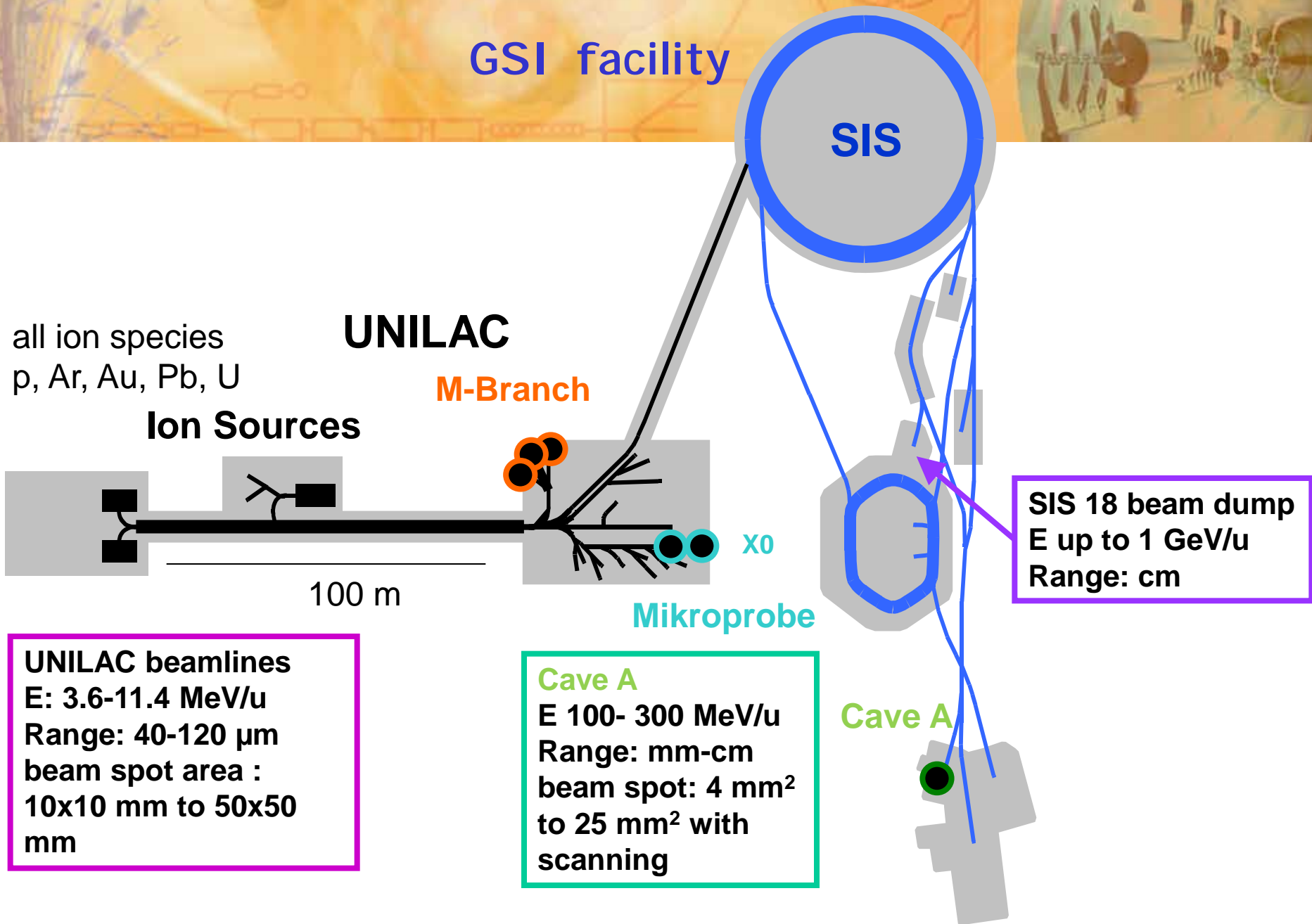




# **GSI tests status and preliminary results**

M. Tomut  
GSI

# GSI facility



**UNILAC**

**M-Branch**

**Ion Sources**

all ion species  
 $p$ ,  $Ar$ ,  $Au$ ,  $Pb$ ,  $U$

100 m

$x_0$

**Mikroprobe**

**SIS 18 beam dump**  
E up to 1 GeV/u  
Range: cm

**UNILAC beamlines**  
E: 3.6-11.4 MeV/u  
Range: 40-120  $\mu\text{m}$   
beam spot area :  
10x10 mm to 50x50 mm

**Cave A**  
E 100- 300 MeV/u  
Range: mm-cm  
beam spot: 4 mm<sup>2</sup>  
to 25 mm<sup>2</sup> with  
scanning

**Cave A**

**SIS**

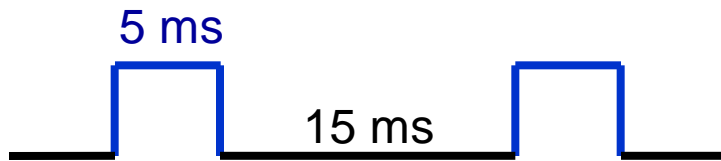
# UNILAC: beam parameters

3.6 / 4.8 / 5.6 / 8.6 / 11.4 MeV/u typical energies

## 50 Hz Mode (Penning, ECR)

50 Hz

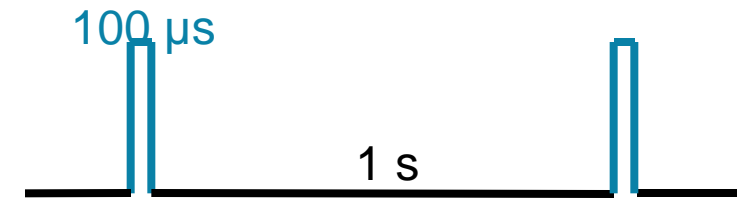
5 ms length of macropulse



## high-current mode (MEVVA source) (for SIS experiments)

1-2 Hz

100-200  $\mu$ s length of macropulse



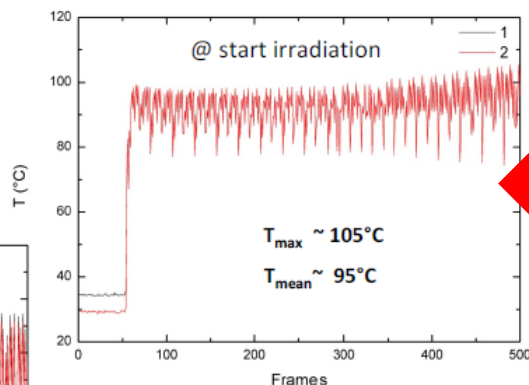
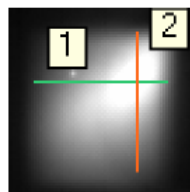
# Thermal camera monitoring of sample temperature

## High duty cycle

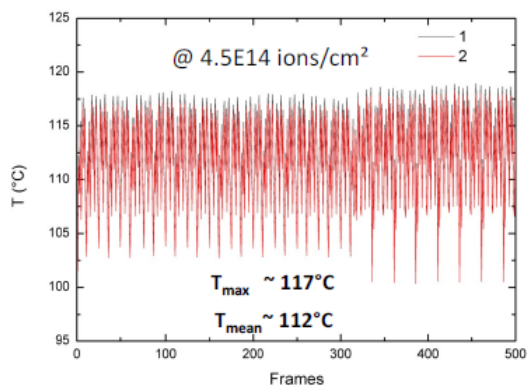
Amorphous Carbon,  $21 \mu\text{g}/\text{cm}^2$  (Targetlab)

$\text{Au}^{25+}$  3.6 MeV/u, 38 Hz, 4ms, defocused beam, beamspot  $1.7 \times 1.7 \text{cm}$  ( $\varnothing$  sample 30mm)

flux:  $1.0 \times 10^{10} \text{ ions}/\text{cm}^2\text{s}$

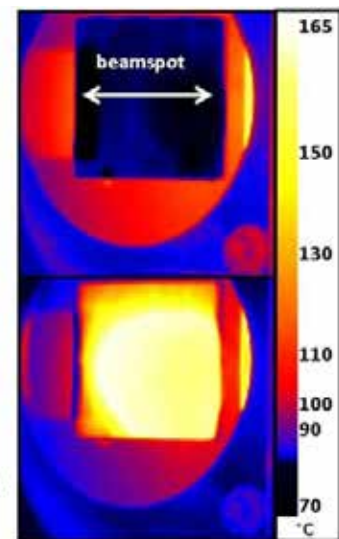
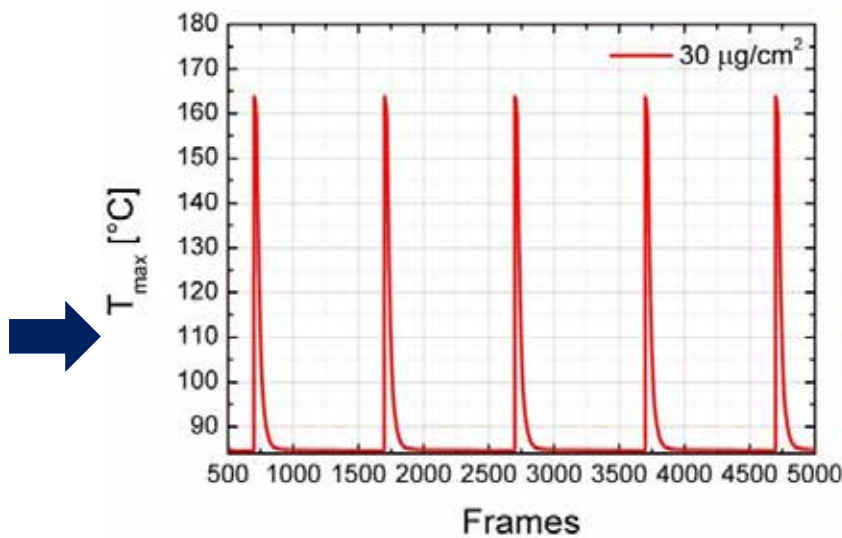


- radiation damage
- thermal effects  $\bar{\sigma}$  stress



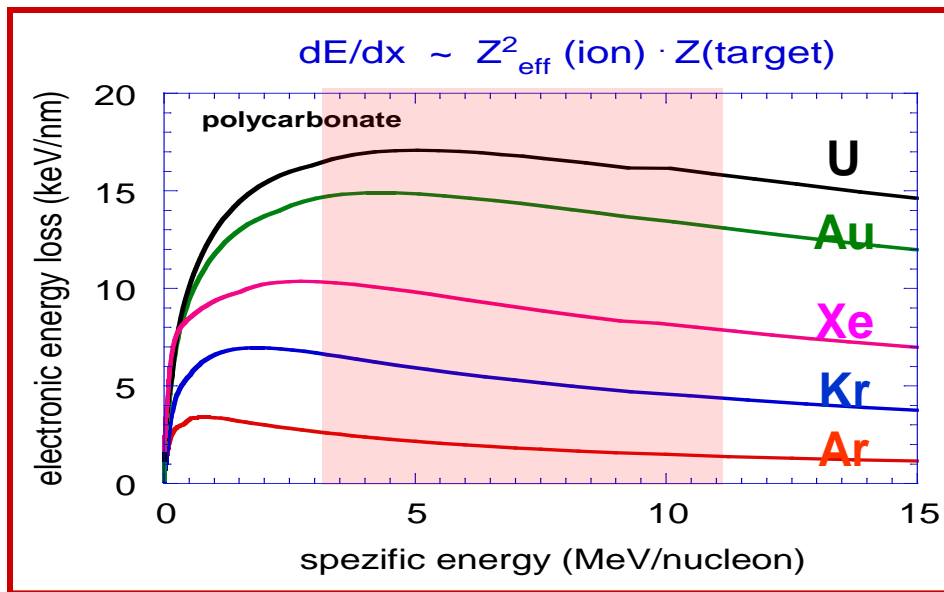
- radiation damage
- short thermal spikes
- $\bar{\sigma}$  stress waves

## Low duty cycle



# Irradiation energy

- energies close to Bragg peak:
  - to maximize energy deposition and damage
  - to avoid activation
- online and in situ monitoring: video camera, fast IR camera, SEM, XRD, IR spectroscopy



SRIM code

ion species ..C...Xe...U

flux:  
up to  $10^{10}$  ions/cm<sup>2</sup> s

# Irradiation Tests at GSI

## 1) Feb-Mar 2014:

§  $^{238}\text{U}$ : 1.14 GeV, 0.5 ms, 0.6 Hz,  $4 \times 10^9$  ions/cm<sup>2</sup>s

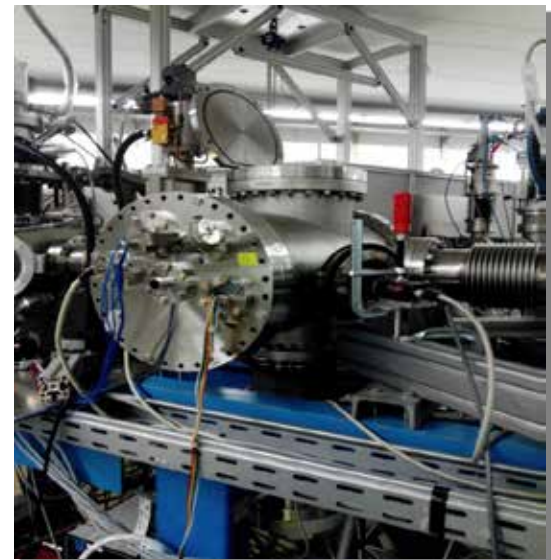
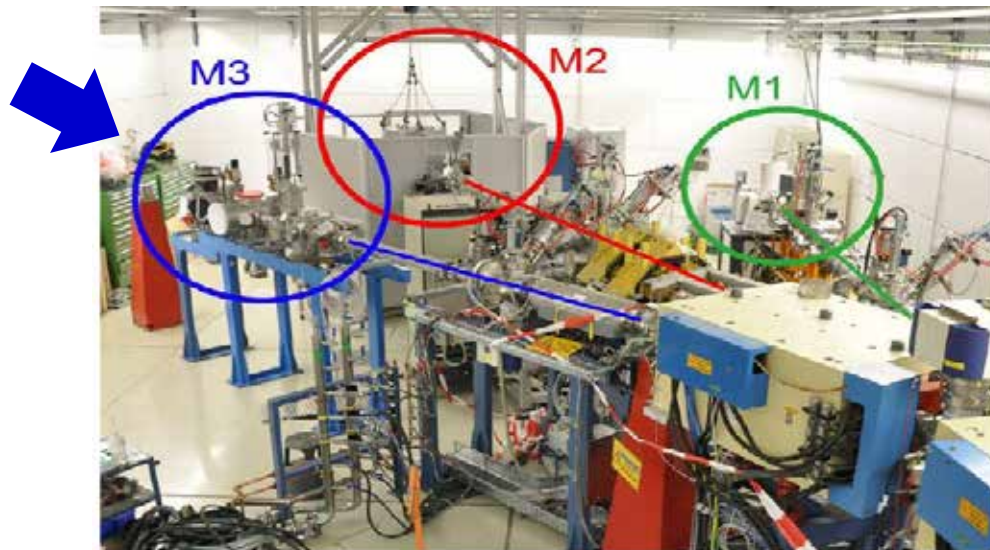
§  $^{208}\text{Bi}$ : 1 GeV, 0.5 ms, 3.4 Hz,  $1.2 \times 10^9$  ions/cm<sup>2</sup>s

