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ARIES

Ion irradiation test at GSI for collimator materials: results and future test plan

ARIES WP17 annual meeting

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With contribution of:

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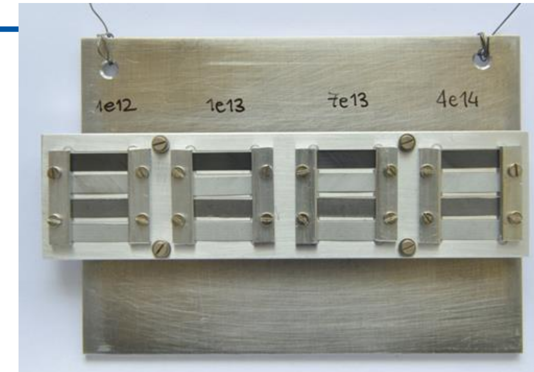
Outline

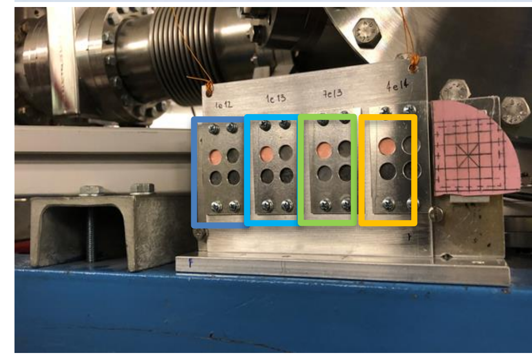
- GSI irradiation 2019
  - Experiment overview
  - Electrical resistivity
  - Raman spectroscopy
- Future test planning
- Conclusions

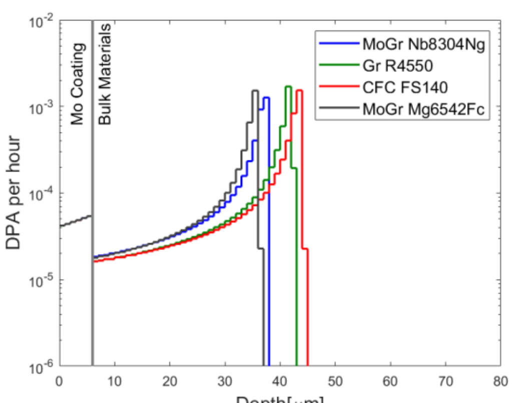
Ion irradiation campaign-GSI2019

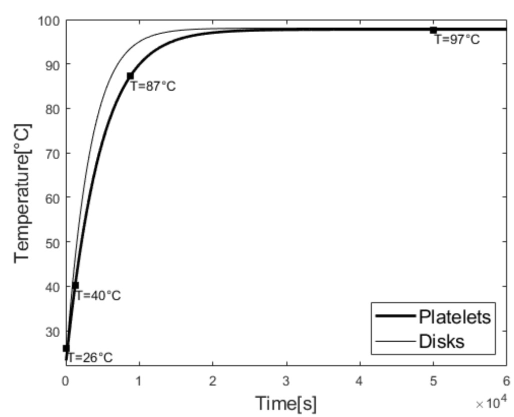
- ^{48}Ca ions at 4.8 MeV/u
- 80 samples of graphitic material tested, half of them coated with thin films
  - Gr R4550
  - Gr R4550+Mo HIPIMS
  - MoGr Nb8304Ng
  - MoGr Nb8304Ng+Mo HIPIMS
  - MoGr Nb8304Ng+Cu HIPIMS,
  - MoGr MG6541Fc+Mo DCMS
  - MoGr MG6541Fc
  - CFC FS140
- FLUKA simulation to compute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

HL-LHC primary and secondary collimators





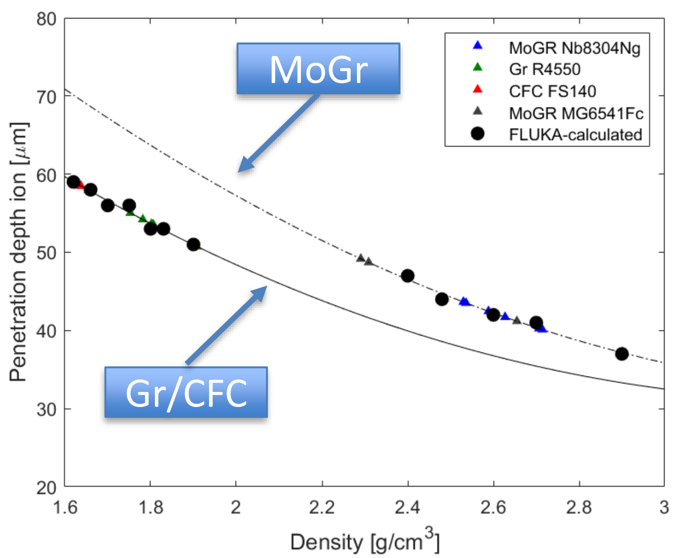




Fluences [ions/cm²]	Peak DPA coating	Peak DPA bulk
$1\cdot10^{12}$	$\sim2.8\cdot10^{-6}$	$\sim1.1\cdot10^{-4}$
$1\cdot10^{13}$	$\sim2.8\cdot10^{-5}$	$\sim1.1\cdot10^{-3}$
$7\cdot10^{13}$	$\sim1.9\cdot10^{-4}$	$\sim7.8\cdot10^{-3}$
$4\cdot10^{14}$	$\sim1.1\cdot10^{-3}$	$\sim4.4\cdot10^{-2}$

