Special Joint HiLumi WP2/WP5 Meeting

Comparison of DC and RF electrical resistivity measurement for ion irradiated samples at GSI

C. Accettura and A. Kurtulus

Acknowledgements: A.Bertarelli, N.Biancacci, H.Bursali, F.Carra, F.Caspers, R.Illian Fiastre, A.T.Perez Fontenla, I.Llamas Garcia, N.Catalan Lasheras, A.Lechner, R.Perez Martinez, E.Métral, L.Mourier, S. Redaelli, B.Salvant, M.Tomut (GSI), W.Vollenberg, C.Vollinger, A.Waets (CERN) and the ATS-IWG team.

CERN, February 23, 2021

Outline

- Irradiation test overview
- DC measurement of electrical resistivity
- RF measurement of electrical resistivity
- Comparison of the two methods
- Conclusions

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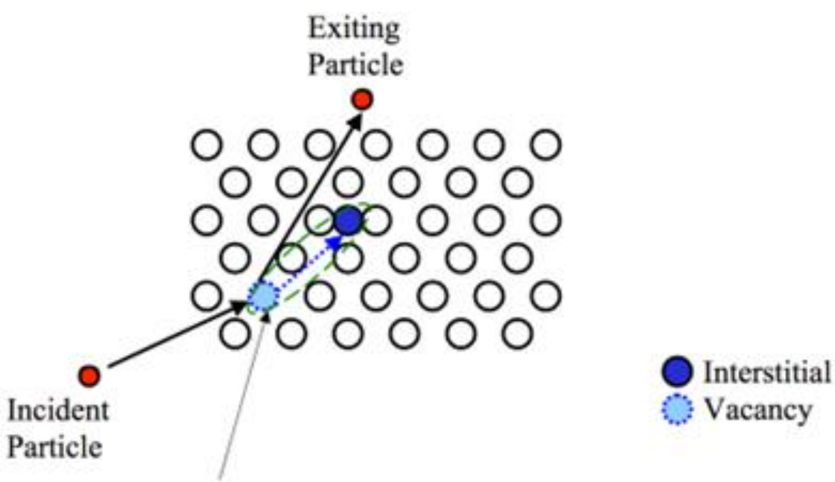
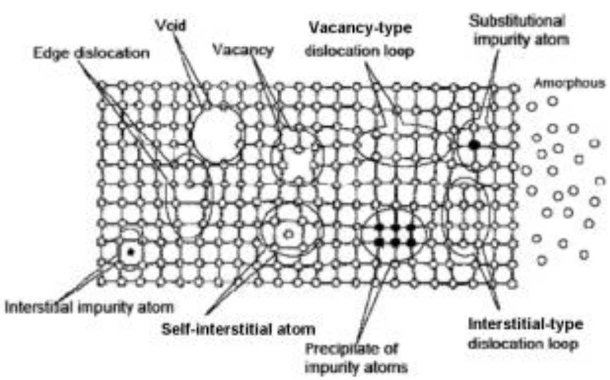
Irradiation test overview

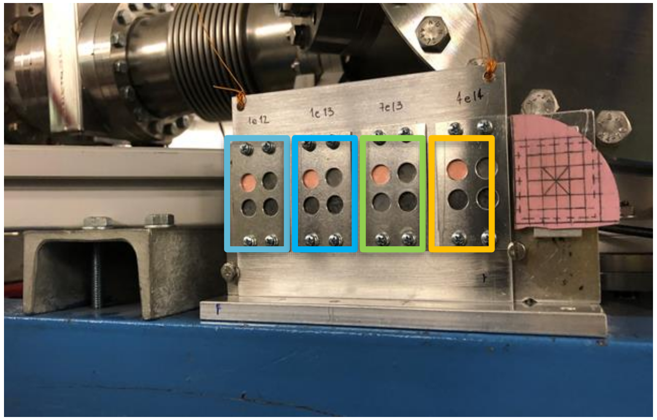
- March, 2019 at UNILAC-GSI (Darmstadt)
- 113h of beamtime granted (ARIES collaboration)
- Aim: investigate the degradation of material for collimators under the effect of radiation damage
  - Focus on the displacement per atom (**dpa**)→used to compare damage level in different experiments





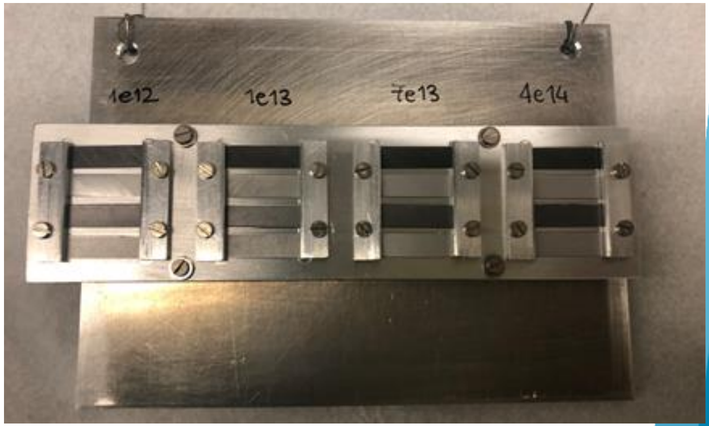
Irradiation test overview

- Ca ions of 4.8 MeV/u
- 4 target station→irradiation time →fluence →dpa
- 2 types of samples



RF

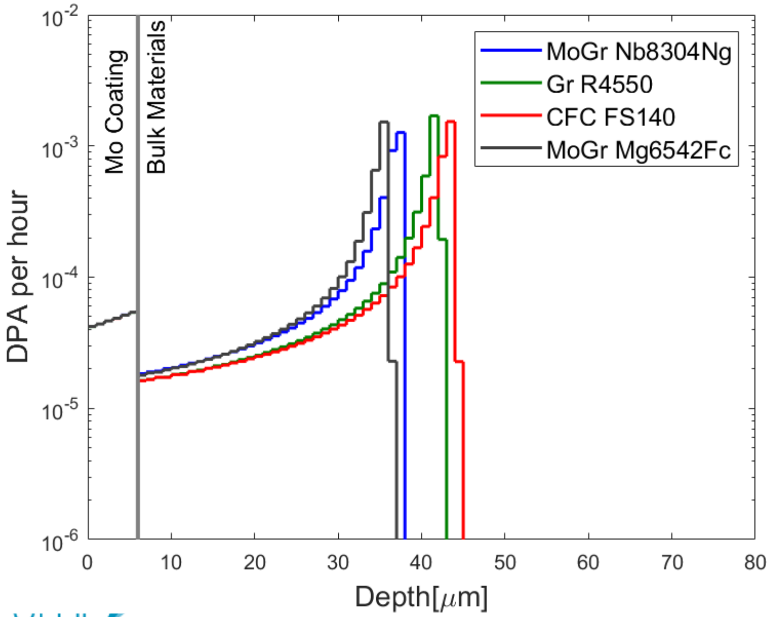
	Fluences [ions/cm²]
	$1\cdot10^{12}$
	$1\cdot10^{13}$
	$7\cdot10^{13}$
	$4\cdot10^{14}$

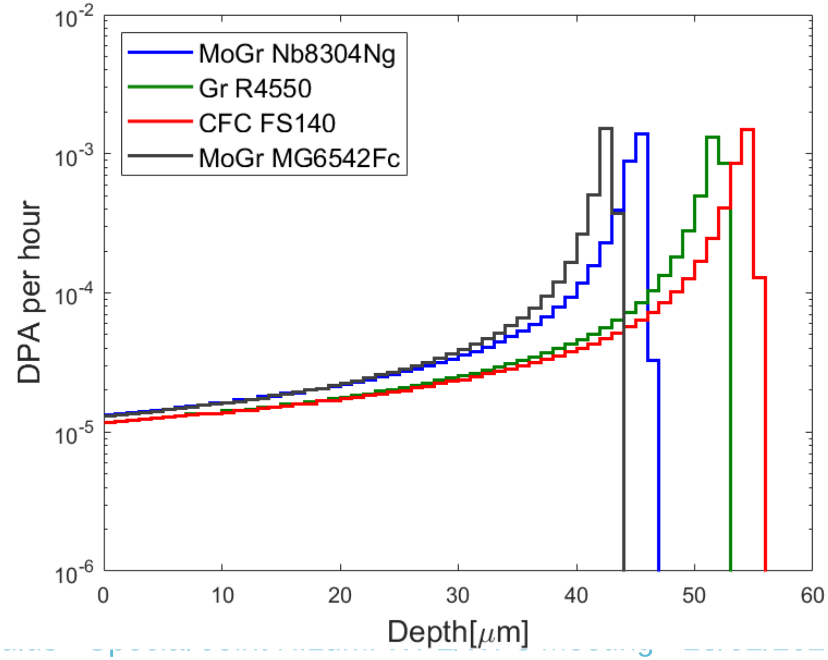


DC

Irradiation test overview

- Ca ions of 4.8 MeV/u
  - Low activation: shipped at CERN 2 months after and measured w/o gloves box/hot cells (dose rate<0.1µSv/h)
  - High damage rate (dpa rate)
  - Non-uniform dpa
  - Small penetration (<50µm)