§ CuCD, CFC (2 orientations), MoGr (MG 3110P, 2 orientations, samples not annealed) irradiated with fuences up to  $1 \times 10^{14}$  ions/cm<sup>2</sup>

## 2) July 2014:

§  $^{197}\text{Au}$ : 945 MeV, 40 Hz, up to  $2 \times 10^9$  ions/cm<sup>2</sup>s

§ C: 11.4 MeV/u,  $5 \times 10^9$  ions/cm<sup>2</sup>s



# Thermal properties degradation

Fluences:  $1e11$ ,  $1e12$ ,  $1e13$ ,  $5e13/1e14$   $i/cm^2$  at fluxes  $\sim 5e9$   $i/cm^2s$

- Samples for LFA: in-plane thermal conductivity measurements
  - Mo-Gr discs in-plane and transversal; U irradiation
  - Cu-CD discs U and Bi irradiation, 4.8 MeV/u



**Cu-CD**

## Mo-Gr I



- Irradiation holders for postirradiation thermal diffusivity tests

- Laser Flash analysis in-plane

# Post-irradiation tests

- Samples for off-line tests:
- Cu-CD,
- Mo-Gr: 2 orientations, CFC: 2 orientations (U, Au, Bi)



Microstructural characterization:

- Raman spectroscopy,
- SEM

Mechanical properties:

- Nanoindentation,

Electrical properties:

- 4-point probe resistivity measurements



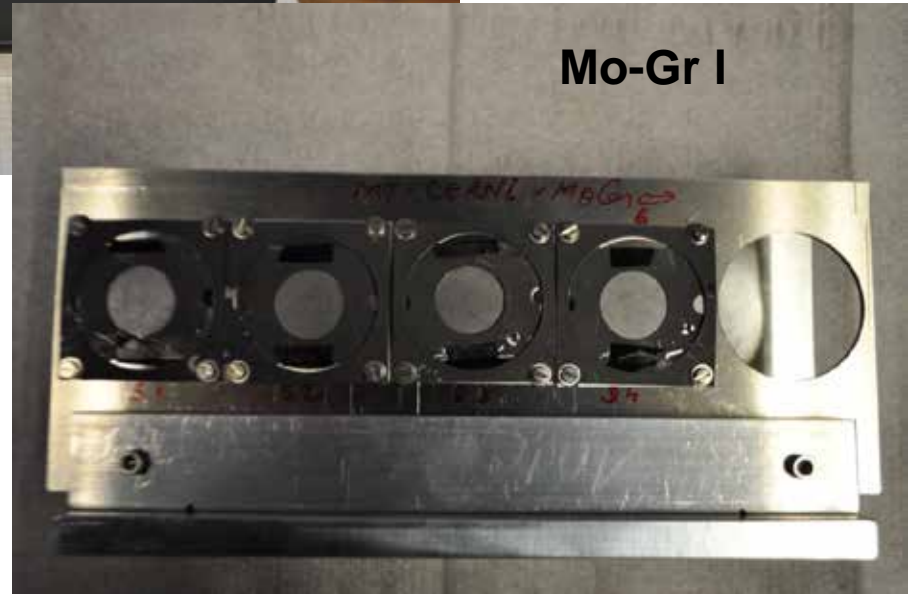
# High energy deposition experiments using focused U beam



Mo-Gr ---



Mo-Gr I



# Irradiation of MoGr and Cu-Dia at high energy

§ CuCD and MoGr samples treated @1800°C for 50 min prior irradiation!

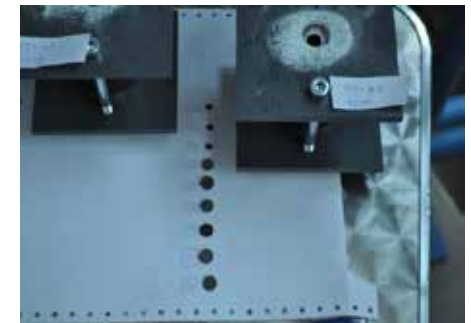
§ Planned for post-irradiation:

- 0 structural analysis
- 0 thermal conductivity measurements
- 0 mechanical test - nanoindentation

...some time after



**Sm<sup>152</sup>**  
**~ 300 MeV/u**  
**range: mm**  
**bulk**  
**samples**



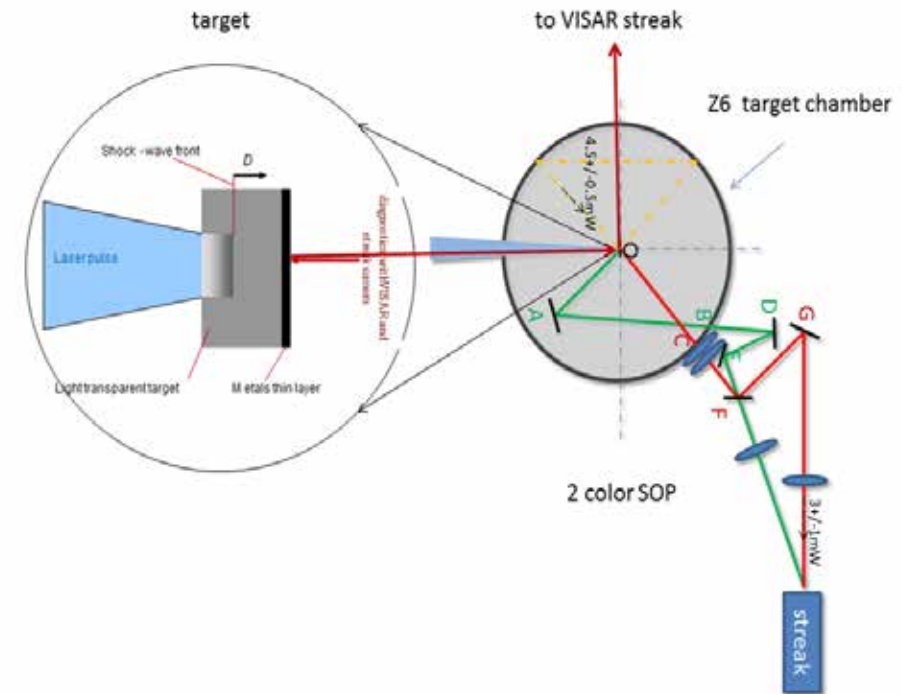
# Shock tests of target and collimator materials using high power PHELIX laser at GSI:

## Laser parameters:

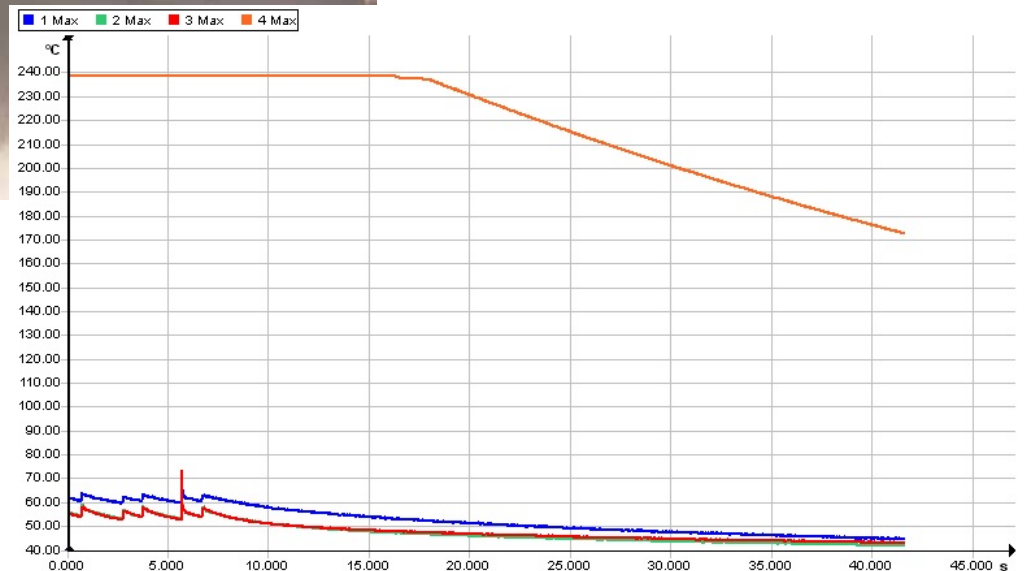
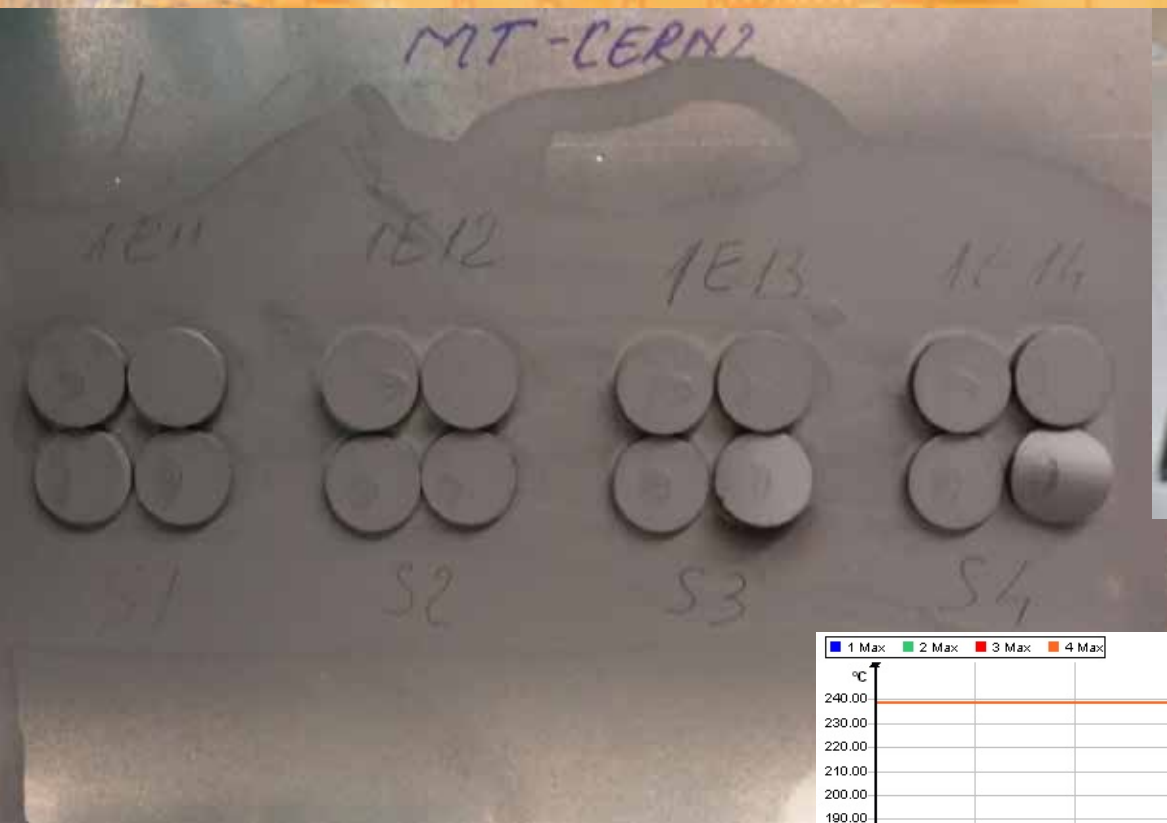
- § Energy: 150 J ( $2\omega$ )
- § Focal spot at the target: 50
- § Laser pulse duration: 7 ns
- § Laser contrast  $\sim 10^{-5}$ - $10^{-6}$
- § Intensity:  $10^{14}$  W/cm<sup>2</sup>

## Diagnostics tested:

- § VISAR-system available
- § Two color SOP (Streak of measurement)



# Deformation of Mo-GR samples transversal cut starting with $6E12$ i/cm<sup>2</sup>



# Thin Mo-Gr samples after irradiation



Mo\_GR in-plane

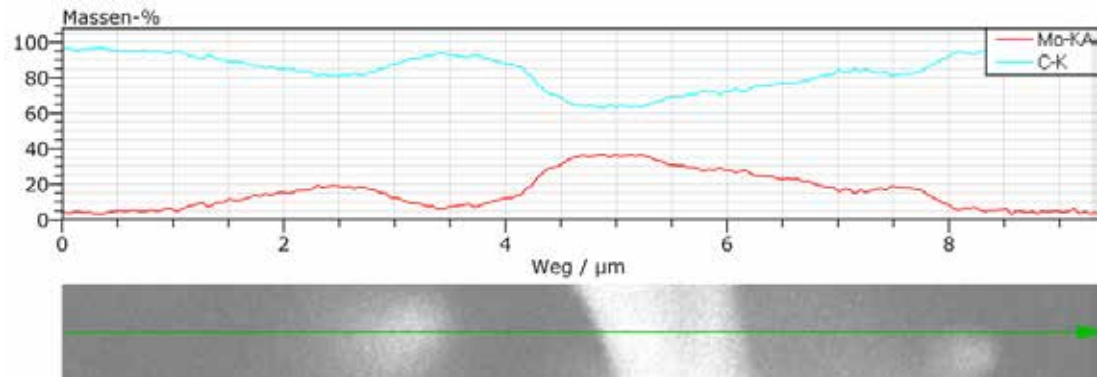
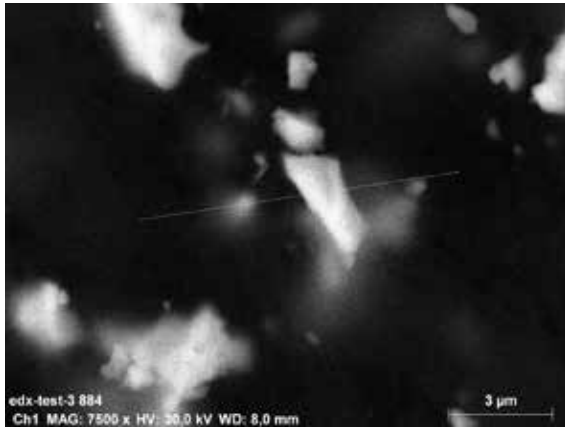
Mo\_GR transversal



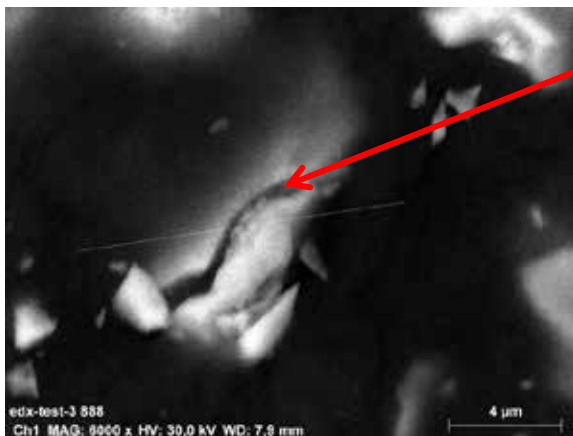
# Microstructure and element distribution - SEM/EDX



