8th HL-LHC Collaboration Meeting

Characterization of graphitic materials for HL-LHC collimators: status and planning

C. Accettura (CERN and Politecnico di Milano)

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With input from WP5

CERN, October 16, 2018

Outline

- Introduction
  - Collimator material requirements
- Irradiation test for graphitic materials
  - Facilities overview
  - Design and planning of ion irradiation at GSI
- Pristine materials characterization
- Conclusions

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  - High electrical conductivity
  - Excellent thermo-mechanical properties
  - UHV compliance
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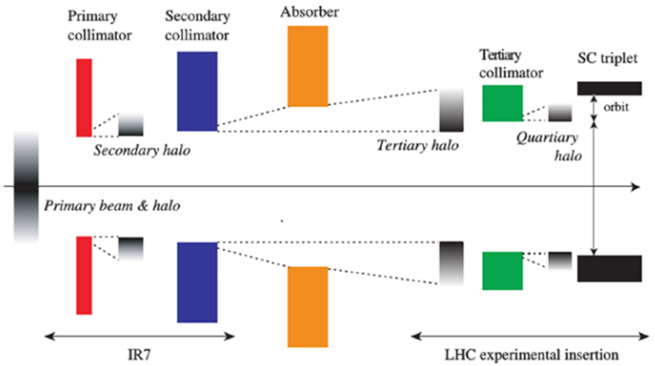
MoGr



MoGr with Mo coating

CuCD

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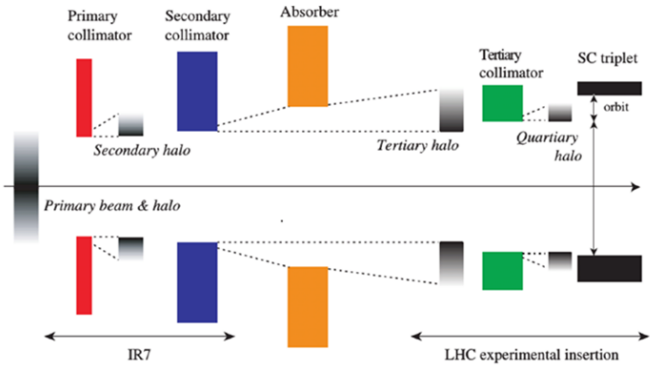
- Increased losses in the collimator system
- Important to assess the degradation of thermo-mechanical and electrical properties induced by radiation

MoGr



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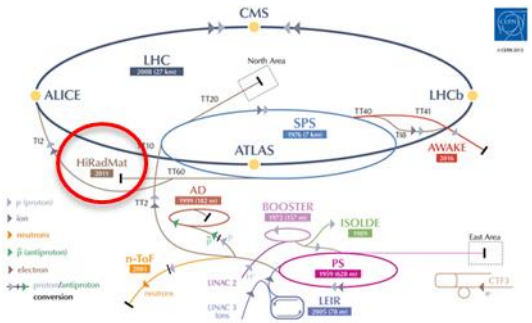
  - High energy density fast interaction→ dynamic thermo-mechanical response→ **influenced by material properties→ radiation damage**



CERN- HRMT beamlines

440 GeV protons

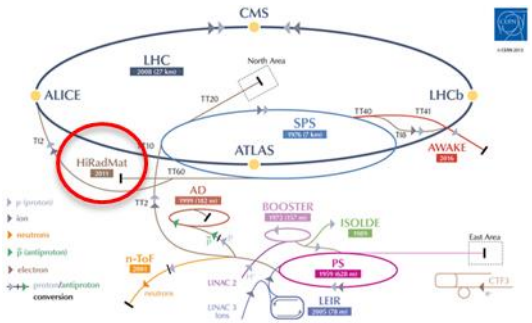
Irradiation test for HL-LHC collimators materials: facilities overview

- For beam-intercepting device, it is important to consider two phenomena:
  - High energy density fast interaction→ dynamic thermo-mechanical response→ **influenced by material properties→ radiation damage**
  - Long-term irradiation→ radiation damage and degradation of properties

1

2-3

1



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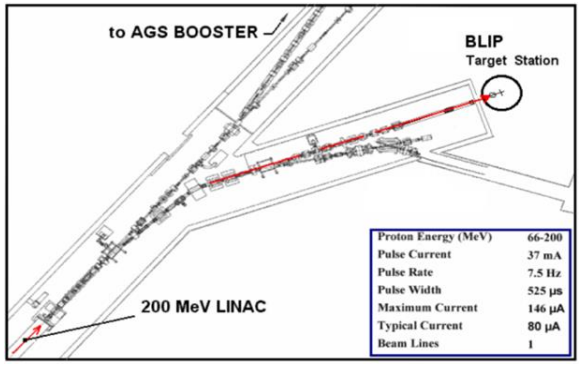
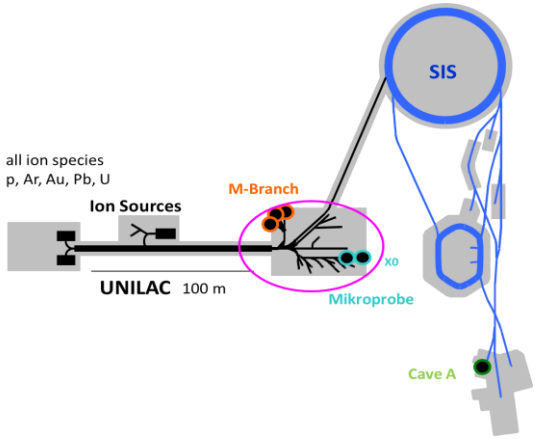


BNL beamlines
Max 200 MeV protons



GSI beamlines
~5 MeV/u ions

See M. Calviani talk

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- For beam-intercepting c… two phenomena:
  - High energy density fa… chanical response → **influence** **n damage**
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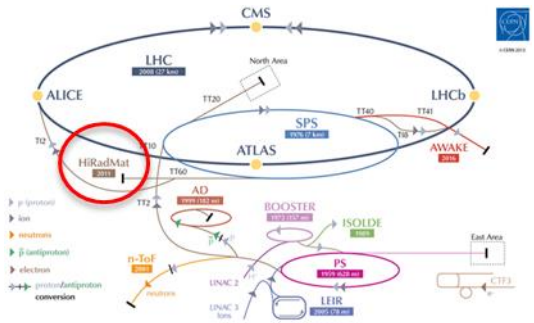
Penetration	Deep	Superficial
Gas production	Yes	No
Activation	High	Zero-low
DPA rate	Medium	High

1



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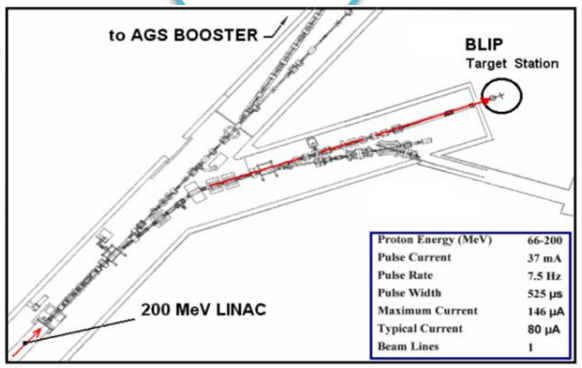

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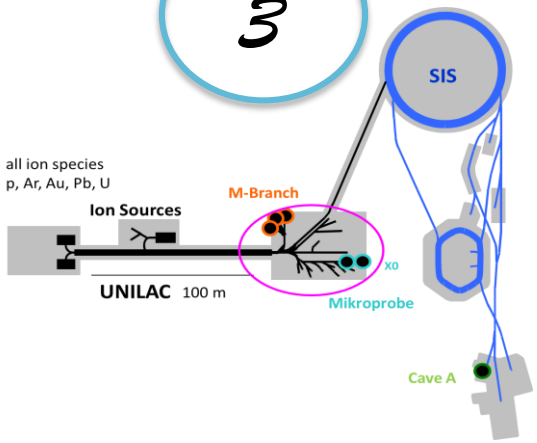
3



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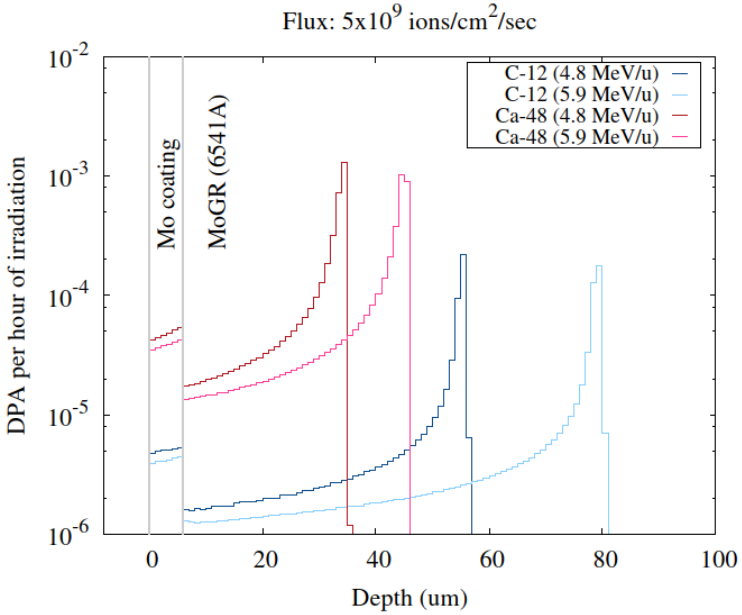
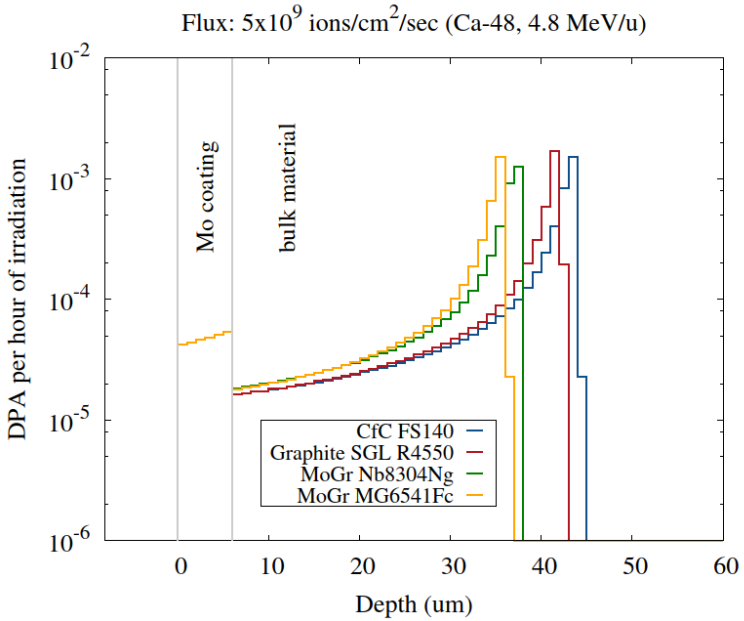
Ion irradiation at GSI: DPA simulation

