



Future irradiation experiment at UNILAC for collimators –tentative planning

ARIES WP17 Progress Meeting

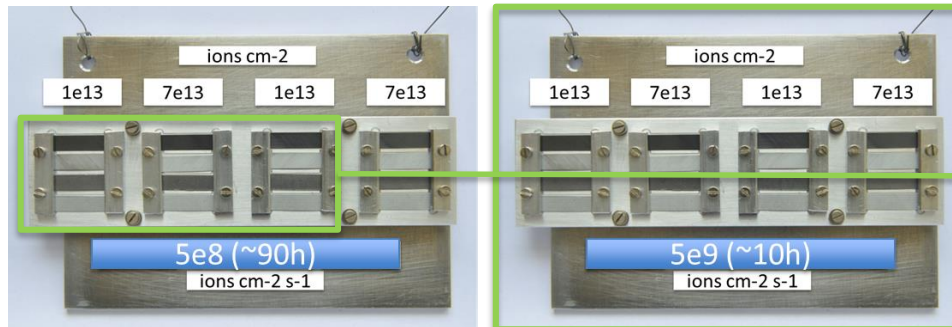
17/11/2020

C. Accettura (CERN and Politecnico di Milano)

On Behalf of WP17.3

Test Overview

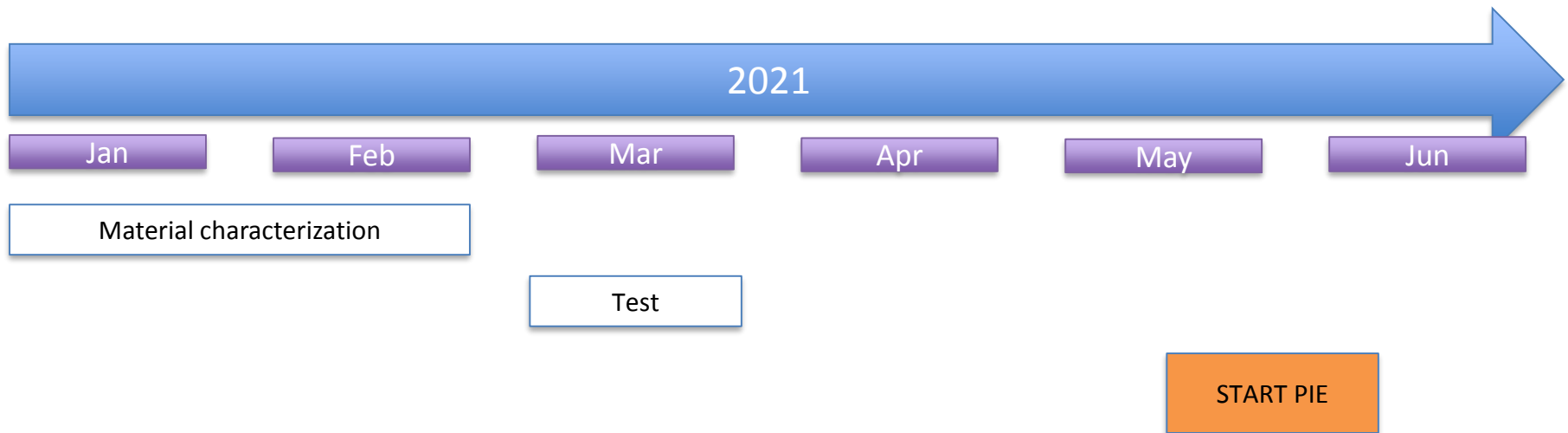
- Initially planned for April, 2020 (18 shifts) → postponed for Covid19
- Proposal re-submitted for 2021-2022 → grant 26,5 shifts as secondary user
 - Approximately **9 shifts** included in the 2020 beamtime
 - Possible during **w.10-11-12** → 5 days of quarantine+test before going to GSI
 - Focus on **flux effect**, 2 fluences and 4 materials (Gr and MoGr with and without coating, 2 samples each)



- To complete all the holders ~97h (+1 holder change) → 13shift
- Considering 9 shifts we can irradiate all the holder at high flux+ 3 target station at low flux → 8 shift
- + try to reach the max fluence on the last one

Planning and timeline

- Material already procured for 2020
- Simulation (dpa, thermo-mechanics) done
- Characterization partly done (to be repeated after coating) → feasible by end of w. 8 (Covid evolution permitting..)



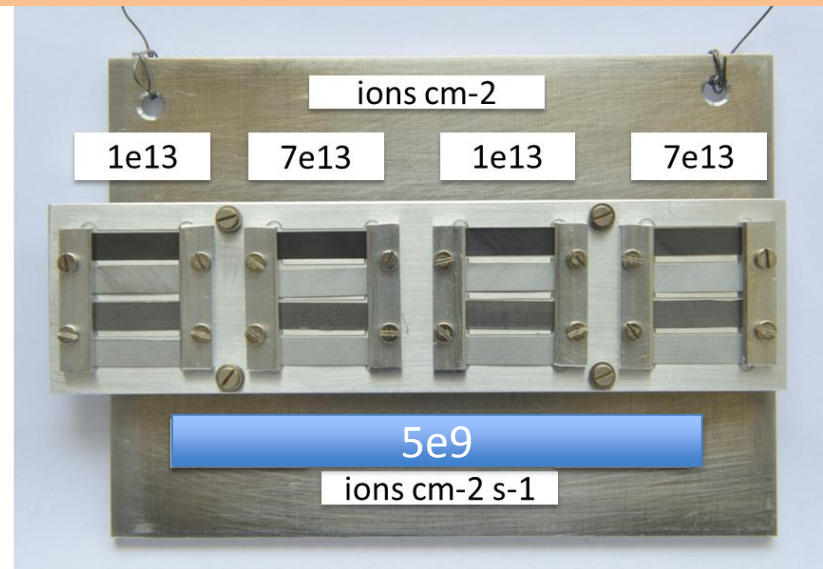
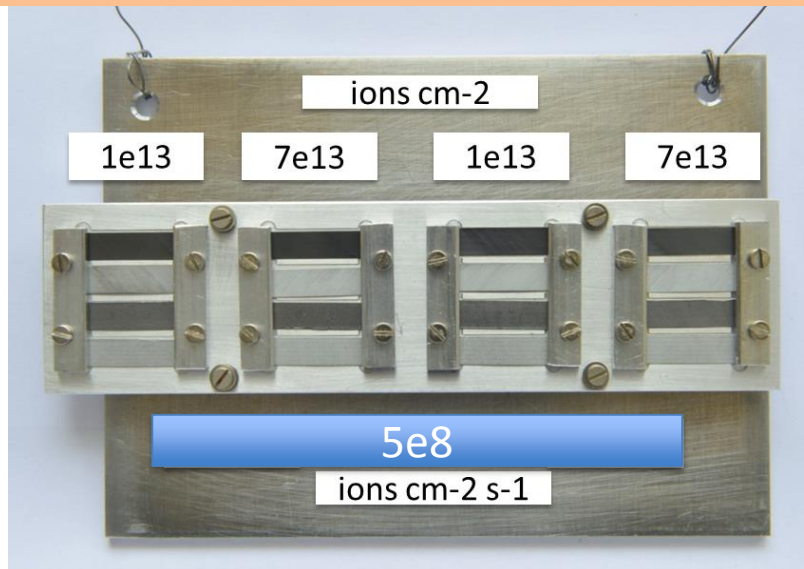
Resources