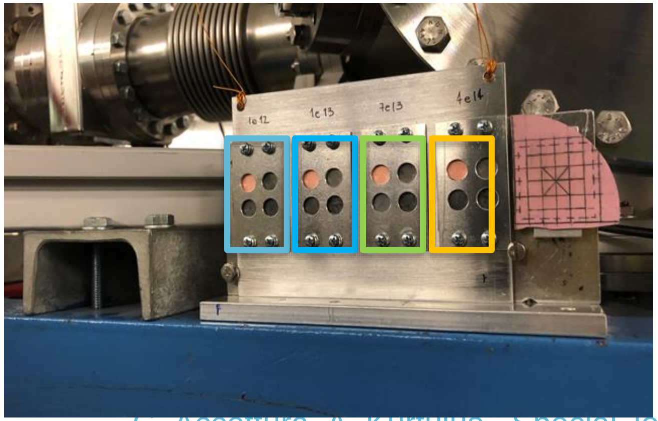


Irradiation test overview

- Test goal: reach peak dpa expected at the end of HL-LHC

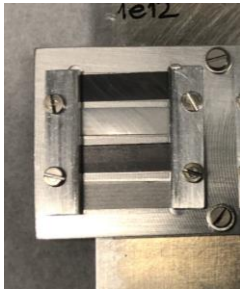
	Peak DPA collimator HL-LHC life
Mo coating	$1 \div 3 \cdot 10^{-3}$
MoGr secondary	$4 \cdot 10^{-4}$
MoGr primary	0.3 (in a small points, to be averaged)

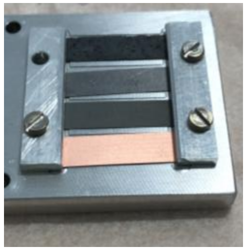
Fluences [ions/cm²]	Peak DPA coating	Peak DPA bulk
$1 \cdot 10^{12}$	$\sim 2.8 \cdot 10^{-6}$	$\sim 1.1 \cdot 10^{-4}$
$1 \cdot 10^{13}$	$\sim 2.8 \cdot 10^{-5}$	$\sim 1.1 \cdot 10^{-3}$
$7 \cdot 10^{13}$	$\sim 1.9 \cdot 10^{-4}$	$\sim 7.8 \cdot 10^{-3}$
$4 \cdot 10^{14}$	$\sim 1.1 \cdot 10^{-3}$	$\sim 4.4 \cdot 10^{--2}$



Irradiation test overview

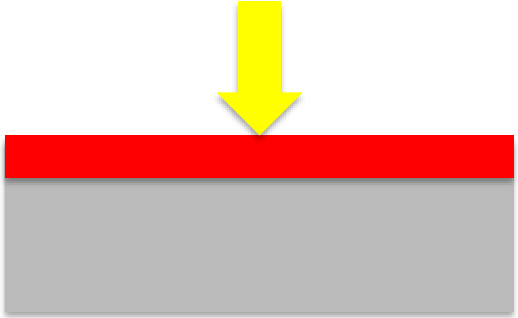
- 4 bulk materials and 4 coatings tested:





- MoGr Nanoker (Nb8304Ng)→ production grade
- Graphite SGL R4550
- Nb8304Ng+ Mo coating DTI
- R4550+ Mo coating DTI

- CFC Tatsuno FS140
- MoGr Brevetti (Mg6541Fc)→ with fibers, tested in HRTM36
- Mg6541Fc+ Mo coating CERN
- Nb8304Ng+ Cu coating DTI



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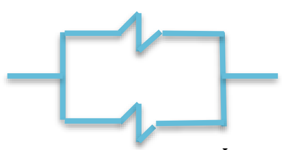
DC measurement of electrical resistivity

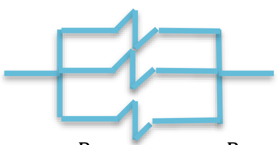
- Four probes method set-up
- Parallel resistance model (2 or 3-layers)
- Minimized sample thickness to see the contribution of the irradiated layer
- Radiation penetration depth from FLUKA simulation

$R_{irradiated} = ?$

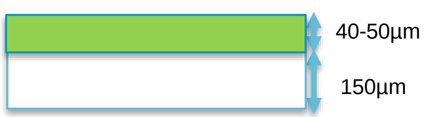


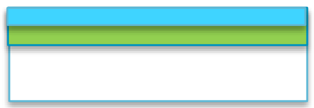
$$R_{bulk} = \rho_{pristine} \frac{L}{d_1 * t_{bulk}}$$

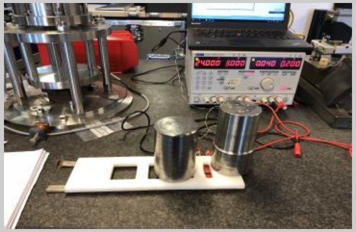
$R_{coating} = ?$

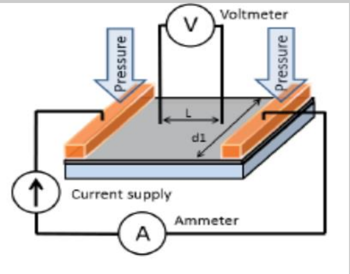


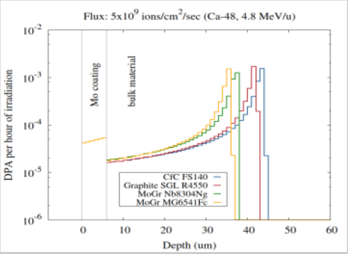
$$R_{bulk} = \frac{R_{bulk\_pristine} \cdot R_{bulk_{irrad.}}}{R_{bulk\_pristine} + R_{bulk_{irrad.}}}$$











Four probe methods measurements @ Impedance meeting: https://indico.cern.ch/event/816840/

- **All the irradiated layer is measured**

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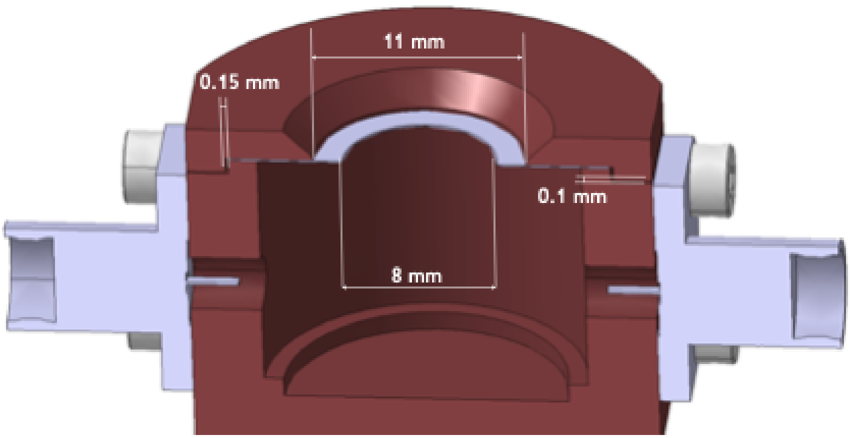
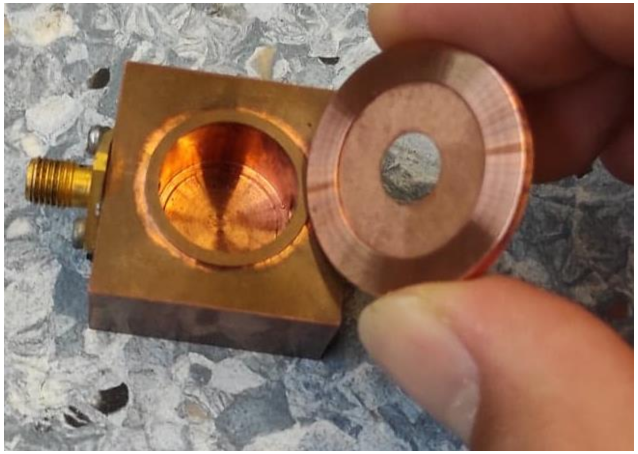
H011 cavity design