- Light ions to minimize electronic stopping power
- Lower energy to minimize activation





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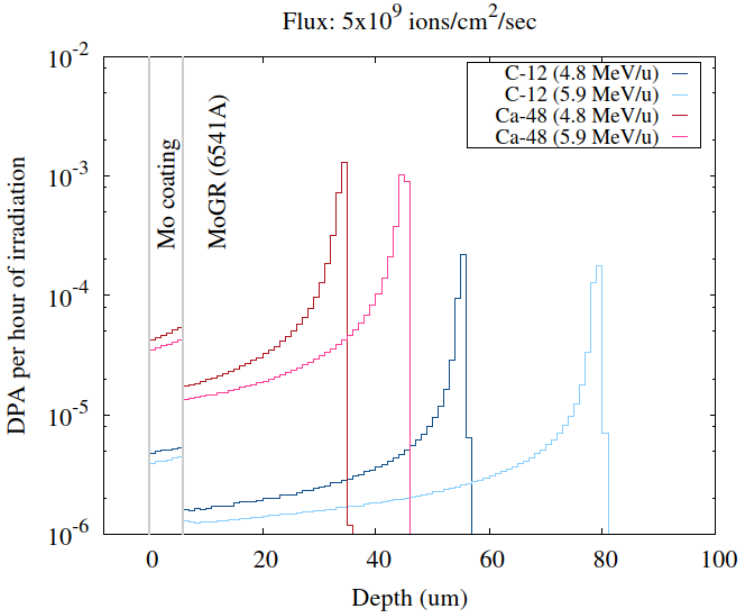

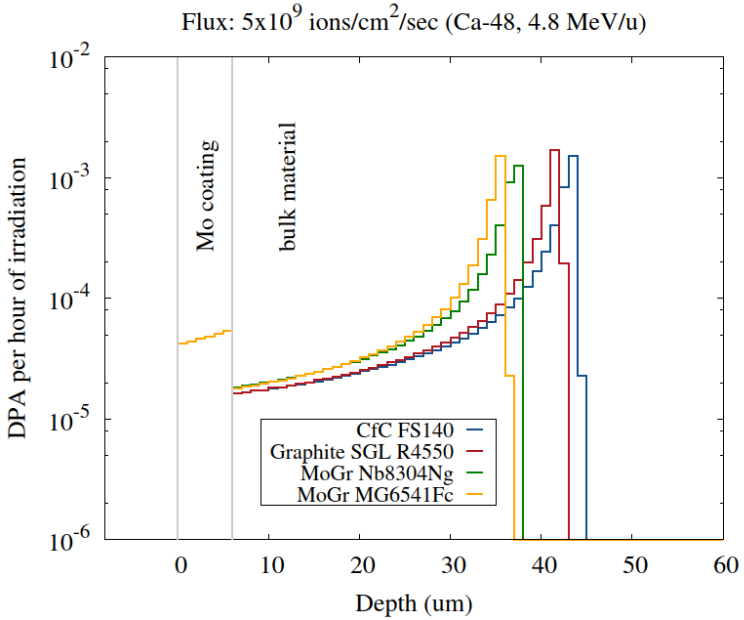

	DPA collimator HL-LHC life
Mo coating	$1 \div 3 \cdot 10^{-3}$
MoGr secondary	$4 \cdot 10^{-4}$
MoGr primary	0.3

	DPA/hour at GSI
Mo coating	$\sim 5 \cdot 10^{-5}$
MoGr	$\sim 1 \cdot 10^{-3}$

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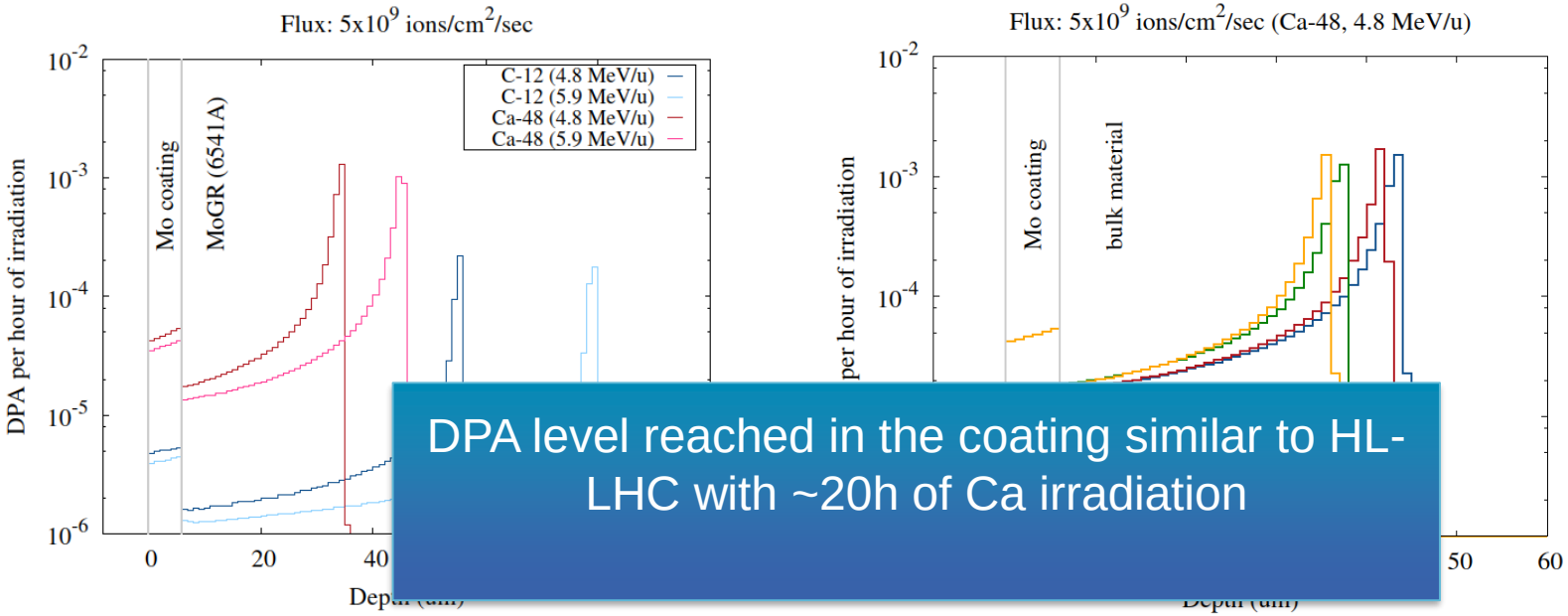

DPA level reached in the coating similar to HL-LHC with ~20h of Ca irradiation

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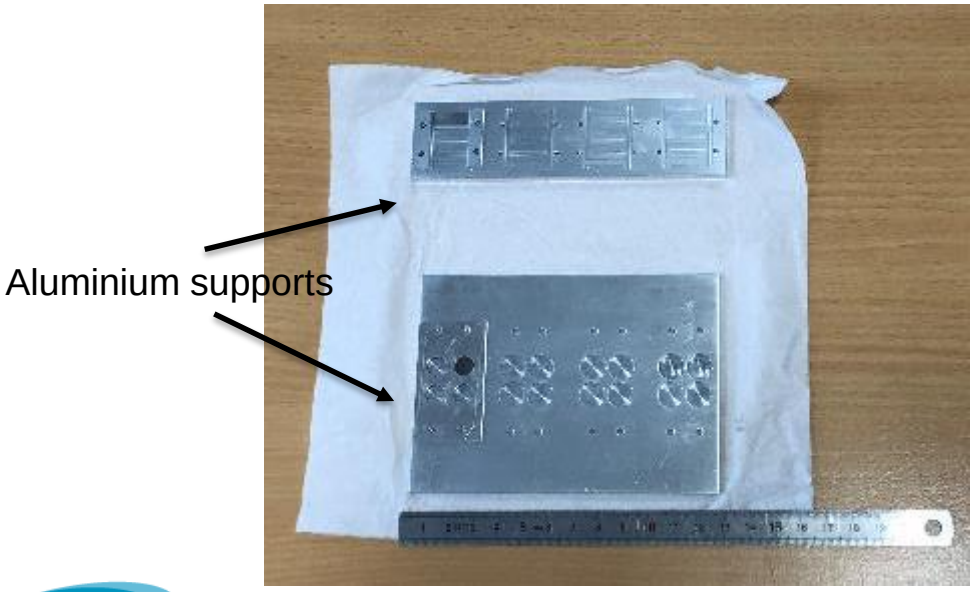
Ion irradiation at GSI: samples and holder

- With the available beam time, we can irradiate 4 holders
- 32 samples irradiated
- 4 materials: MoGr, MoGr (with C fibers), CFC, Graphite
- Each material will be irradiated bare and with a Molybdenum coating of 6 μm (done at CERN-TE/VSC)



- 2 holders for rectangular samples 20x5x0.150mm
- 2 holders for round samples D10x1mm

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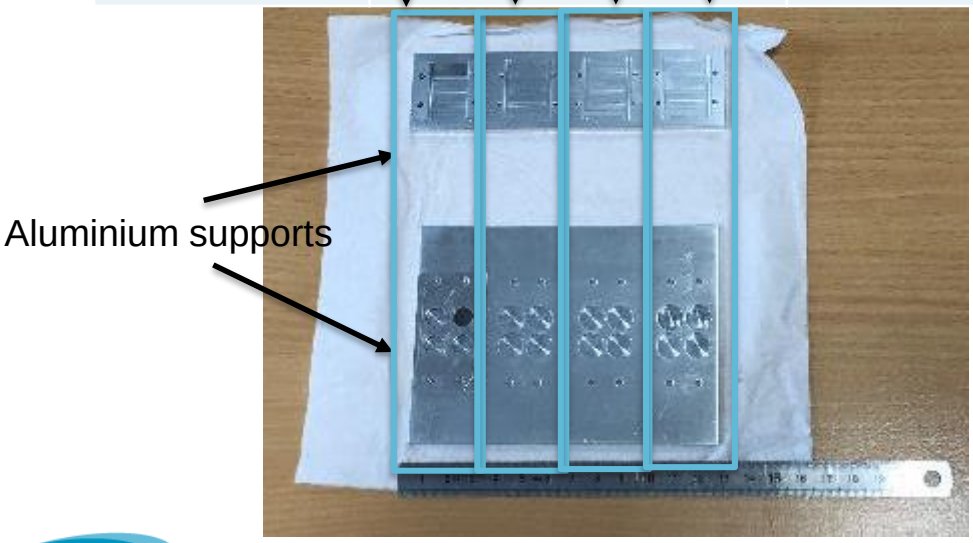
- [illegible]a Molybdenum