- If the procedure is confirmed (5d of quarantine+test)→ minimize the travels
- Only 2 sample holders→ 1 change of holder
- If GSI supports us with 1 person/shifts→ max 3 people involved by CERN (1/shift, 1 shift/day), only 1 holder changes

New test overview

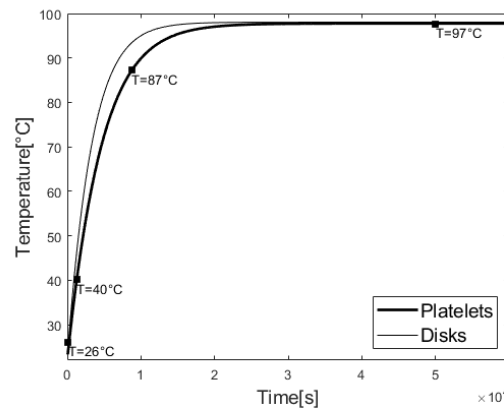
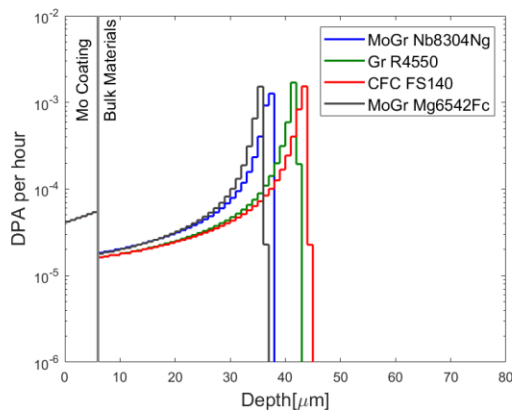
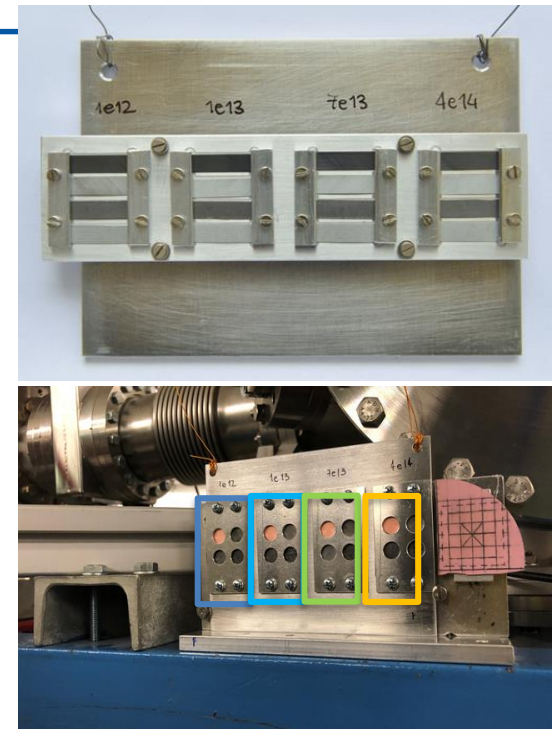
Novelty of the test

- New grade of MoGr (Nb8404Ng) with new powders
- 2 samples per material per fluence
- Same materials (Gr, MoGr and coating) selected for proton irradiation @BLIP→ possible comparison between ion and proton
- Investigation of flux effect→ DPA rate effect (damage evolution→ scaling to HL-LHC?)



Ion irradiation campaign-GSI2019

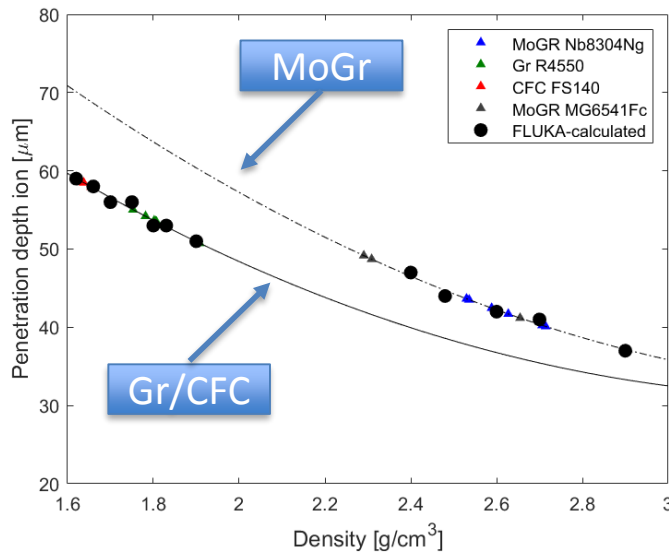
- ^{48}Ca ions at 4.8 MeV/u
- 80 samples of graphitic material tested, half of them coated with thin films
 - Gr R4550
 - Gr R4550+Mo HIPIMS
 - MoGr Nb8304Ng
 - MoGr Nb8304Ng+Mo HIPIMS
 - MoGr Nb8304Ng+Cu HIPIMS,
 - MoGr MG6541Fc+Mo DCMS
 - MoGr MG6541Fc
 - CFC FS140
- FLUKA simulation to compute DPA rate
- $T_{\text{max}} \sim 100^\circ\text{C}$ (simulated)
- Movable sample holder to irradiated 4 target station at different fluence (max DPA in the coating=HL-LHC)



Fluences [ions/cm ²]	Peak DPA coating	Peak DPA bulk
$1 \cdot 10^{12}$	$\sim 2.8 \cdot 10^{-6}$	$\sim 1.1 \cdot 10^{-4}$
$1 \cdot 10^{13}$	$\sim 2.8 \cdot 10^{-5}$	$\sim 1.1 \cdot 10^{-3}$
$7 \cdot 10^{13}$	$\sim 1.9 \cdot 10^{-4}$	$\sim 7.8 \cdot 10^{-3}$
$4 \cdot 10^{14}$	$\sim 1.1 \cdot 10^{-3}$	$\sim 4.4 \cdot 10^{-2}$

Electrical resistivity studies

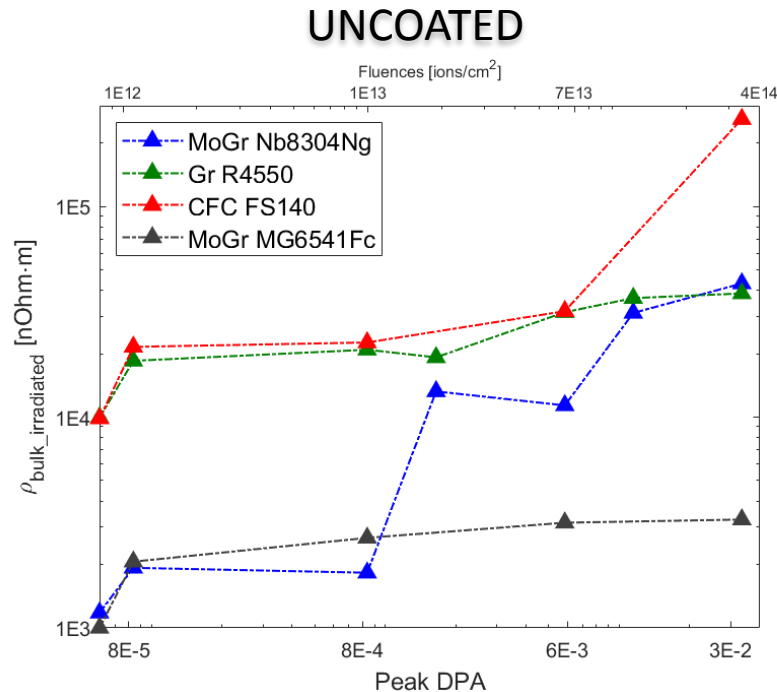
- DC measurements with the 4-probe method and multi-layer model (~1/3 of the sample irradiated)→ important to know the irradiated layer thickness



- FLUKA simulation for sample with different density: penetration depth in function of the density→ analytical correlation to know the irradiated layer for every samples
- Density as important as the composition!

