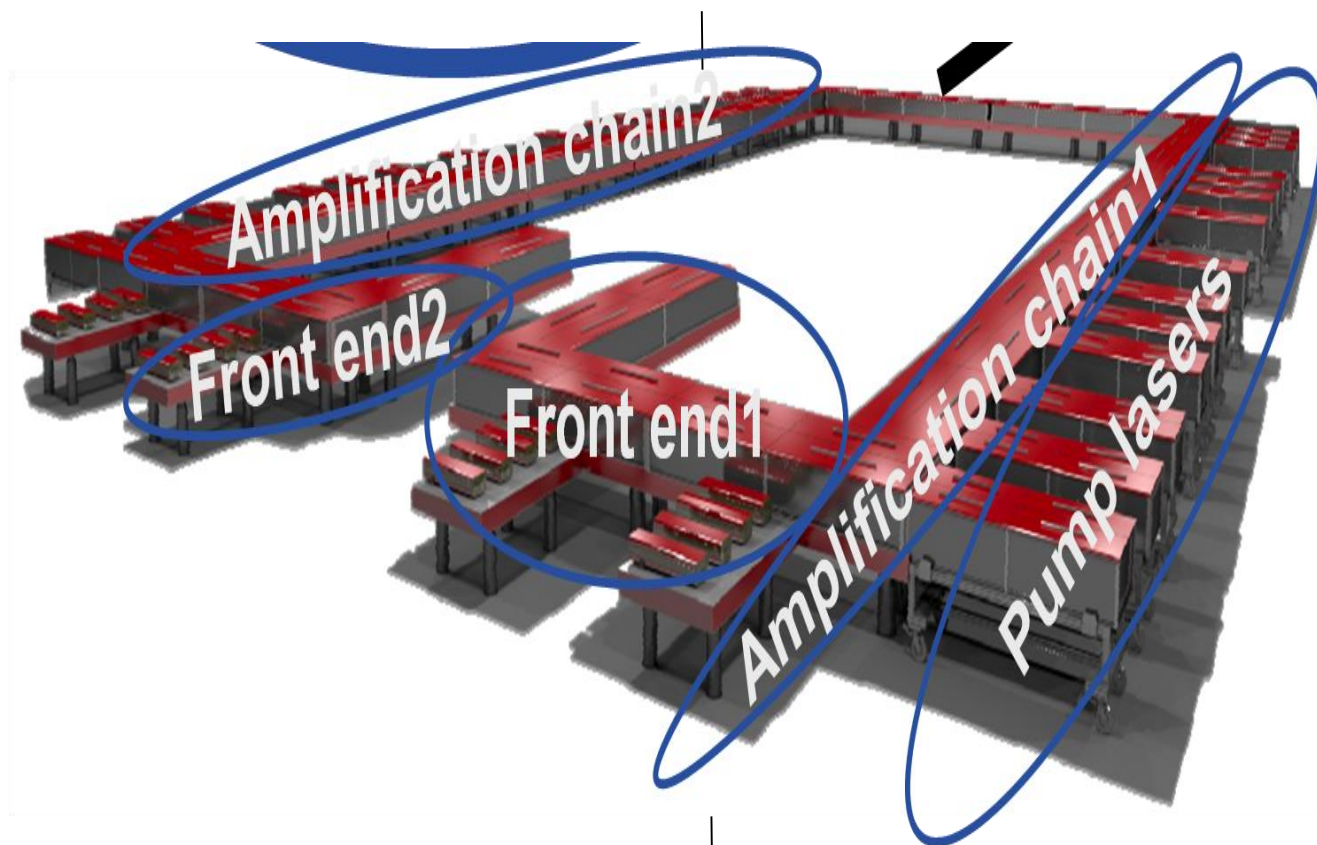
High power laser system
- Power 15 TW
- Pulse width 25 fs
- Wavelength 800 nm
- Rep. Rate 10 Hz

*Provided by THALES -
France*

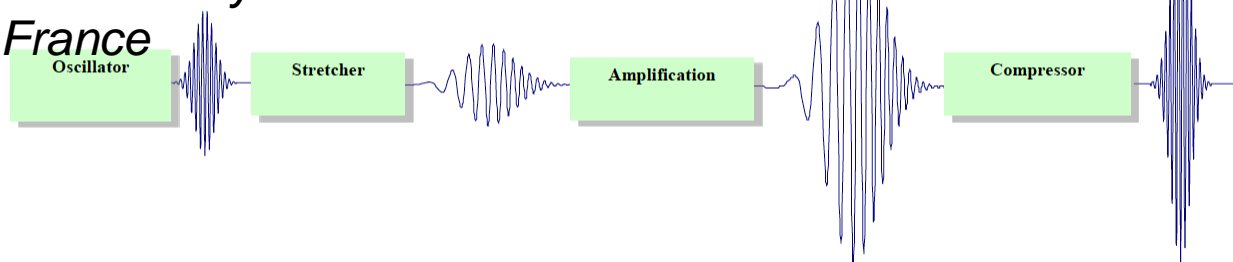
Laser Facilities - Magurele

ELI-NP

- High power laser system, 2 x 10PW maximum power
- Gamma beam, high intensity, up to 20MeV, produced by Compton scattering of a laser beam on a 700 MeV electron beam produced by a warm LINAC



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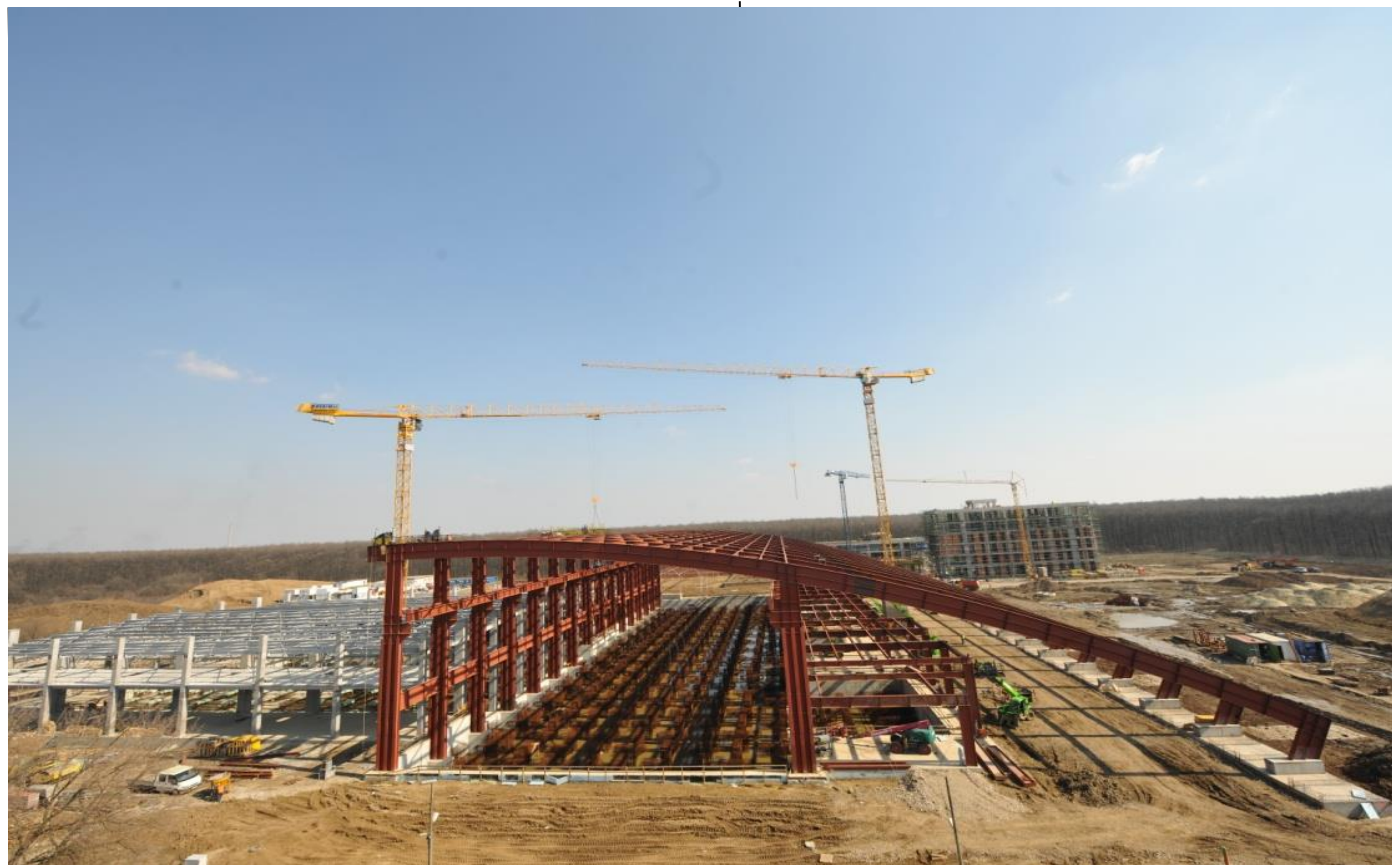


2 x 0.1 PW
2 x 1 PW
2 x 10 PW

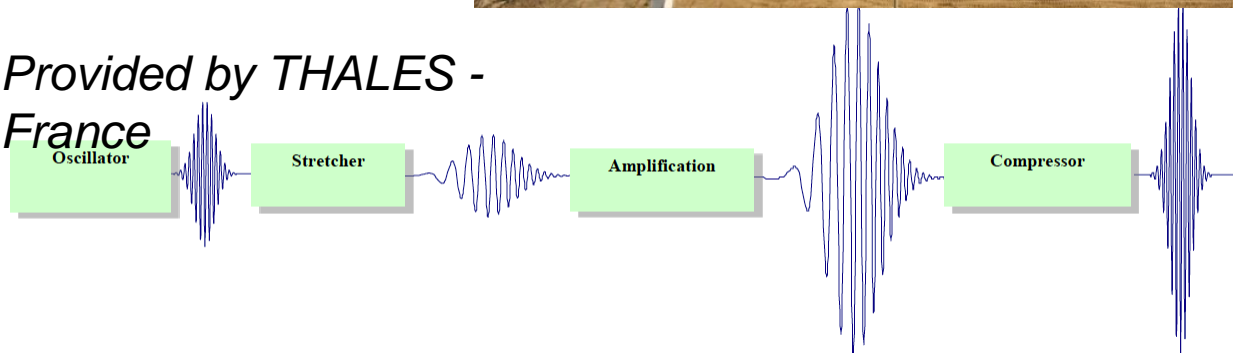
Laser Facilities - Magurele

ELI-NP

- High power laser system, $2 \times 10\text{PW}$ maximum power
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France