Irradiation time [hours]	Fluences [ions/cm^2]	DPA coating	DPA bulk
0.1	~1·10^{12}	~5·10^{-6}	0.0001
0.6	~1·10^{13}	~7.5·10^{-5}	0.0006
4	~7·10^{13}	~2·10^{-4}	0.004
20	~3·10^{14}	~1·10^{-3}	0.02



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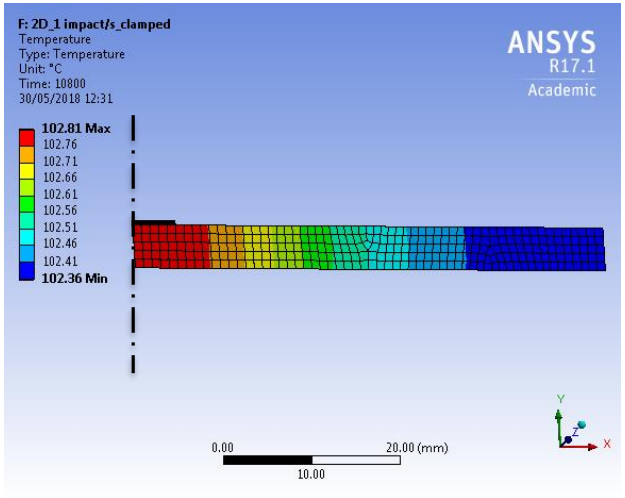
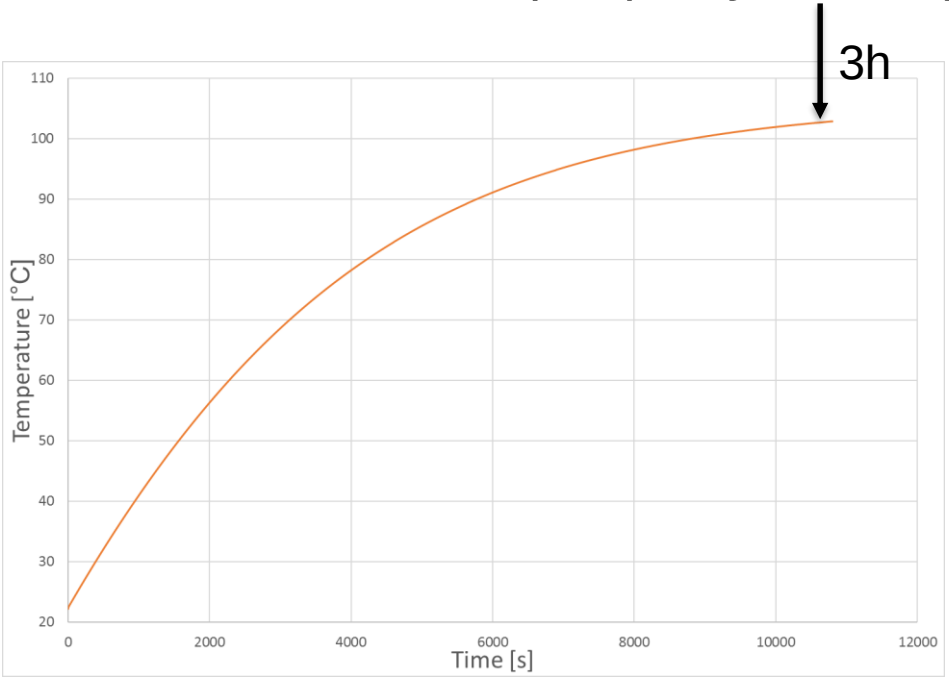
Ion irradiation at GSI: thermo-mechanical simulation

- Thermomechanical simulation of 1mm thick sample (axisymmetric)



Transient thermal analysis



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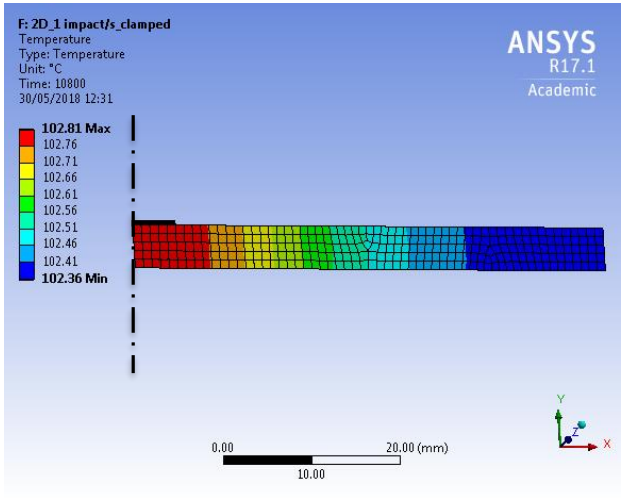

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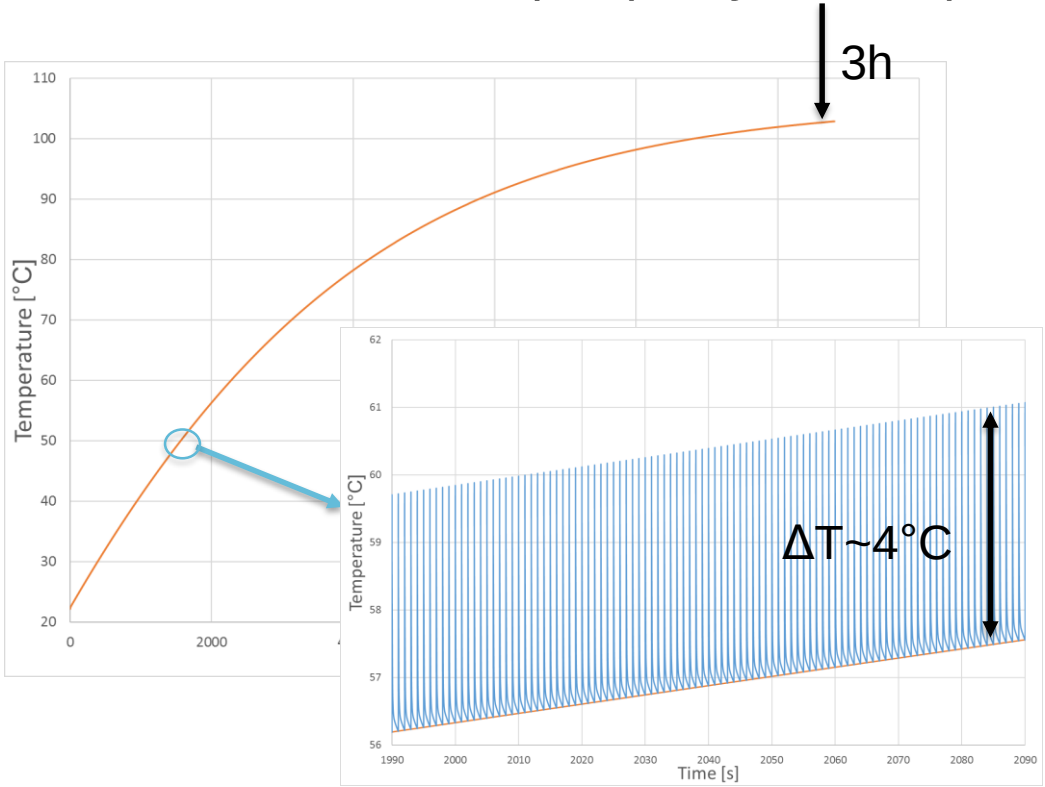

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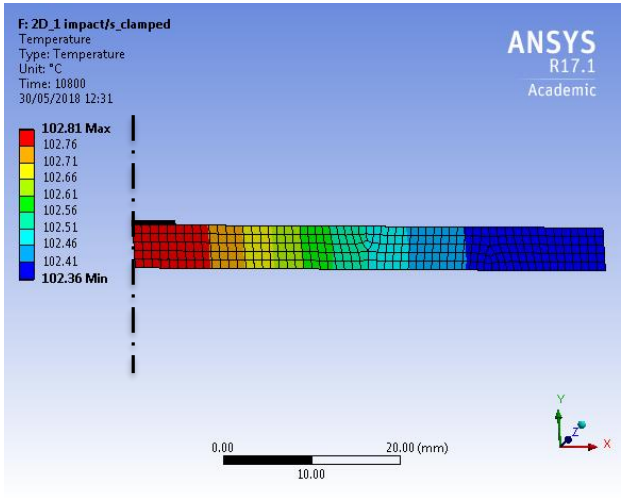

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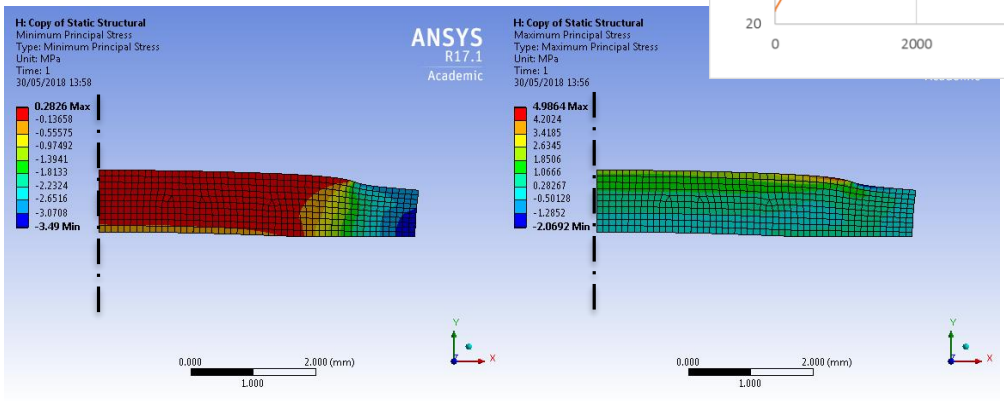
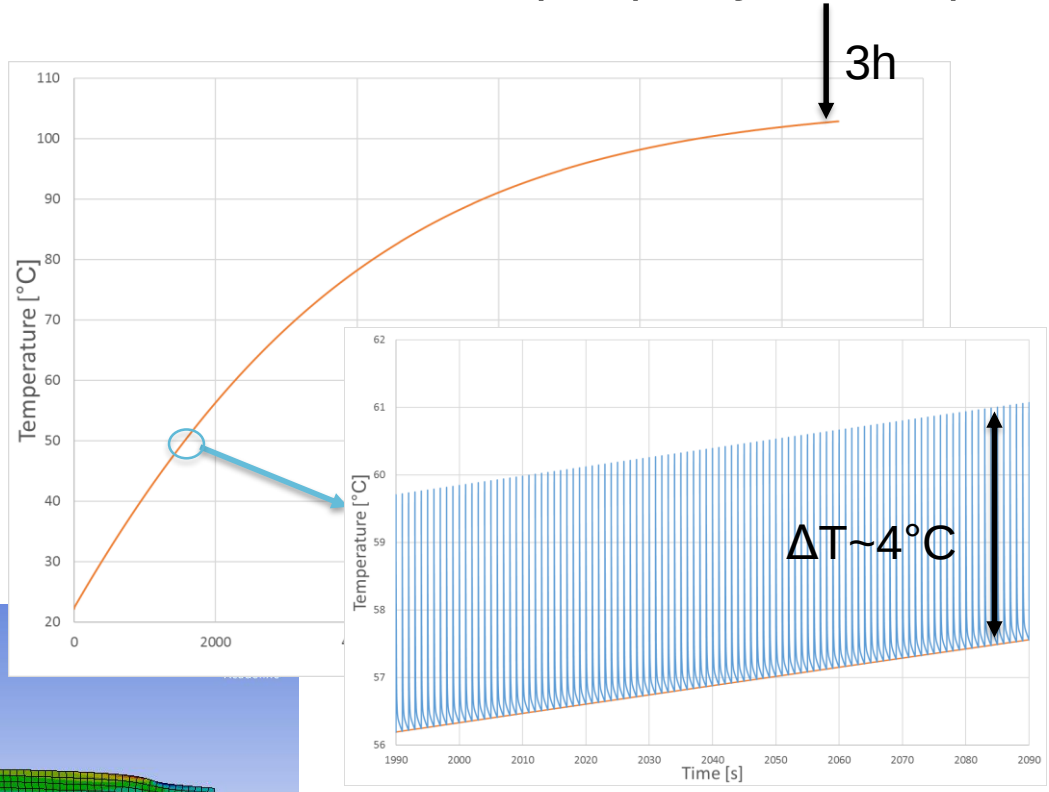