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

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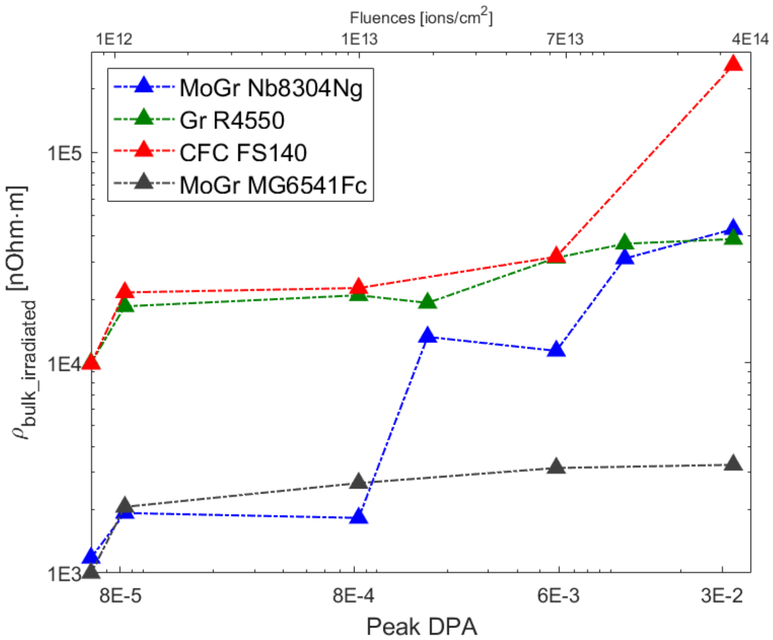
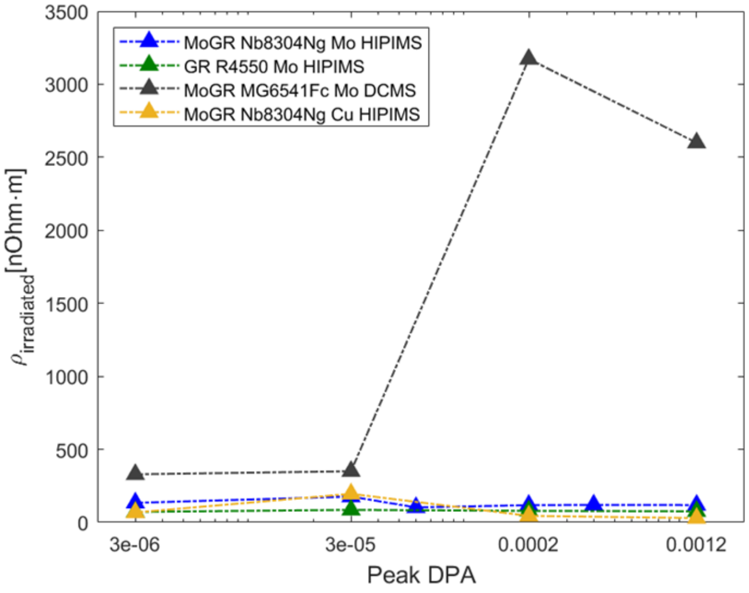


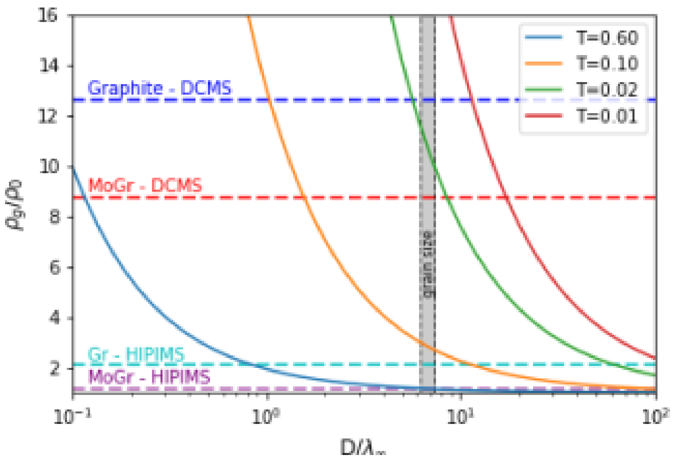
- Electrical resistivity decrease faster in material with higher graphitization (MoGr)
- MoGr with fibers more radiation resistant

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





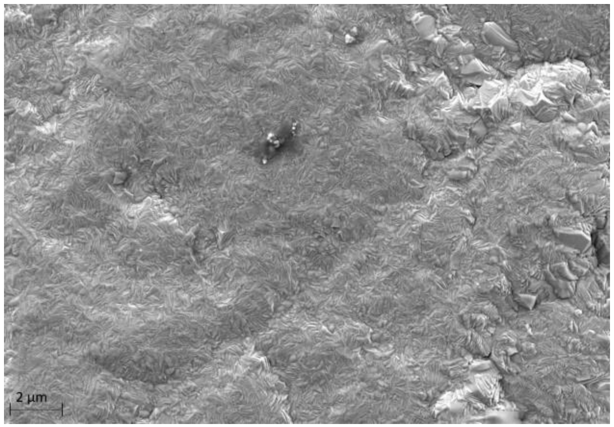
C. Accettura et al. "Resistivity Characterization of Molybdenum-Coated Graphite-Based Substrates for High-Luminosity LHC Collimators." Coatings 10.4 (2020): 361.