$2 \times 0.1\text{ PW}$
 $2 \times 1\text{ PW}$
 $2 \times 10\text{ PW}$

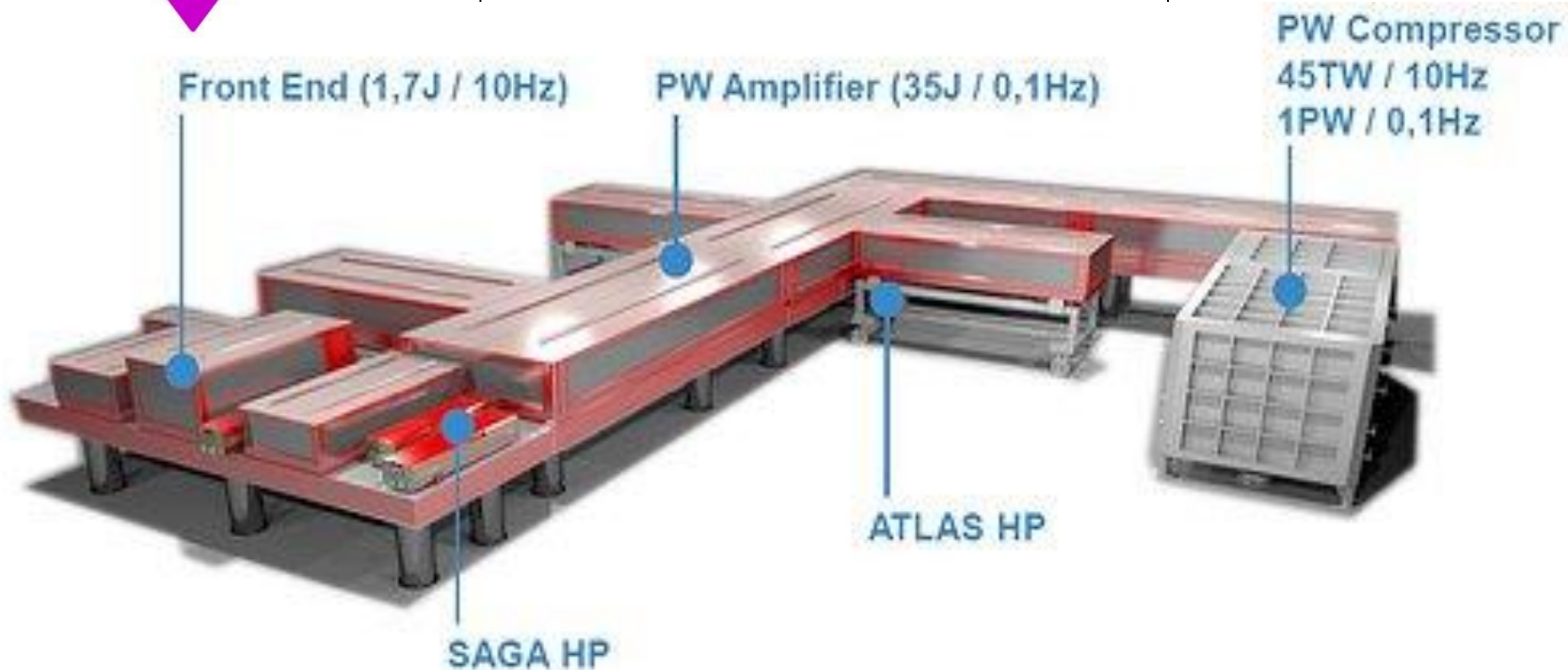
Laser Facilities - Magurele



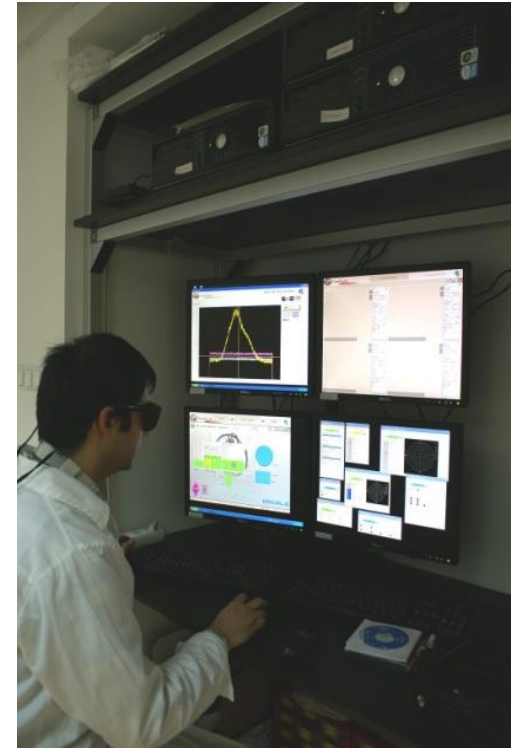
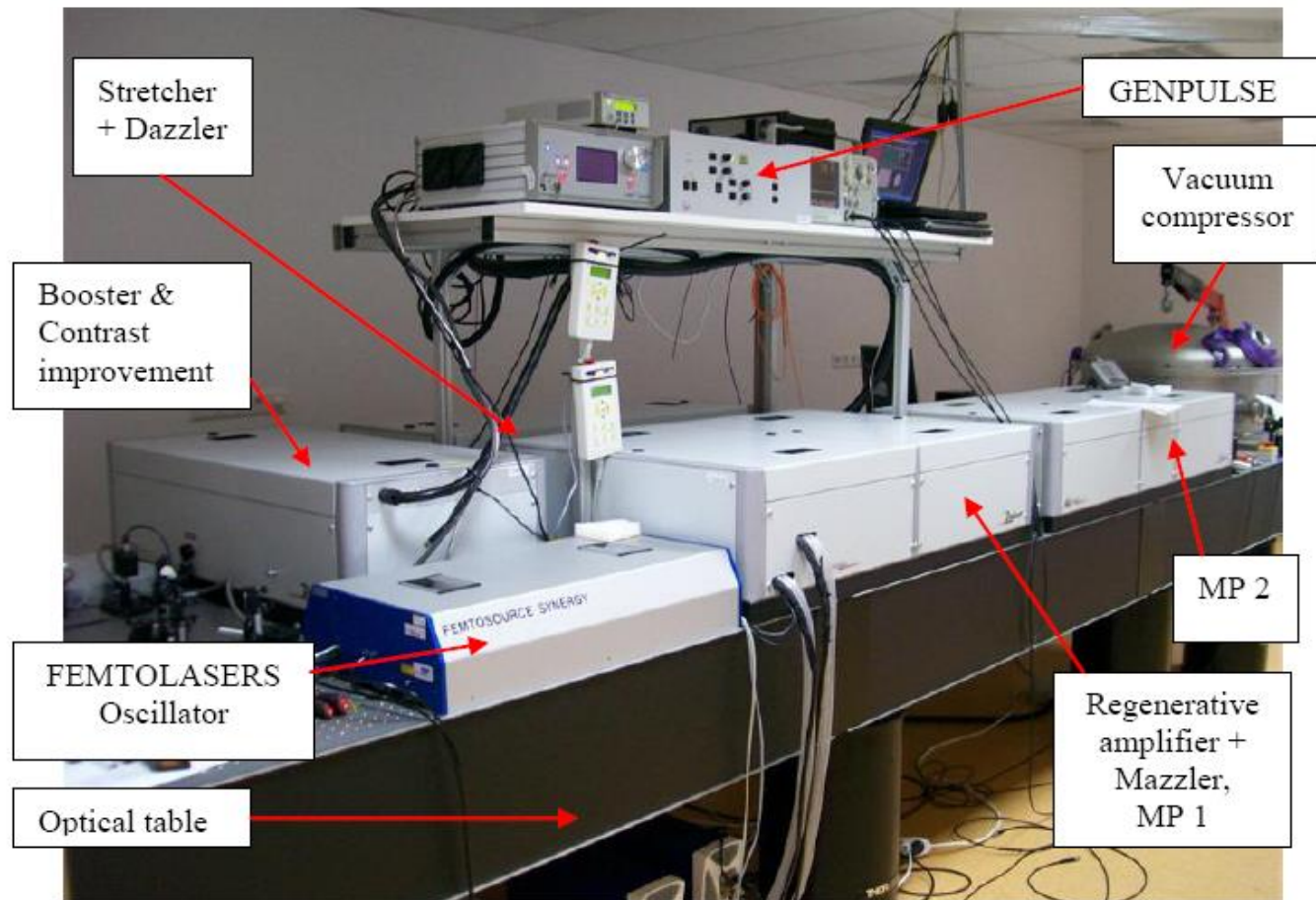
CETAL

High power laser system

- Power 1 PW,
- Pulse width 100 fs
- Wavelength 800 nm
- Rep. Rate 0.1 Hz (1PW)