Structural analysis



- Tmax~100°C
- Maximum stresses (mainly due to clamping) far from material limits

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Material characterization

- One of the drawbacks of light ion irradiation is the small penetration depth (~37−45 μm)
  - Superficial technique
  - 2-layer model
  - Thin sample

- Material roughness
  - Impossible to use techniques based on sample reflectivity (thermoreflectance, Brillouin spectroscopy, substrate curvature)

Material characterization

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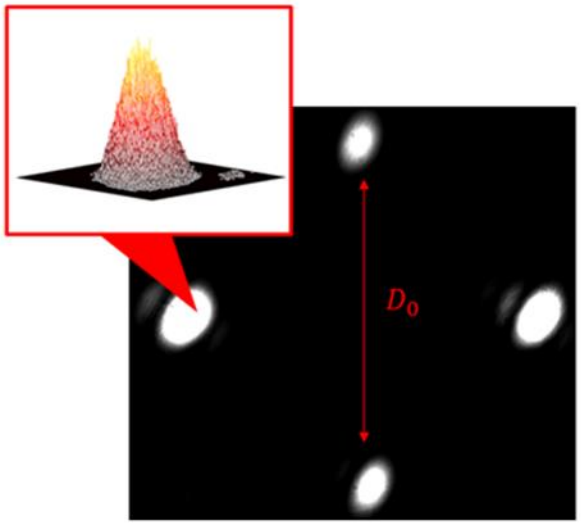
  - 2-l
  - Thin sample
- Mate
  - Ir vity (t curvature)

Substrate curvature method: No reflection of the beam (high roughness), diffusion forming a pattern→ impossible to determine D0 (the curvature)



Reflection by polished surface

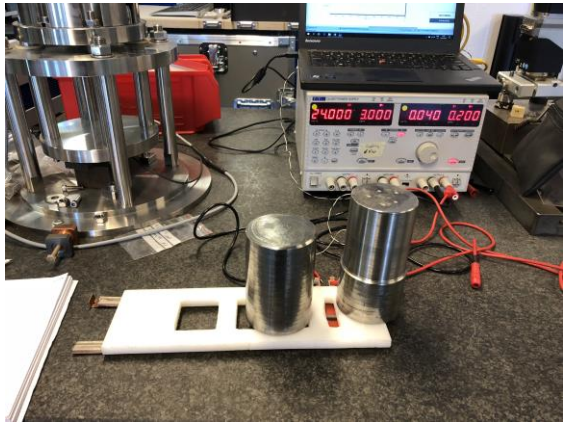
Pattern produced by Mo on MoGr

Courtesy of E. Besozzi (Energy Department, Politecnico di Milano)

Electrical conductivity-four probes method

Material	Grade	# sample measured	Average σ* [MS/m]	STDev [MS/m]	average uncertainty [%]
CFC	FS140	25	0.10	0.01	28
Gr	R4550	26	0.12	0.03	27
MoGr	Nb8304Ng	16	0.70	0.16	34
MoGr	MG6541Fc	18	1.23	0.34	26

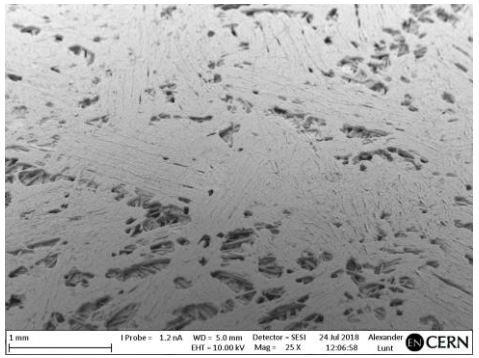
**pessimistic with respect to AC measurements (sample to small for that!)*



Four probes method set-up (CERN)

- The thickness was optimized in order to measure the thin Mo films by applying parallel resistance model→ the same sample measured before and after the coating

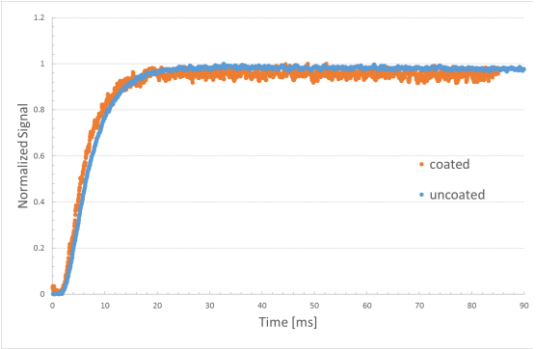
Material	Grade	# sample measured	Average σ [MS/m]	STDev [MS/m]	average uncertainty [%]
Mo on CFC	FS140	11	2.57	0.56	37
Mo on Gr	R4550	10	3.19	1.47	39
Mo on MoGr	Nb8304Ng	9	10.2	4.6	48
Mo on MoGr	MG6541Fc	9	3.68	1.48	68



Discontinuities on CFC coated surface

Thermal diffusivity-laser flash analysis

- Pristine characterization underlines that it is not possible to investigate the coating by applying a two-layers model→ negligible resistance



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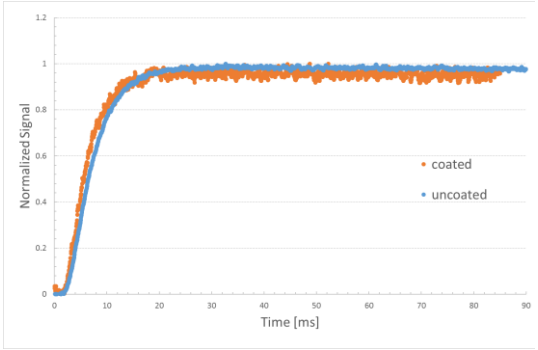
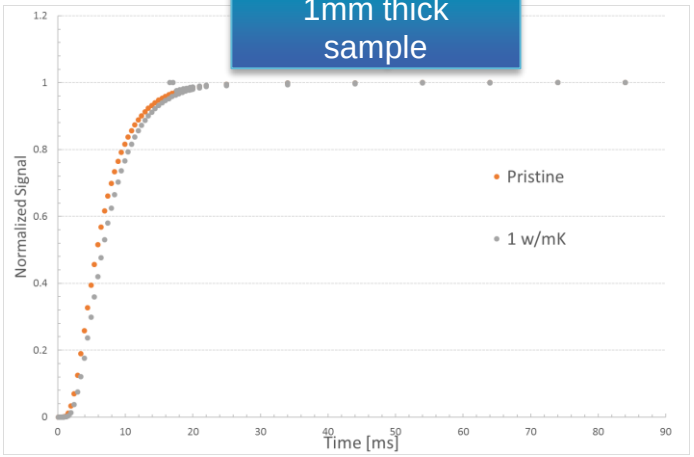
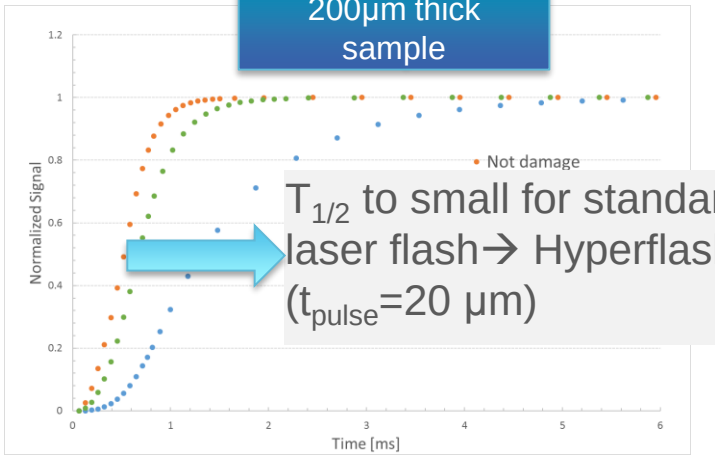


- If we assume 1mm thick sample, and 35 μm of ion penetration, the contribution of the damaged layer is so small that it is very difficult to see, even in the ideal case (simulation).