Electrical resistivity studies

- DC measurements with the 4-probe method and multi-layer model (~1/3 of the sample irradiated) → important to know the irradiated layer thickness

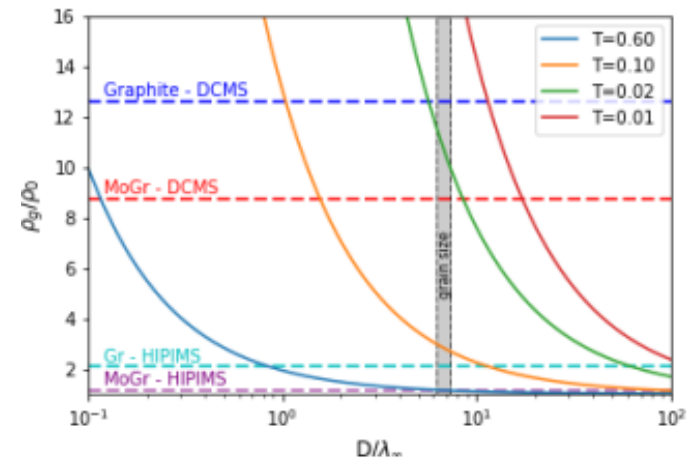
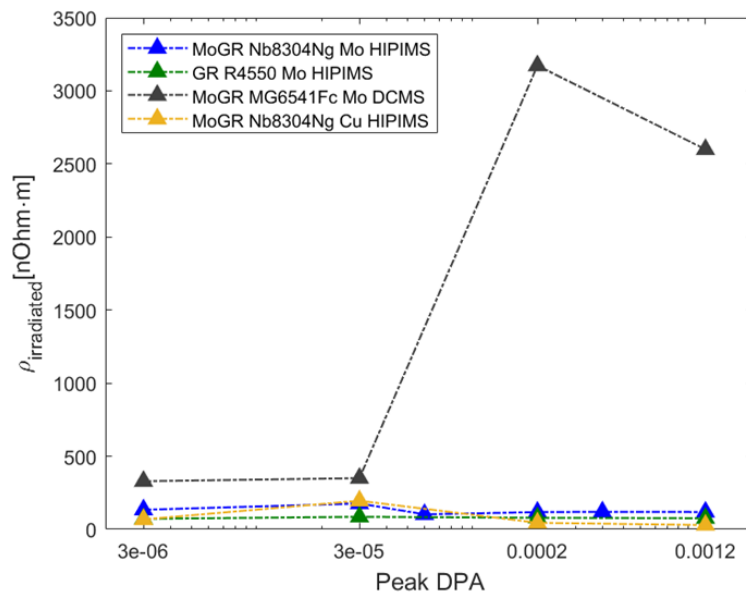


- Electrical resistivity decrease faster in material with higher graphitization (MoGr)
- MoGr with fibers more radiation resistant

Electrical resistivity studies

- DC measurements with the 4-probe method and multi-layer model (~1/3 of the sample irradiated) → important to know the irradiated layer thickness

COATED

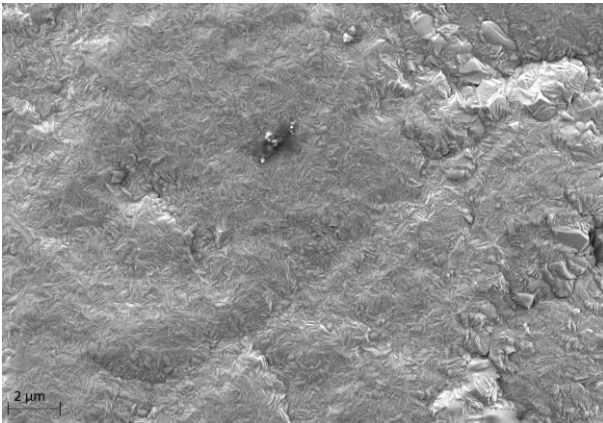


C. Accettura et al. "Resistivity Characterization of Molybdenum-Coated Graphite-Based Substrates for High-Luminosity LHC Collimators." *Coatings* 10.4 (2020): 361.

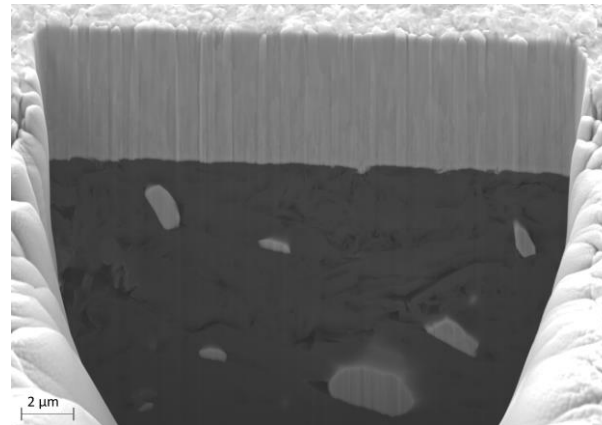
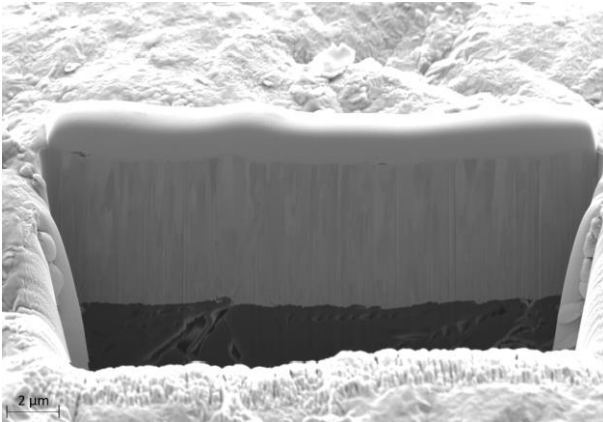
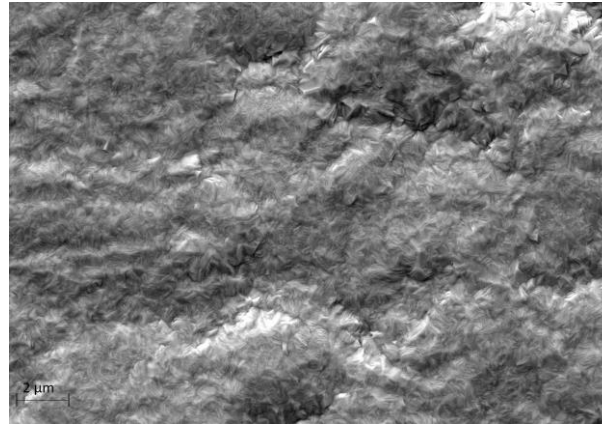
- DCSM coating more resistant before the irradiation and it is losing more than HIPIMS
- DCSM has lower transmission coefficient, but similar grain size → difference at the GBs