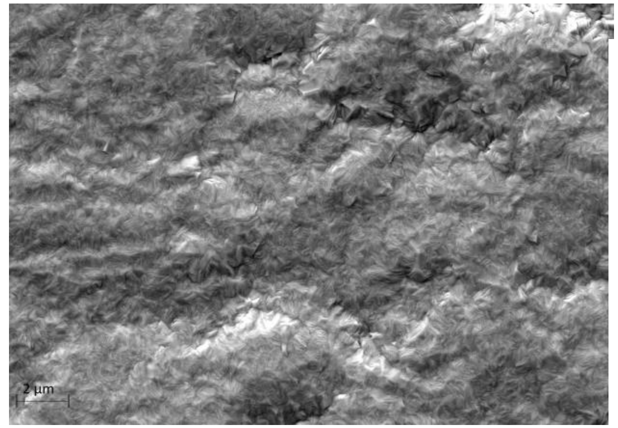
- DCSM coating more resistant before the irradiation and it is loosing more then HIPIMS
- DCSM has lower transmission coefficient, but similar grain size→ difference at the GBs

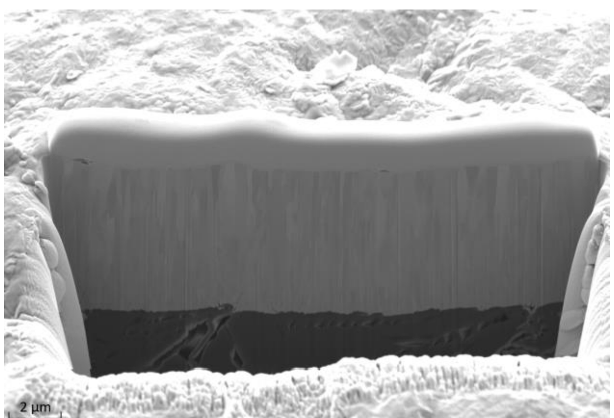
Microscopic observation of coatings

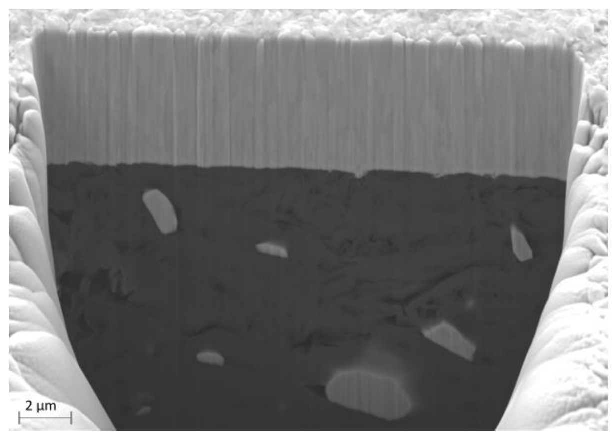
Pristine

Irradiated at 4e14 ions/cm^2 → max DPA



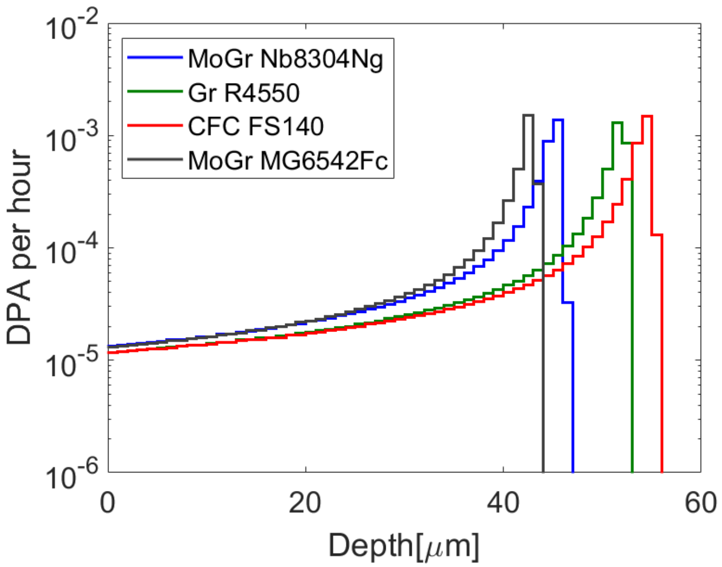






- Mo coating produced with HIPIMS on MoGr (baseline for HL-LHC collimators)
  - Columnar and dense structure kept after irradiation
  - No cracks
  - Good contact with the bulk
- Important to evaluate effect of gas production

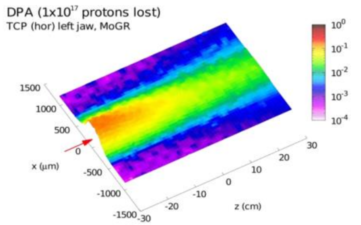
Peak vs average dpa

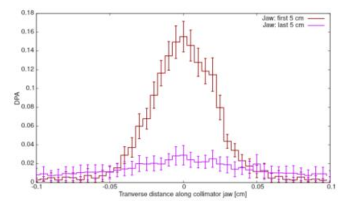


- Peak dpa coating~average
- Peak dpa 1 order of magnitude higher with respect to the average, 2 order of magnitude higher with respect to the surface