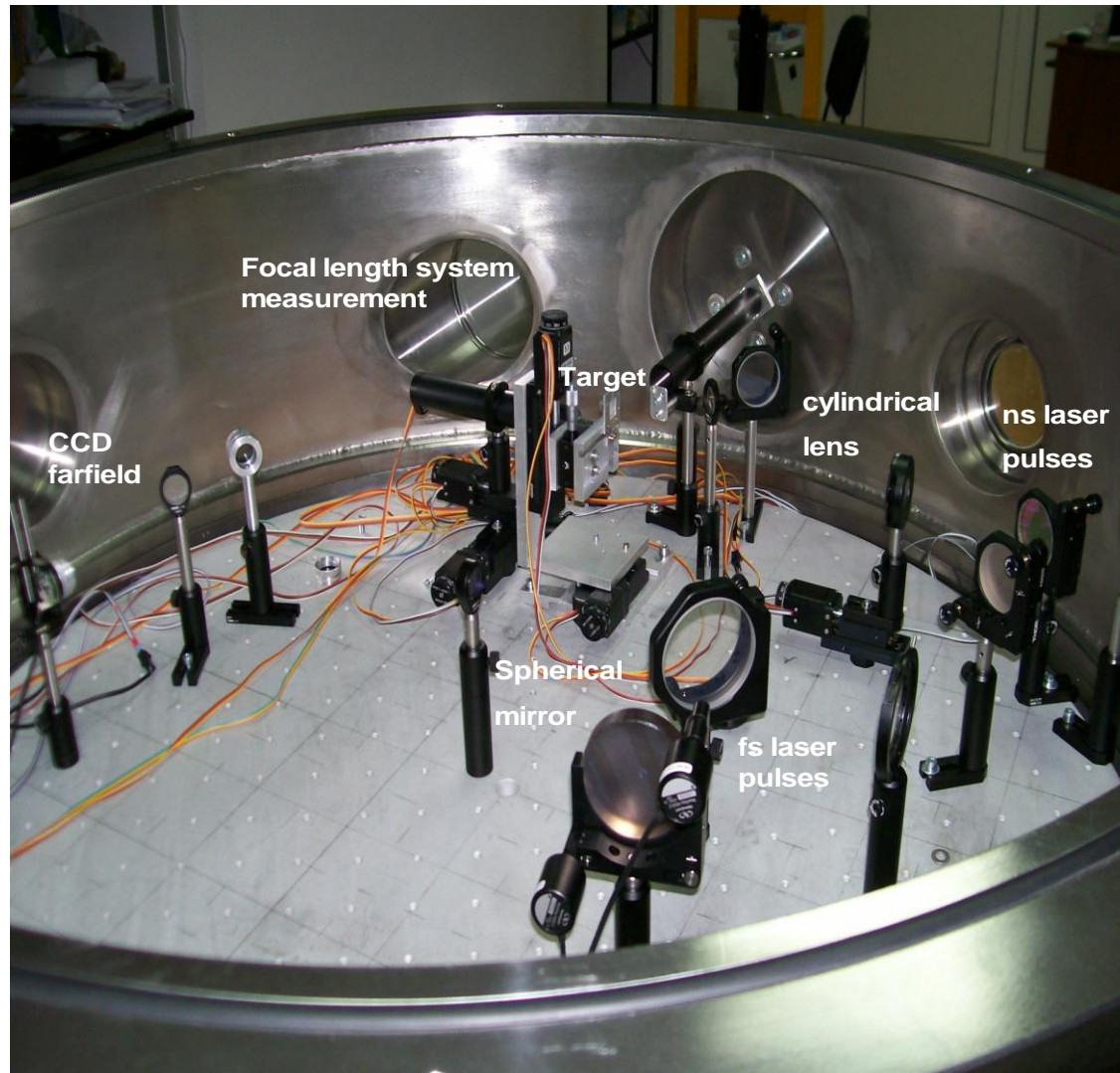


TEWALS -Laser facility -



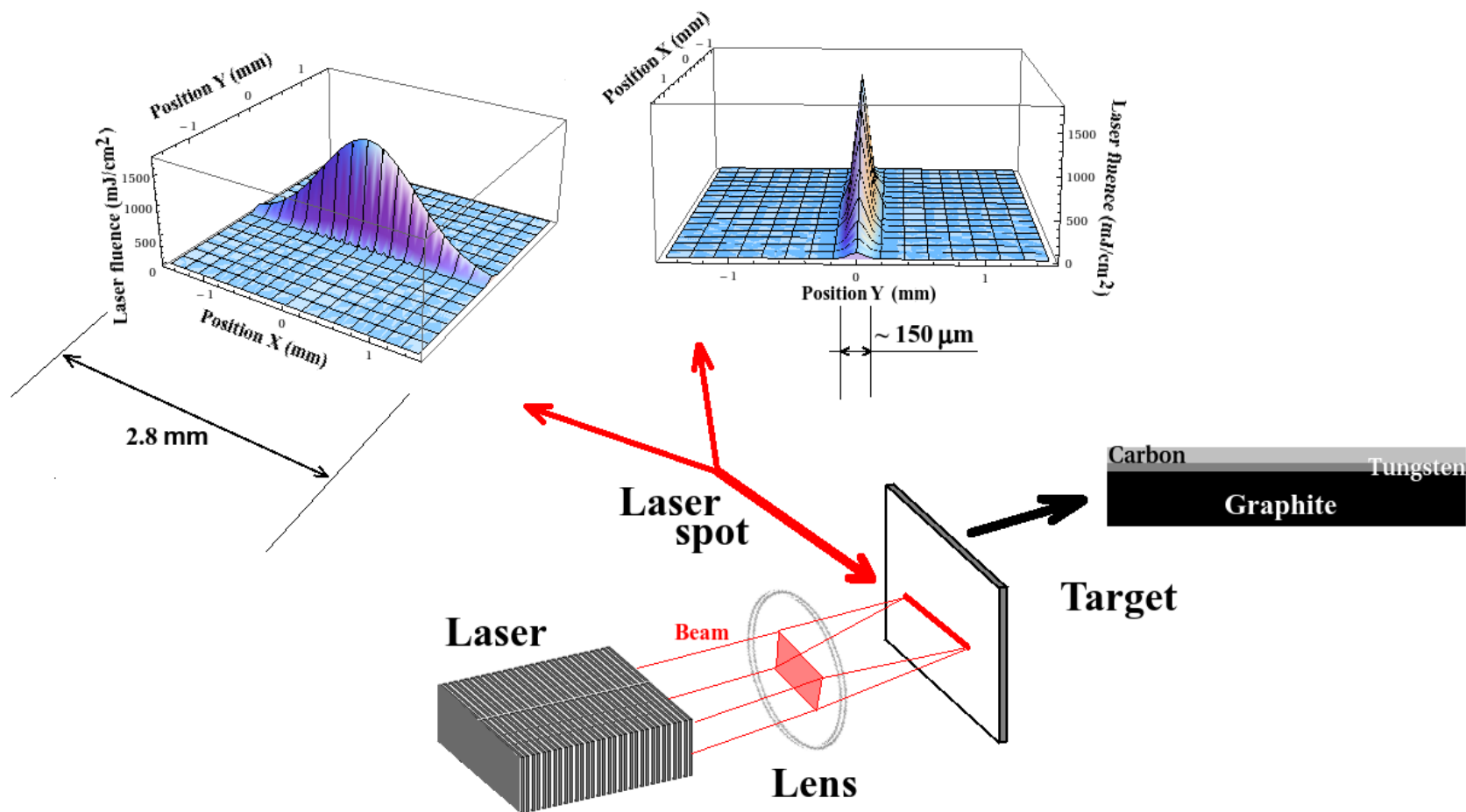
Interaction chamber

Interaction Chamber



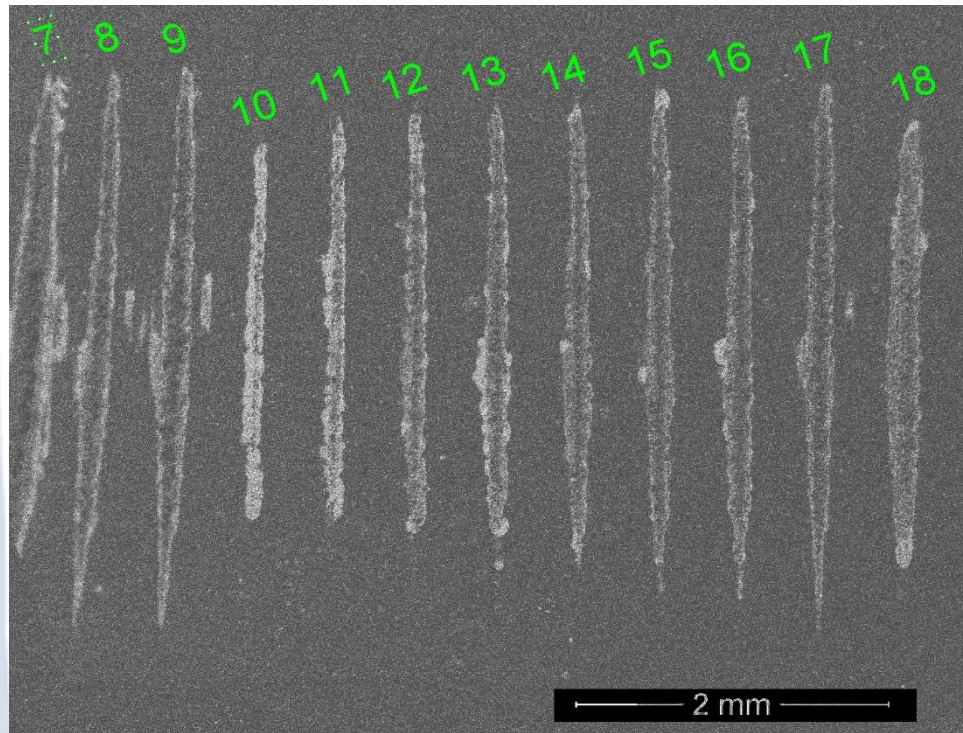
Experimental Setup

- Direct laser irradiation -

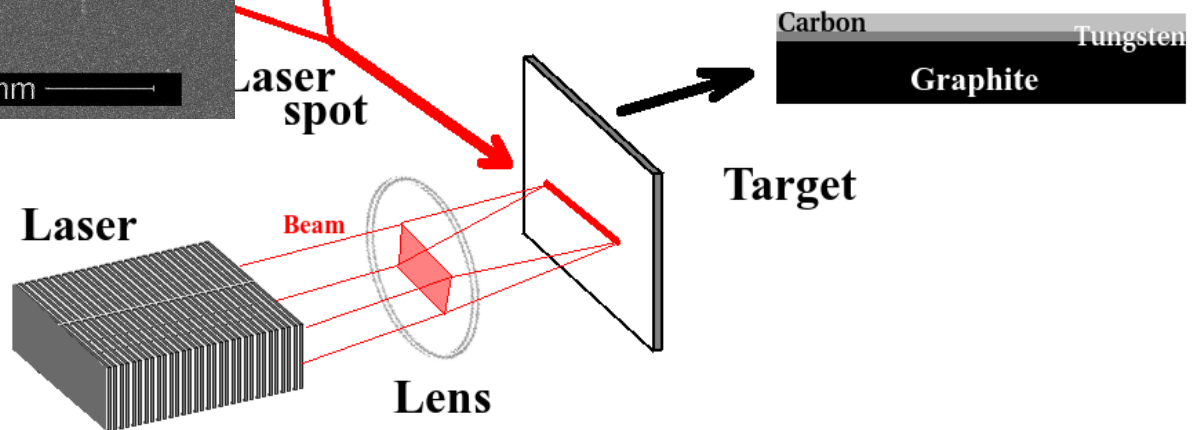
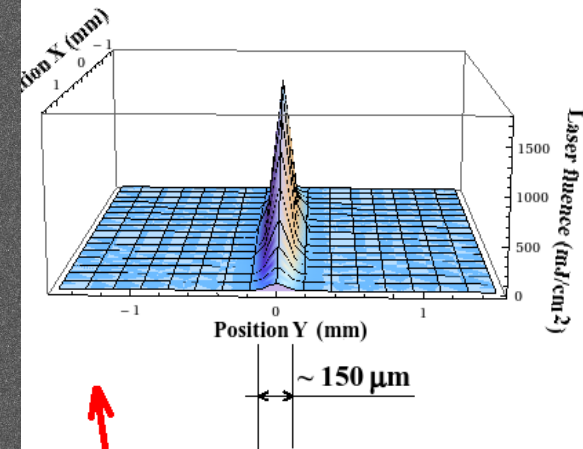