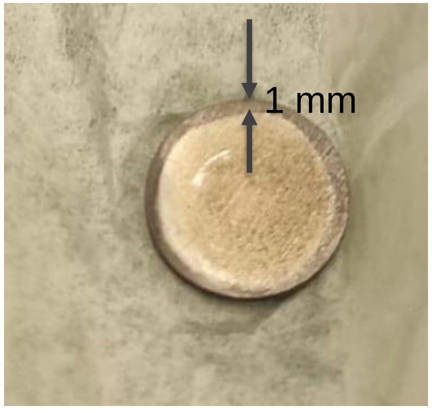
- Resonators can be used to probe the electrical resistivity of materials:
  high operating frequency → small skin depth → thin layers
- With IWG-team, we designed a dedicated H011 cavity working at 24.7 GHz.
- H011 mode is allows to measure samples on the top of the cavity and it is insensitive to contacts.
- The cavity is suitable to measure Ø10mm samples (GSI) and larger samples thanks to an adapting cover.
- The cavity has been fabricated by EN-STI (many thanks!) and successfully tested.

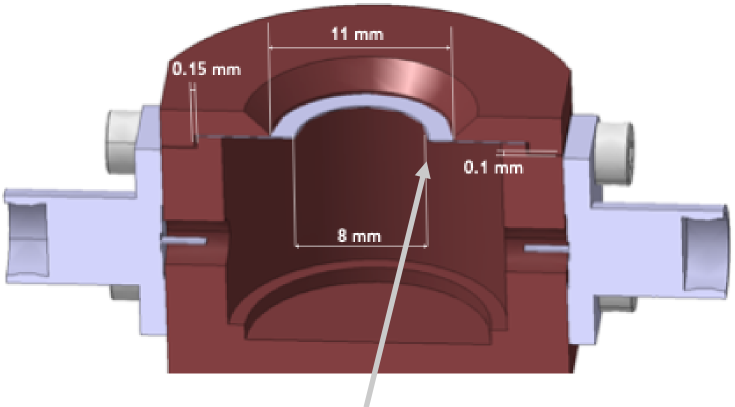




Adapting mask

- The irradiated samples at GSI have a diameter of 10mm with 1mm of circular uncoated surface on the coated face.
- A thin foil masks the uncoated surface and exposes the coated one to the field of the cavity.
- The use of a mask allows to keep the operating frequency low enough without compromising the measurement sensitivity.



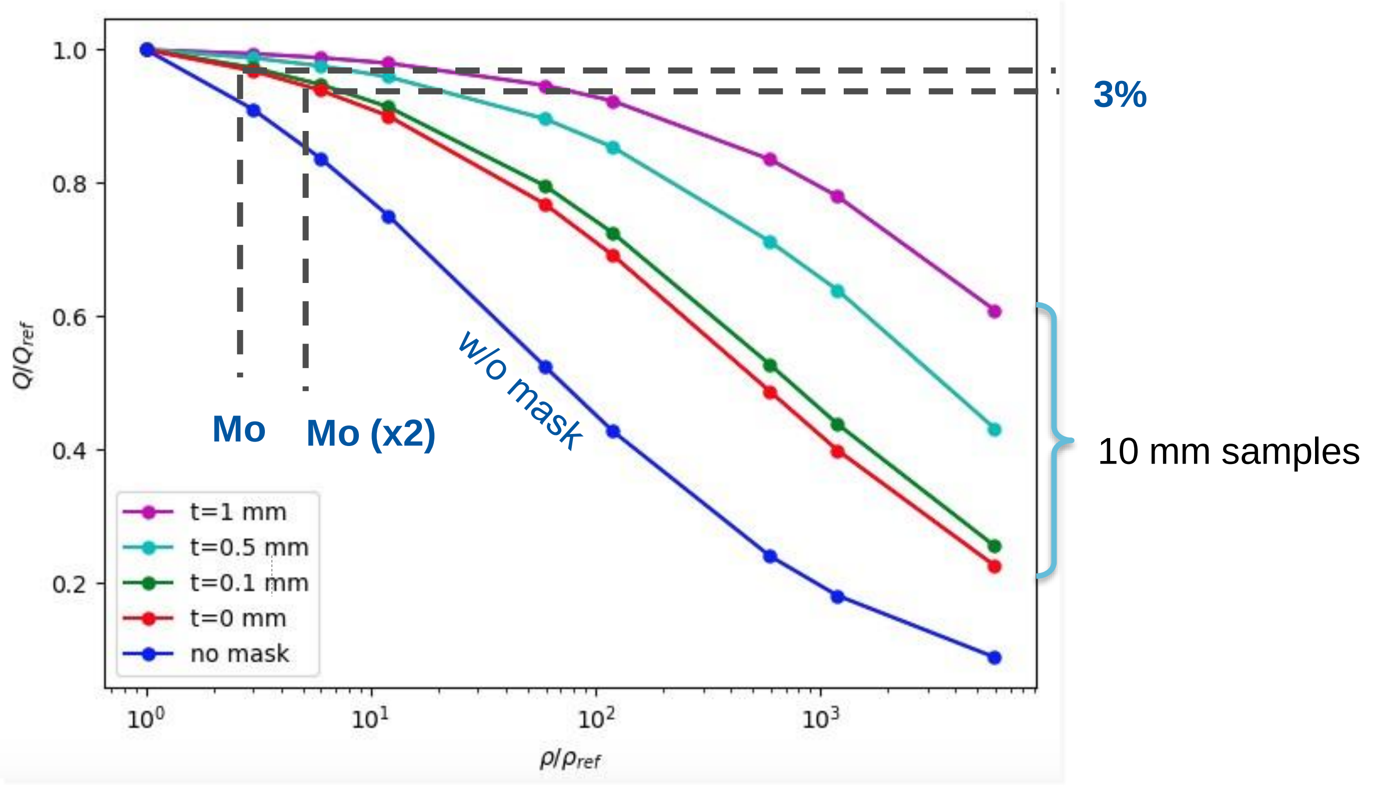


A mask is needed to reduce aperture size

Detailed explanation of the resonant cavity method can be found: https://doi.org/10.3390/coatings10040361