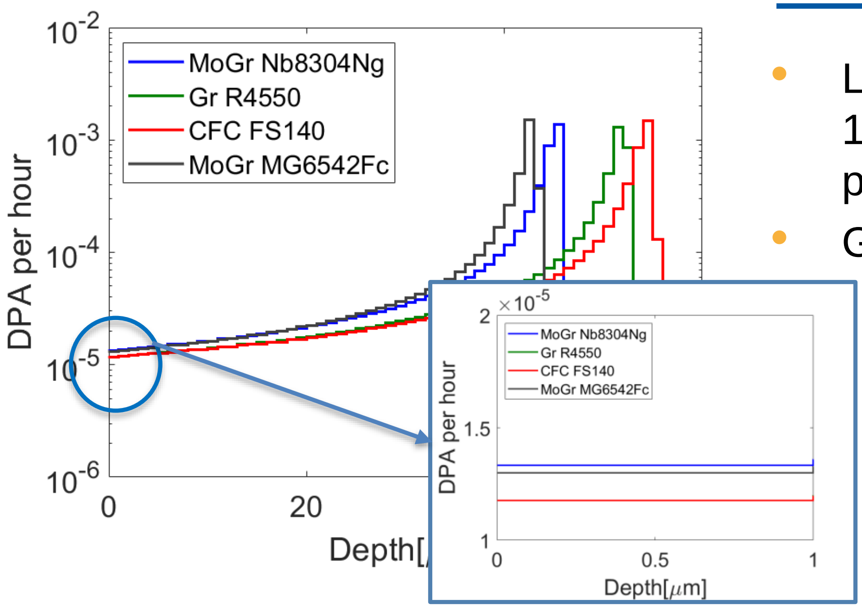
	PEAK DPA		Average DPA	
Fluences	Nb	Gr	Nb	Gr
1.00E+12	7.66E-05	7.25E-05	5.14E-06	4.75E-06
1.00E+13	7.66E-04	7.25E-04	5.14E-05	4.75E-05
7.00E+13	0.0053	0.0051	0.00036	0.000333
4.00E+14	0.0306	0.029	0.002056	0.001901

- 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

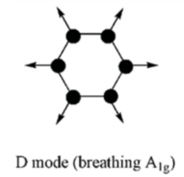




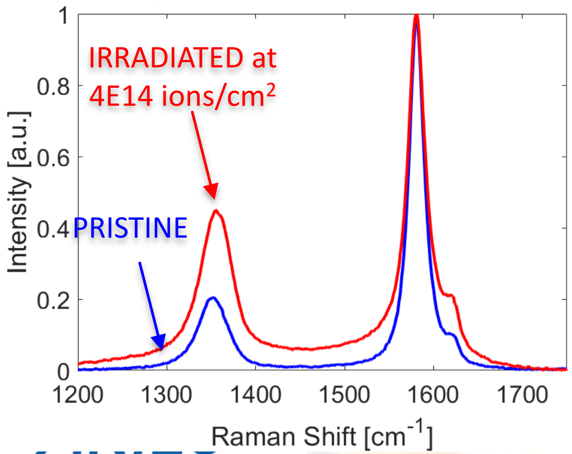
Raman spectroscopy on irradiated sample



- 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→ in-plane information
  - 2D double peak becomes single (MoGr)→ through-plane information