Experimental Setup

- Direct laser irradiation -

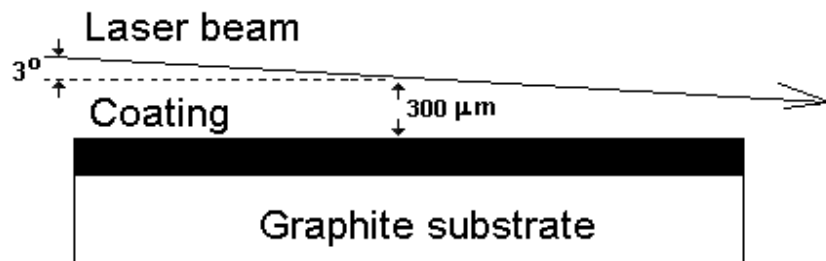


SEM image of the Sample traces



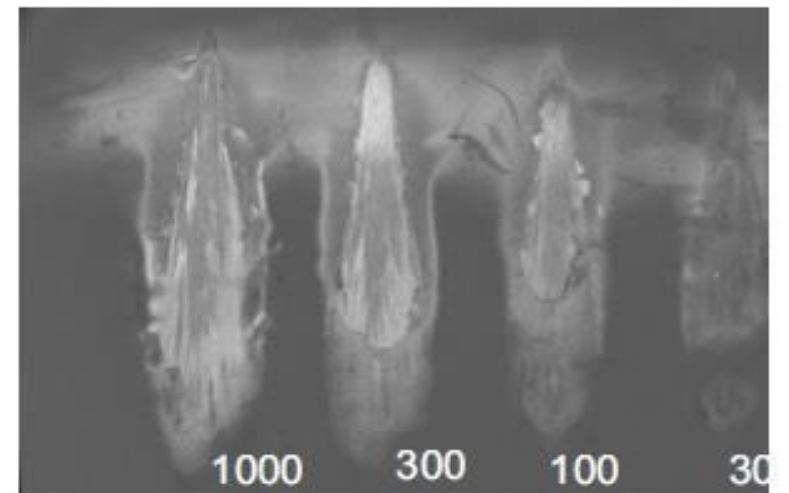
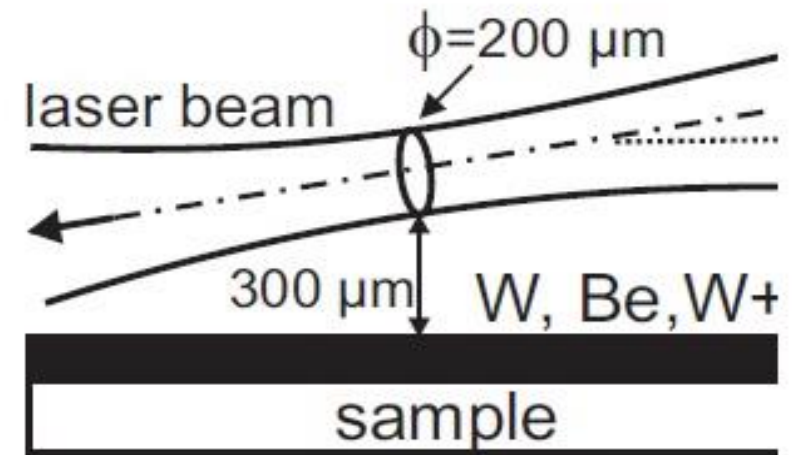
Experimental Setup

- Indirect laser irradiation -



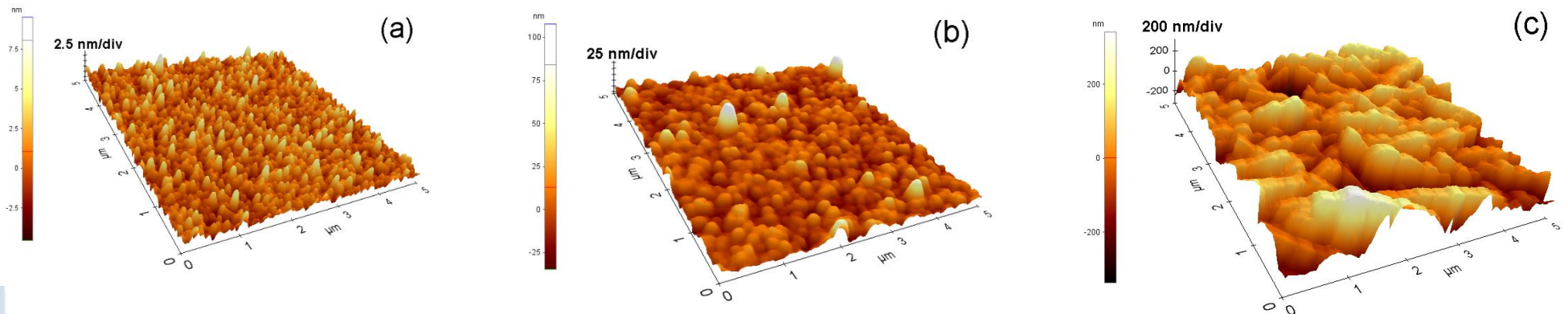
Experimental Setup scheme

SEM image of the
Sample traces



Results: Sample Roughness - before irradiation-

The AFM analyses highlighted the formation of very smooth films of carbon (Ra: 1.68 nm), smooth films of W (Ra: 9.28 nm) and rough films of Be (Ra: 72.26 nm). Due to the specific growth of Be films, the 2D growth planes are obvious.