1mm thick sample



200μm thick sample



$T_{1/2}$ to small for standard laser flash→ Hyperflash (t_{pulse}=20 μm)

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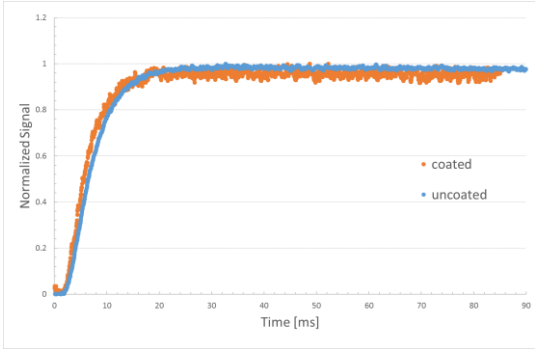

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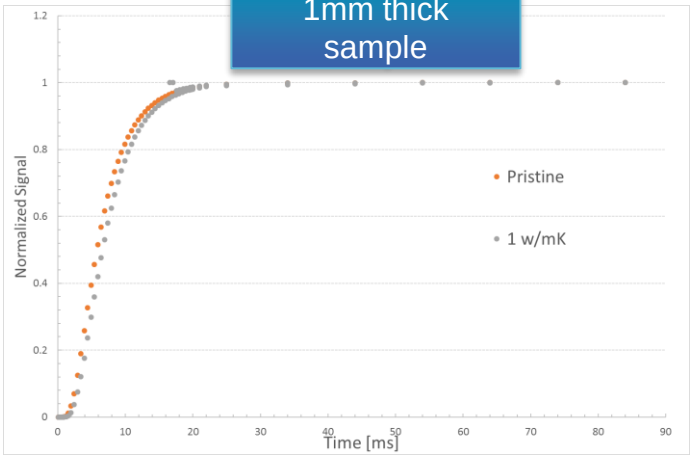

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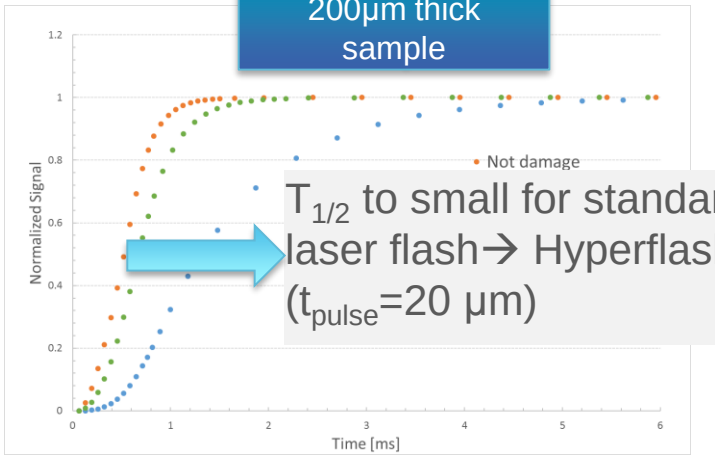
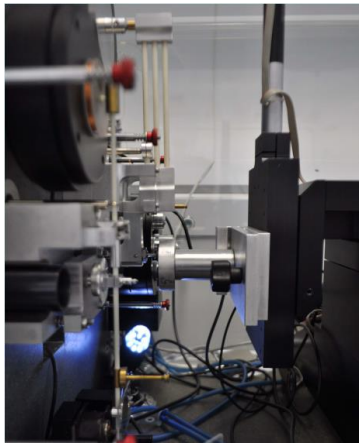


$T_{1/2}$ to small for standard laser flash→ Hyperflash (t_{pulse}=20 μm)

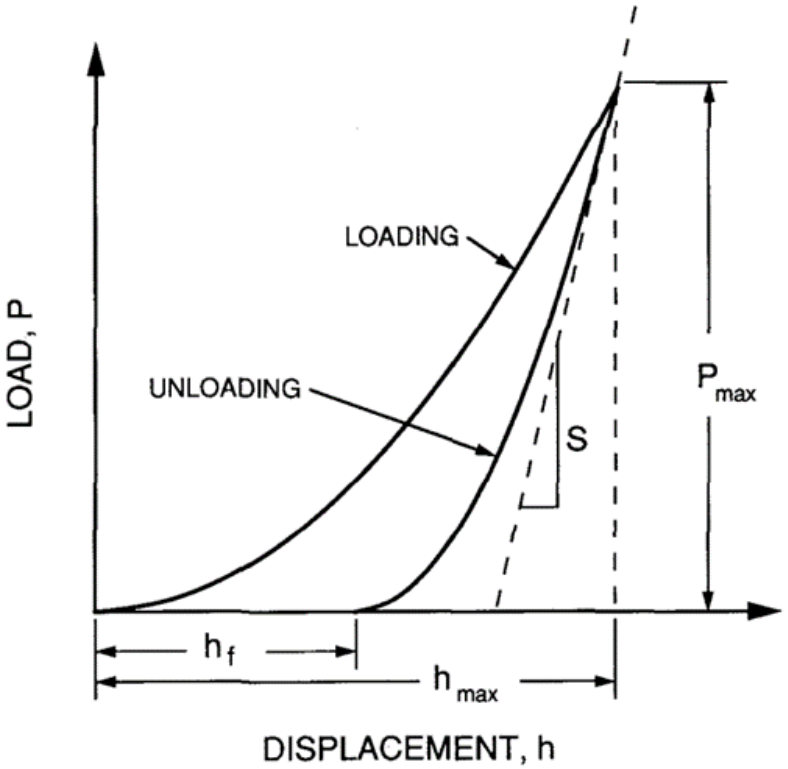
- Coating contribution still to small→ assume constant dependence from electrical conductivity (Wiedemann-Franz law)

Indentation−Bulk

- Each sample indented in 25 points with penetration-controlled (nano) and load controlled (micro)



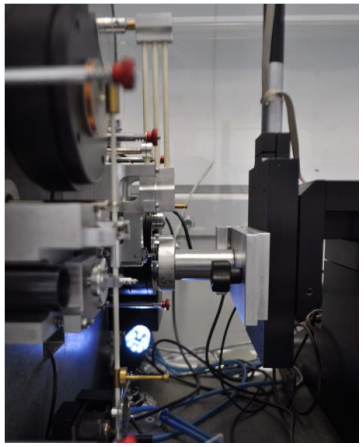
Nano-Micro indentation set-up (GSI)



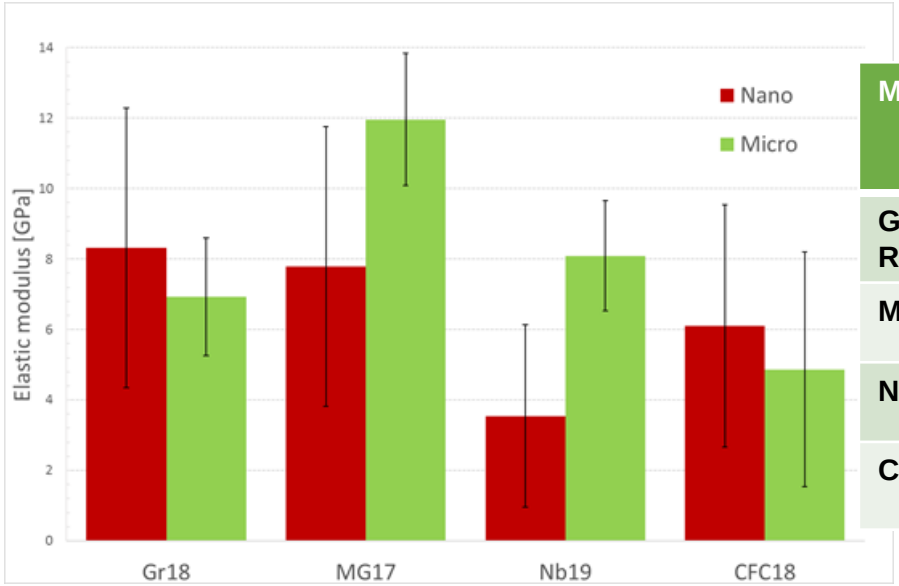
	Max load [mN]	Max depth [nm]
Nanoindentation	2	500
Microindentation	400	8000

Indentation–Bulk

- Each sample indented in 25 points with penetration-controlled (nano) and load controlled (micro)
- Microindentation allows a reduction of the standard deviation→ useful to observe radiation-induced hardening and increase of the elastic modulus



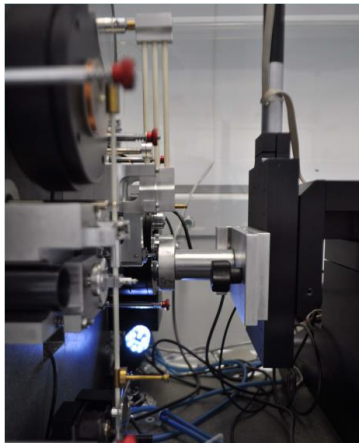
Nano-Micro indentation set-up (GSI)



Material	Average E [GPa]	Standard deviation [GPa]	IET [GPa]
Graphite R4550	6.9	1.7	11
MG6541Fc	12	1.9	5
Nb8304Ng	8.1	1.6	4
CFC FS 140	4.9	3.3	3

Indentation–Bulk

- Each sample indented in 25 points with penetration-controlled (nano) and load controlled (micro)
- Microindentation allows a reduction of the standard deviation→ useful to observe radiation-induced hardening and increase of the elastic modulus



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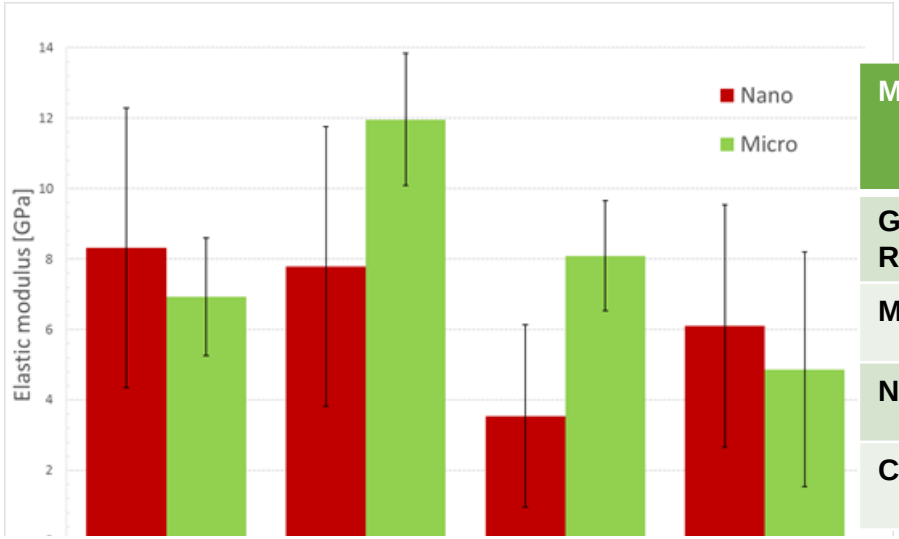

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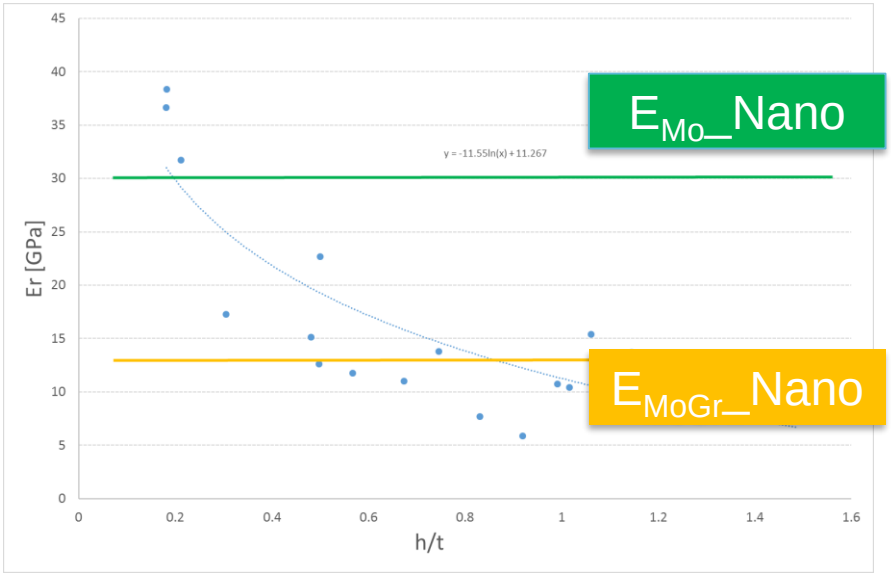
- Differences with respect other method (e.g. IET) related to factors such as anisotropy and non-linearity.