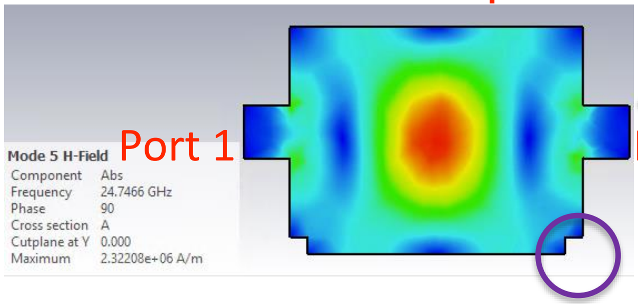
Sensitivity analysis

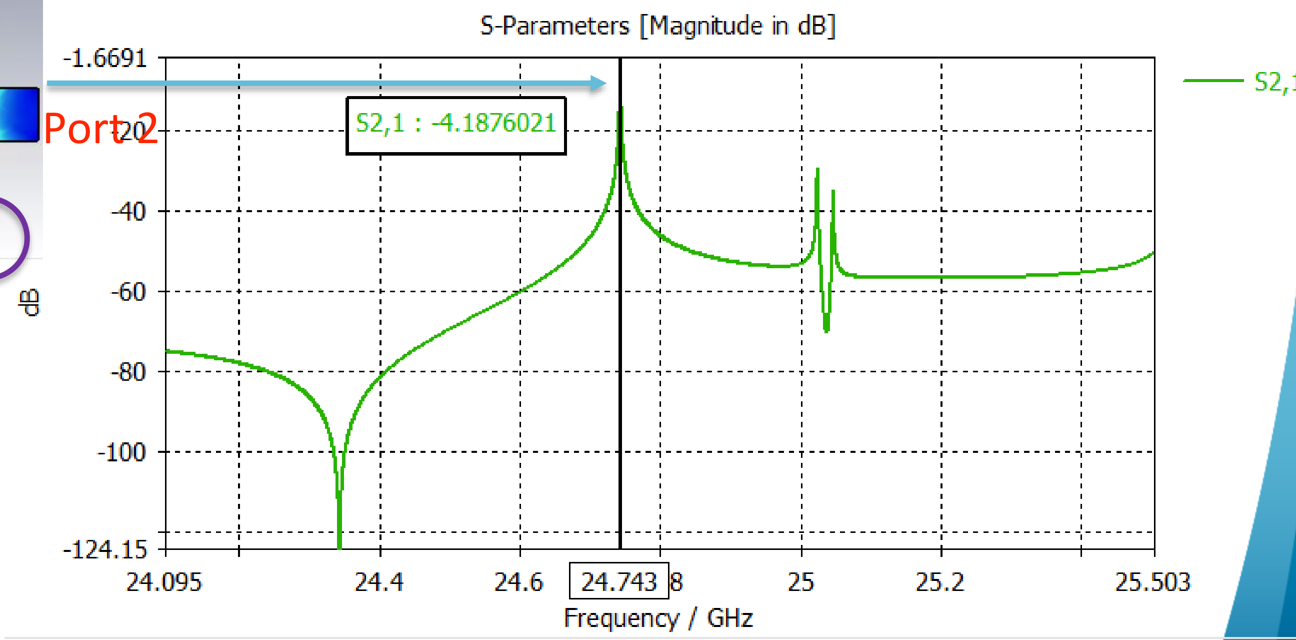
- The effect of mask thickness is studied on sensitivity of the cavity.
- With 0.1mm mask thickness ~ 3% sensitivity to detect x2 Mo.



H011 cavity simulations

- The main mode H field resonates at 24.76 GHz with a $Q_0^{Cu} = 17260$
- The adjacent TM mode is detuned by the bottom chamfer.
- The operating frequency translates in:
  - Skin depth for Mo: ~0.7um
  - Skin depth for MoGr: ~1um

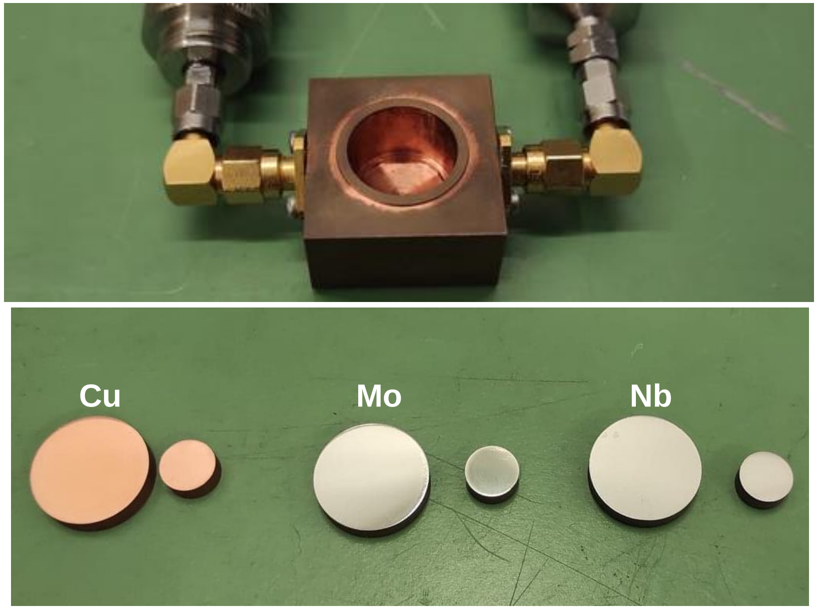


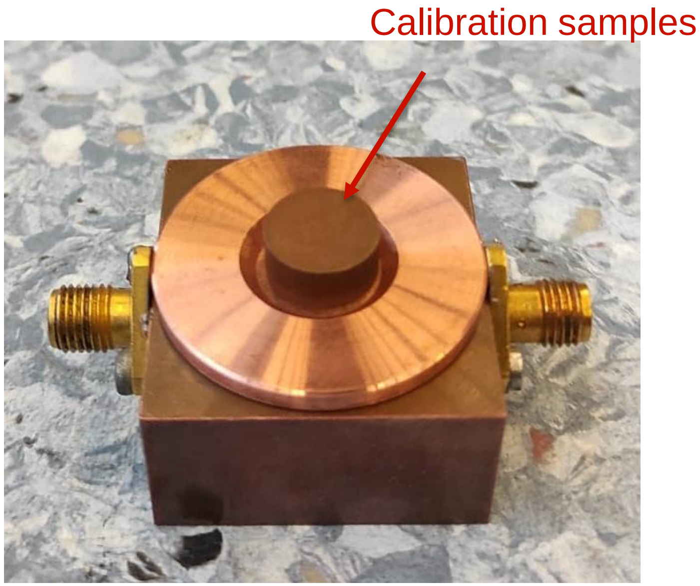


Calibration

- Calibration samples are needed to be able to deduce the resistivity of unknown materials.
- Chosen Cu (17.1 nOhm.m), Mo (53.4 nOhm.m), Nb (152nOhm.m)

  → We can span the range of resistivity of interest!
- Calibration samples have been polished to minimize the effect of surface roughness.

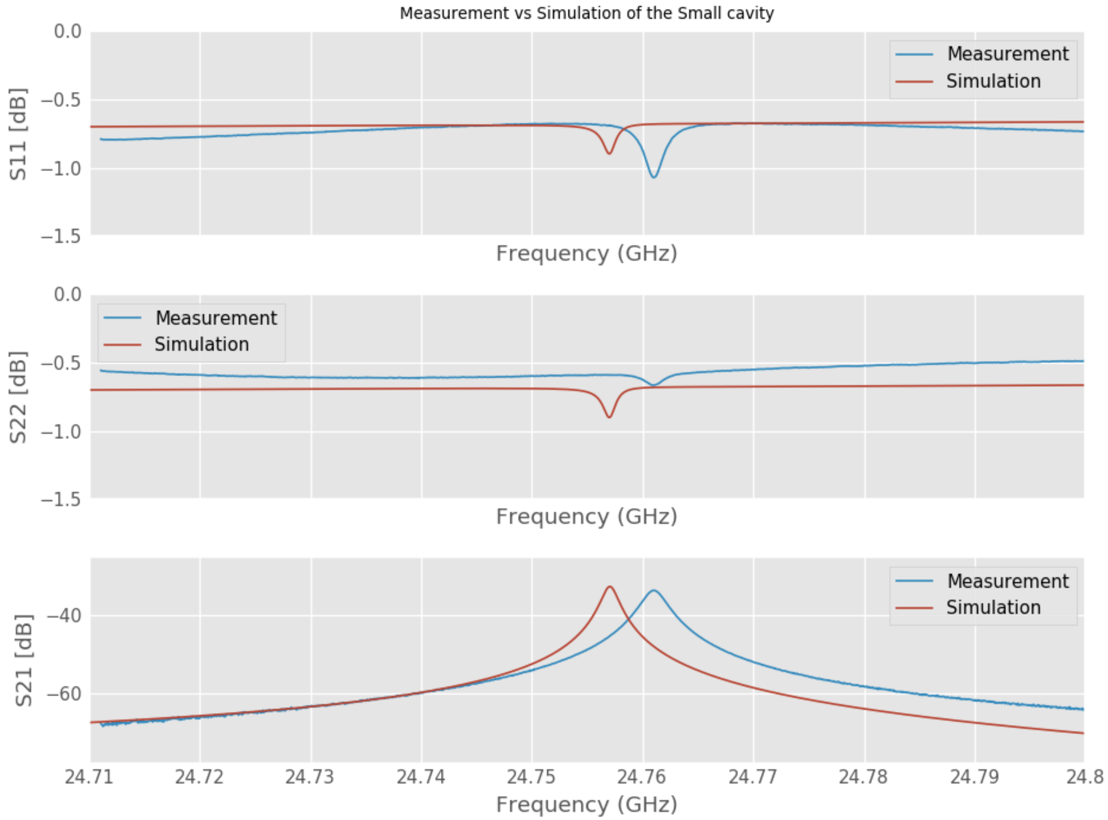




Measurements vs Simulations

- We compare measurements and simulations for Cu calibration sample.
- Small (~8MHz) frequency shift on resonant mode due to probes modeling.
- The measured Q is smaller than the simulated one:

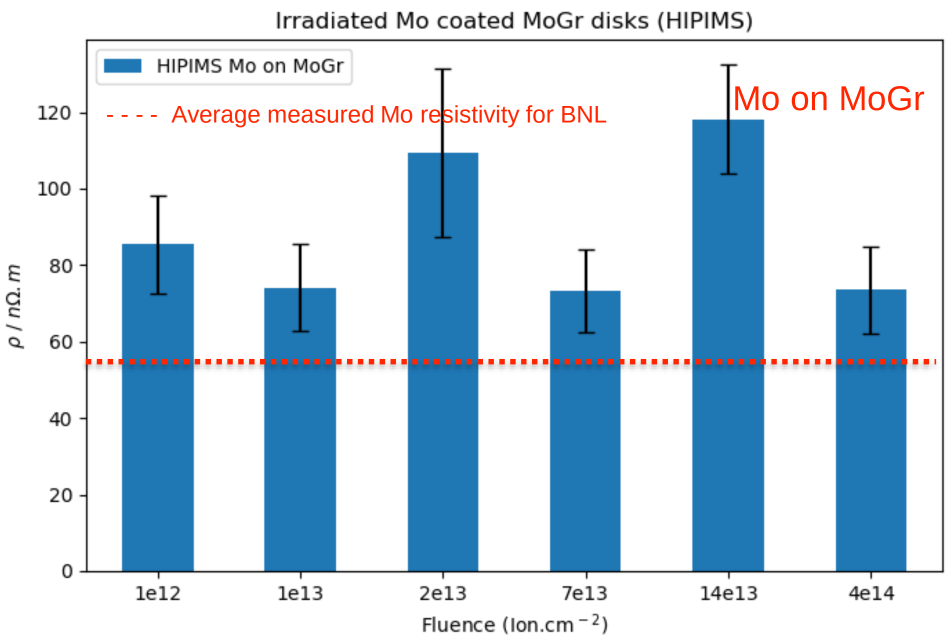
→ mainly due to the residual roughness losses on cavity walls: not a problem as this contribution is the same for all the samples.

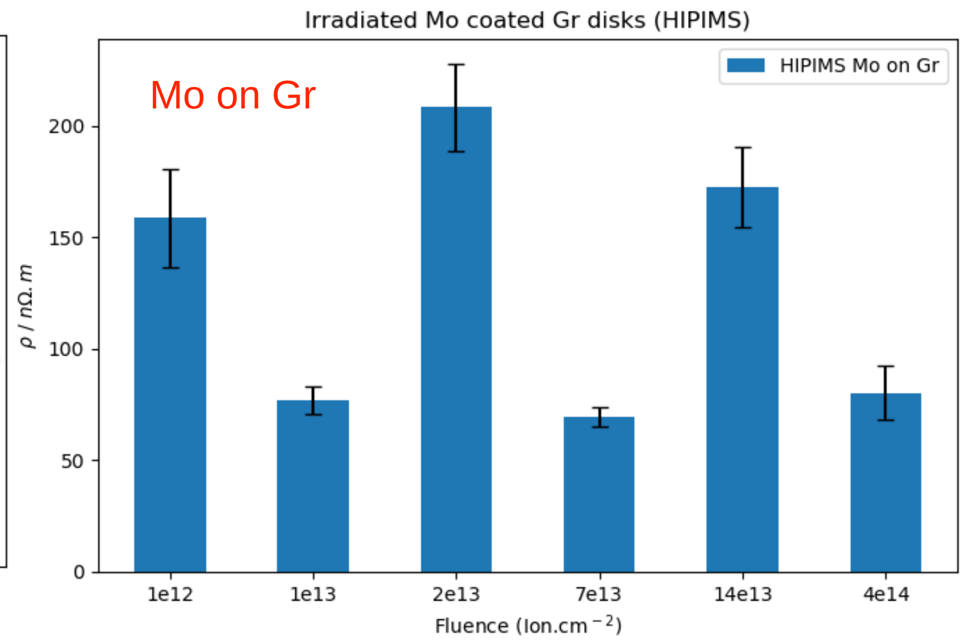


Q_{sim}= 17260
Q_{meas}= 11160

GSI-2019 irradiated Ø10mm samples

- Ion irradiation at GSI.
- All samples irradiated at 6 fluences: 1e12, 1e13, 2e13, 7e13, 14e13, 4e14.
- Measurement results of HIPIMS Mo on MoGr (left) and Mo on Gr (right) are given below with six different fluencies levels.

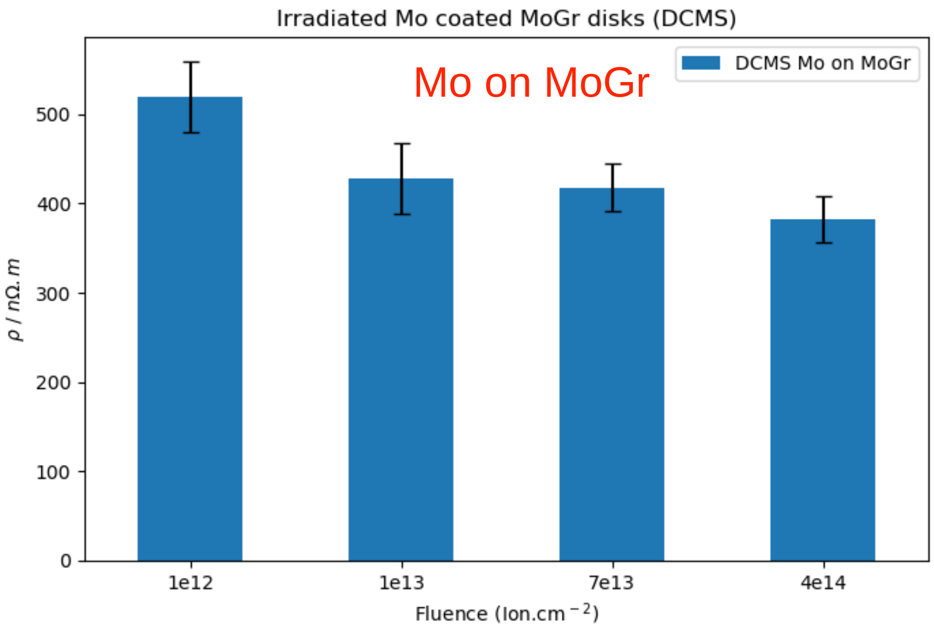


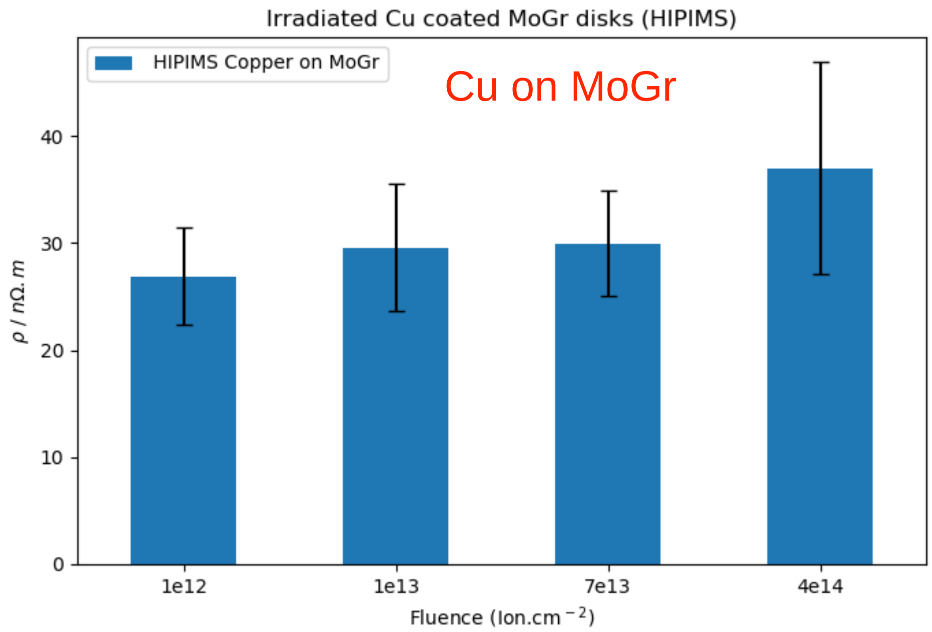


- Increase of resistivity of Mo on MoGr within factor 2 with radiation.
- No clear trend with fluence.
- For Mo on Gr no clear reference available: more studies needed.