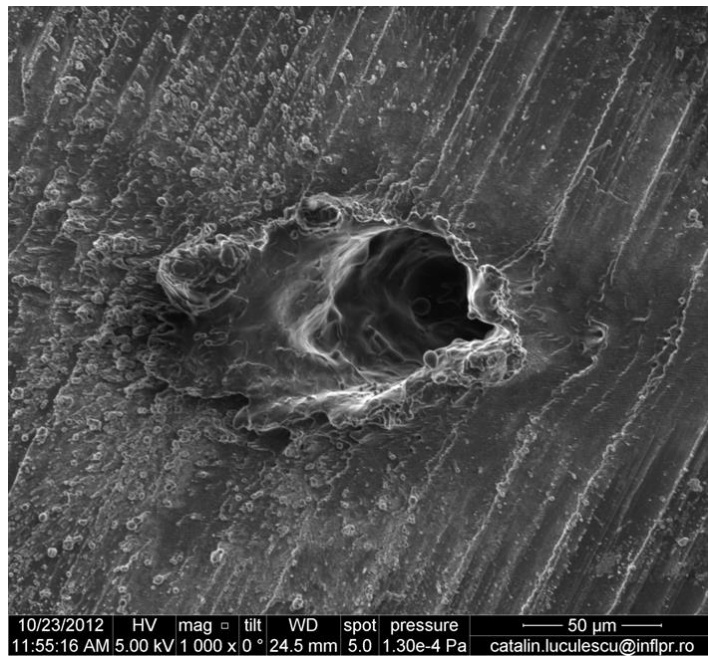


**3D AFM images: C film, Ra: 1.7 nm (a), W film, Ra: 9.3 nm (b)
and Be film, Ra: 72.3 nm (c)**

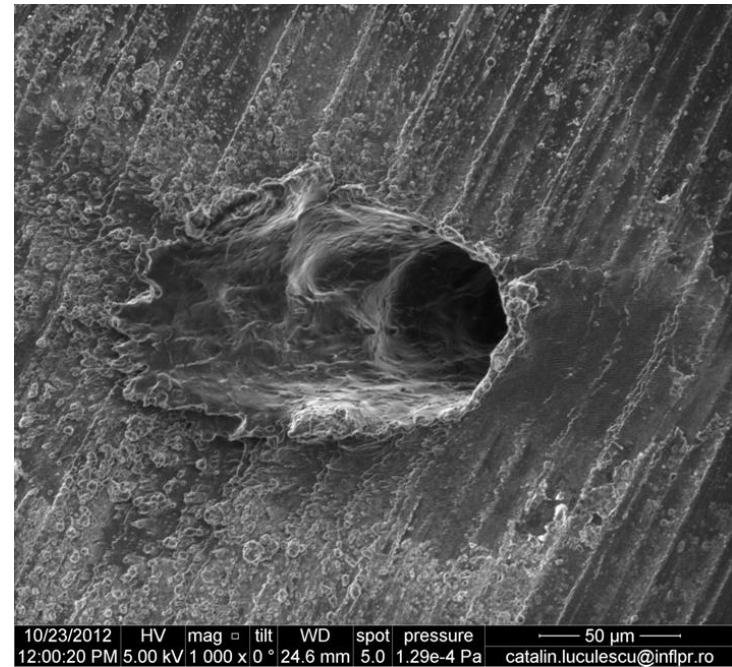
Direct Laser Ablation of Be

- Morphology -

10 Pulses



100 Pulses

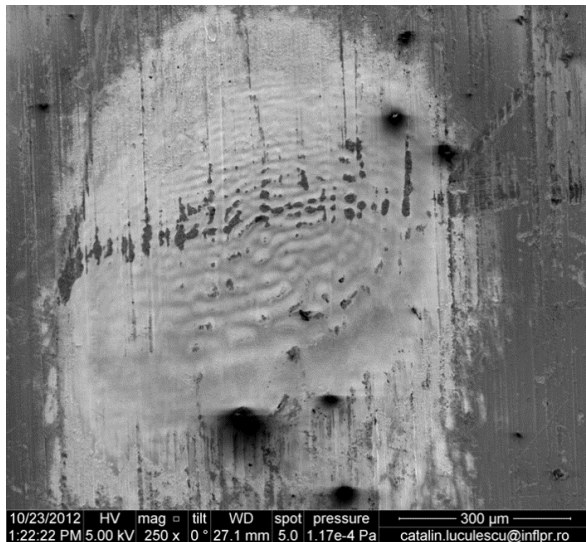


SEM Image of the Be irradiated area after 10 Pulses and 100 Pulses

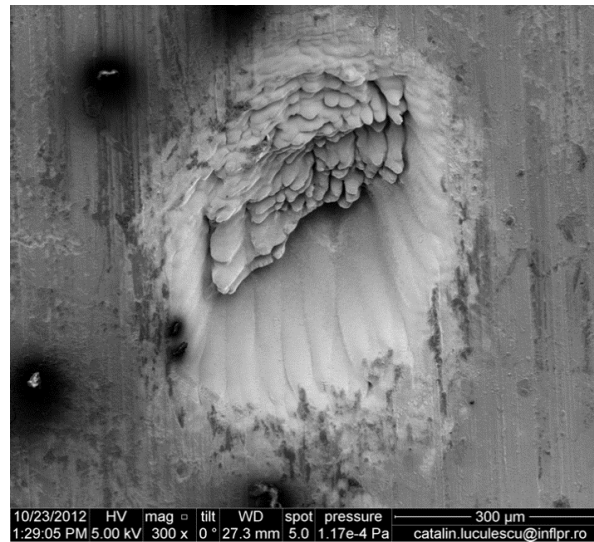
Direct Laser Ablation of W

- Morphology -

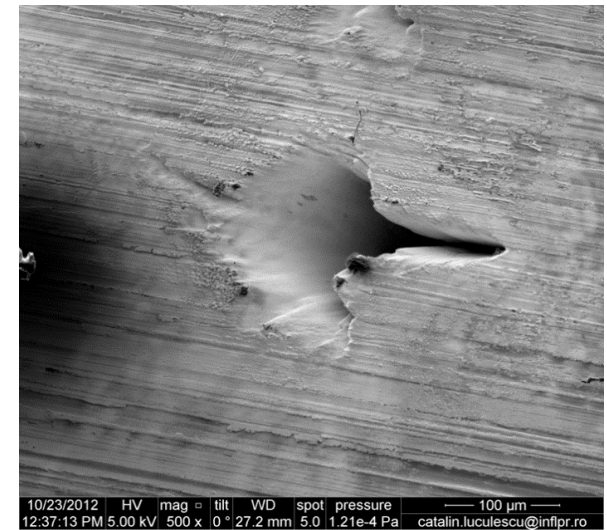
1 Pulse



10 Pulses

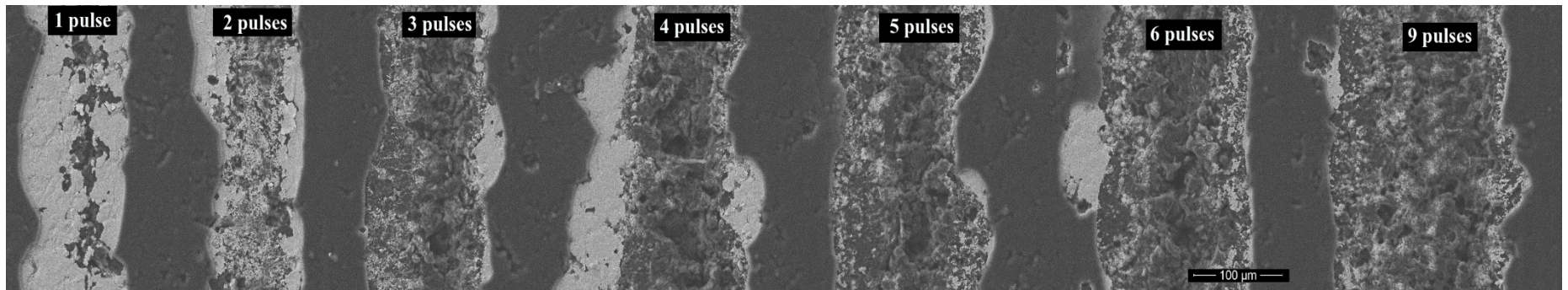


100 Pulses

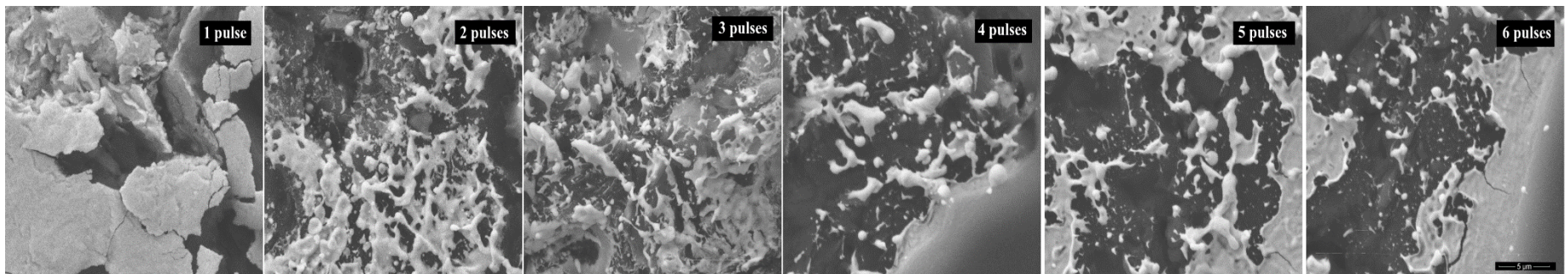


SEM Image of the W irradiated area after 1 Pulse, 10 Pulses and 100 Pulses

Direct Laser Ablation of Carbon(/Tungsten) - Morphology -



SEM Image of the target surface in the center of the irradiated area

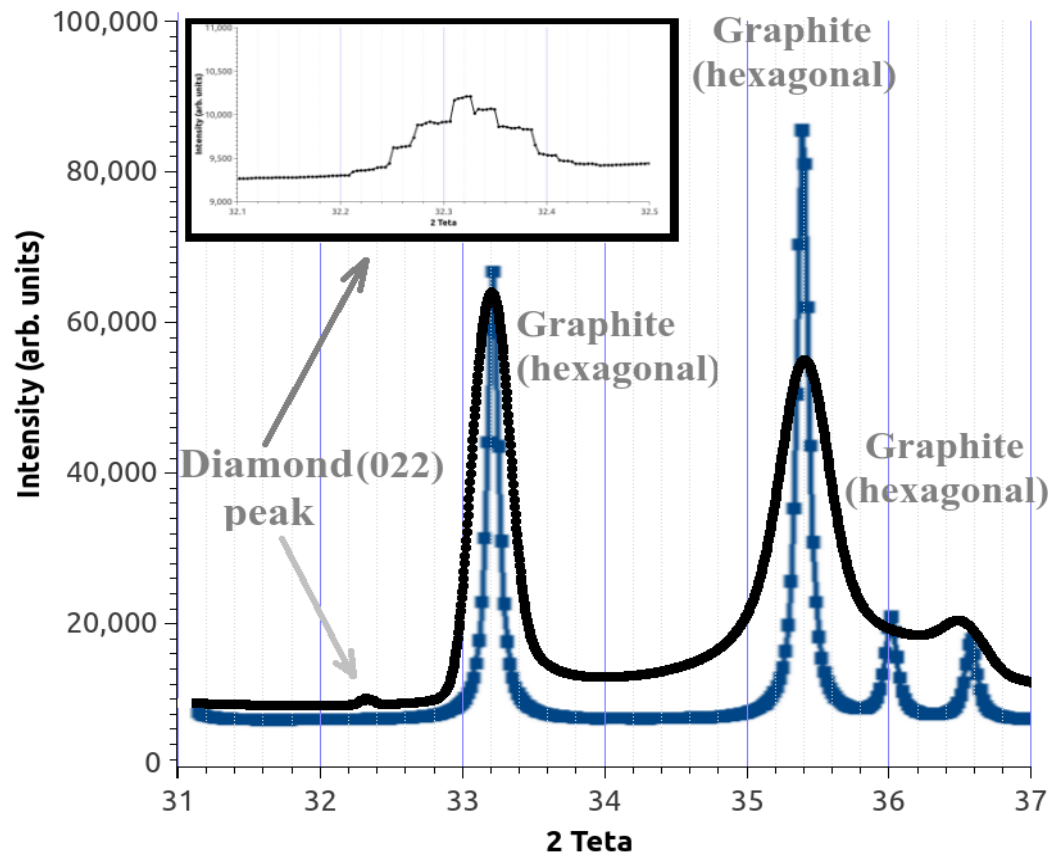


SEM Details of central irradiated area surface (SEM)

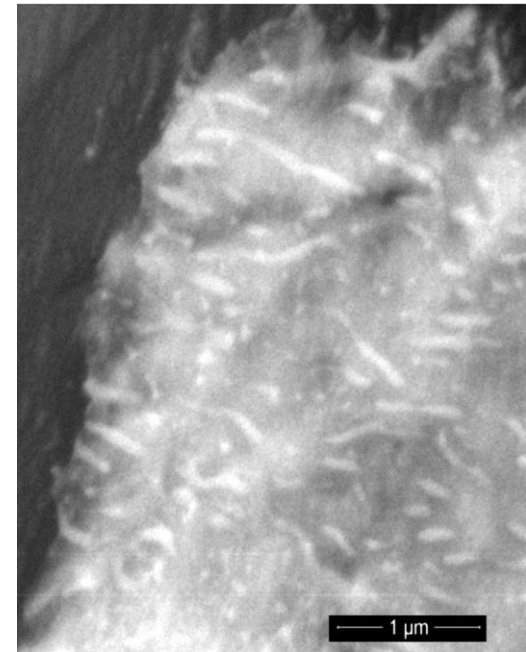


Direct Laser Ablation of Carbon

- Diamond formation -



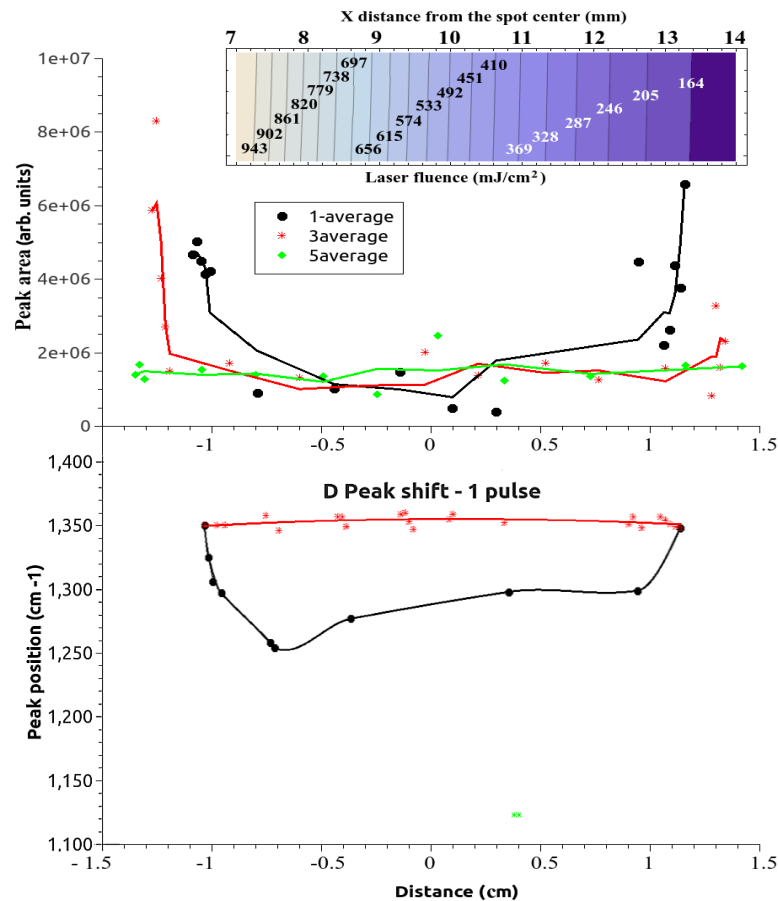
Accidental diamond signals on Synchrotron XRD



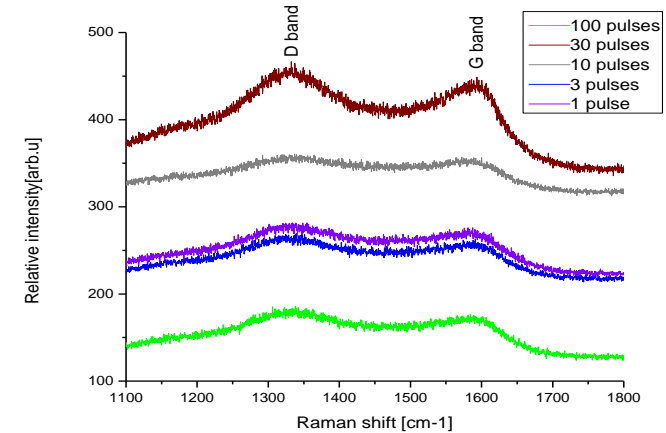
There are morphological signs of possible material nucleation and recrystallization.