Microscopic observation of coatings

Pristine

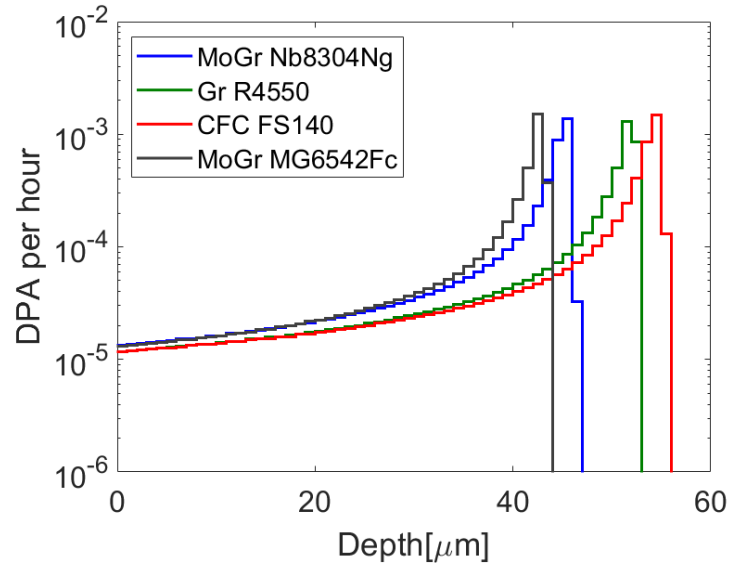


Irradiated at $4e14$ ions/cm² → max DPA



- Mo coating produced with HIPIMS on MoGr (baseline for HL-LHC collimators)
 - Columnar and dense structure kept after irradiation
 - No cracks
 - Good contact with the bulk
- Important to evaluate effect of gas production

Peak vs average dpa

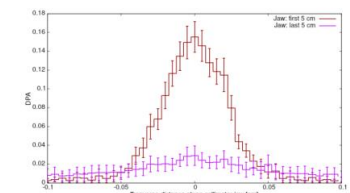
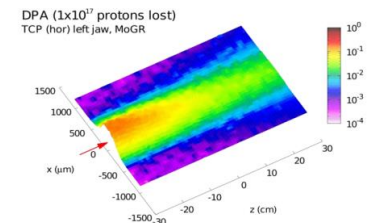


- Peak dpa coating~average
- Peak dpa 1 order of magnitude higher with respect to the average, 2 order of magnitude higher with respect to the surface

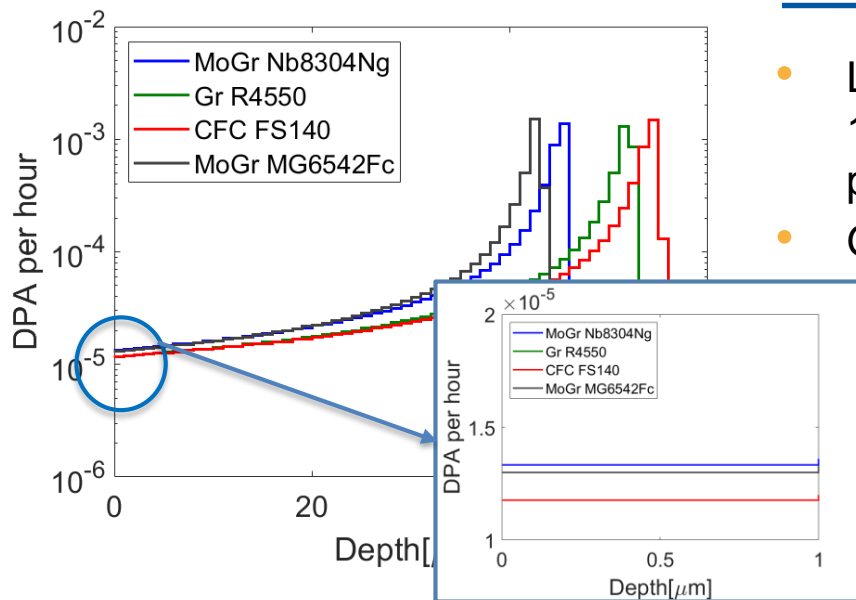
Fluences	PEAK DPA		Average DPA	
	Nb	Gr	Nb	Gr
1.00E+12	7.66E-05	7.25E-05	5.14E-06	4.75E-06
1.00E+13	7.66E-04	7.25E-04	5.14E-05	4.75E-05
7.00E+13	0.0053	0.0051	0.00036	0.000333
4.00E+14	0.0306	0.029	0.002056	0.001901

How to plot the properties degradation?

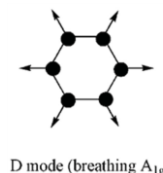
- Reference in the past used the peak dpa
- If I consider only the peak in the bulk → underestimating the damage
- If I consider the average: not complete information, average dpa with higher peak can give different results, especially if threshold of more severe damage exist
- When considering the comparison with the collimators, consider a larger affected area



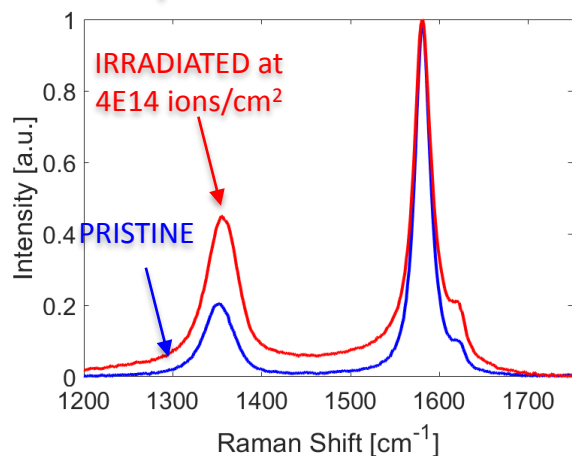
Raman spectroscopy on irradiated sample



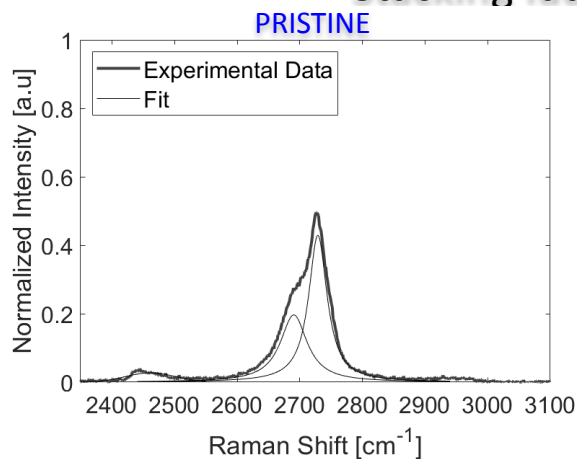
- Laser skin depth (514.5nm) for graphite~50-100nm: investigated region with a dpa << peak dpa, but flat
- General findings:
 - I_D , $I_{D'}$, $FWHM_G$ increase \rightarrow in-plane information
 - 2D double peak becomes single (MoGr) \rightarrow through-plane information