GSI-2019 irradiated Ø10mm samples

- Ion irradiation at GSI.
- All samples irradiated at 4 fluences: 1e12, 1e13, 7e13, 4e14.
- Measurement results of DCMS Mo on MoGr (left) and HIPIMS Cu on MoGr (right) are given below with four different fluencies levels.



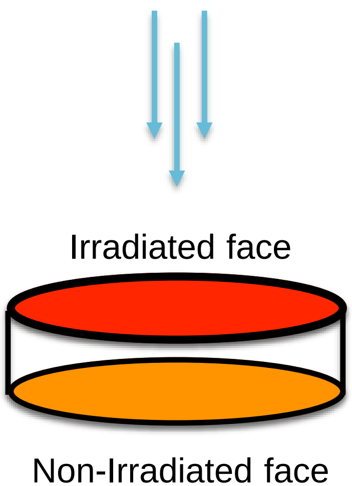


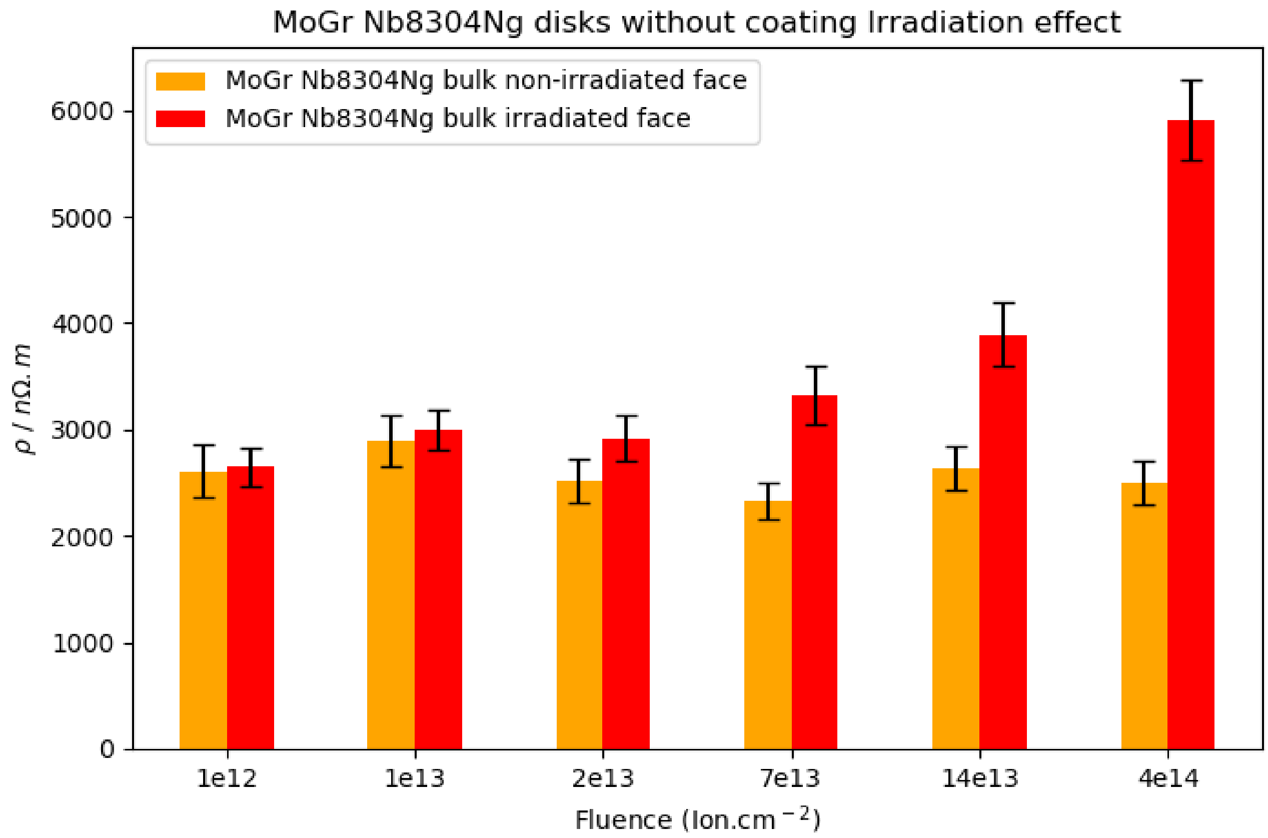
- Confirmed larger resistivity by using DCMS technique.
- No trend with fluence.

GSI-2019 irradiated Ø10mm samples

- Ion irradiation at GSI.
- Uncoated Ø10mm MoGr Nb8304Ng samples comparisons with irradiated and non-irradiated faces are shown below.

→ Increment of resistivity observed on the irradiated face.

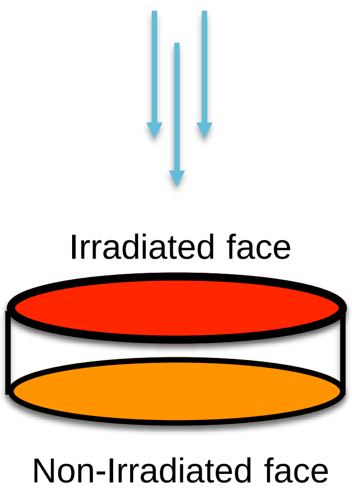


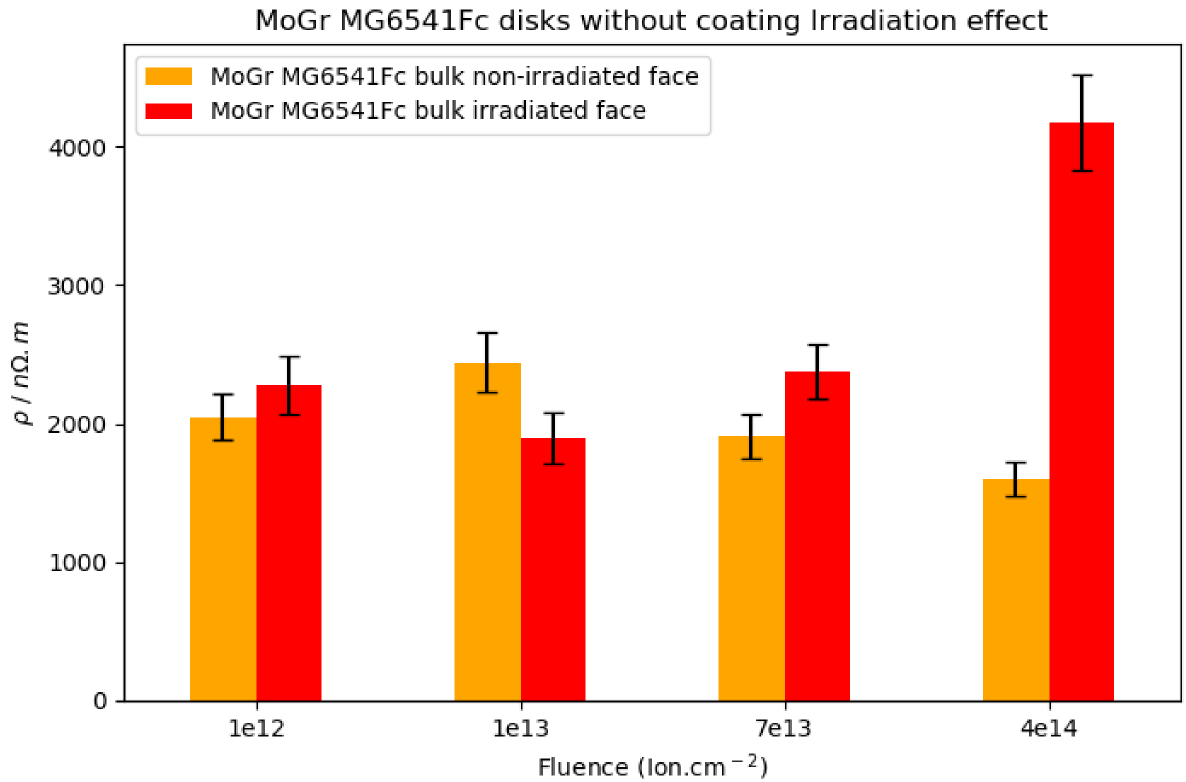


GSI-2019 irradiated Ø10mm samples

- Ion irradiation at GSI.
- Uncoated Ø10mm MoGr MG6541Fc samples comparisons with irradiated and non-irradiated faces are shown below.