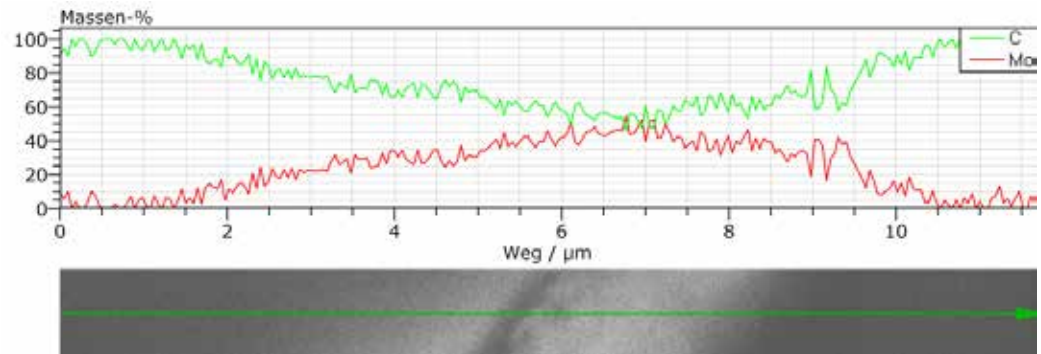
## Pristine Mo-Gr-transversal orientation



## Mo-Gr-transversal orientation at 1E13 U ions/cm<sup>2</sup>

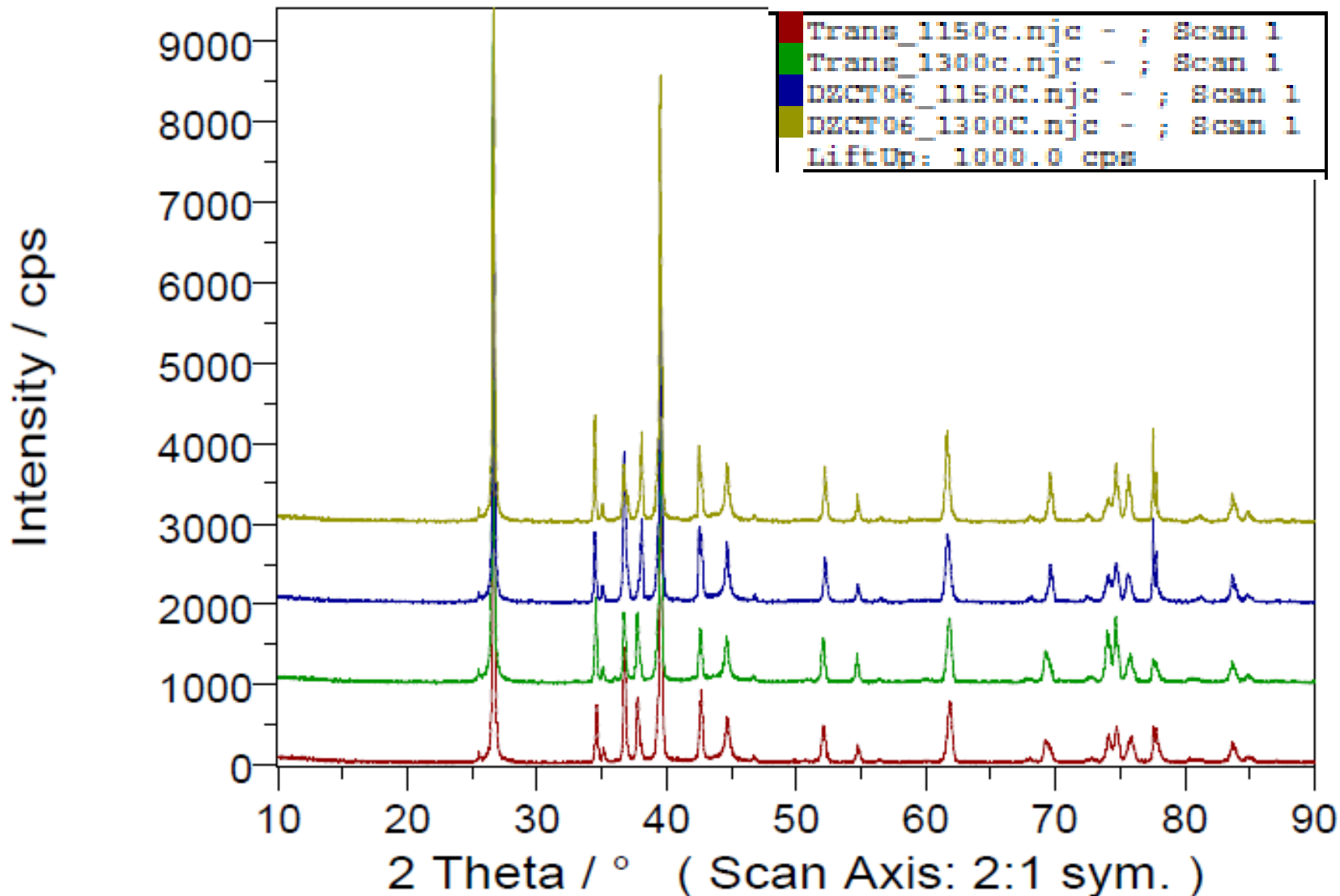


Cracks In MoC particles - irradiation induced stress



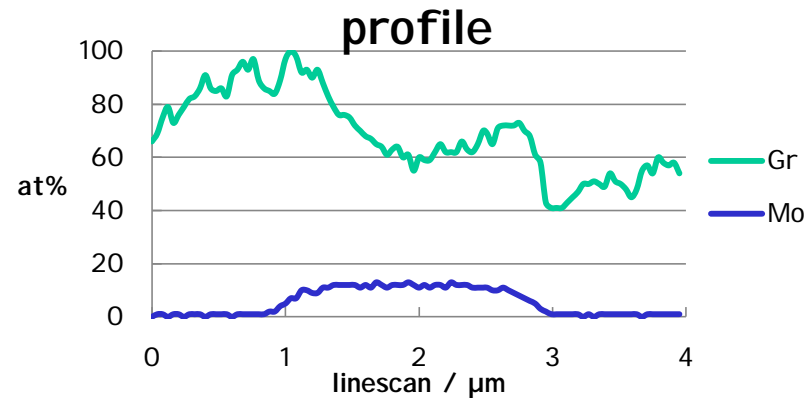
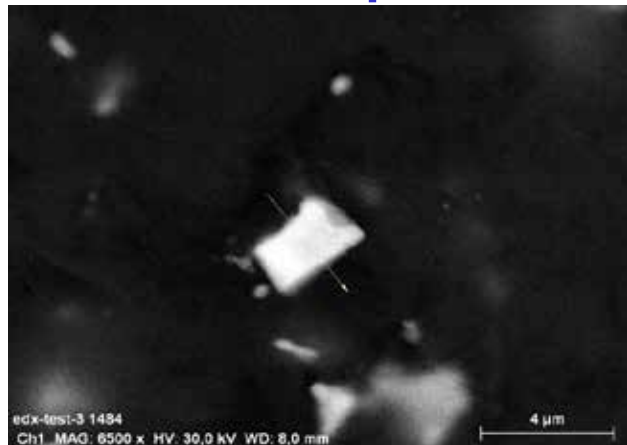
# XRD analysis of optimized Mo-Gr samples: pristine and high fluence

Comparison of 4 Scans

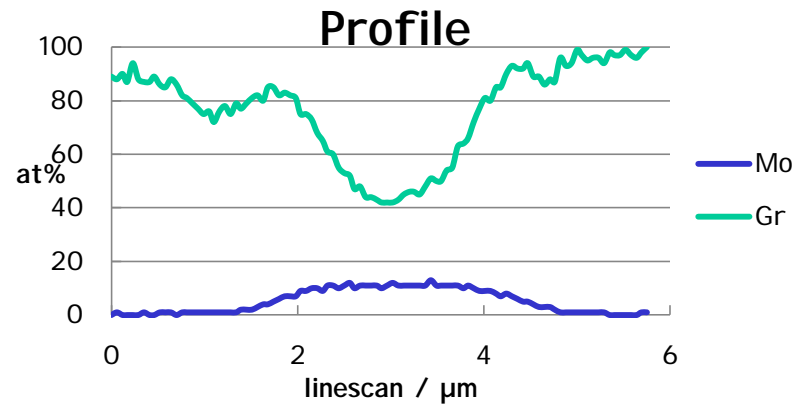
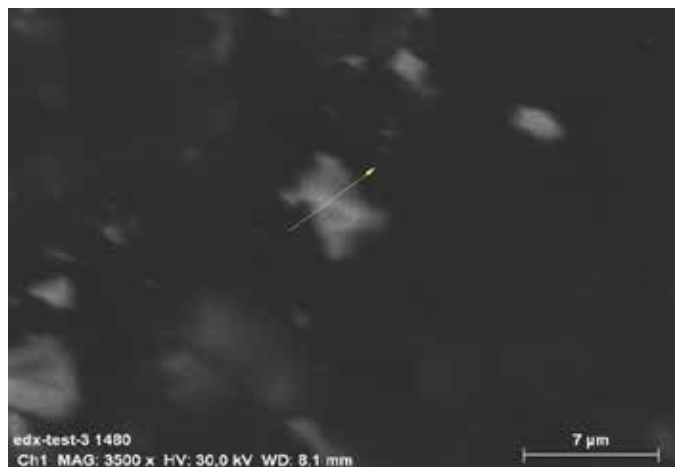


# Microstructure and element distribution in optimized Mo-Gr- SEM/EDX

sample: MoGr-5220S-T-1150-1e11- Au

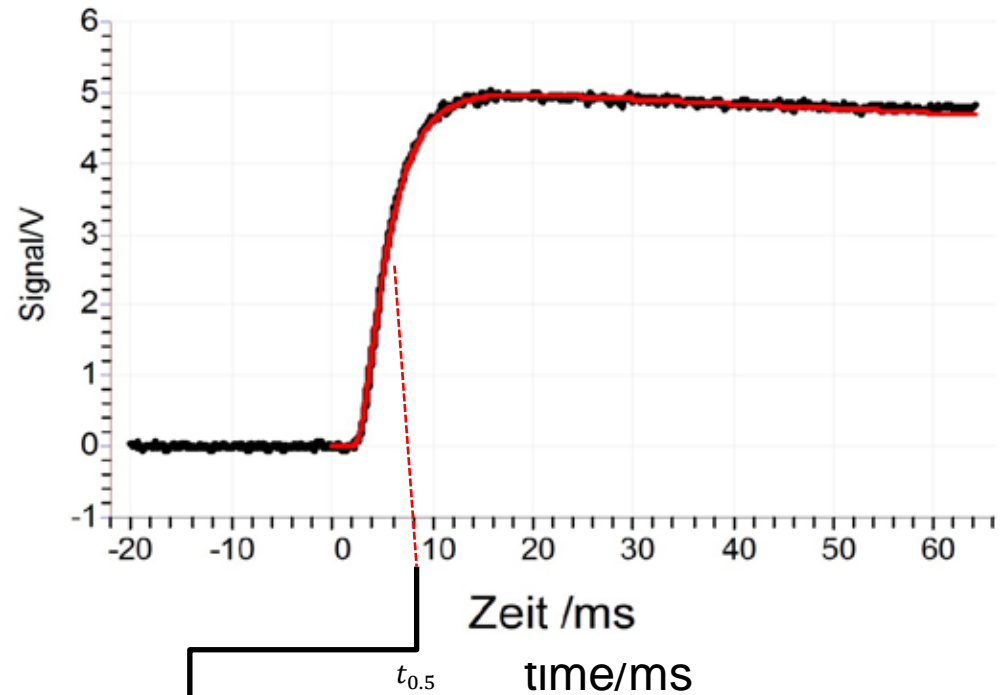
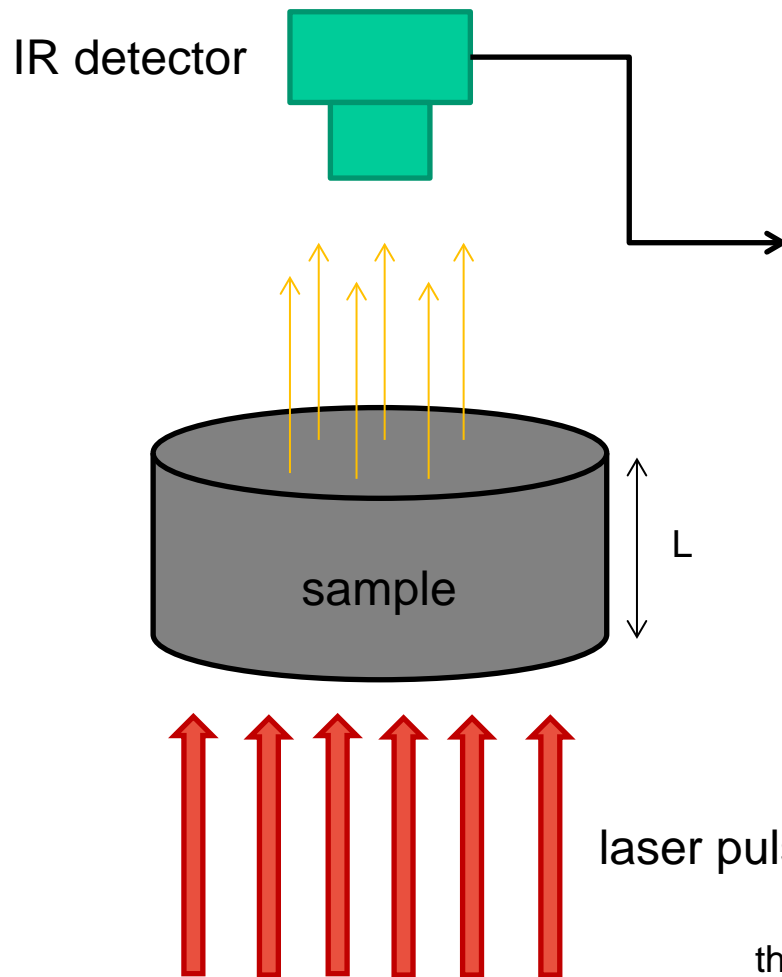


sample: MoGr-5220S-T-1150-1e12- Au





# Thermal diffusivity degradation -Laser flash analysis



thermal diffusivity

$$\alpha = \frac{0.1388 \cdot L^2}{t_{0.5}}$$

thermal conductivity

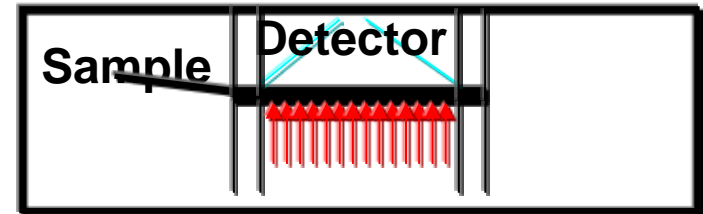
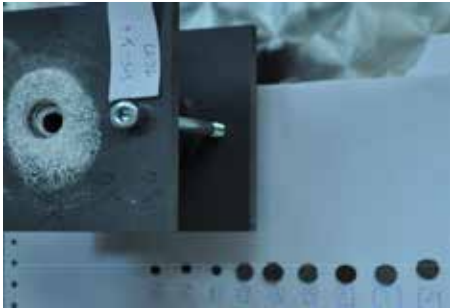
$$\lambda = \alpha \cdot \rho \cdot c_p$$