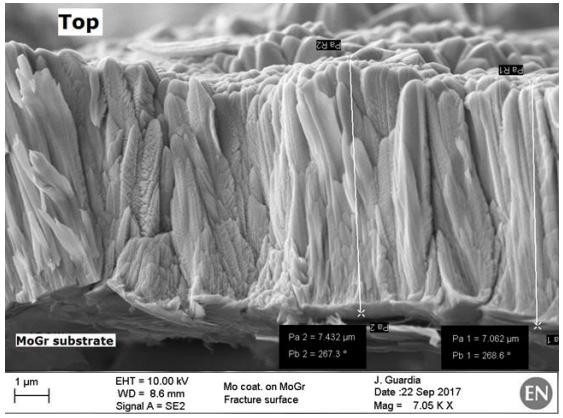
Indentation–Coating

- The substrate influences the indentation modulus of the coating if the penetration depth is 1/10 of the film thickness→ extrapolate the data to h/t=0.1



Material	Grade	$E_{reduced}$ Extrapolated [GPa]
MoGr	MG6541Fc	38
CFC	FS140	29
Gr	R4550	46
MoGr	Nb8304Ng	34

- Differences with respect Mo bulk value probably related to coating anisotropy.



Courtesy of J. Guardia

Coating adhesion-Scratch test

- An increasing load is applied by the nanoindenter, possible coating failure can be detected with:

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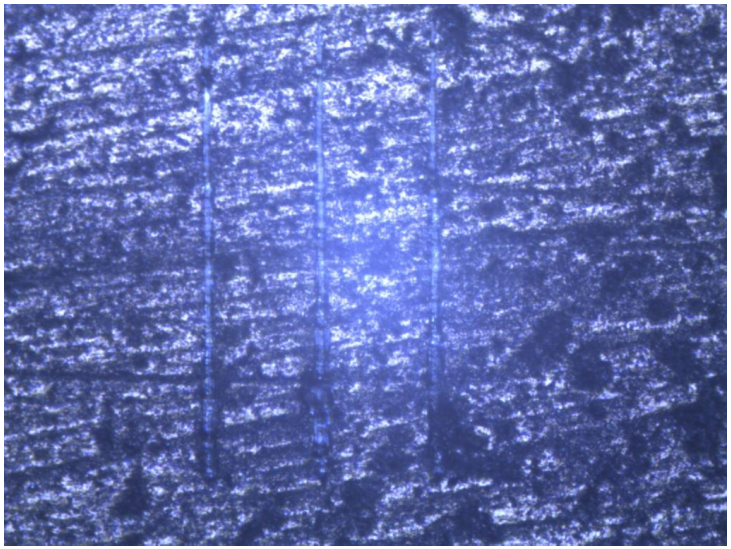
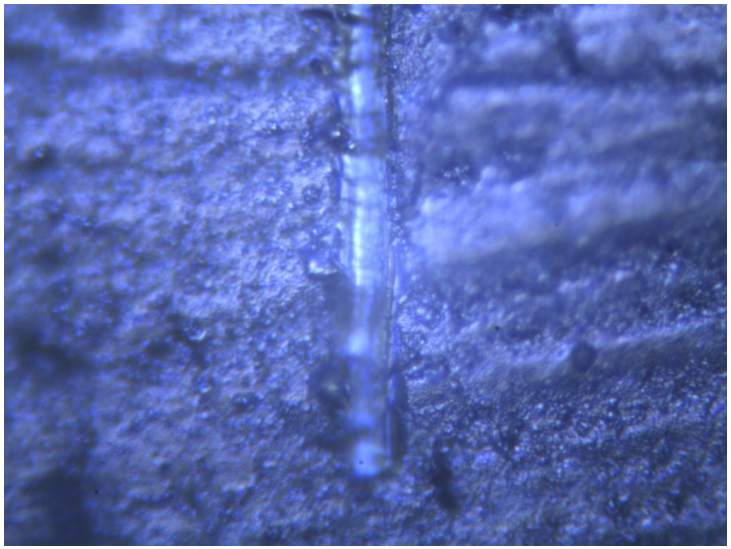
  - Optical microscopy images→ difficult for material roughness and coating/bulk colours





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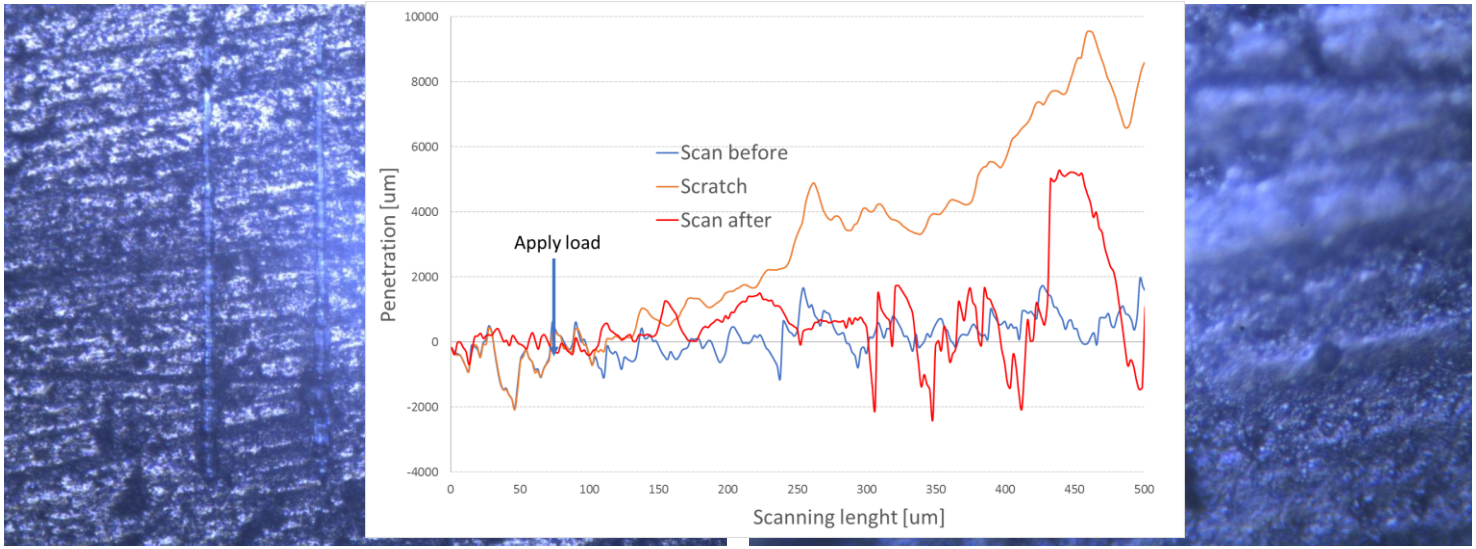

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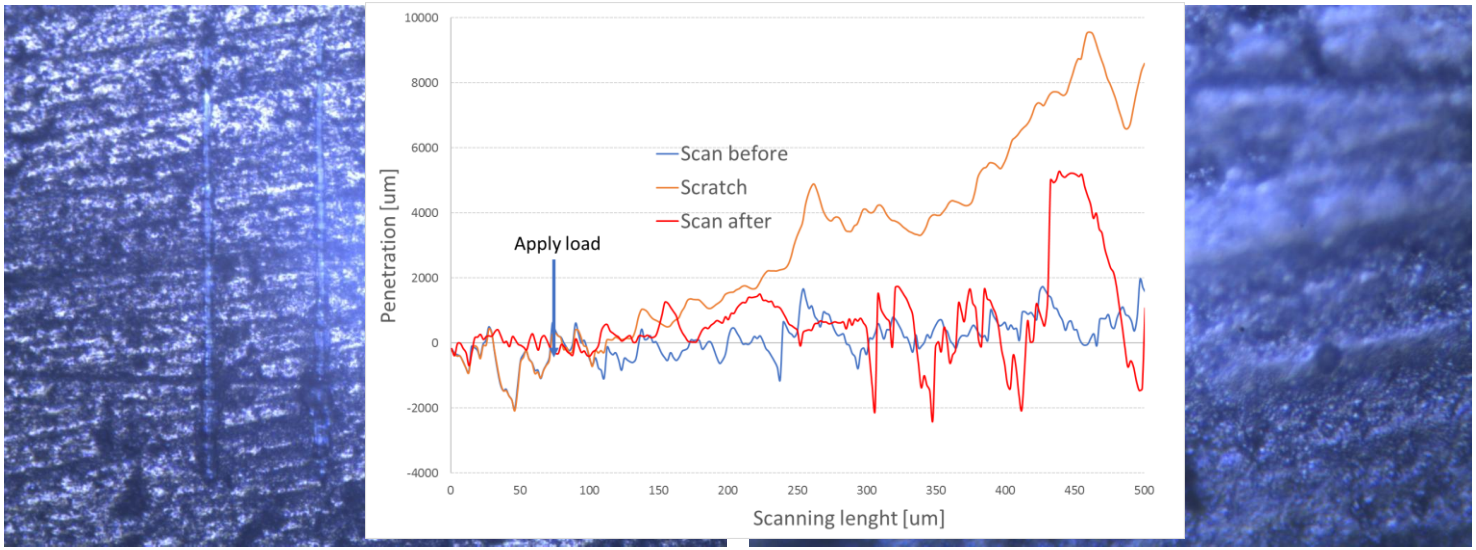
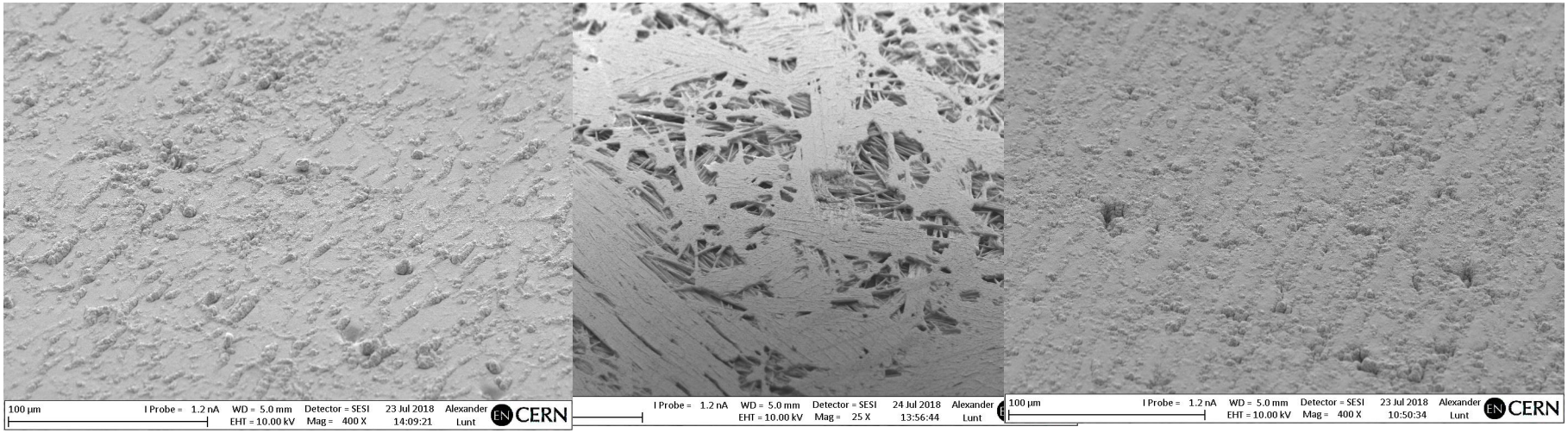
  - Optical microscopy images→ difficult for material roughness and coating/bulk colours
  - Abrupt variation in the load curve→ irregularities due to roughness already present
  - Acoustic emission→ not available in the used set-up