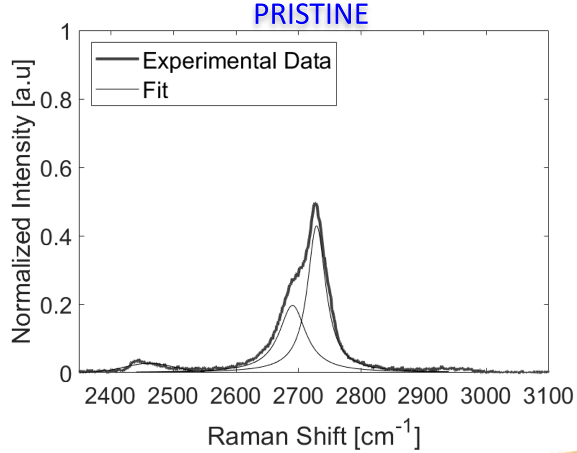


In-plane defects in CFC

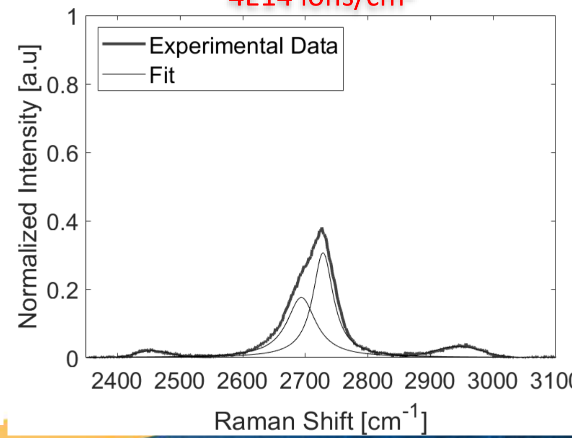


Stacking fault in MoGr

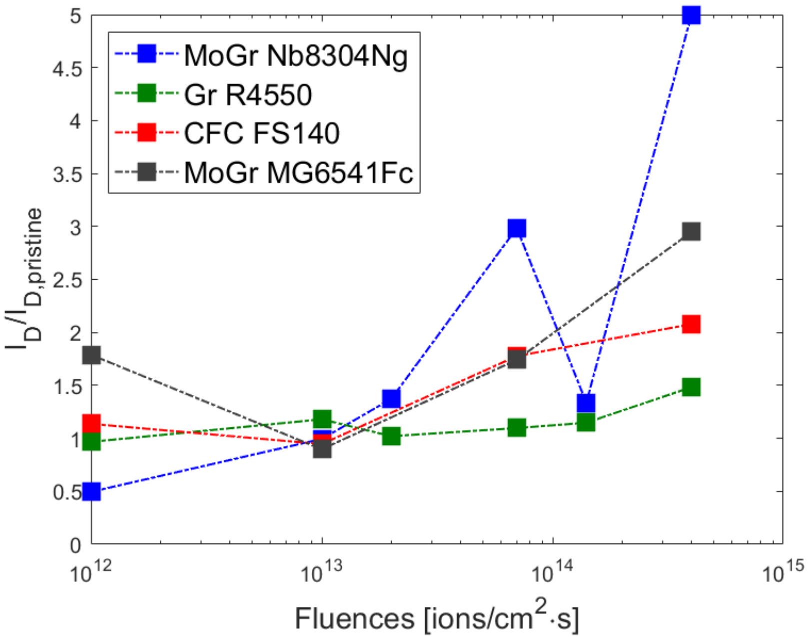
PRISTINE

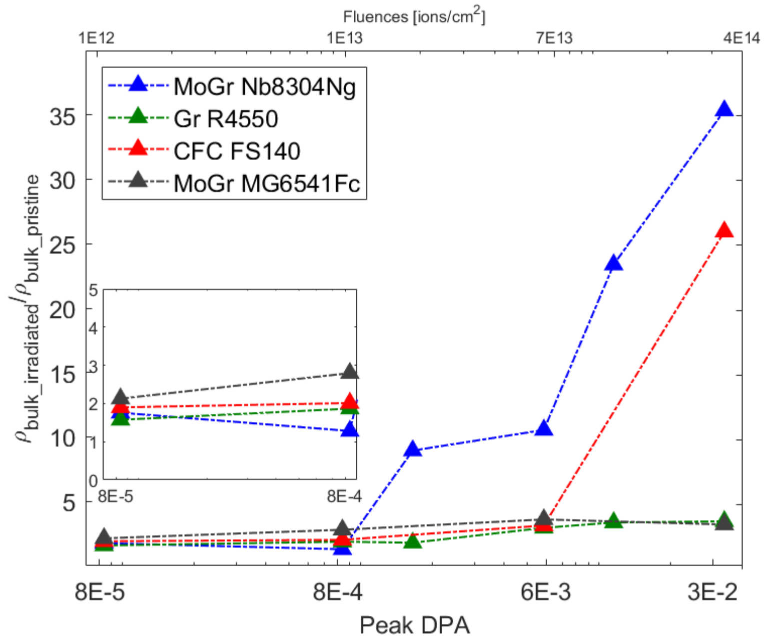


IRRADIATED at 4E14 ions/cm²



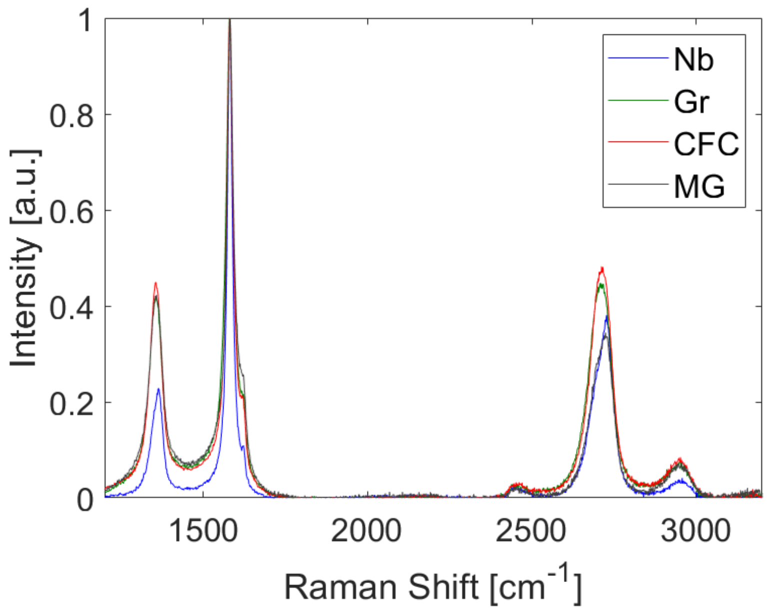
Microscopic vs electrical resistivity





- 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)?

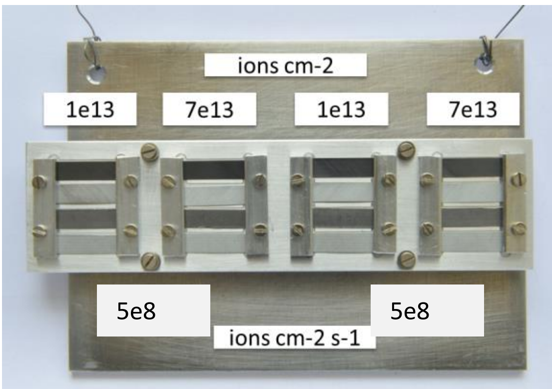
Raman spectroscopy



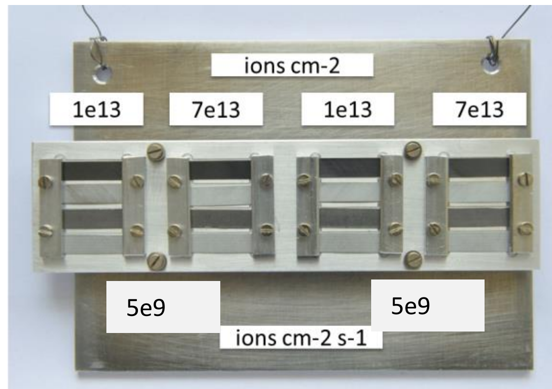
- MoGr remains with a more order structure clearly different with respect to Gr and CFC, especially the AB stacking fault
- RS useful to see difference response of materials and coherent with electrical resistivity
- **Test proposal**: investigate RS evolution after annealing of material at different 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?)