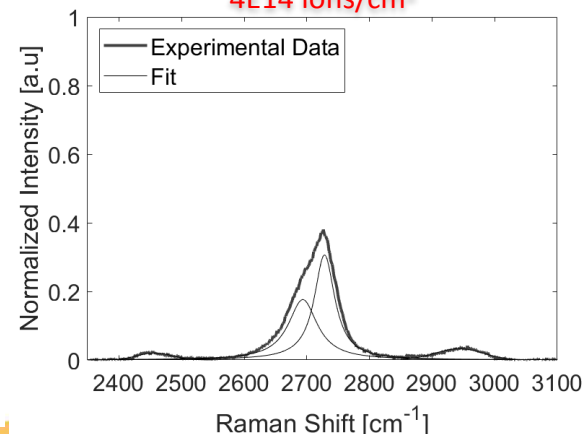
In-plane defects in CFC



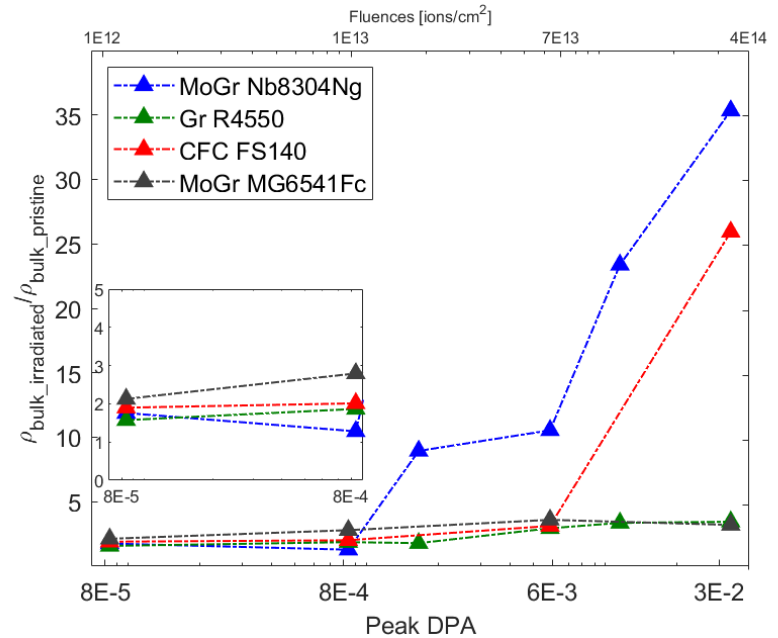
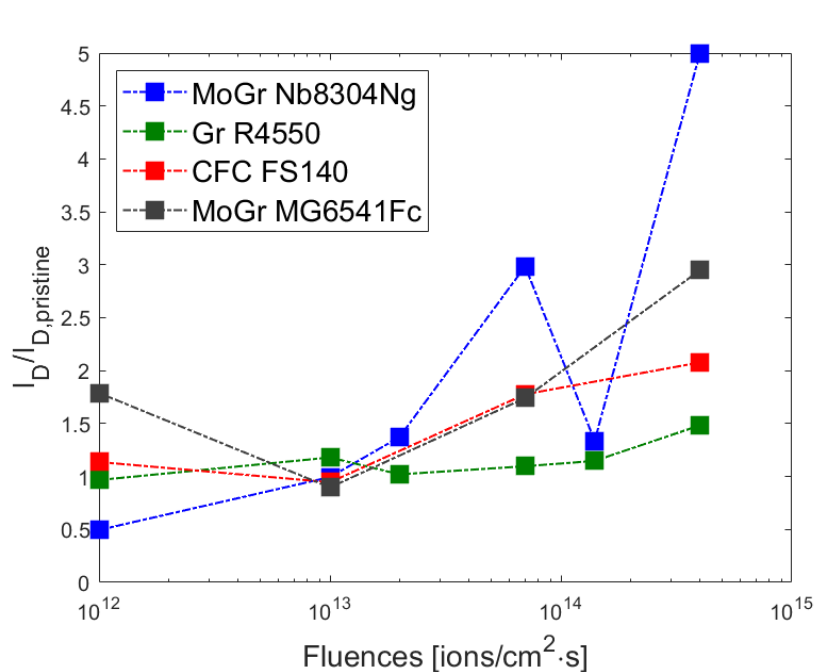
Stacking fault in MoGr



IRRADIATED at
4E14 ions/cm²

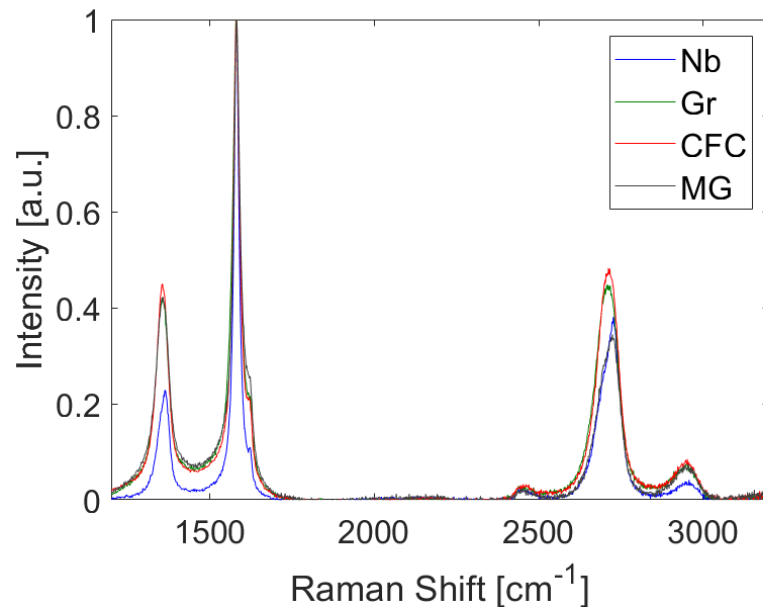


Microscopic vs electrical resistivity



- ID increasing with fluences → accumulation of defects
- Increase of electrical resistivity coherent with in-plane defects concentration found with RS: MoGr Nb more affected and Gr less affected (relative values)
- MoGr with fibers higher D peak, but less affected than CFC → phenomenon related to higher dpa (not detected by RS)?