Microscopic characterization

Focused Ion Beam (FIB)

- Important to check bulk-coating interface and coating microstructure
- Qualitative assessment of coating adherence



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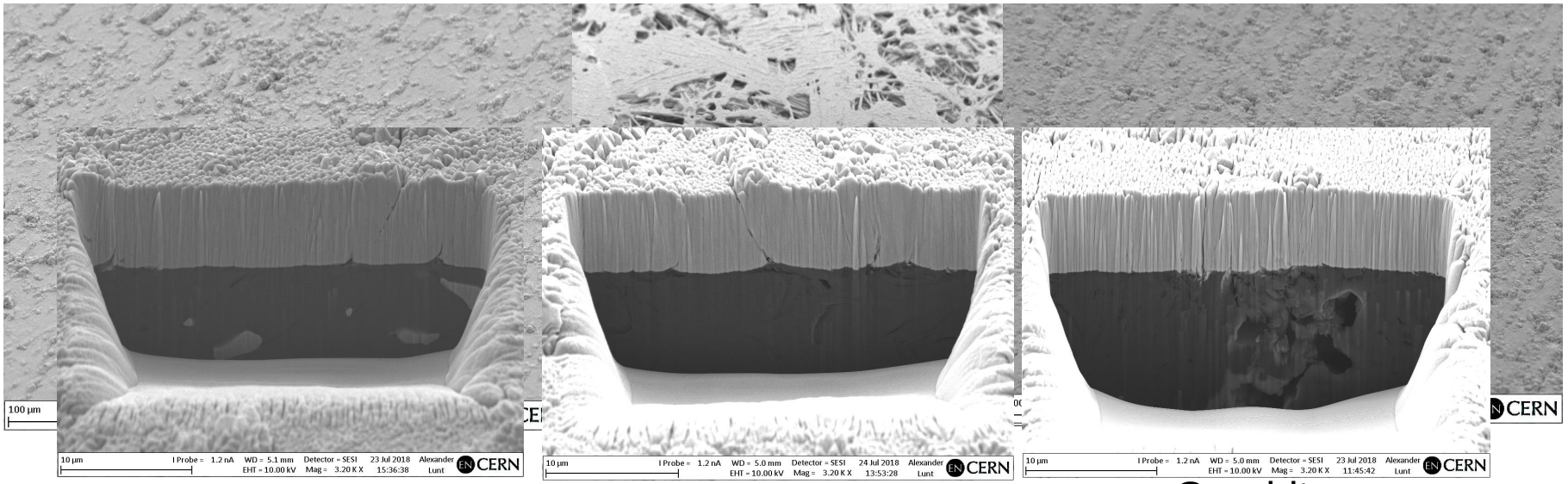

MoGr CFC Graphite

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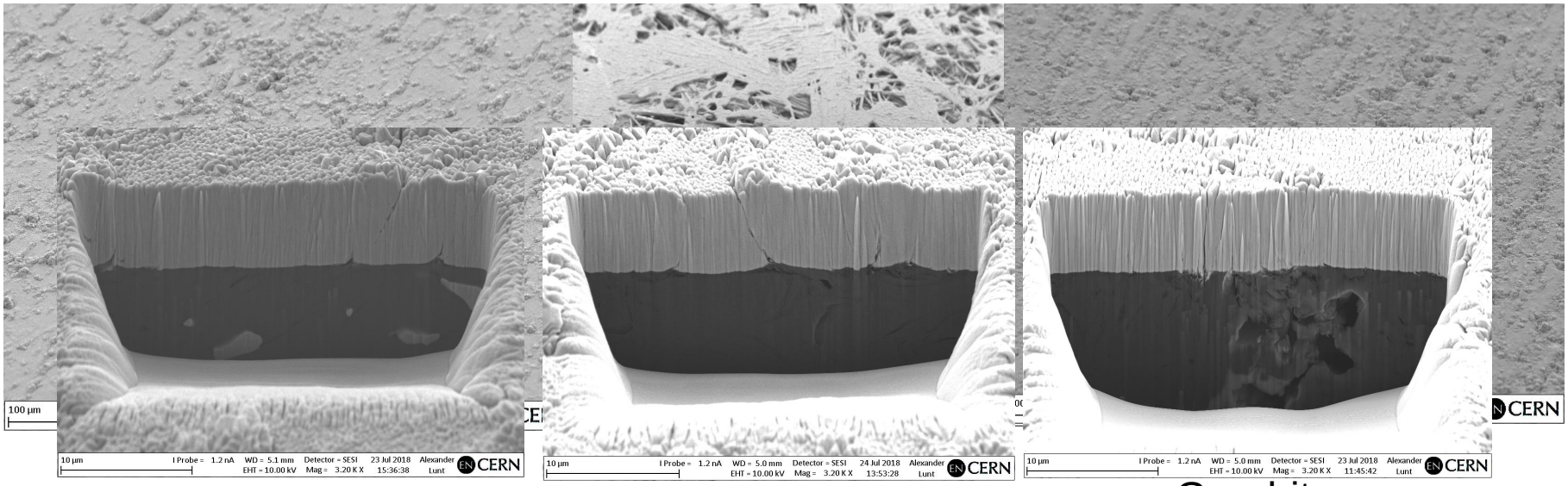


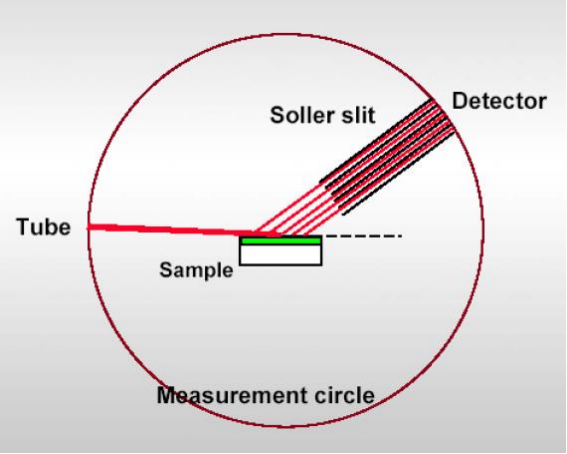
MoGr CFC Graphite

- Very local→ important to check the same milling before and after irradiation
- Milled location will see different irradiation condition→ the milling is repeat after irradiation

Other measurements (ongoing)

X-rays diffraction

- Useful to investigate radiation-induced changes of phase, crystal lattice parameter, grain size
- Penetration range of x-rays in graphite >> ion range→ use thin-film XRD configuration to detect only the damaged surface



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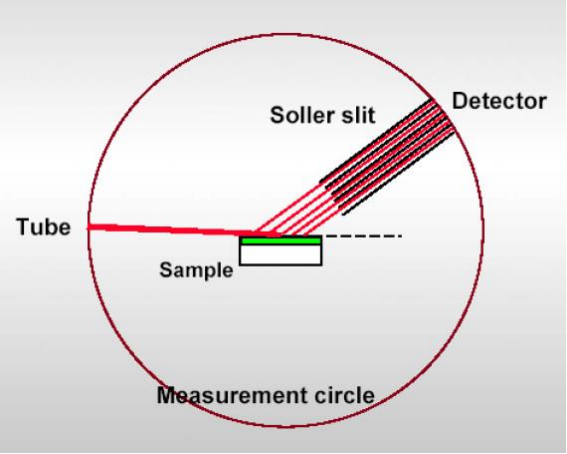

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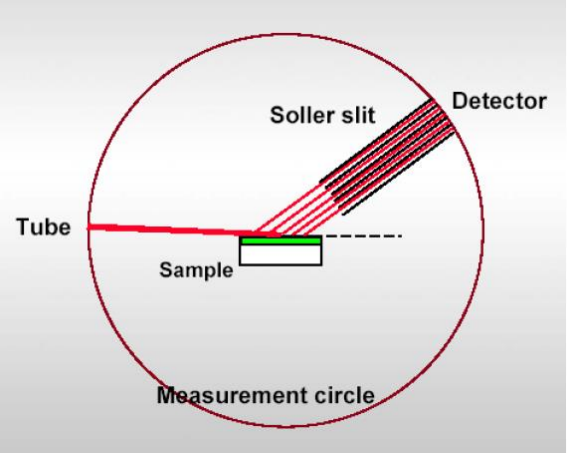

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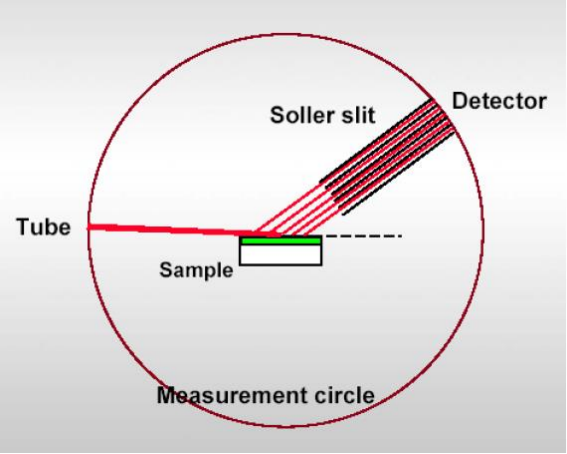

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X-rays photoelectron spectroscopy (superficial)

- Investigation of chemical changes

Thermal desorption spectroscopy

- Desorption behaviour modified by microstructural changes induced by radiation

Conclusions

- Radiation effects on materials for BID must be checked under different conditions:
  - Material functionality after high-energy fast interaction→ deep characterization will be launched **first half next year** on a wide set of materials and coating (HRMT36)
  - High-energy proton irradiation at BNL→ See M. Calviani presentation
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  - Small penetration
    - Thin sample
    - Superficial measurement techniques
  - No gas production
    - Comparison with p-irradiated material data + theoretical correlation ?