5e8 (~90h)



5e9 (~10h)

- 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**

Timeline

Jan-Feb 2020: Design, Sample procurement — done

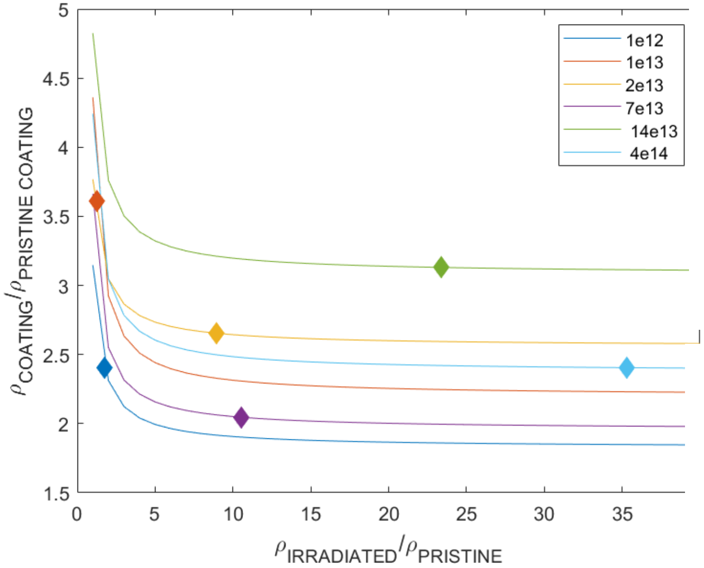
Mar-Apr 2020: Sample characterization, Test — postponed

Beginning 2021?