→ Increment of resistivity observed on irradiated face (except 1e13).

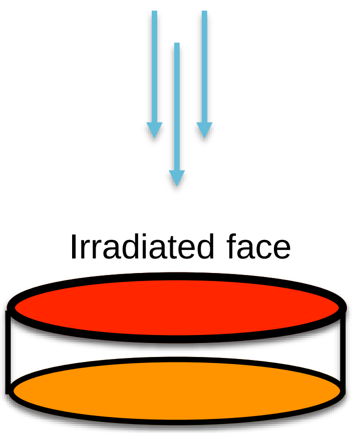


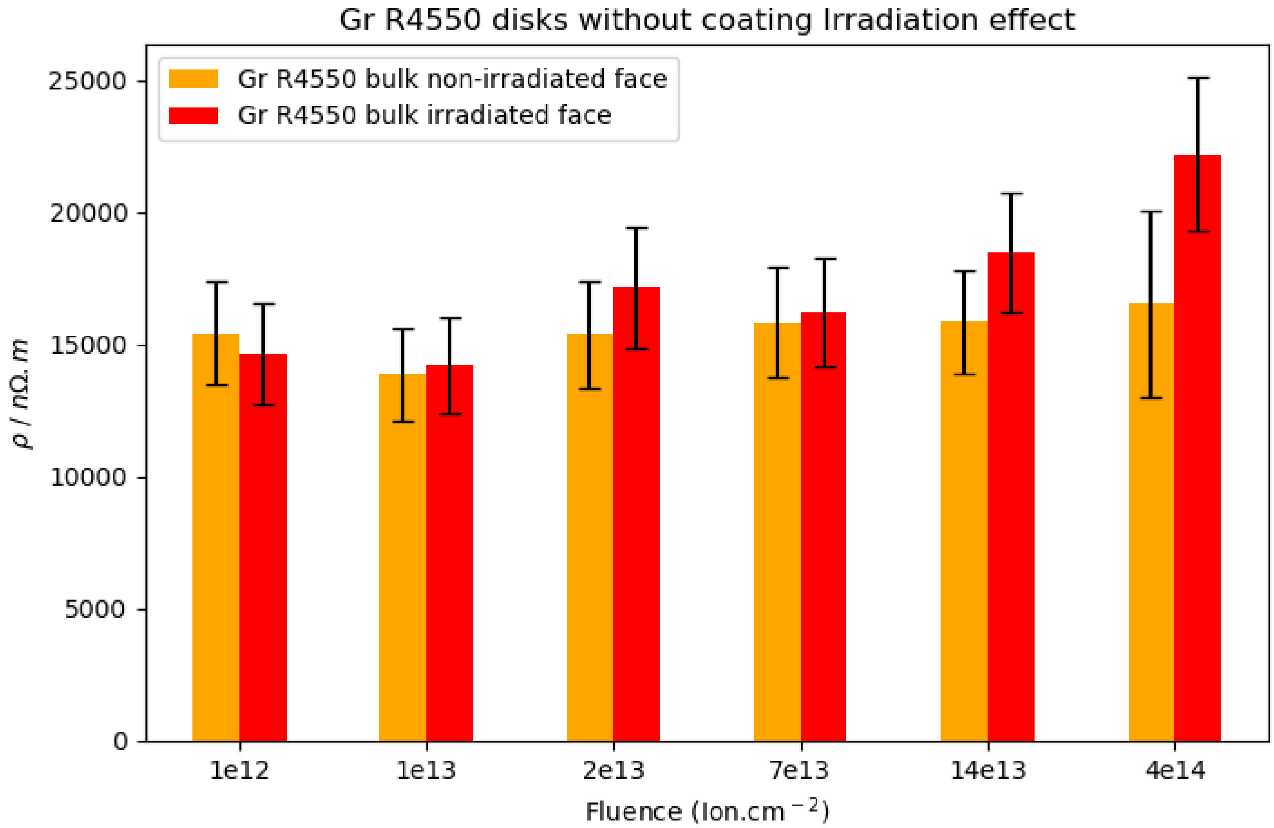


GSI-2019 irradiated Ø10mm samples

- Ion irradiation at GSI.
- Uncoated Ø10mm Gr R4550 samples comparisons with irradiated and non-irradiated faces are shown below.

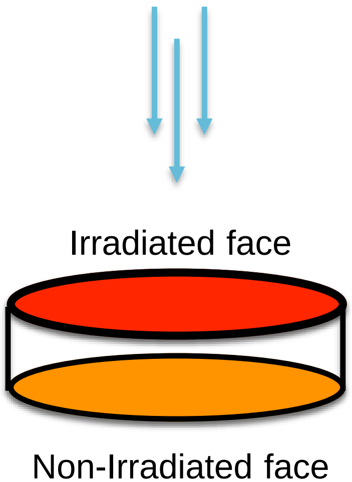
→ Increment of resistivity observed on irradiated face (except 1e12).

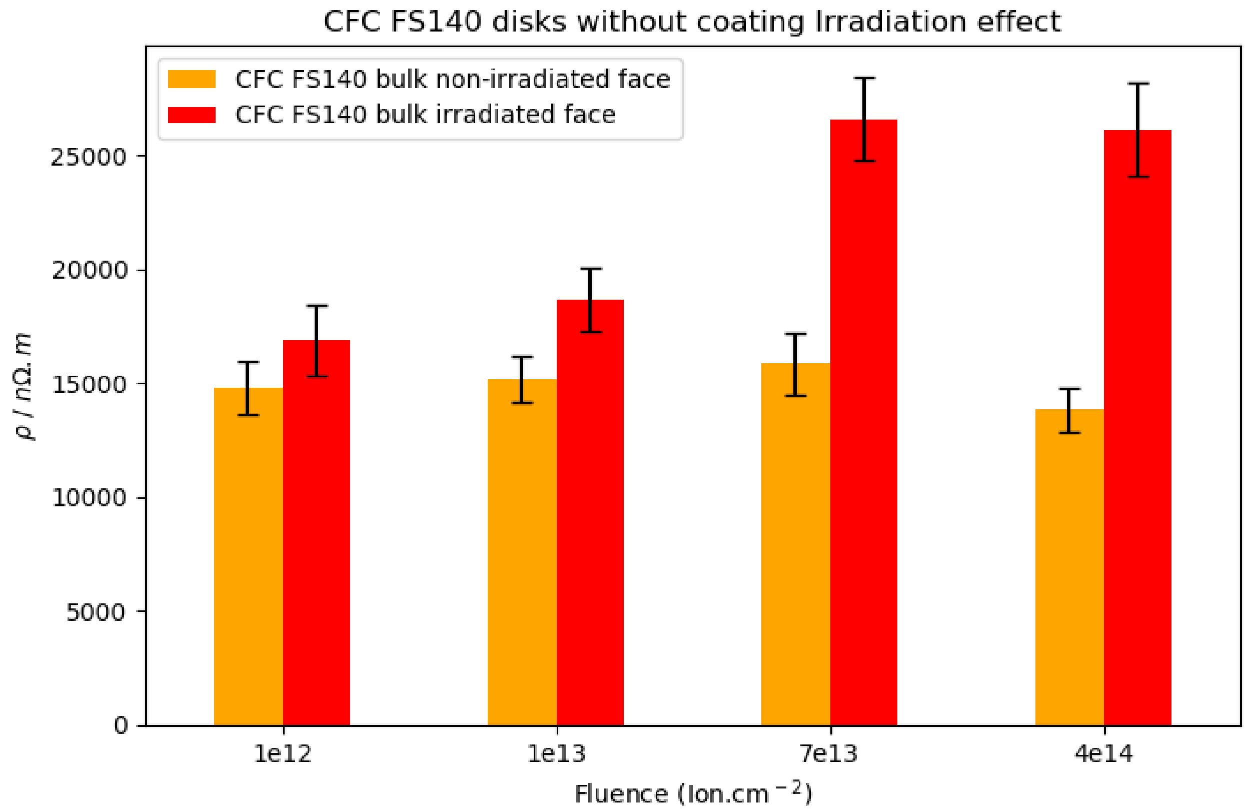




GSI-2019 irradiated Ø10mm samples

- Ion irradiation at GSI.
- Uncoated Ø10mm CFC FS140 samples comparisons with irradiated and non-irradiated faces are shown below.
- Increment of resistivity observed on irradiated face.