# Thermal properties degradation -post-irradiation evaluation

fluences:  $1e11$ ,  $1e12$ ,  $1e13$ ,  $5e13/1e14$   $i/cm^2$  at fluxes  $\sim 5e9$   $i/cm^2s$

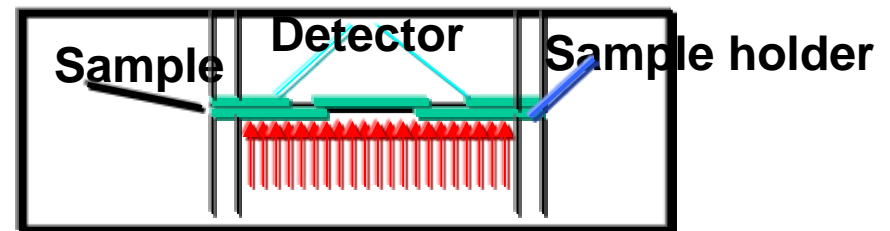
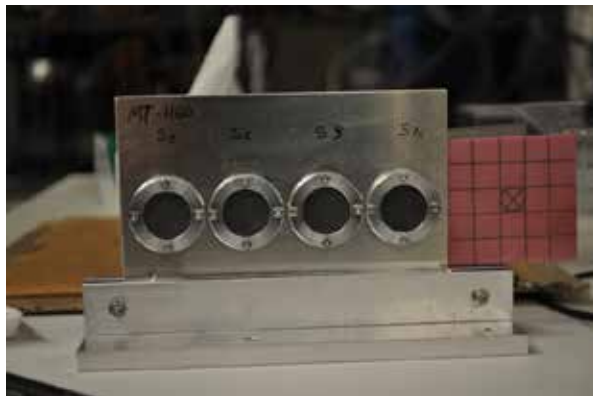
## Samples for LFA: Isotropic graphite and flexible graphite

- classical transmission measuring geometry



Transmission

- in-plane measuring geometry



In-plane

# Post-irradiation tests

- Samples for off-line tests:
- Cu-CD,
- Mo-Gr: 2 orientations, CFC: 2 orientations (U, Au, Bi)



Microstructural characterization:

- Raman spectroscopy,
- SEM

Mechanical properties:

- Nanoindentation,

Electrical properties:

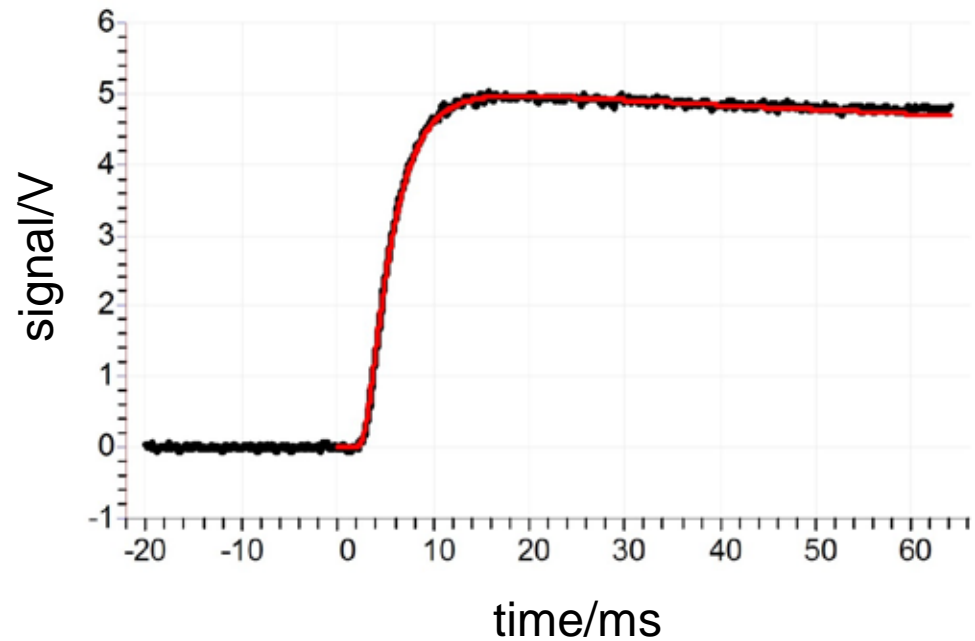
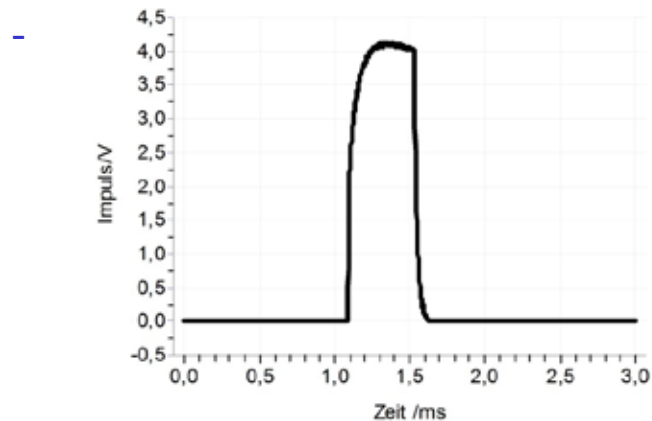
- 4-point probe resistivity measurements

# Mathematical models - heat loss

- Cape and Lehman (1963)

- included a heat loss term in the initial equation derived from Stefan-Boltzmann radiation law:

- $$\nu = \frac{4\sigma\epsilon T_0^3}{\lambda}$$

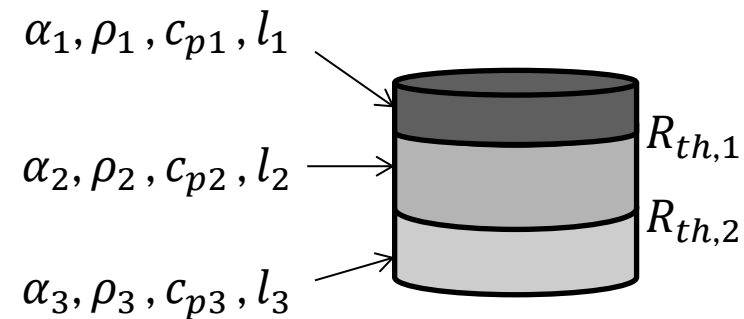
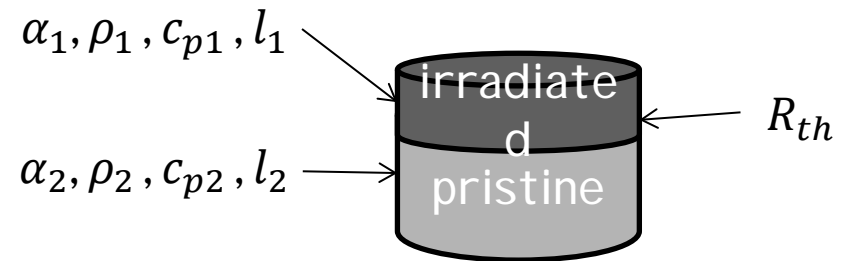


$$\alpha = 44.103 \text{ mm}^2/\text{s}$$

# Layered composites

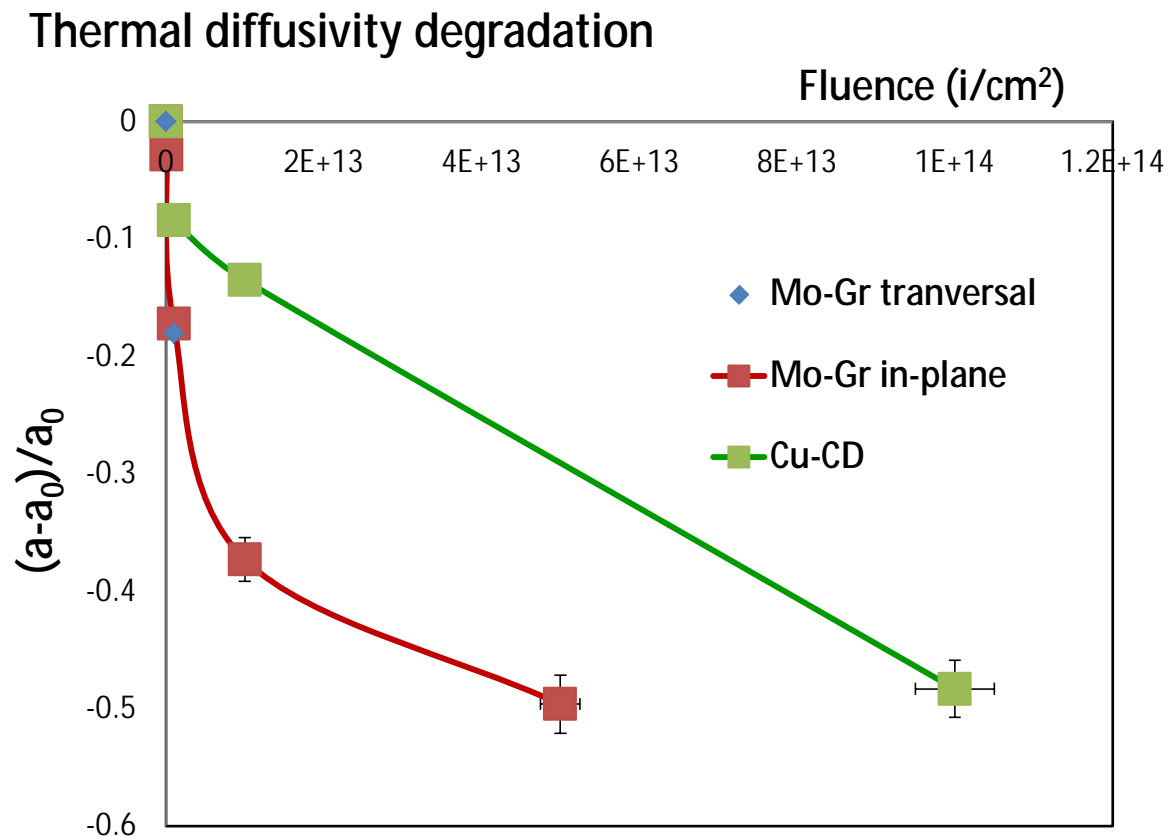
- contact resistance or
- diffusivity of the unknown layer values can be calculated from  $t_{0.5}$

- more difficult for more layers:
  - effective thermal conductivity is independent of the layer order
  - but not the temperature history (boundary conditions)
- $\rightarrow t_{0.5}$  depends on the order



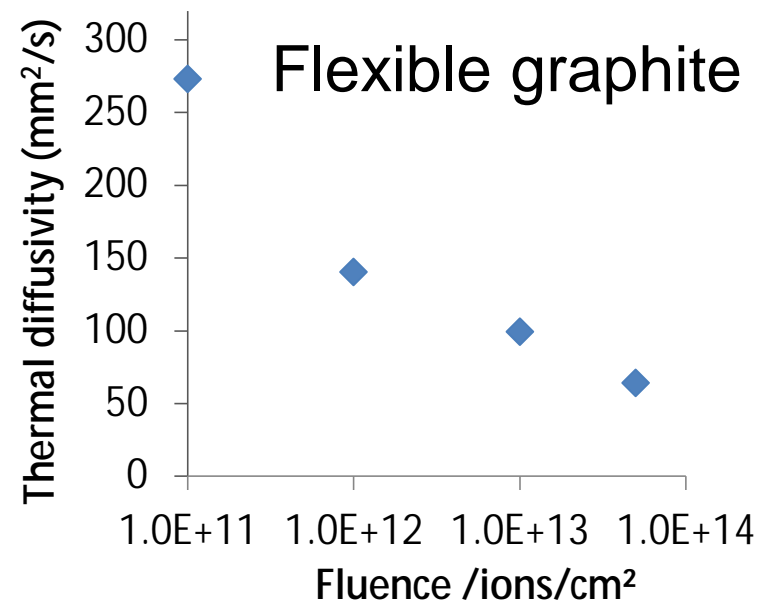
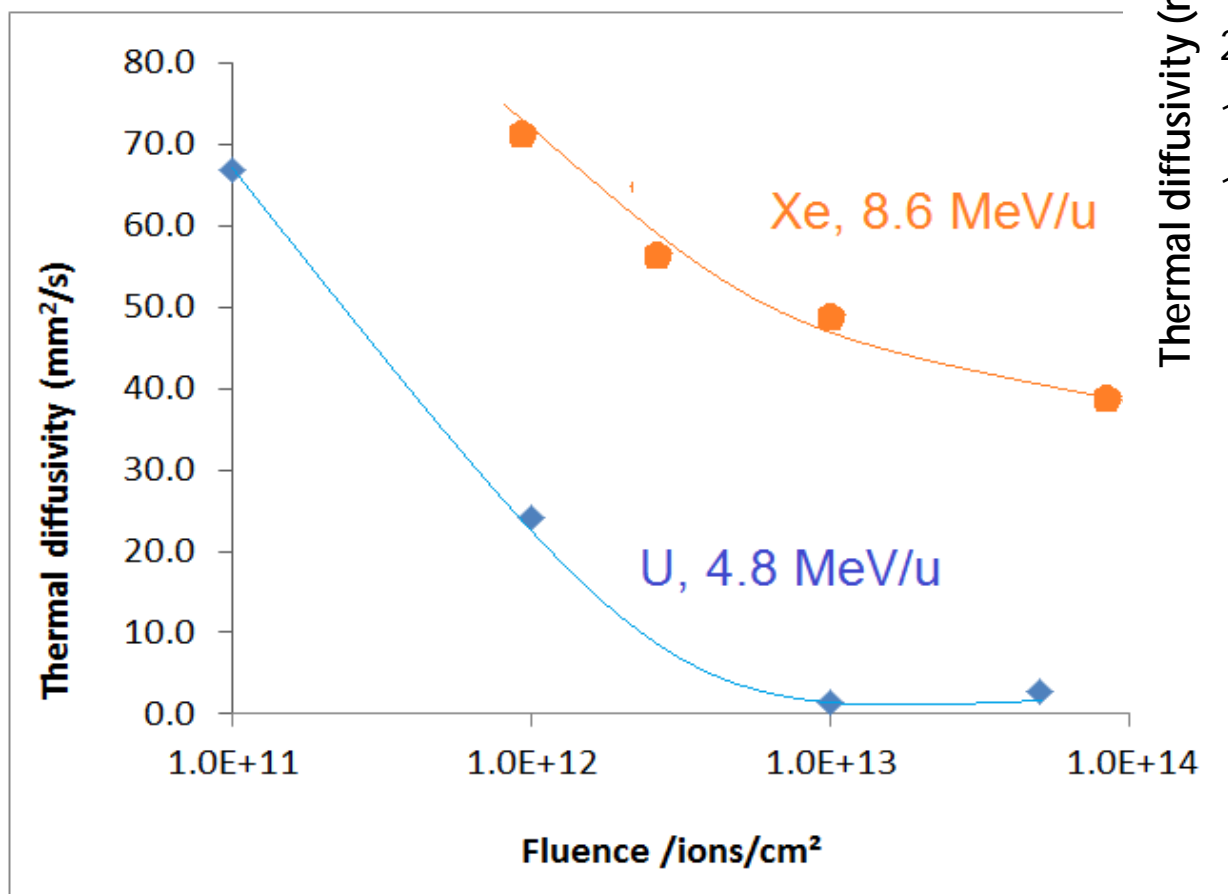
# Thermal diffusivity degradation - composites