Direct Laser Ablation of Carbon/Tungsten

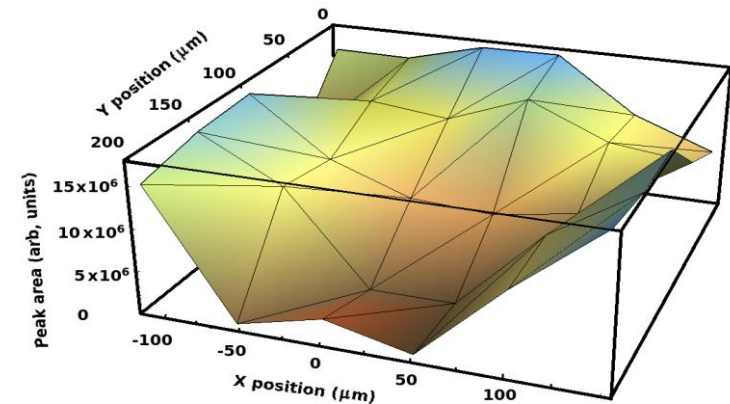
- Raman -



Variation of the 'D' peak integrate intensity
Longitudinal variations on the 1, 3 and 5 pulses
irradiated zones



Raman analysis on the zones irradiated directly
with 1, 3, 10, 30 and 100 pulses

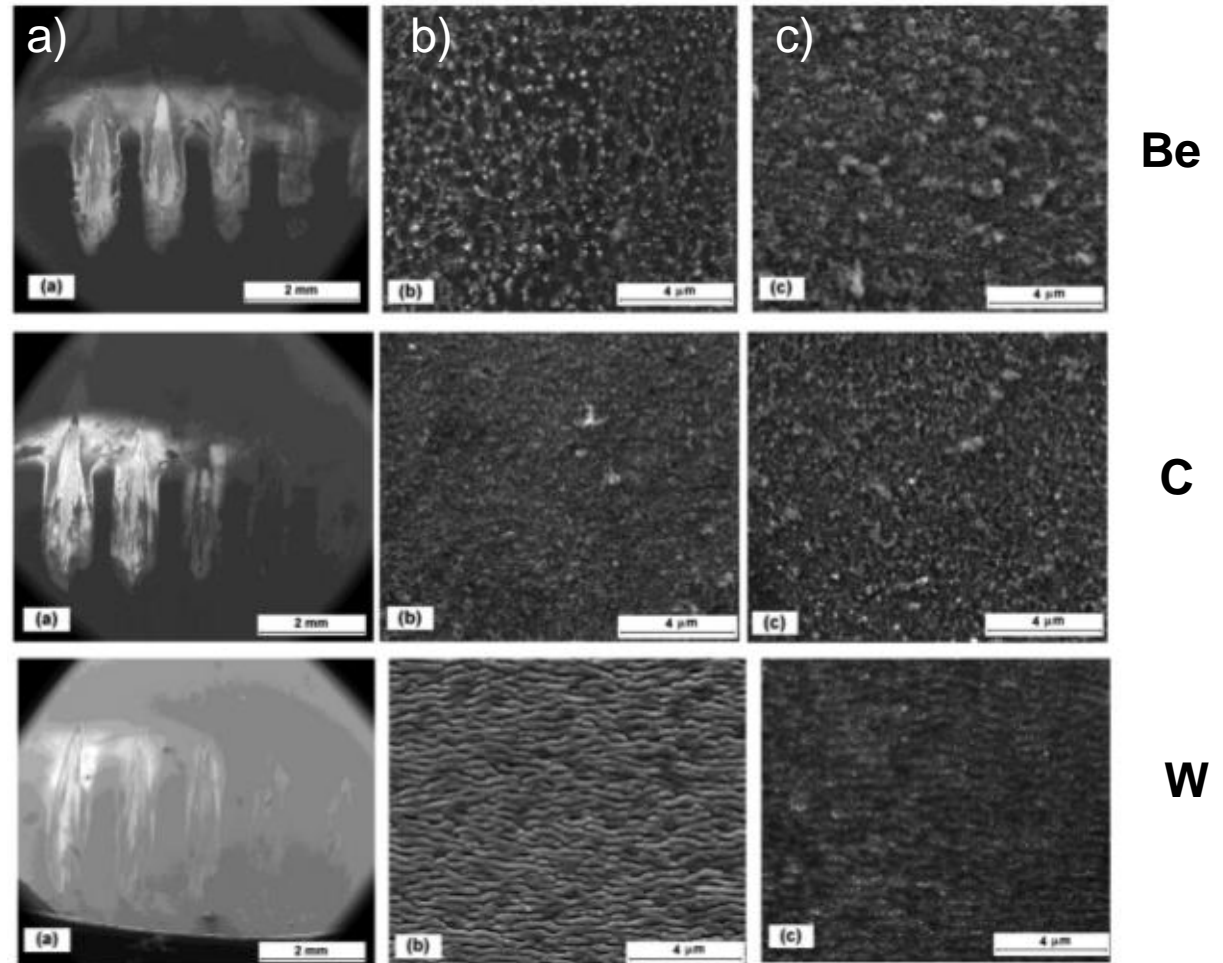


2 D mapping on the edge of 3 pulses irradiated
zone.

Indirect Laser Irradiation – Air - - morphology -

The pulse duration was 70 fs at energy of 6mJ and 10 Hz repetition rate.

The laser beam was focused by a 300 mm parabolic mirror. The diameter of the focus point was about 200 μm leading to a laser fluence of 19.1 J/cm².



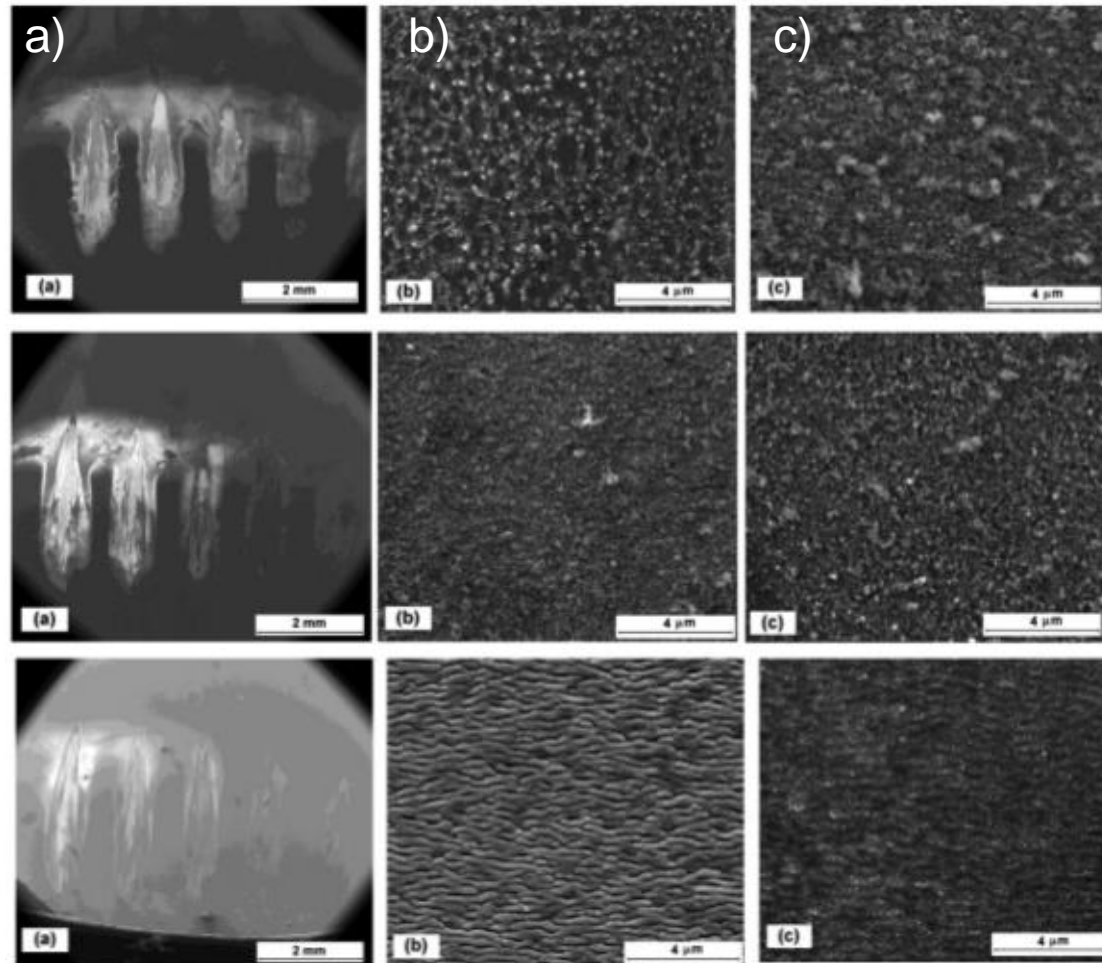
SEM images of: films irradiated with: 1000; 300; 100; 30 and 1 pulses (a), film irradiated with 1000 pulses (b), film irradiated with 300 pulses (c).

Indirect Laser Irradiation – Air - - morphology -

The pulse duration was 70 fs at energy of 6mJ and 10 Hz repetition rate.

The laser beam was focused by a 300 mm parabolic mirror. The diameter of the focus point was about 200 μm leading to a laser fluence of 19.1 J/cm².