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



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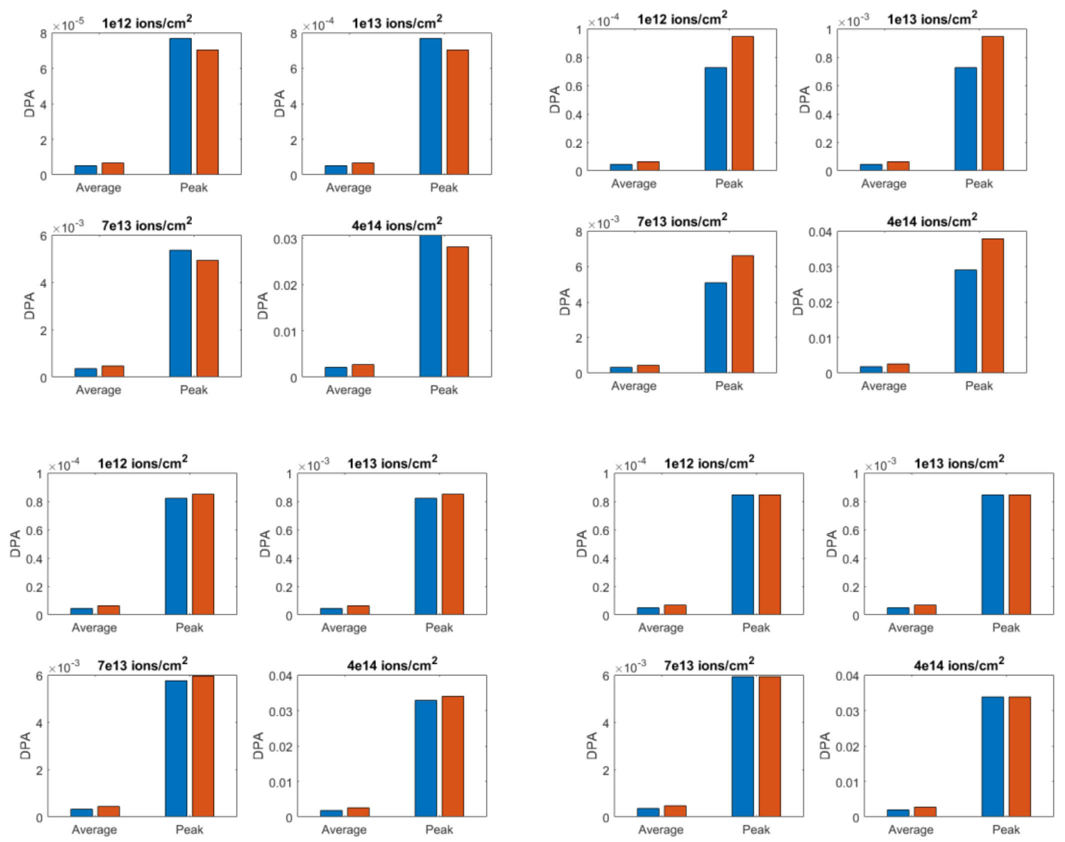
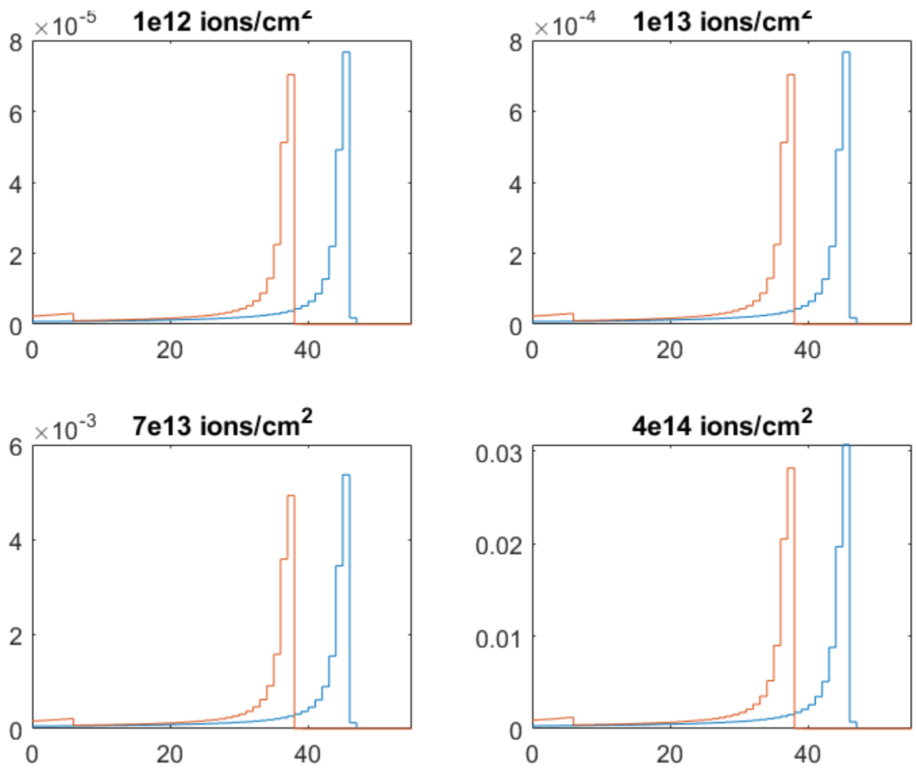
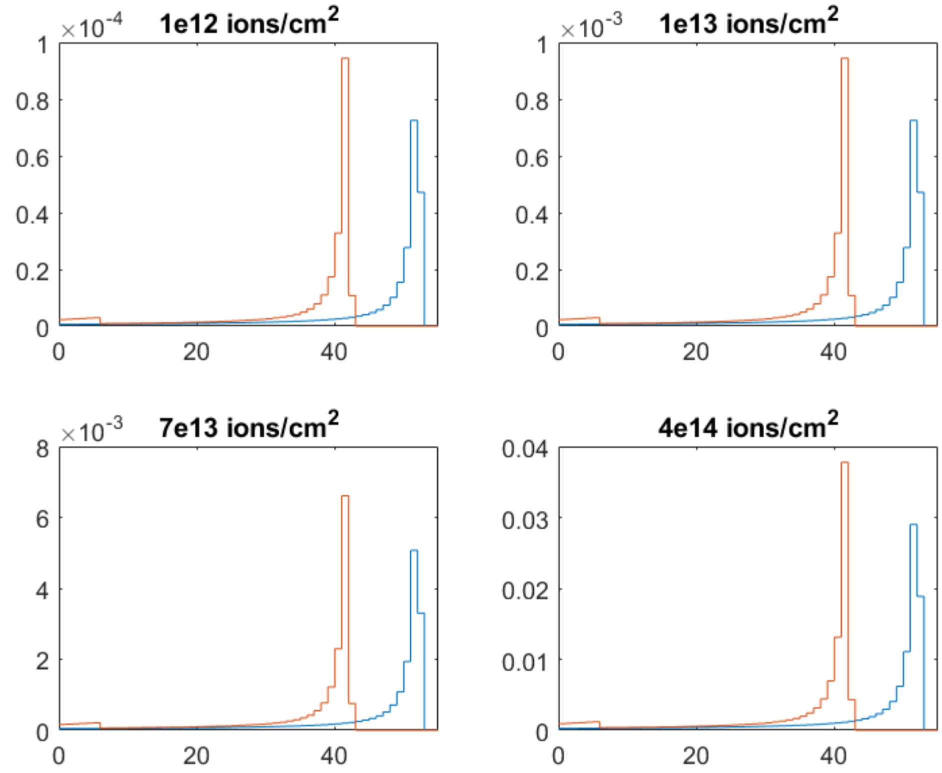