## ion- irradiated Mo-Gr (non-optimized) and Cu-CD



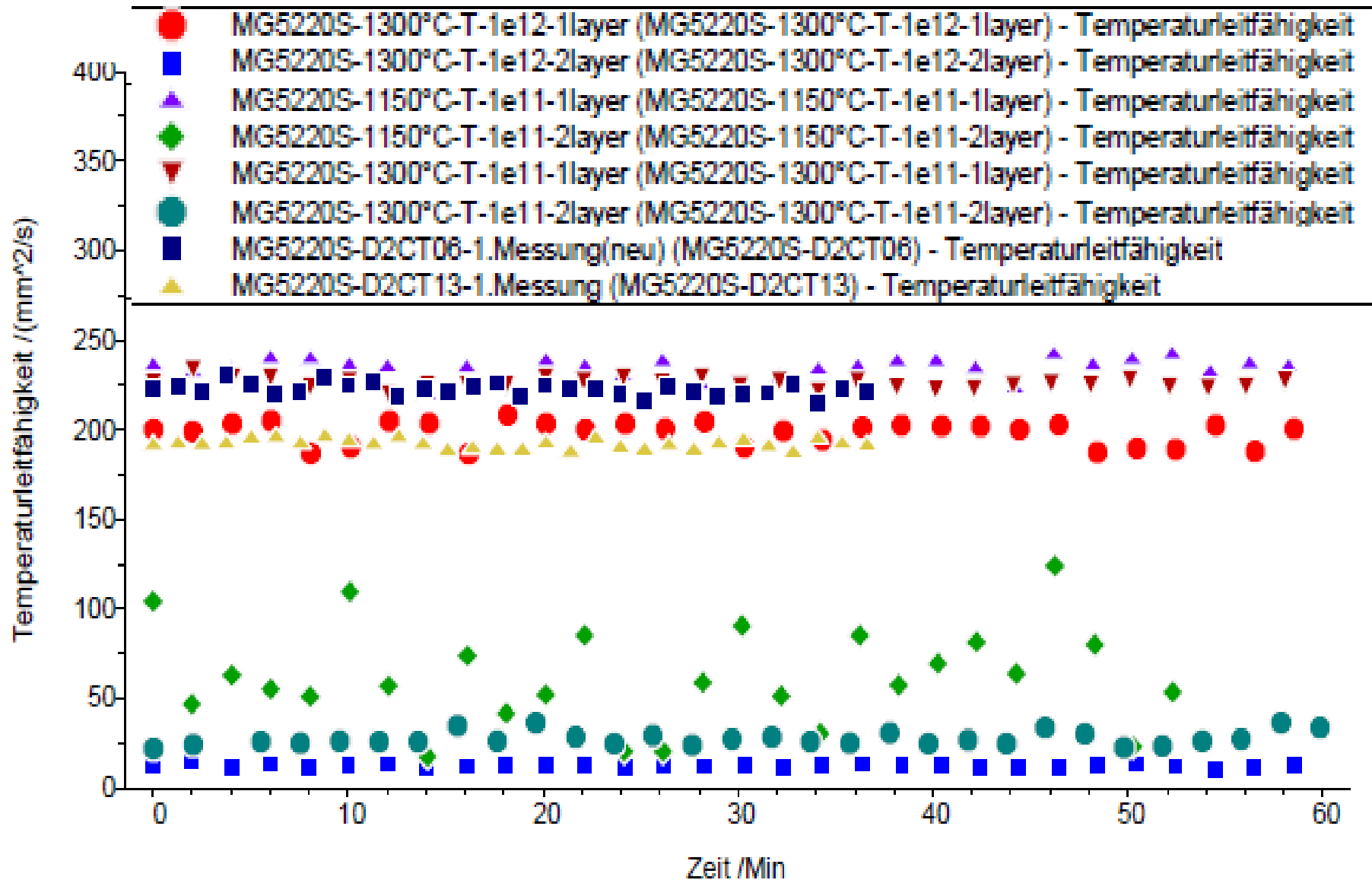
# Ion-induced thermal diffusivity degradation of graphite

## Comparison U vs Xe irradiation graphite vs flexible graphite



Isotropic graphite

# Thermal diffusivity degradation – optimized Mo-Gr

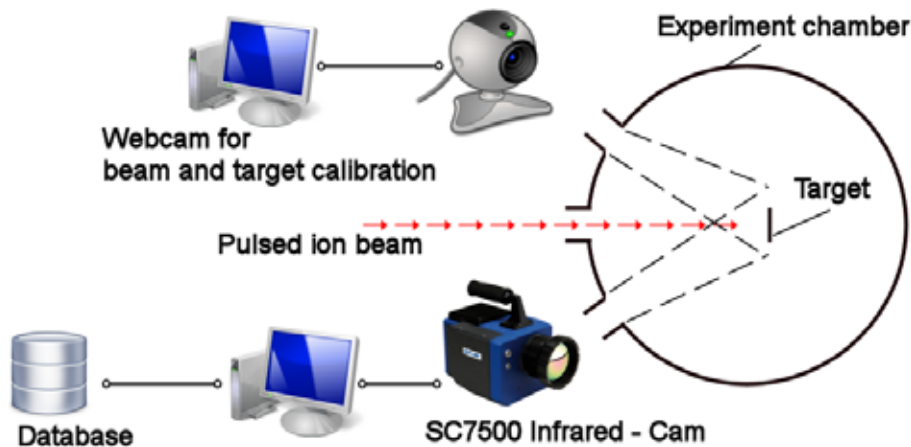




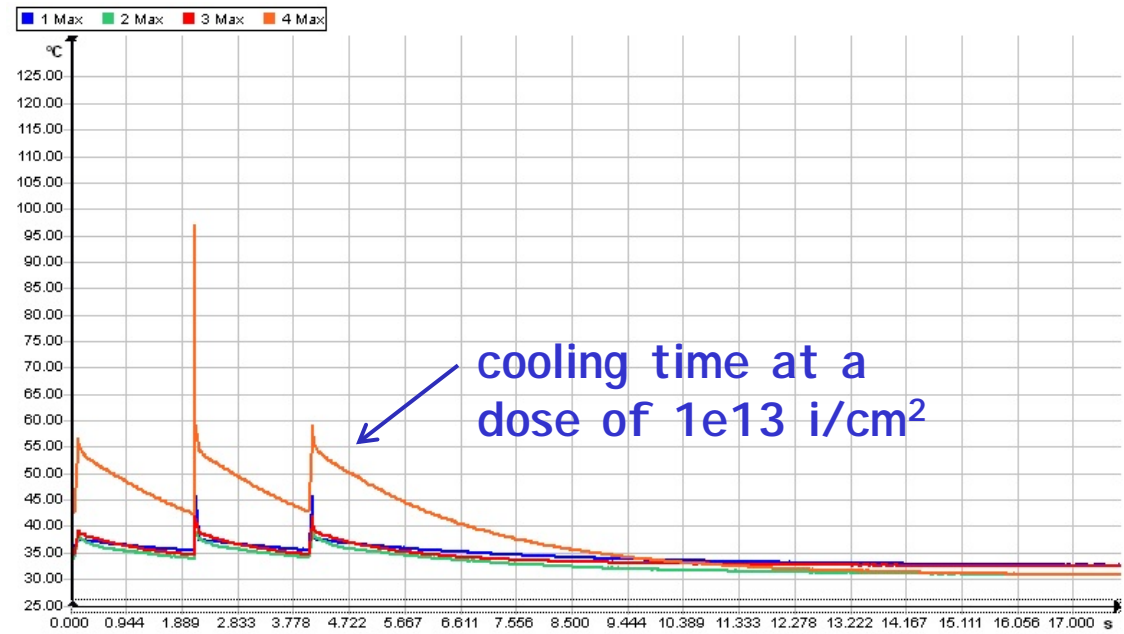
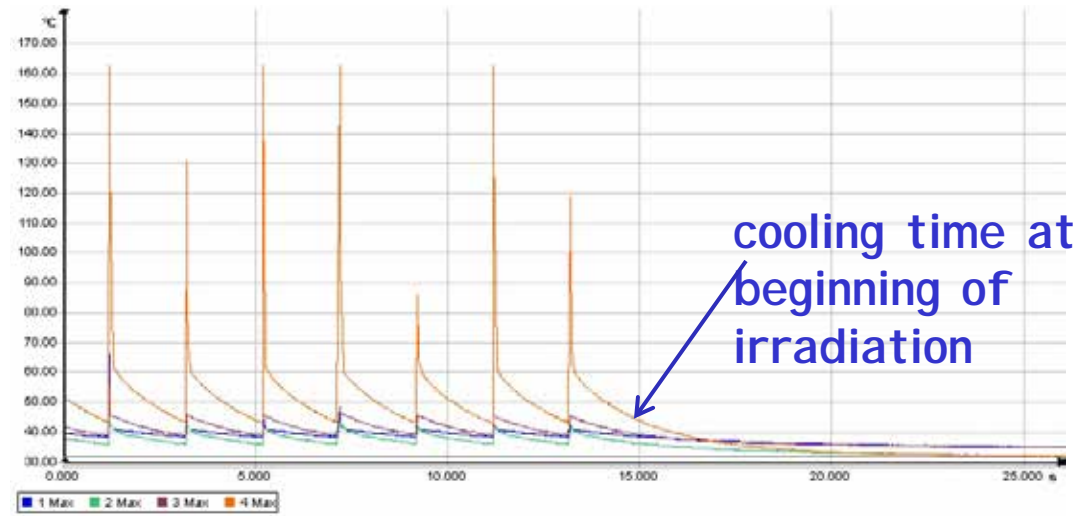
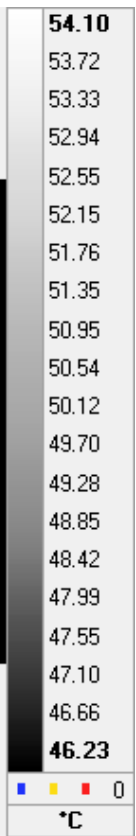
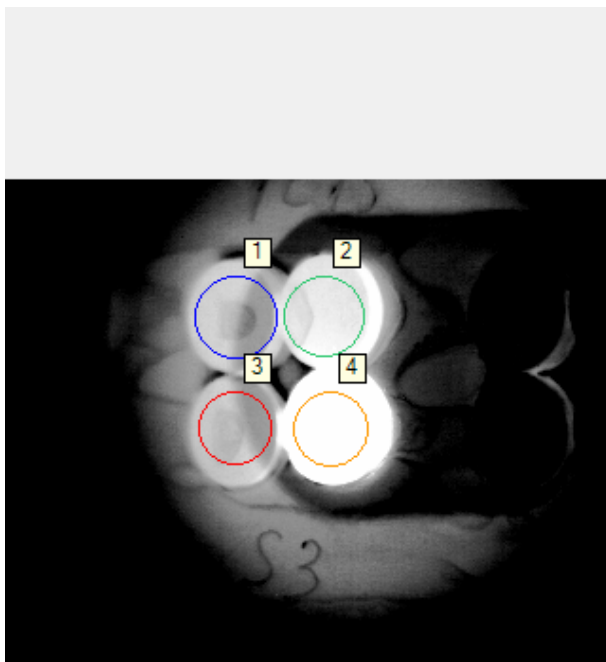
# Thermal properties degradation - online

fluences:  $1e11$ ,  $1e12$ ,  $1e13$ ,  $5e13/1e14$  i/cm<sup>2</sup> at fluxes  $\sim 5e9$  i/cm<sup>2</sup>s

- Thermal conductivity degradation monitoring (on-line using thermal camera: estimation of time constant at cooling)
  - Cu-CD, Mo-Gr: 2 orientations, CFC: 2 orientations (U, Bi)



# Thermal camera monitoring of sample temperature during cooling

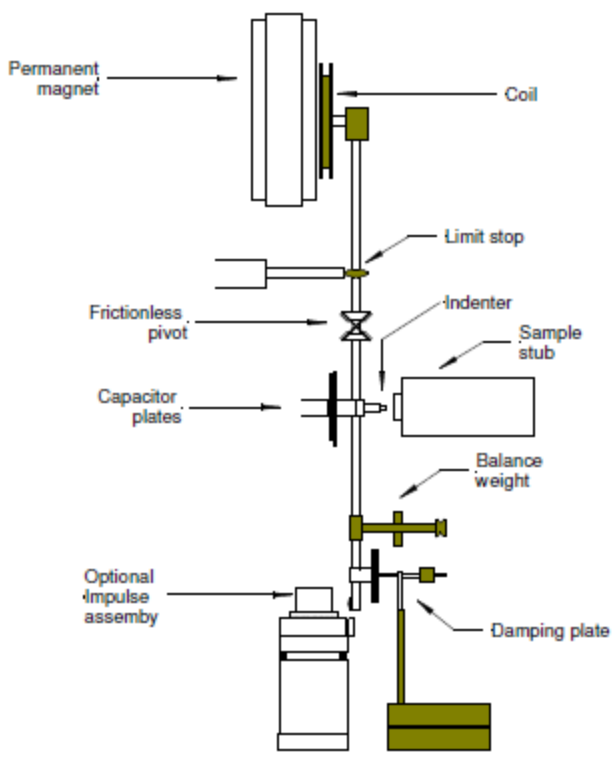


Temporal evolution of maximum temperature in irradiated samples

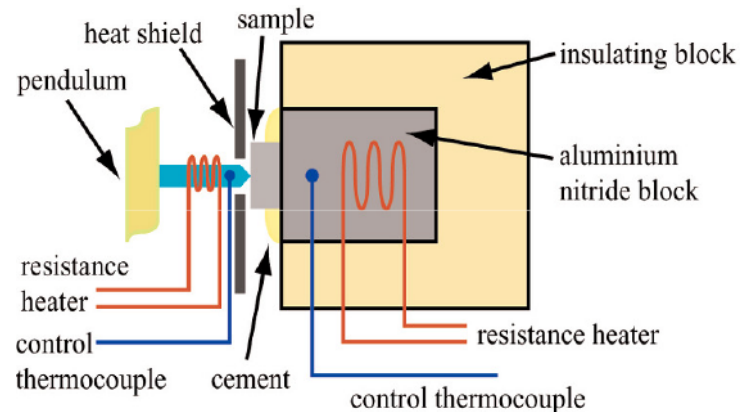
# Mechanical properties degradation-nanoindentation

investigations of hardening and E modulus change of irradiated layers

high temperature



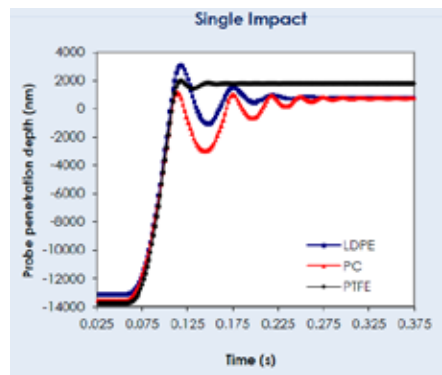
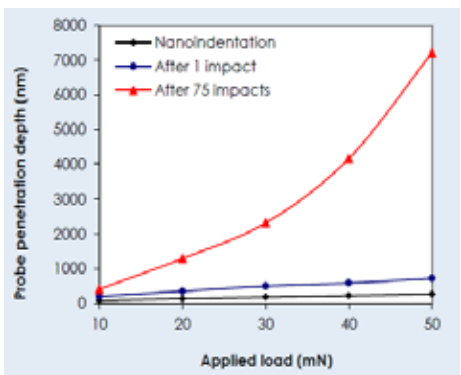
Courtesy LOT Quantum Design