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- Low-no activation of the sample→ extensive characterization and cheap

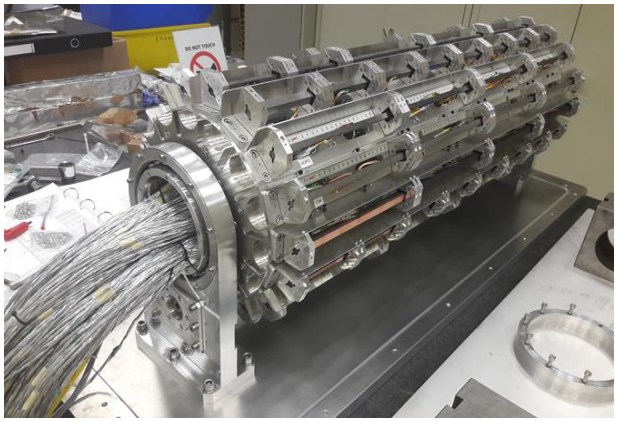
Acknowledgements

- Mechanical Measurements Lab of EN-MME (CERN)
- Metallurgy Lab of EN-MME (CERN)
- Surface preparation and coating team of TE-VSC (CERN)
- Impedance measurements (BE-ABP)
- Material research group (GSI)

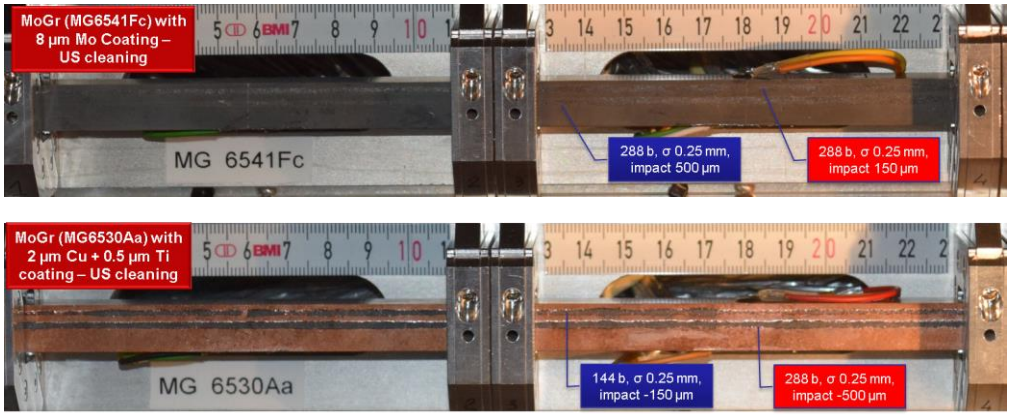
Thank you for your attention!

MultiMAT: HRTM36 experience

- HRTM36: assessment of thermo-mechanical response of 18 materials, including metallic coatings (Mo, Cu, TiN) on CFC, MoGr, Graphite



HRTM36 rotating barrel

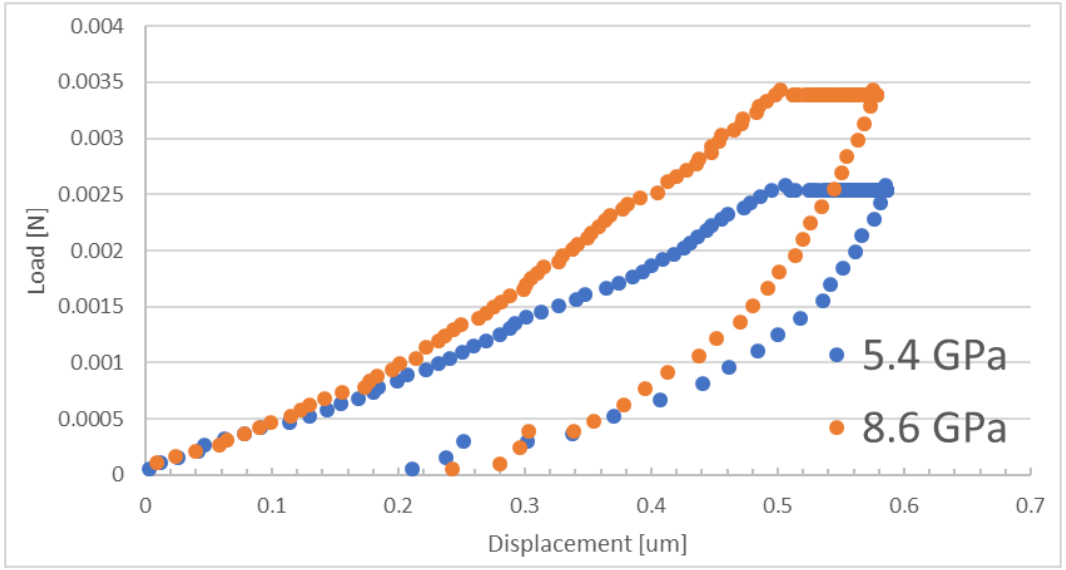


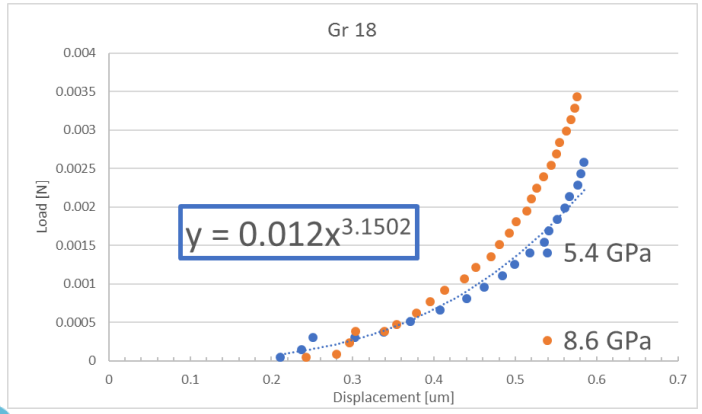
Impacted Cu and Mo coating on MoGr

- Activation level (October 2018) 210µSv/h (contact)
- Planned opening first half of 2019
- Possible PIE at CERN: electrical conductivity , topography, indentation, scratch test

Indentation-Graphite

- The software uses the Oliver-Pharr method to find the stiffness.





Interpolation of the unloading curve with a power function:

$$P = A(h - h_f)^m$$

$$S = \left(\frac{dP}{dh}\right)_{h_{max}}$$

$$h_c = h_{max} - \varepsilon \frac{P_{max}}{S}$$

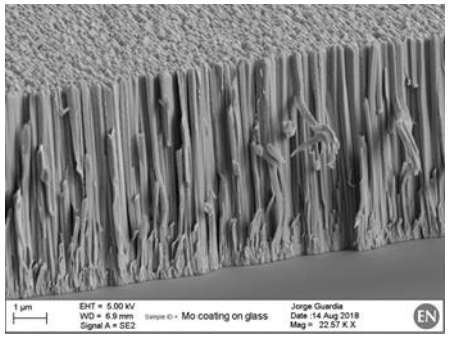
$$E_{reduced} = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{24.5 h_c^2}}$$

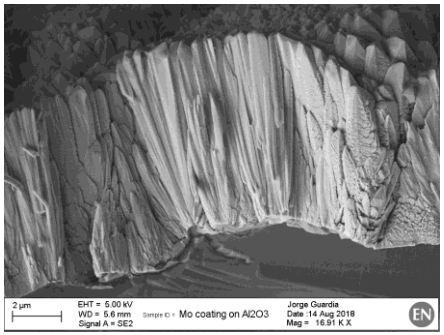
Point	Esoftware [GPa]	Ecurve * [GPa]
1	5.4	9.2
2	8.6	12.2

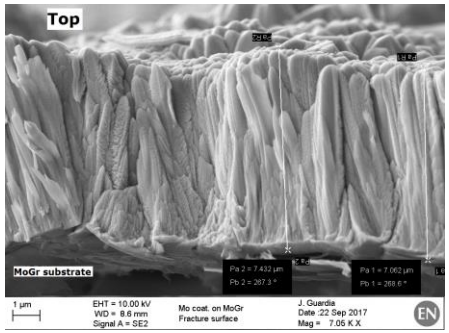
Coating electrical conductivity

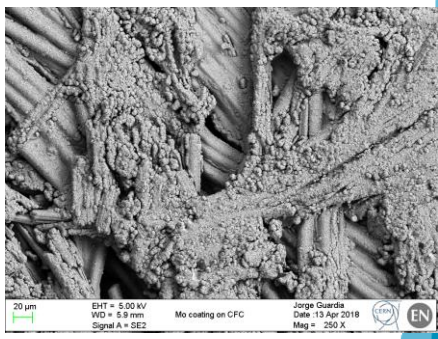
- The resistivity of the coating is affected by the combination of grain size and defects (discontinuities).

	Substrate roughness	Mo grain size (average)	Amount of coating discontinuities	Coating conductivity (MS/m)		Coating resistivity (nΩ.m)
Glass	~0	+	no	+ 😐	4.3 [DC] 5.0 [RF]	232 [DC] 200 [RF]
Alumina	+++	++	++	+ 😐	4.6 [DC] 4.1 [RF]	218 [DC] 244 [RF]
MoGr	+	++	+	+++ ☺	- 14.3-16.7 [RF]	- 60-70 [RF]
CFC	++++	++	(big voids)	- ☹	n.d. (≈substrate)	n.d. (≈substrate)









Courtesy of J. Guardia EN/MME
Impedance meeting 24-08-2018