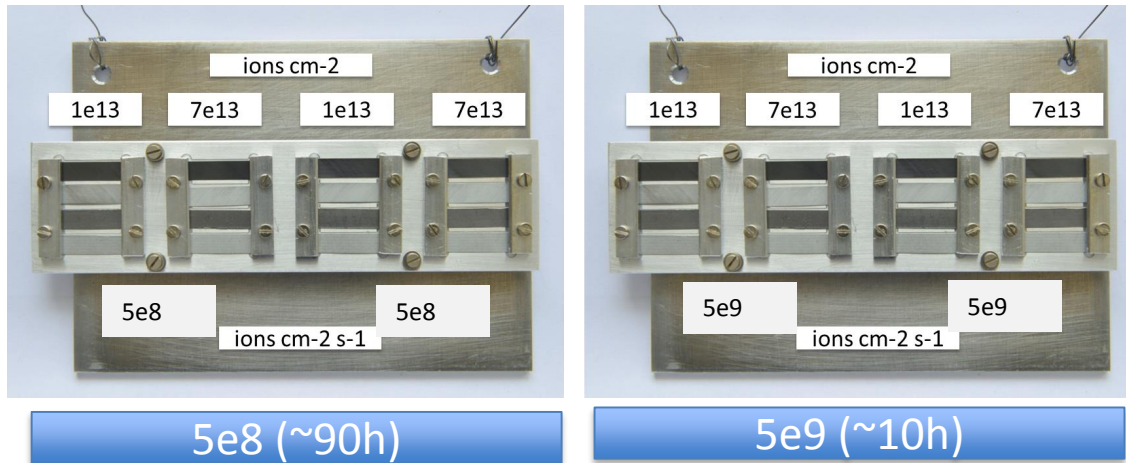
Raman spectroscopy



- MoGr remains with a more order structure clearly different with respect to Gr and CFC, especially the AB stacking fault
- RS useful to see difference response of materials and coherent with electrical resistivity
- **Test proposal:** investigate RS evolution after annealing of material at different temperature
 - Useful to study defects mobility
 - Useful to study influence of material structure on defects recombination
 - Useful to estimate properties recovery (**Bake-out temperature in LHC!**)

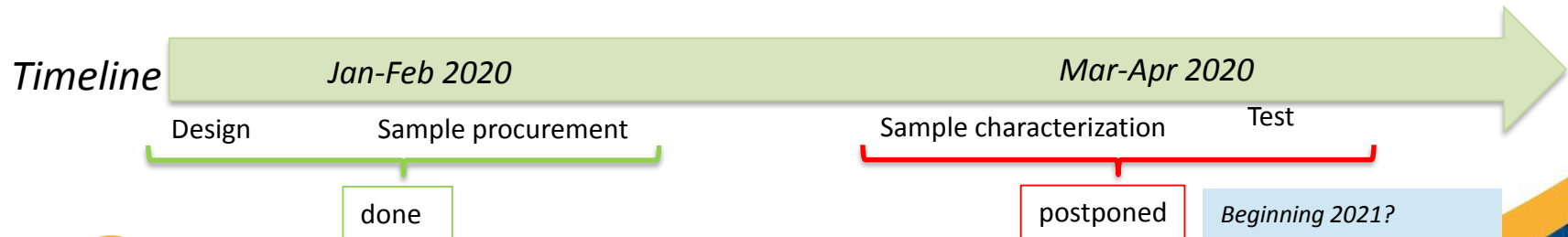
Planning for GSI2020 campaign (postponed)

- Test postponed for the Covid-19 emergency → new proposal for 2021 beamtime submitted
- Aim: Investigation of flux effect → DPA rate effect (damage evolution → scaling to HL-LHC?)



- Focus on MoGr (new grade) and Gr with Mo coating
- Investigate 2 fluences at 2 flux
- Reduce the heat exchange on the low flux to have the same temperature on the two holders

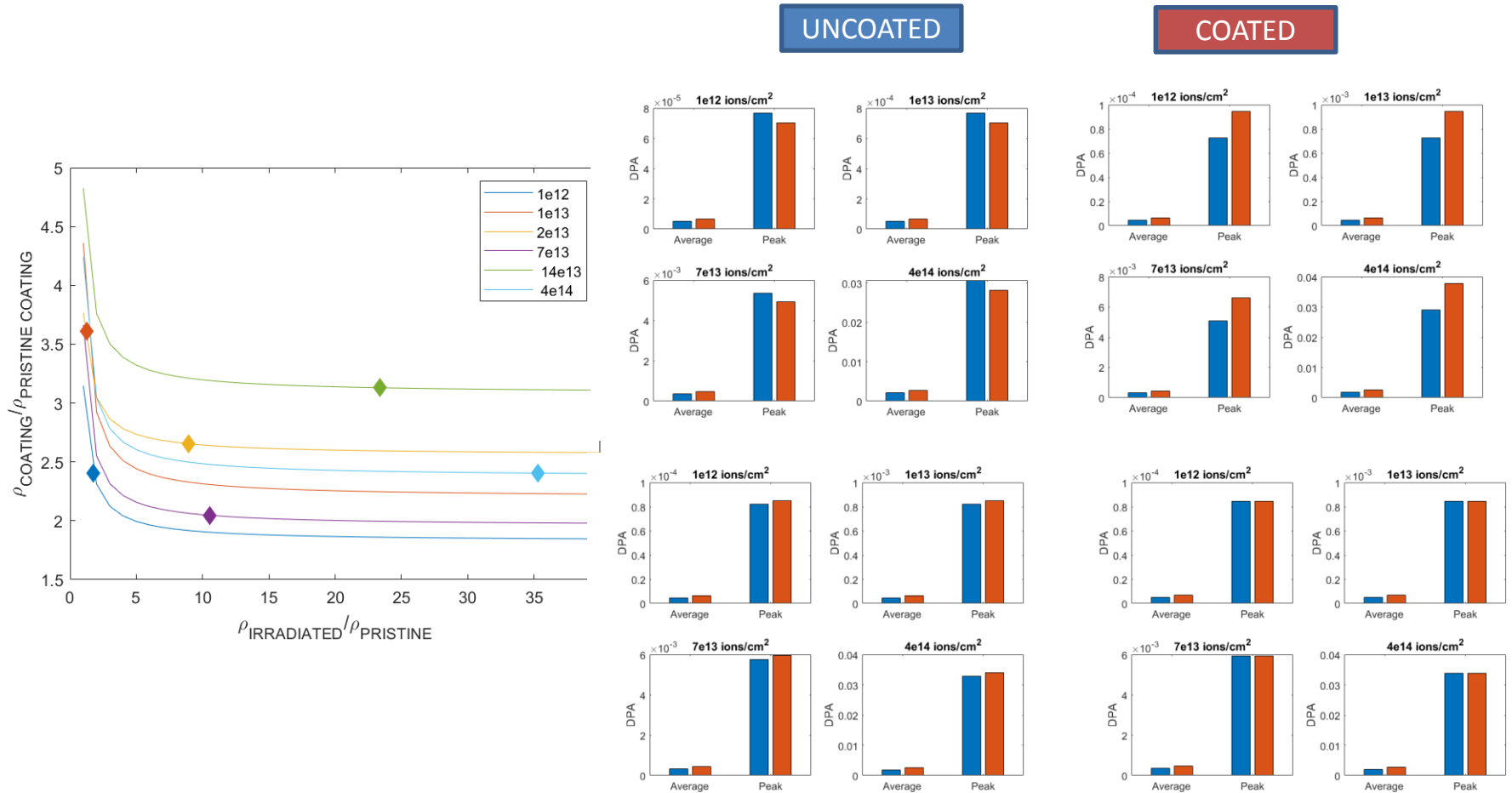
- **Same materials (Gr, MoGr and coating) selected for proton irradiation @BLIP (see N. Solieri presentation) → additional effect of gas production**