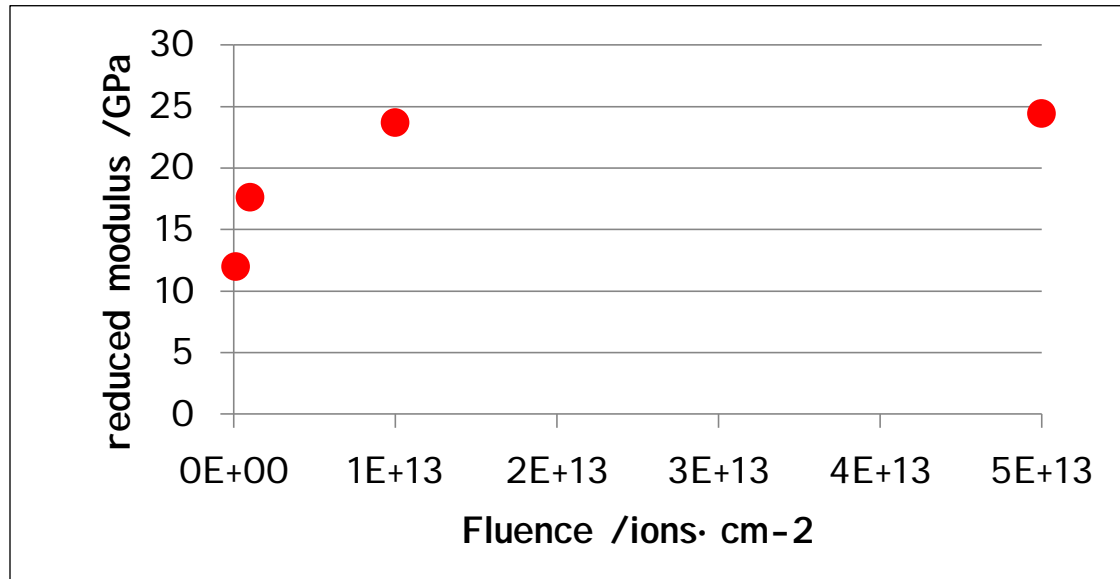
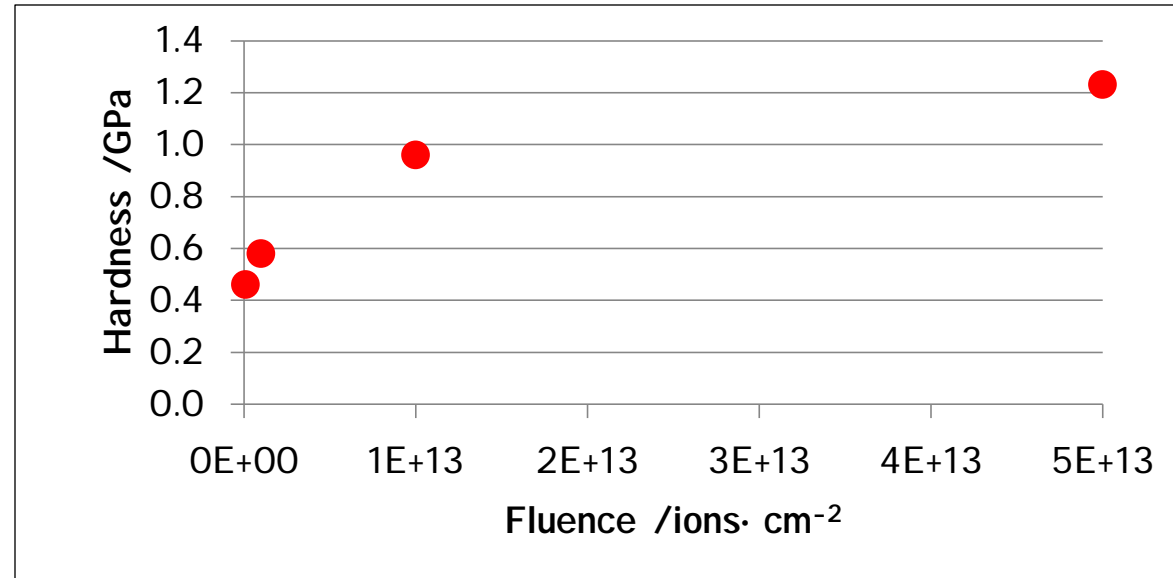
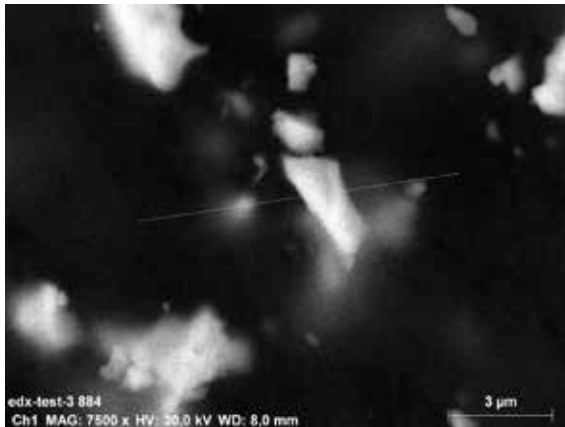
Impact:

fatigue

damping



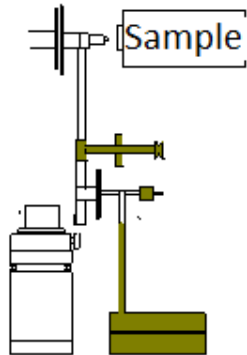
# Molybdenum Graphite composite



- **Bi**, 4.8 MeV/u
- Harder materials due to carbides
- Less radiation induced hardening than CFC