Clear evidence of striation formation by an indirect irradiation produced by a femtosecond type laser

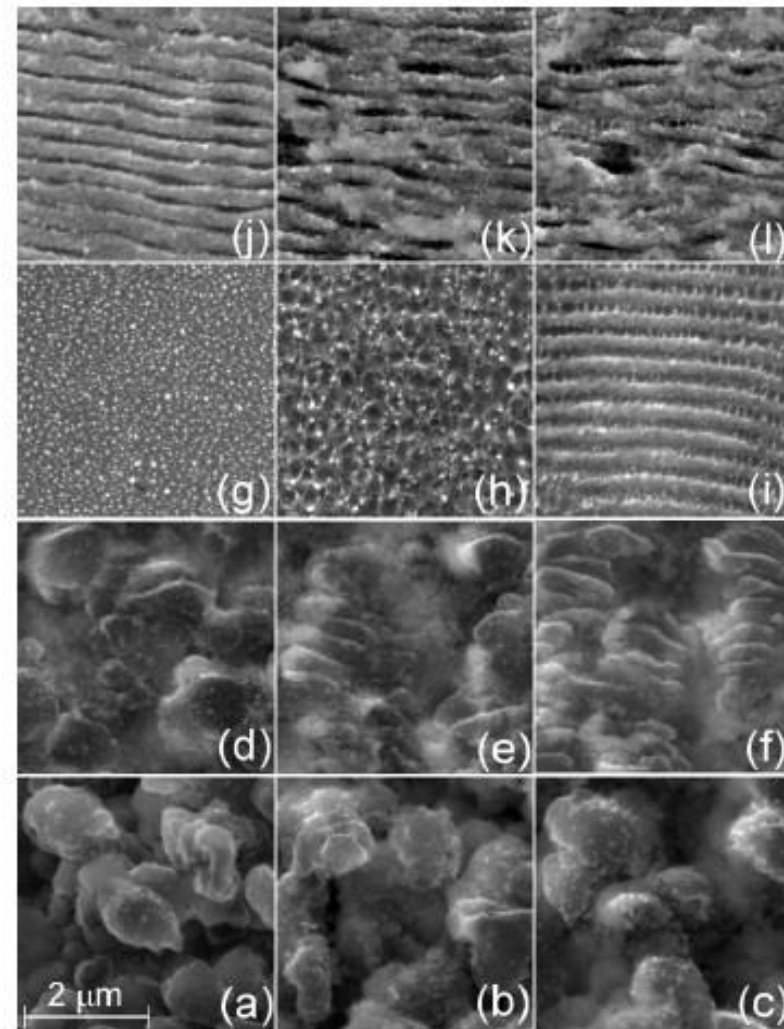


SEM images of: films irradiated with: 1000; 300; 100; 30 and 1 pulses (a), film irradiated with 1000 pulses (b), film irradiated with 300 pulses (c).

Indirect Laser Irradiation – Be-W

The mechanism for surface striations can be explained as the result of interference between the incident laser beam and the surface plasmons although neither Be and nor W have the real part of the dielectric permittivity negative

Other factors such as surface roughness and transfer of heat from the plasma to the surface during the ripple formation have to be accounted for.



Exposed samples made of Be-W mixture in air in (a) to (f) and in deuterium in (g) to (l), after 1, 10, 30, 100, 300 and 1000 shots, respectively

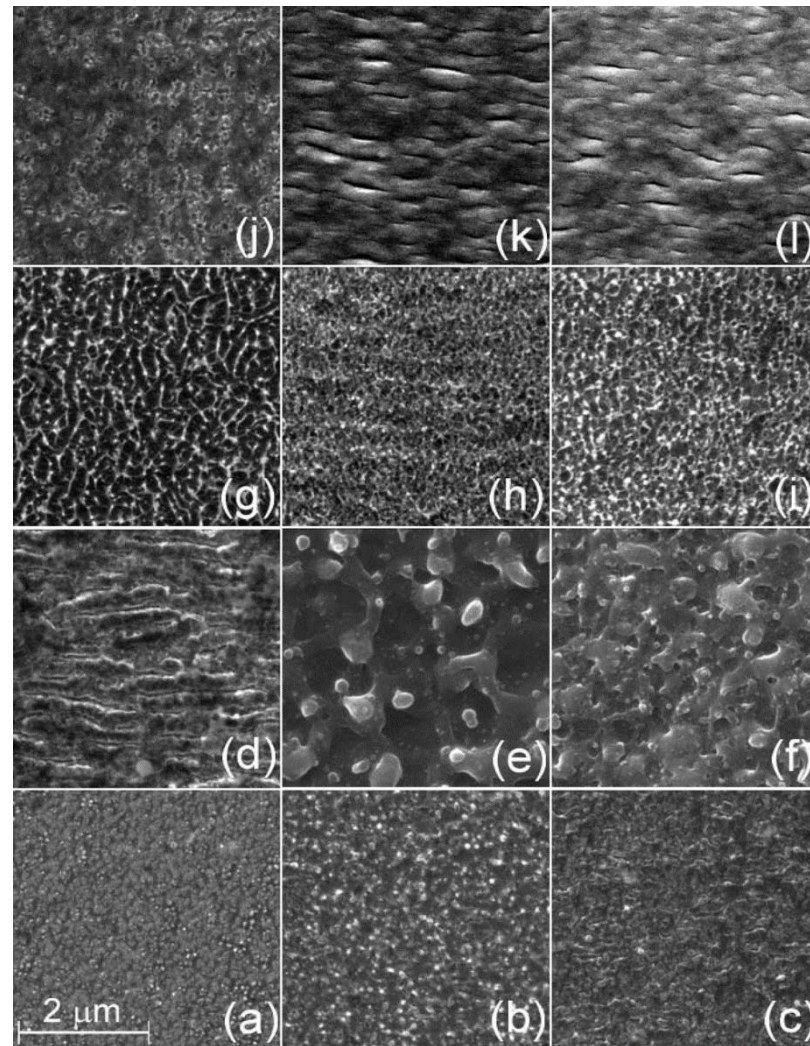
Indirect Laser Irradiation – D - Be and W -

The morphology of the analyzed striations is similar to that observed in experiments which used a laser beam incident on a W target [1-3]

1 A. Y. Vorobyev, C. Guo, J. Appl. Phys. 104, 063523 (2008).

3 Q.-Z. Zhao, S. Malzer, L. J. Wang, Opt. Lett. 32, 1932 (2007).

4 L. Xue, J. Yang, Y. Yang, Y. Wang, X. Zhu, Appl Phys A 109,357 (2012).



Exposed Be samples in (a) to (f) and W samples in (g) to (l) in D at 20 Torr after 1, 10, 30, 100, 300, and 1000 shots, respectively

Indirect Laser Irradiation – D

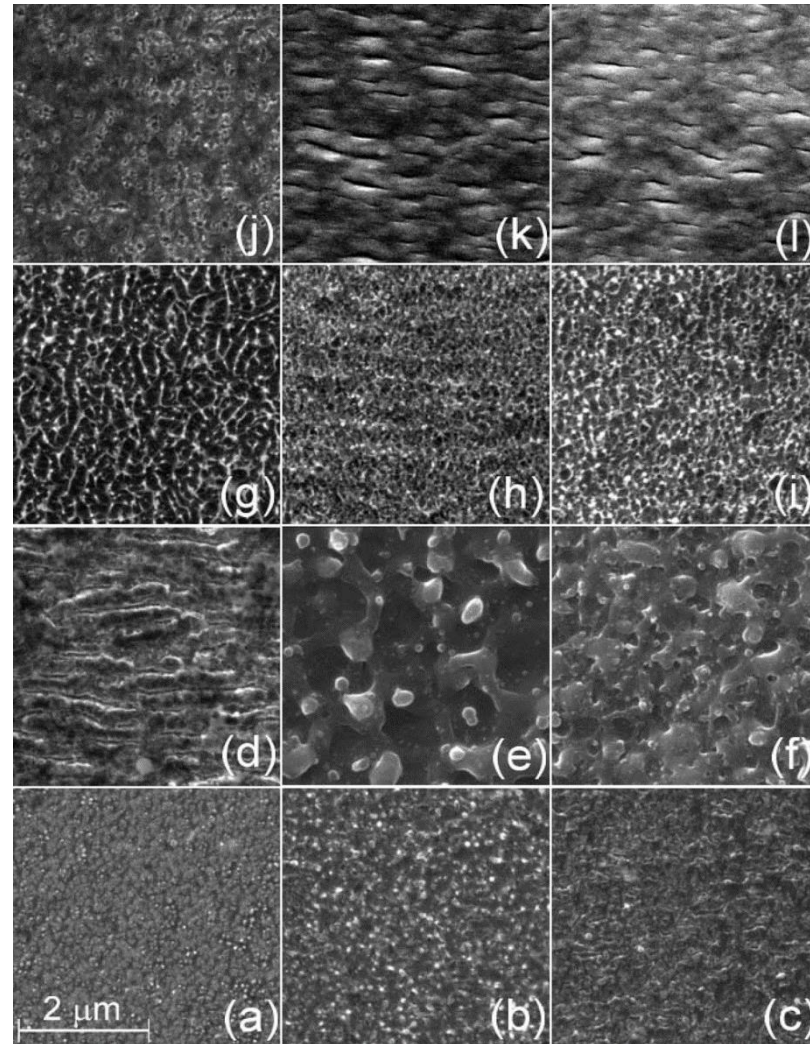
- Be and W -

Vorobyev and Guo [1] reported a period $\Lambda = 289$ nm in air at $\lambda = 400$ nm and a fluence of 0.35 J/cm², while at $\lambda = 800$ nm the period depended on the number of laser shots: $\Lambda = 560$ after 40 shots and 470 nm after 800 shots, respectively, at a fluence of 0.44 J/cm², concluding that $\Lambda > \sim \lambda/2$.

In our case the laser beam does not hit the sample surface and the measured period is in the range $\Lambda = 290$ nm to 400 nm for both W and Be, thus $\Lambda < \sim \lambda/2$.

On the other hand, ripples with periodicity $\Lambda = 30$ to 100 nm $\ll \lambda = 800$ nm were observed for a higher fluence of 3 J/cm² and after 10 shots [4].

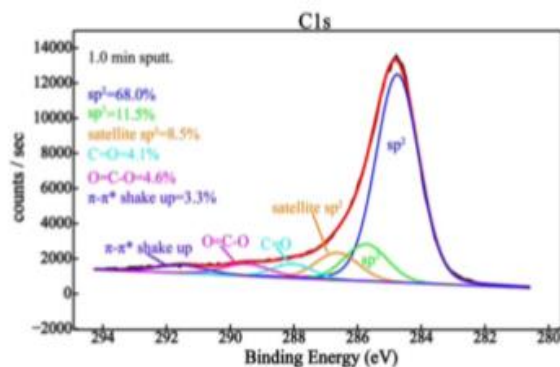
4 Q.-Z. Zhao, S. Malzer, and L.-J. Wang,
Opt. Expr. 15, 15741 (2007).