Conclusion

- GSI irradiation campaign in 2019 very useful: coating and new MoGr grade irradiated for the first time and accessible 1 month after the test
- Electrical resistivity degradation:
 - Interesting results indicate possible positive effect of C-fibers
 - Coating production process influence the results
- RS gives microscopic information coherent with the macroscopic → interesting to perform annealing studies to understand defects mobilities for different materials
- Possibility to compare outcome of ion irradiation with proton irradiation (RADIATE collaboration)

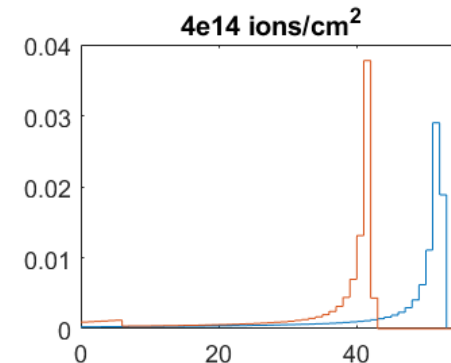
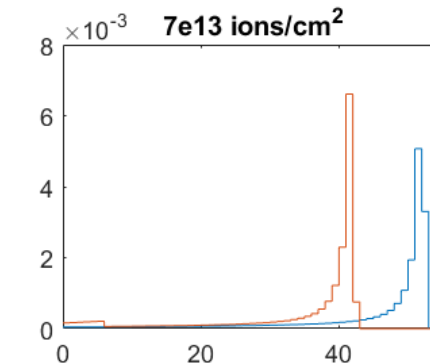
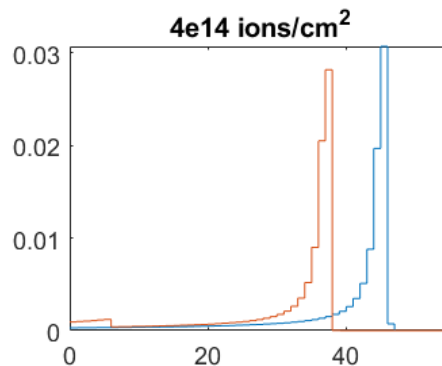
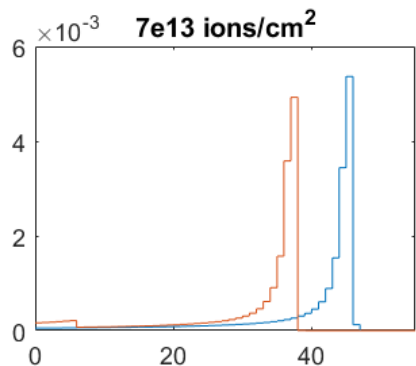
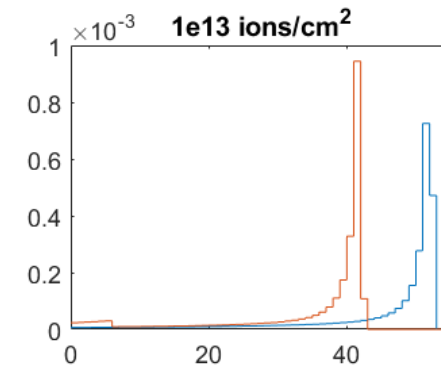
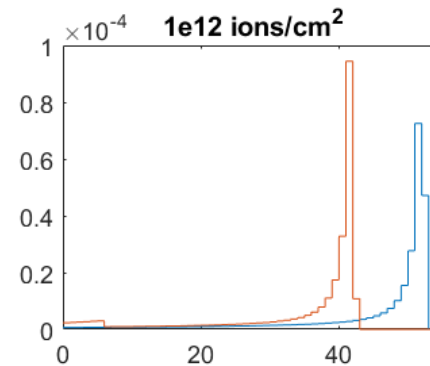
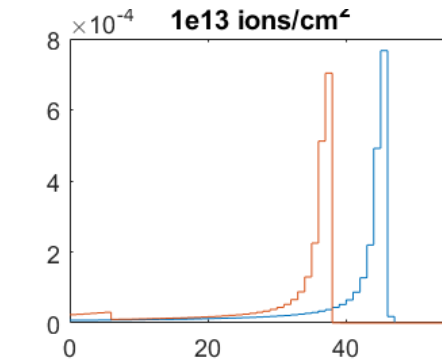
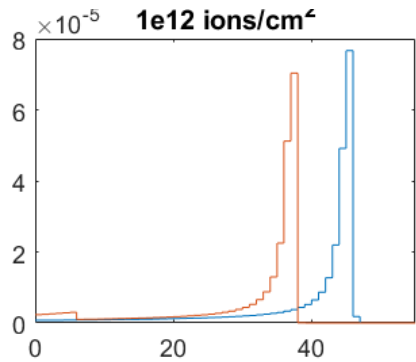
Electrical resistivity coating