Electrical resistivity coating

Nb

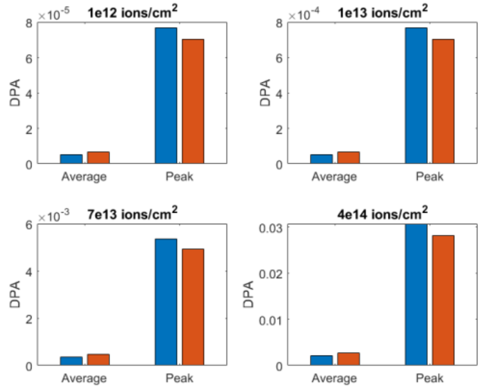


Gr

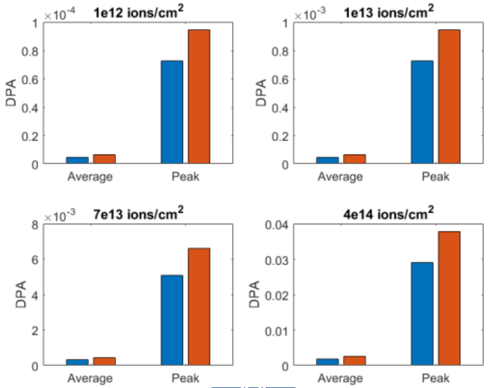


Uncoated and coated

Nb



Gr



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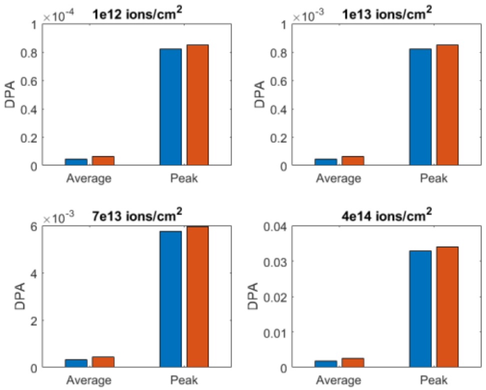
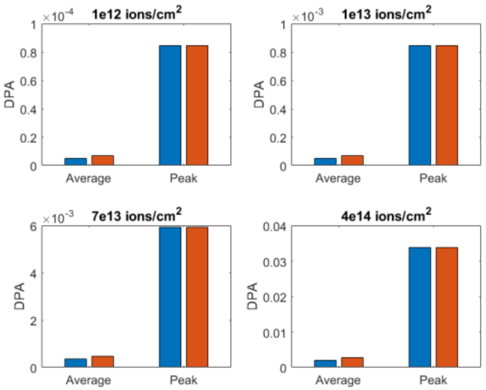
CFC



Mg



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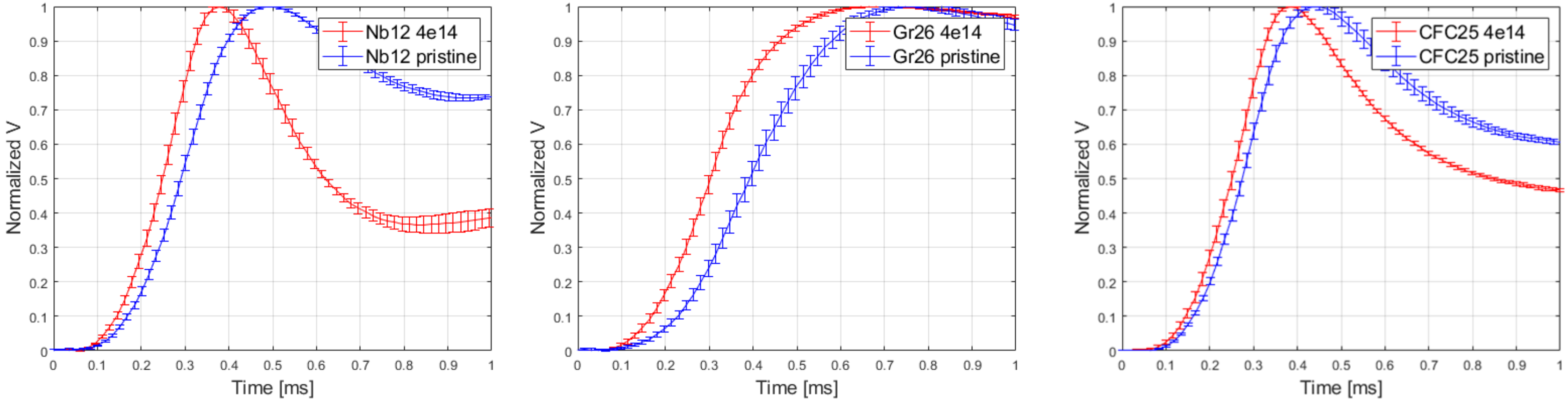
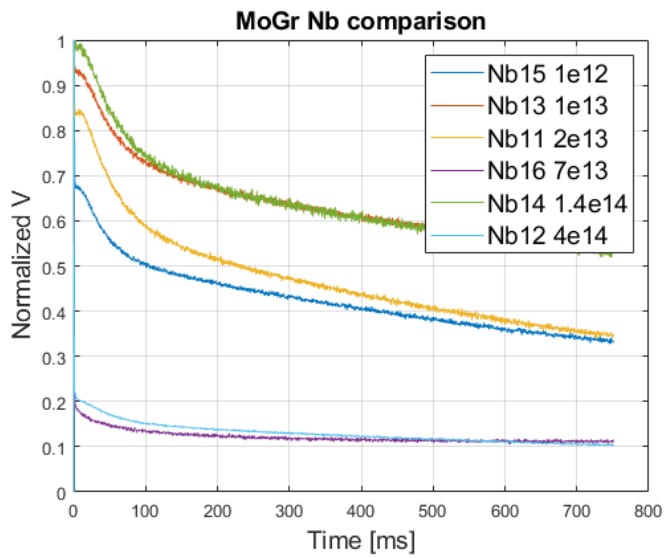
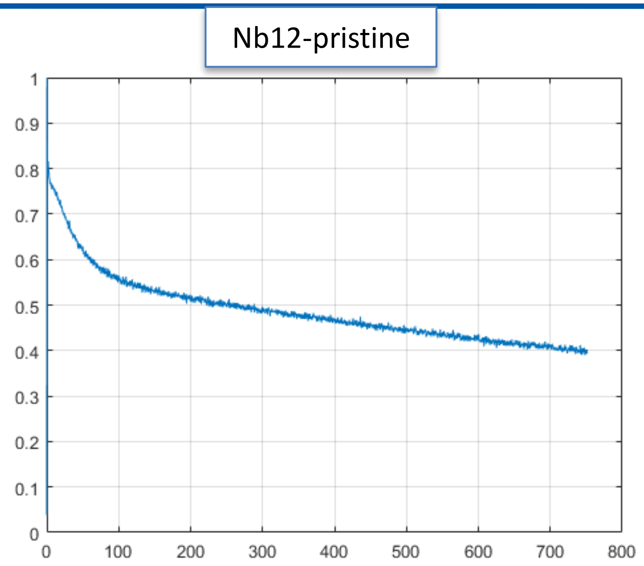
GSI 2019-Thermal conductivity



- 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