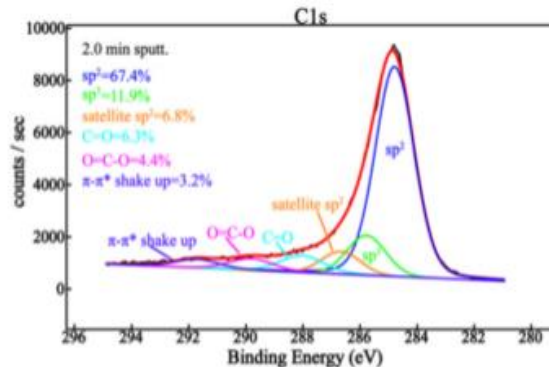
Exposed Be samples in (a) to (f) and W samples in (g) to (l) in D at 20 Torr after 1, 10, 30, 100, 300, and 1000 shots, respectively

Indirect Laser Irradiation of C – in air

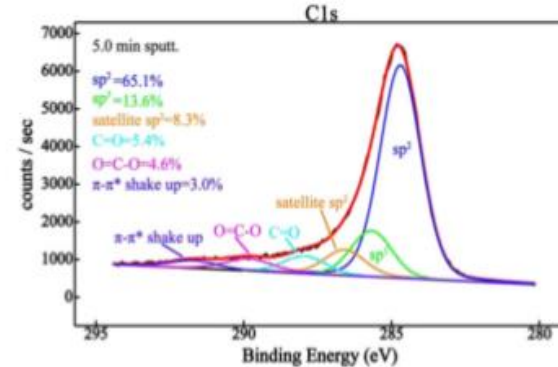
- XPS -



C1s XPS spectra deconvoluted after 1 min Ar sputtering.



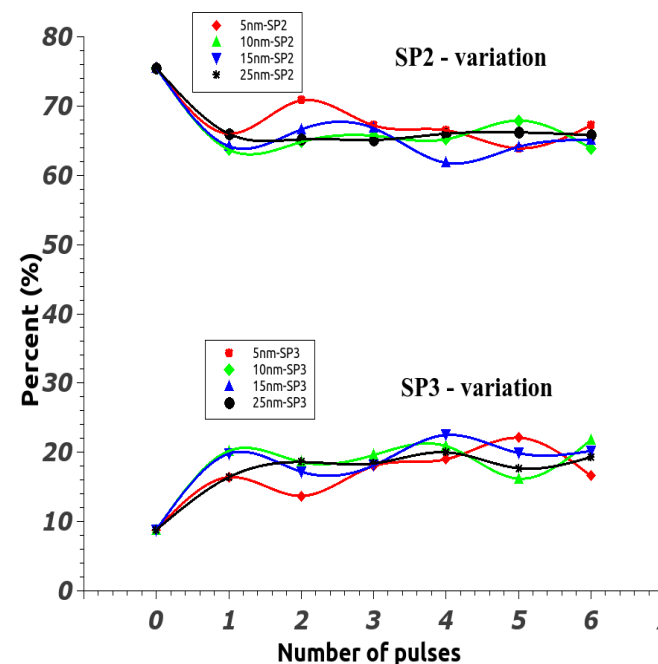
C1s XPS spectra deconvoluted after 2 min Ar sputtering.



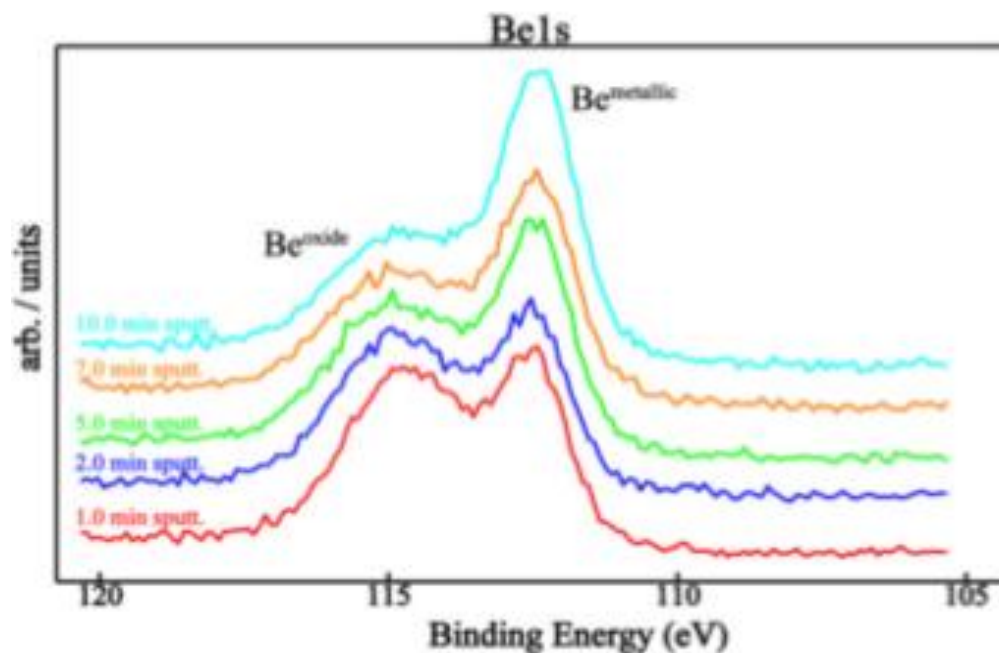
C1s XPS spectra deconvoluted after 5 min Ar sputtering.

Center: A close inspection of the deconvoluted spectra exhibit that the sp^3 component shows a tendency of increasing with the sputtering time (i.e. with sampling depth) from 11.5% (1 min Ar etching) to 13.6% (5 min Ar etching). This behavior is accompanied by the corresponding decrease of the sp^2 feature from 68.0% (1 min Ar etching) to 65.1% (5 min Ar etching). Our estimations for depth profiling are, as follows: 1 min sputtering ~10 nm; 2 min sputtering ~12.5 nm; 5 min sputtering ~22 nm. The noisy O1s XPS spectra suggest a small amount of oxygen incorporated into the sample matrix. After 5 min sputtering we notice that a tiny amount of oxygen is still dissolved into the sample's matrix.

Periphery: A weak cumulative tendency could be assumed for few nm thick layers



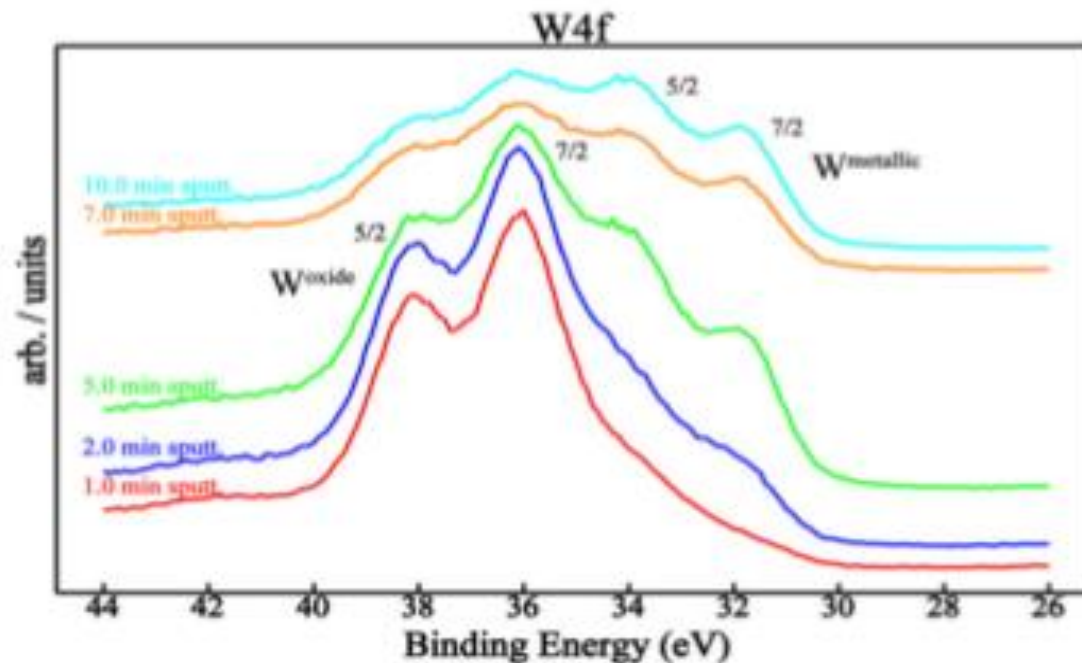
Indirect Laser Irradiation of Be – in air - XPS -



The superimposed Be1s XPS spectra for the Be irradiated sample.

The Be1s band-like XPS spectra were recorded under the circumstances labeled on the spectra (after 1, 2 and 5 min Ar etching time). The spectra clearly display a mixture of metallic and oxide Beryllium with the metallic feature increasing with the sampling depth as a result of diminishing the amount of oxygen.

Indirect Laser Irradiation of W – in air - XPS -



W 4f XPS spectra measured after 1, 2, 5, 7 and 10 min Ar etching of W sample.

The W 4f XPS spectra exhibit only the oxide layer on the outermost layer of the sample. By etching the sample the depth characteristic W 4f doublet of metallic tungsten shows a major oxide component still remaining

Conclusions

- Structures -

The laser fluency threshold for a complete removal of a 1.5 μm carbon layer from several laser pulses is estimated around 1.7 J/cm² which is about 5 times higher than the carbon ablation threshold with a femtosecond laser beam.

At direct irradiation, tungsten cracks, melts and eventually evaporation could still be produced for a higher number of laser pulses.

While higher laser fluency could lead to diamond crystal formation in the graphite surface, at values between 0.2-0.5 mJ/cm², i.e. around the carbon ablation threshold could still produce an increase of the sp³ bonds percentage at the expenses of the sp² bonds.

Only a decrease of oxygen content could be noticed by in depth XPS investigations for Be and W, while a weak cumulative effect through the increase of the sp³ percent could be assumed for the carbon (direct) irradiation.

Conclusions

- Morphology -

Striations were observed on the surface of samples made of Be, W and a mixture of Be-W immersed in air at atmospheric pressure and in deuterium at 20 torr after exposure to plasma created by focusing a high power ultrashort laser pulses within the nearby gas, at 300 μm from the surfaces.

The morphology of the surface structures is similar to that observed in experiments with direct laser irradiation of the surfaces.

For a coating made of Be-W, the striations were localized within areas of 1 to 2 μm well delimited from each other. This observation could be of interest for the creation of surfaces with variable morphology at the micron level in which periodic structures alternate with regions with no particular structuring.