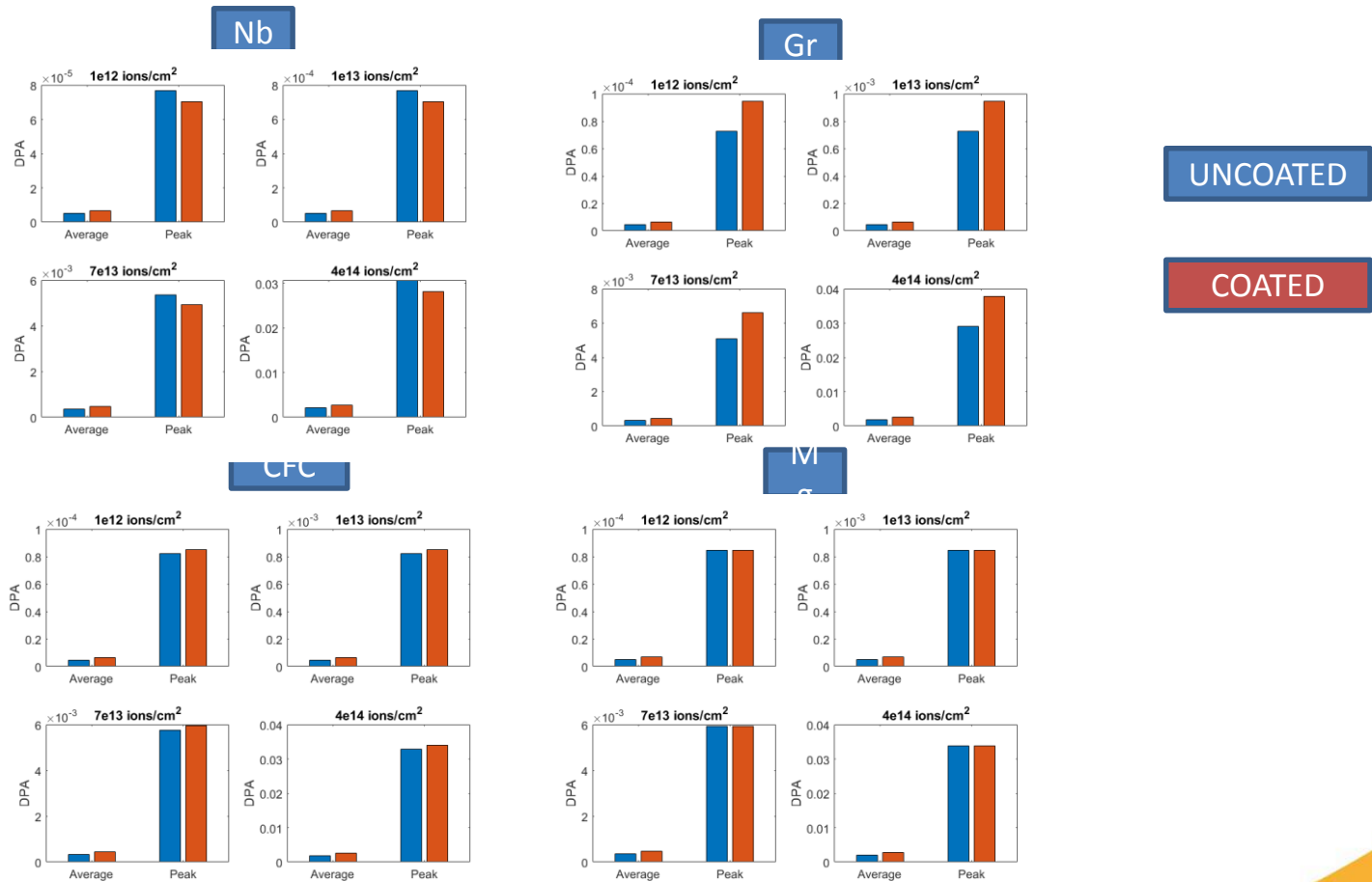
Electrical resistivity coating

Nb

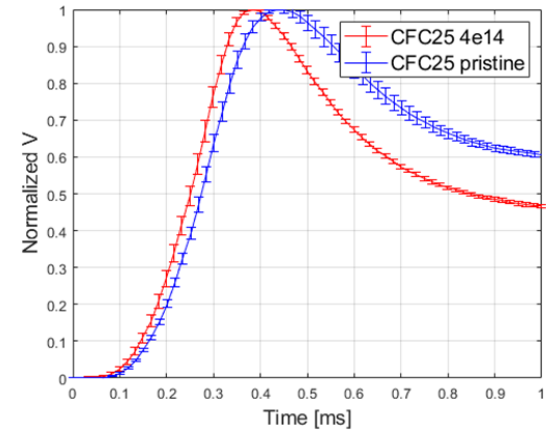
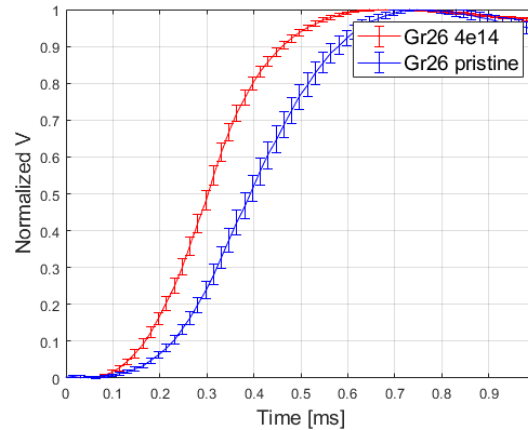
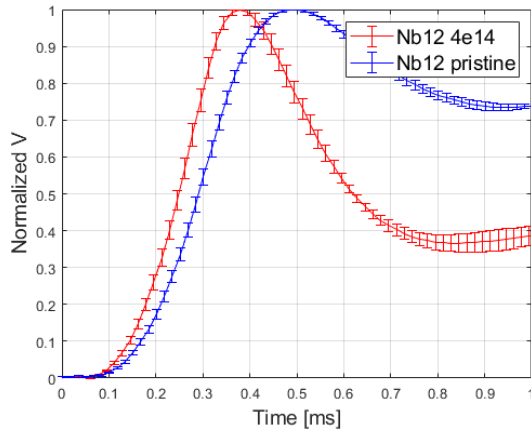
Gr



Uncoated and coated

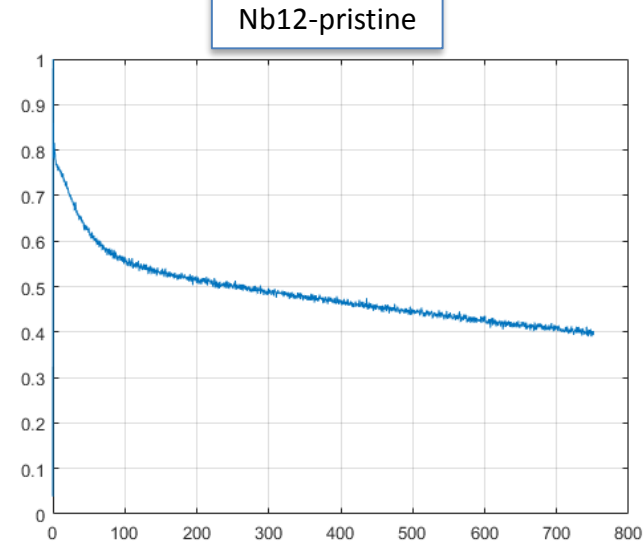
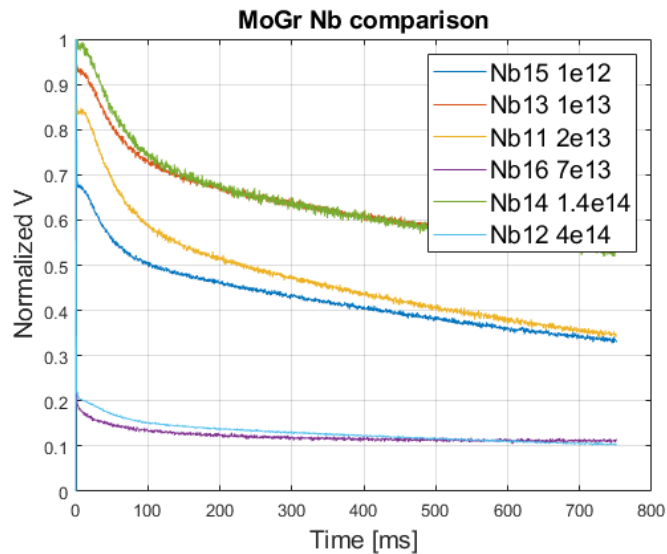


GSI 2019-Thermal conductivity



- All the samples tested
- Systematic (for all the samples) increase of the heating ramp → not coherent with literature
- More test planned to understand this behavior (Aluminum foil)

GSI 2019-Thermal conductivity



- Long acquisition time measurement indicate also a decrease in the in-plane thermal conductivity, but difficult to quantitative estimate without the fitting the first part of the curves.

Methods

- Deep investigation of possible thermal losses:
 - Convection/radiation
 - Gaussian flux distribution
 - Laser power penetration depth
 - Thermoelastic temperature oscillation