# Mechanical behaviour of irradiated Mo-Gr

## Nanoindentation

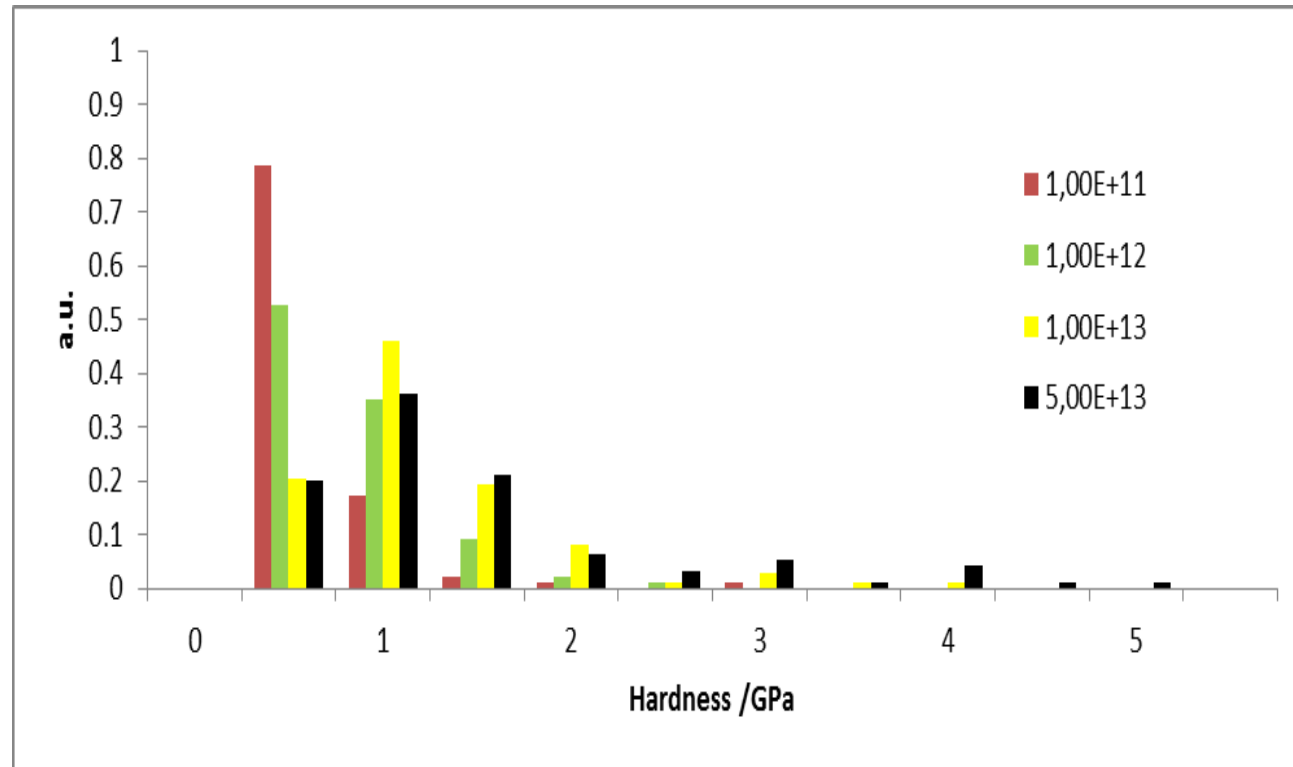


### Histograms of

- Hardness
- Young modulus

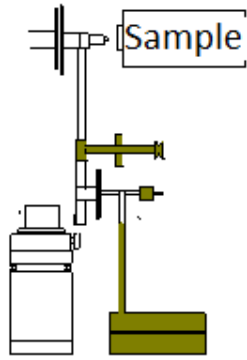
• evolution with dose

• statistical approach for composites



# Mechanical behaviour of irradiated Mo-Gr

## Nanoindentation

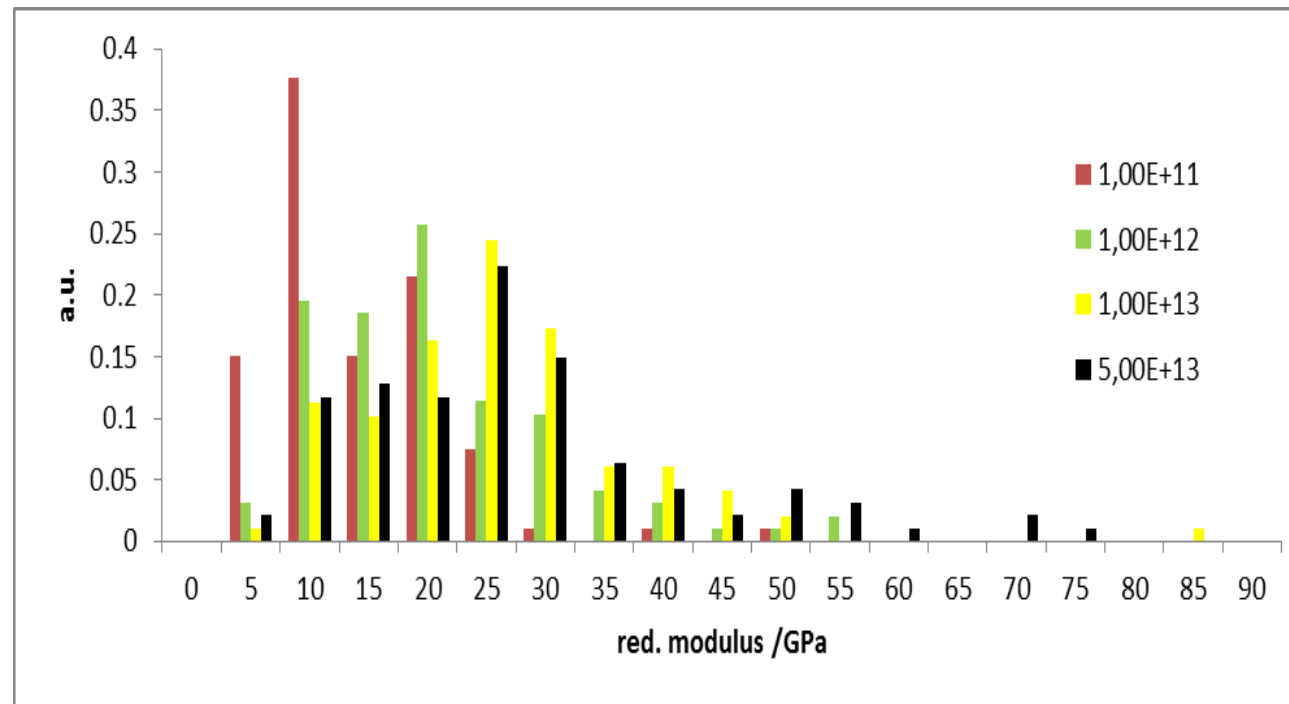


### Histograms of

- Hardness
- Young modulus

• evolution with dose

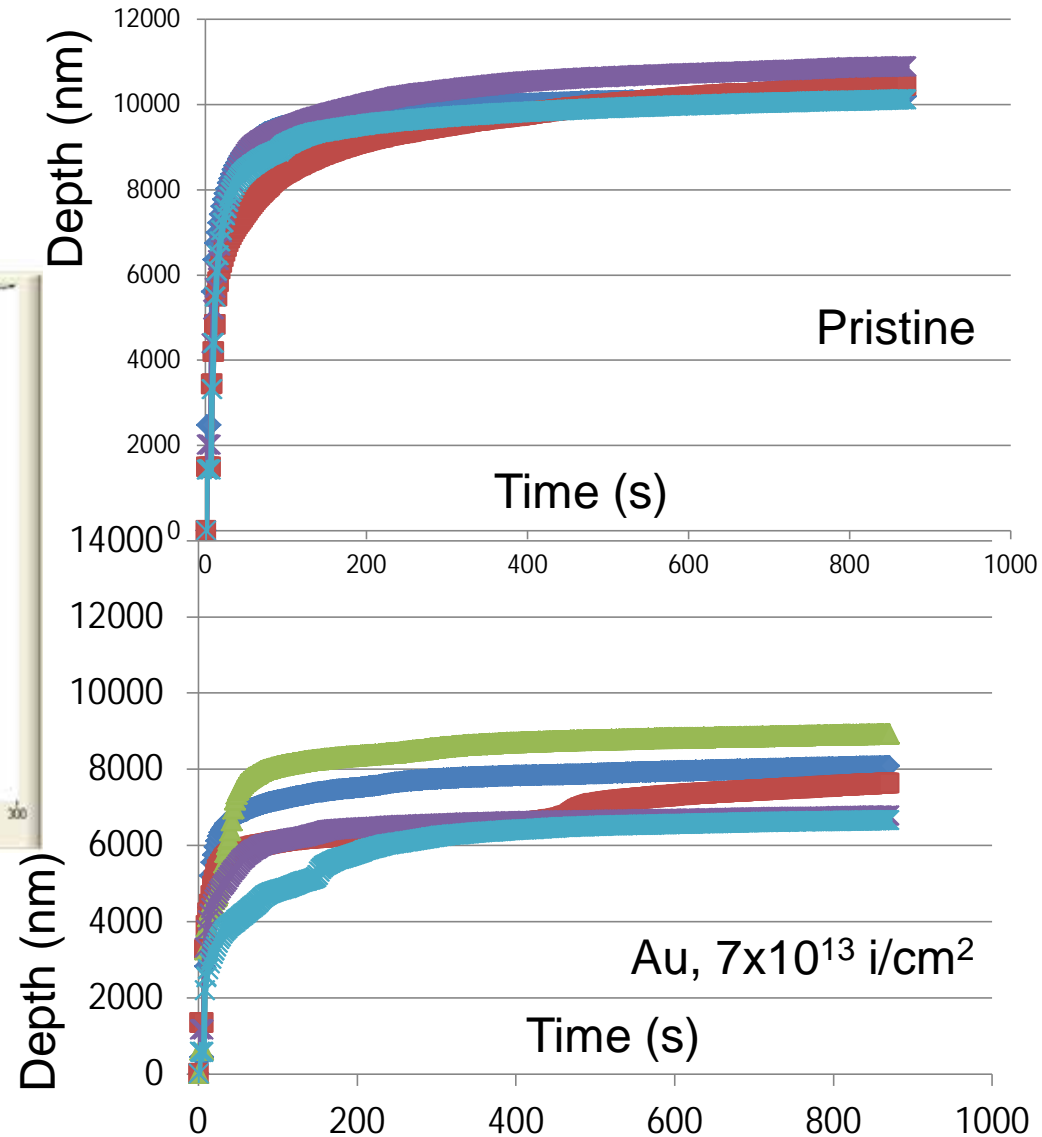
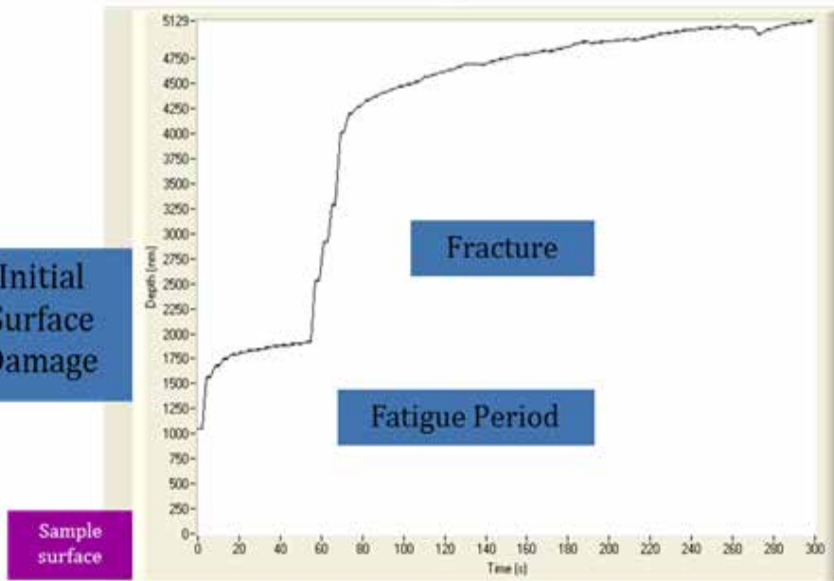
• statistical approach for composites



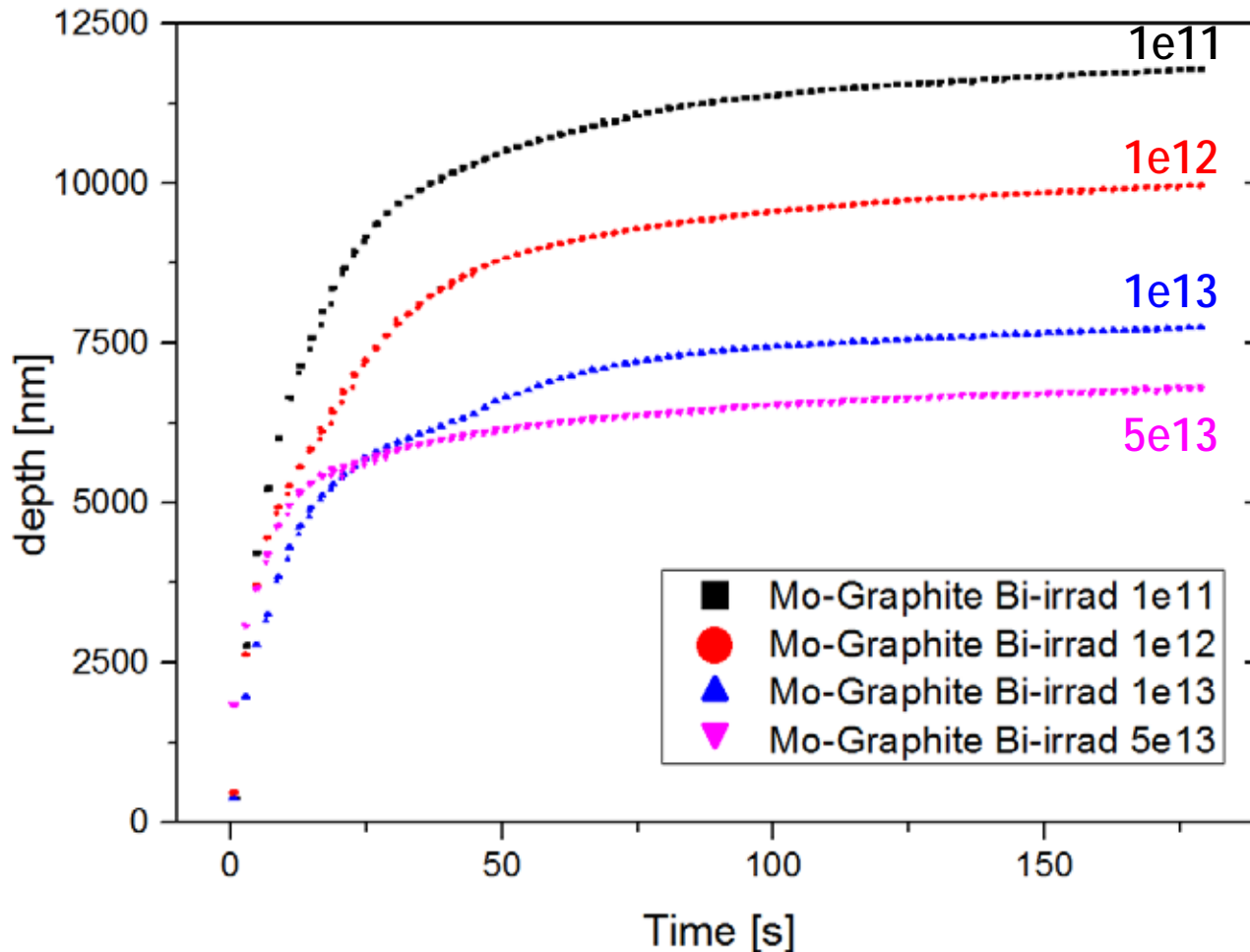
# Impact nanoindentation study of fatigue behaviour of irradiated isotropic graphite

## Cube Corner:

- 5 mN load,
- 28  $\mu\text{m}$  acceleration distance



# Impact/Woodpecker on Molybdenum Graphite



Irradiation:

Ion: Bi 27+

Energy: 4.8 MeV/u

Flux:  $2 \times 10^9$  ions/cm<sup>2</sup>s

Frequency: 2 Hz

Pulse length: 0.3 ms

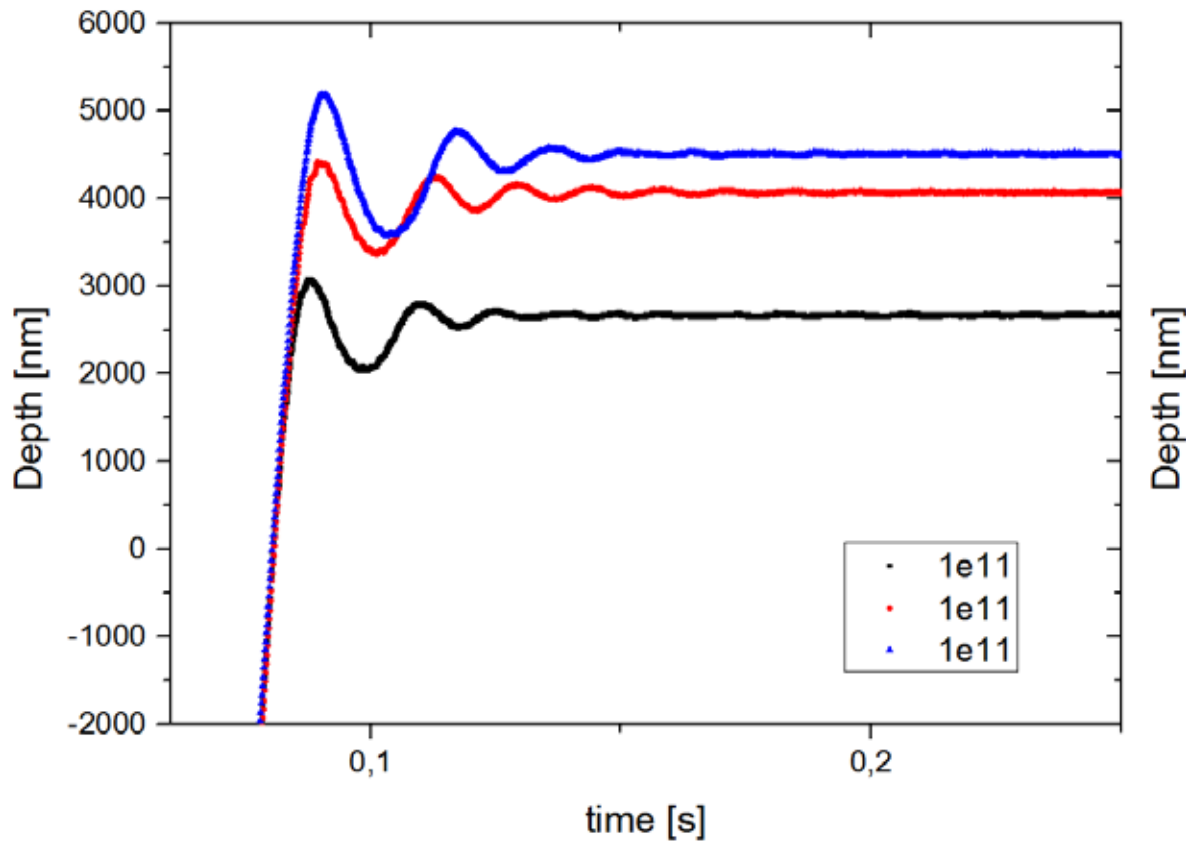
Impact frequency: 0.5 Hz

Impact load: 2mN

Impact distance: 15000 nm



# Impact/dynamic hardness on Molybdenum Graphite



Irradiation:

Ion: Bi 27+

Energy: 4.8 MeV/u

Flux:  $2 \times 10^9$  ions/cm<sup>2</sup>s

Frequency: 2 Hz

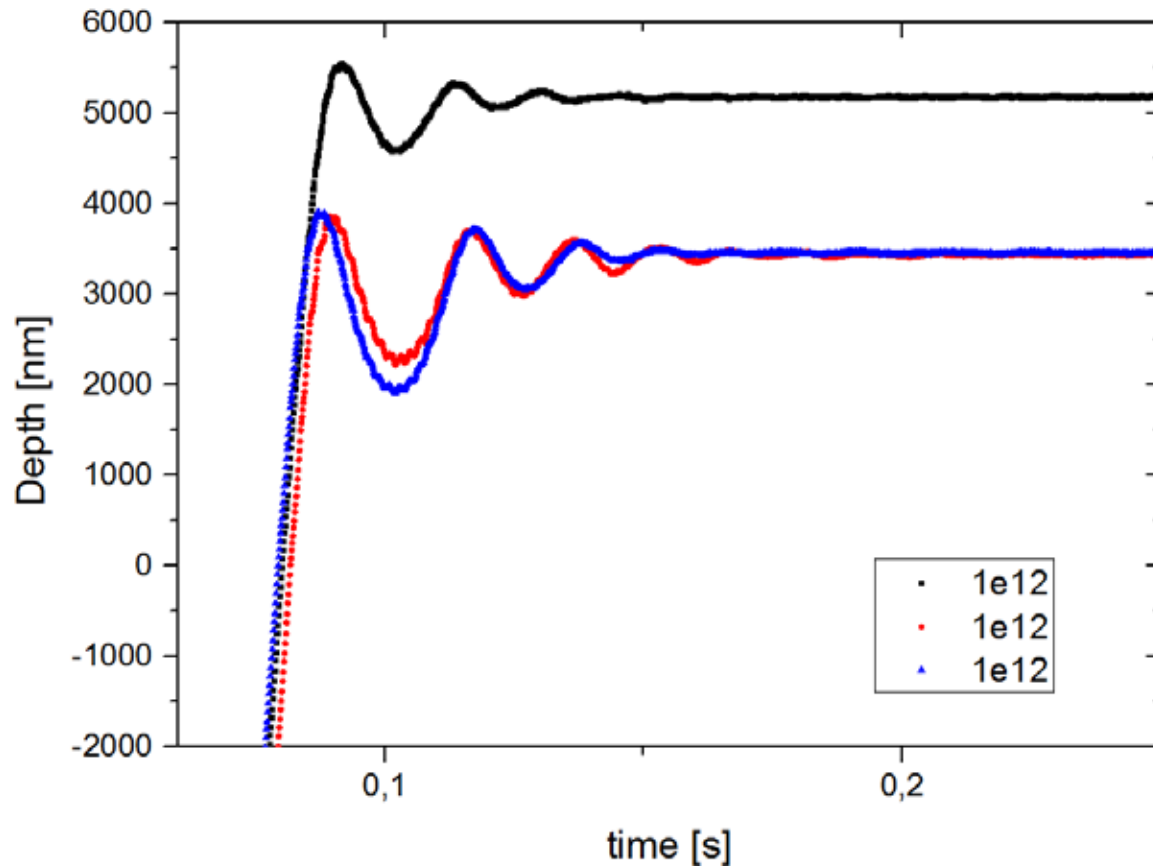
Pulse length: 0.3 ms

Sampling time: 0.5 s

Impact load: 2mN

Impact distance: 15000 nm

# Impact/dynamic hardness on Molybdenum Graphite



Irradiation:

Ion: Bi 27+

Energy: 4.8 MeV/u

Flux:  $2 \times 10^9$  ions/cm<sup>2</sup>s

Frequency: 2 Hz

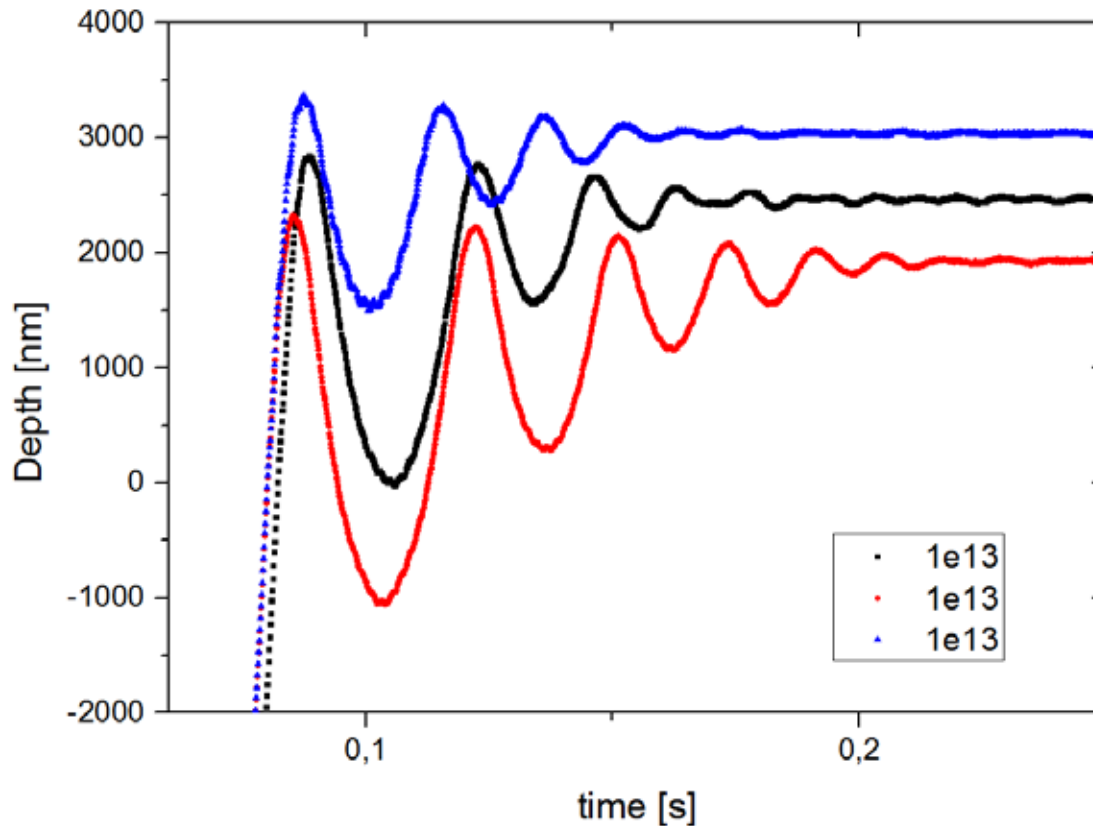
Pulse length: 0.3 ms

Sampling time: 0.5 s

Impact load: 2mN

Impact distance: 15000 nm

# Impact/dynamic hardness on Molybdenum Graphite



Irradiation:

Ion: Bi 27+

Energy: 4.8 MeV/u

Flux:  $2e9$  ions/cm<sup>2</sup>s

Frequency: 2 Hz

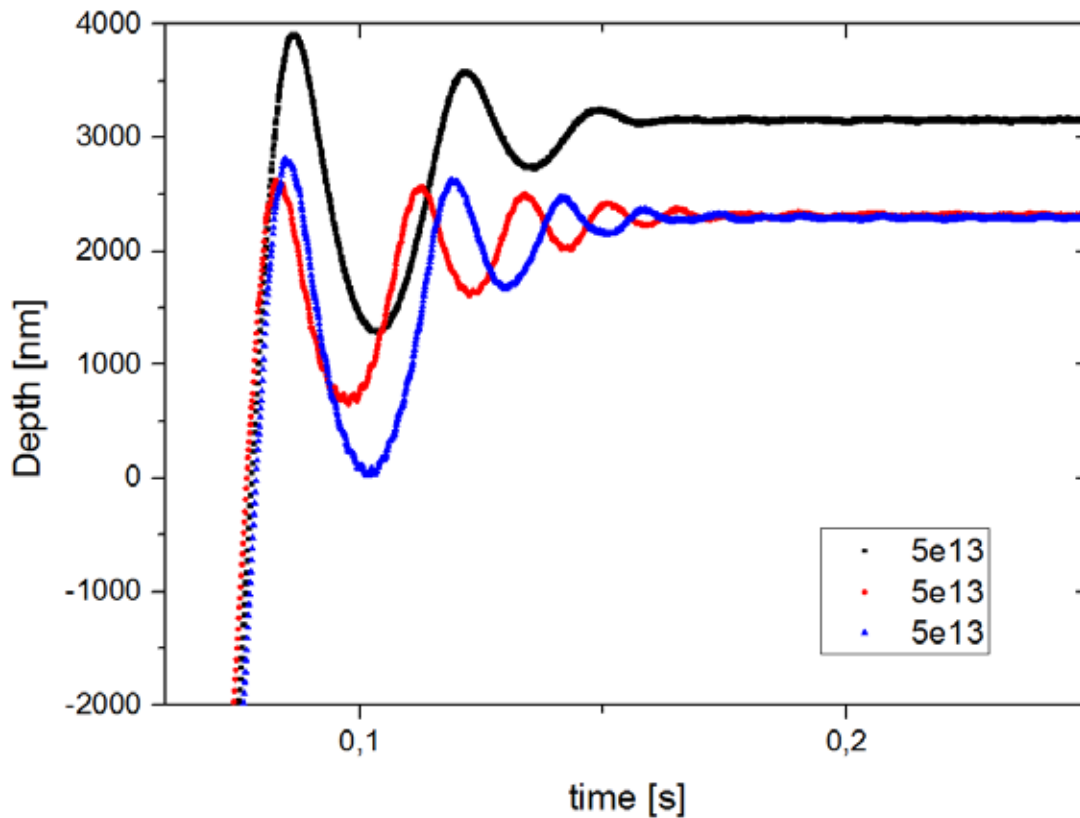
Pulse length: 0.3 ms

Sampling time: 0.5 s

Impact load: 2mN

Impact distance: 15000 nm

# Impact/dynamic hardness on Molybdenum Graphite



Irradiation:

Ion: Bi 27+

Energy: 4.8 MeV/u

Flux:  $2 \times 10^9$  ions/cm<sup>2</sup>s

Frequency: 2 Hz

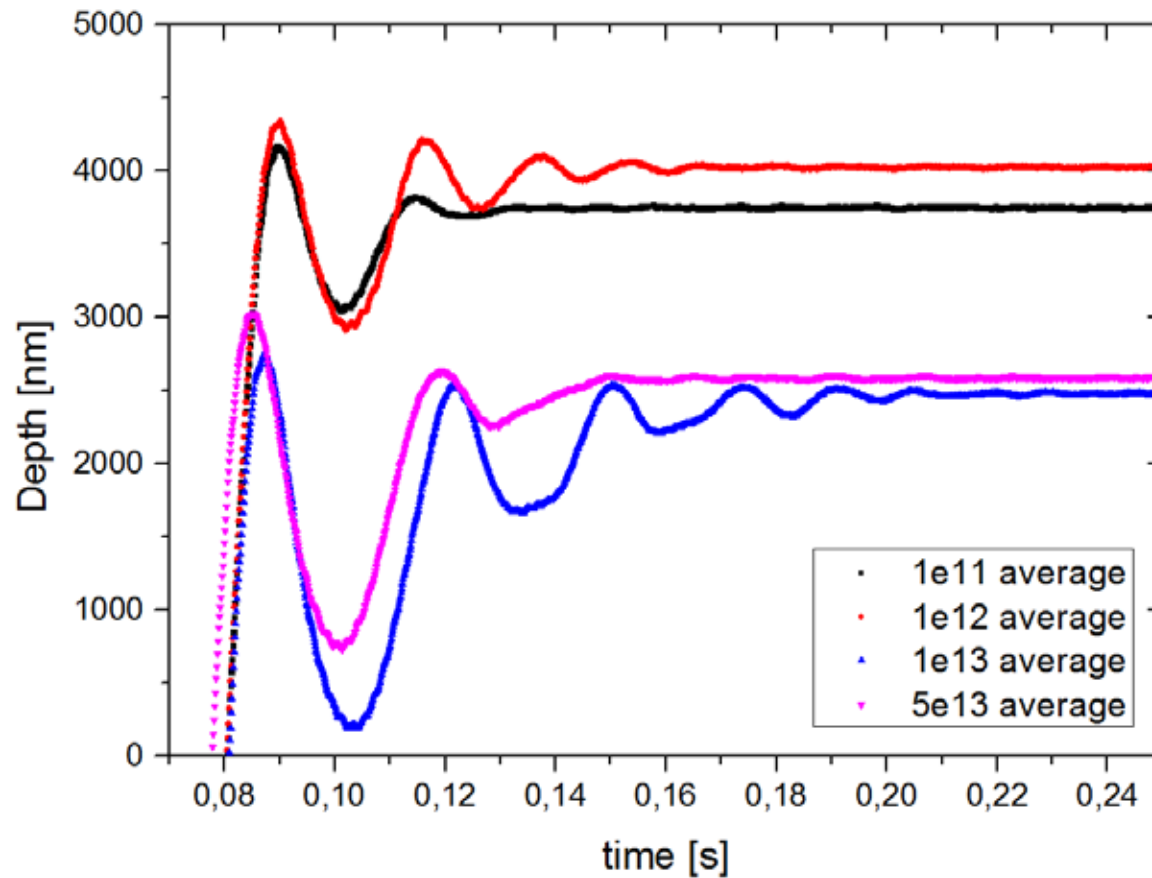
Pulse length: 0.3 ms

Sampling time: 0.5 s

Impact load: 2mN

Impact distance: 15000 nm

# Impact/dynamic hardness on Molybdenum Graphite



1e12  
1e11

Irradiation:

Ion: Bi 27+

Energy: 4.8 MeV/u

Flux: 2e9 ions/cm<sup>2</sup>s

Frequency: 2 Hz

Pulse length: 0.3 ms

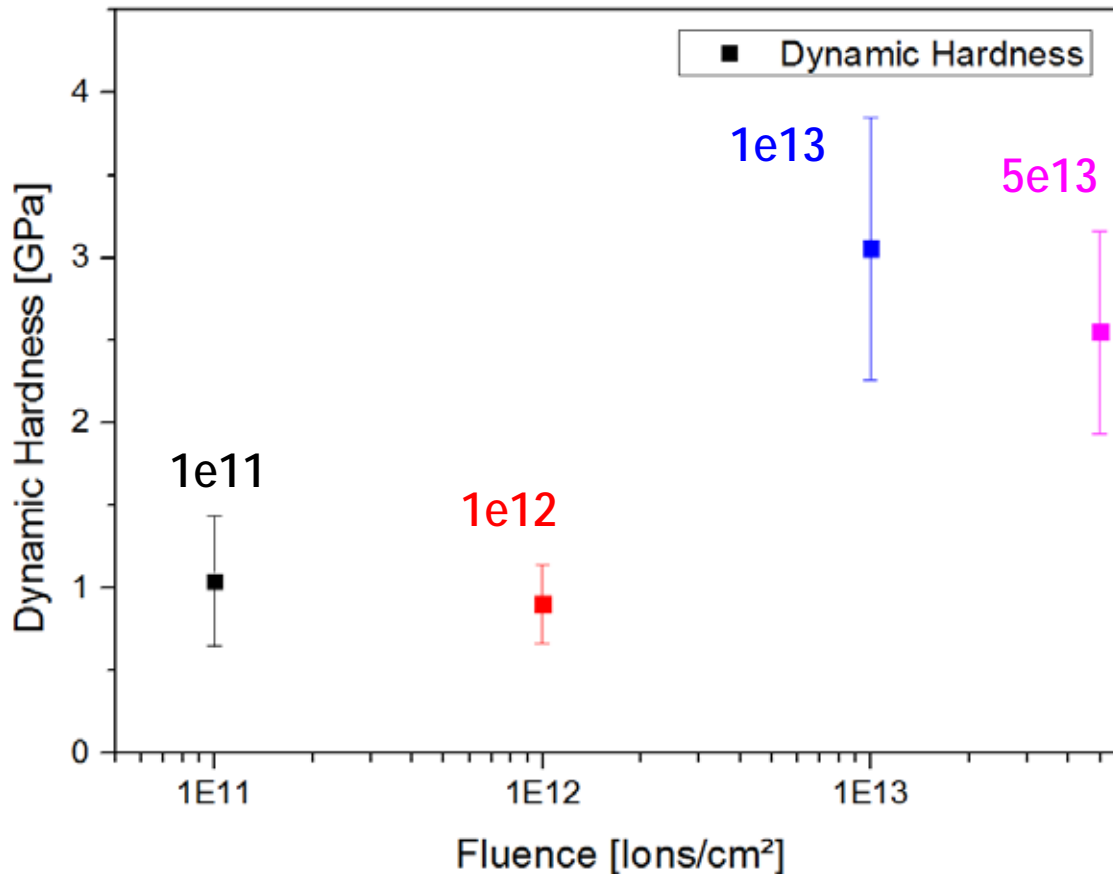
5e13  
1e13

Sampling time: 0.5 s

Impact load: 2mN

Impact distance: 15000 nm

# Impact/dynamic hardness on Molybdenum Graphite



Irradiation:

Ion: Bi 27+

Energy: 4.8 MeV/u

Flux: 2e9 ions/cm<sup>2</sup>s

Frequency: 2 Hz

Pulse length: 0.3 ms

Sampling time: 0.5 s

Impact load: 2mN

Impact distance: 15000 nm

# Conclusions and Outlook

- First irradiation tests of novel composite materials for LHC collimators at GSI
- For Mo-Gr the feed-back from irradiation results have been used to adjust the processing of the material to improve mechanical strength on transversal direction :
  - annealing at temperatures where vacancies are mobile in graphitic planes and stress relaxation
  - test samples without long pitch carbon fibers
  - in-situ heat treatments
- Completion of thermo-mechanical and structural characterization of irradiated samples
- analysis of online thermal camera monitoring with increased frame rate
- Impact response of the novel material pristine and irradiated (fast extracted ion beam, laser beam, and nanoindenter )

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Thank you for your attention!