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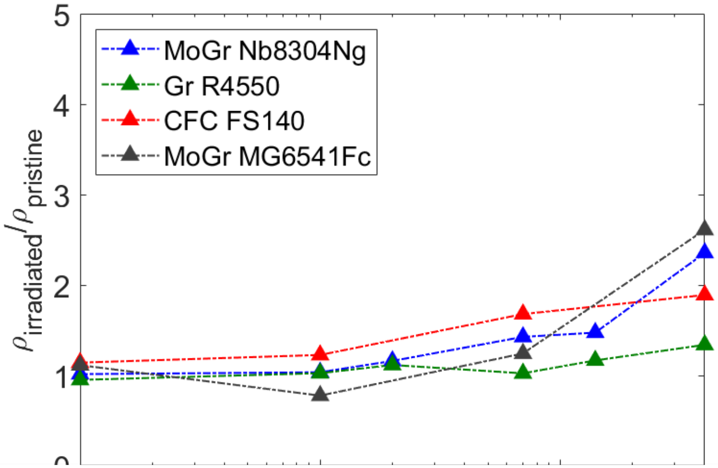
Comparison materials-bulk

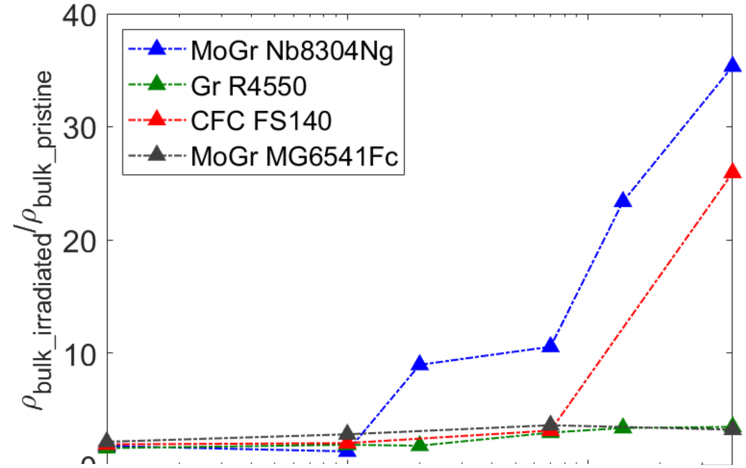
- Comparison as a function of
  - Fluence
  - Average dpa
  - Peak dpa

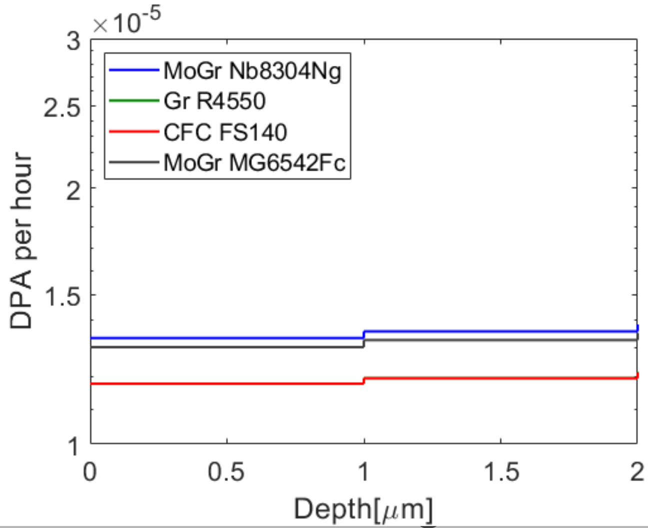
Comparison materials-bulk

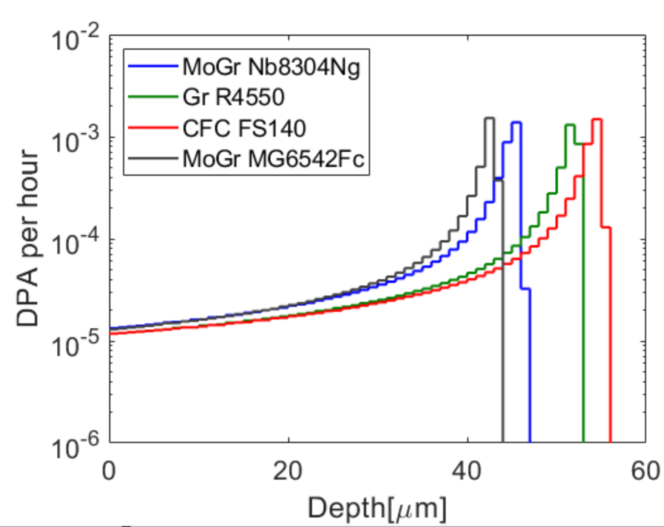
RF



DC

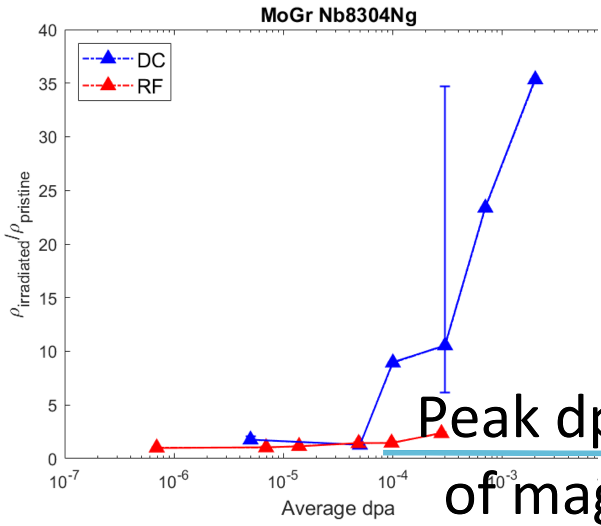


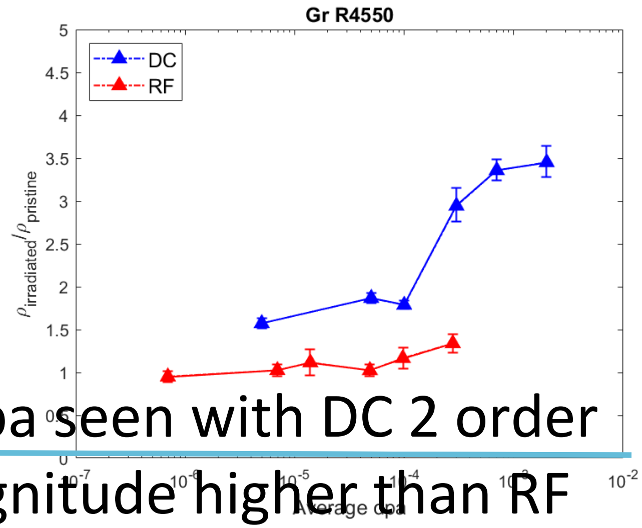




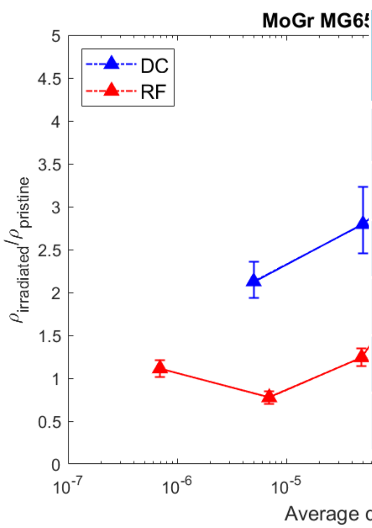
- RF is measuring only 1μm (dpa<<than whole layer)

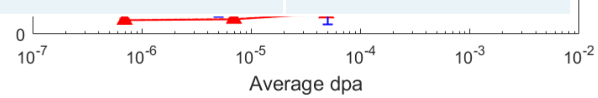
Resistivity vs average dpa





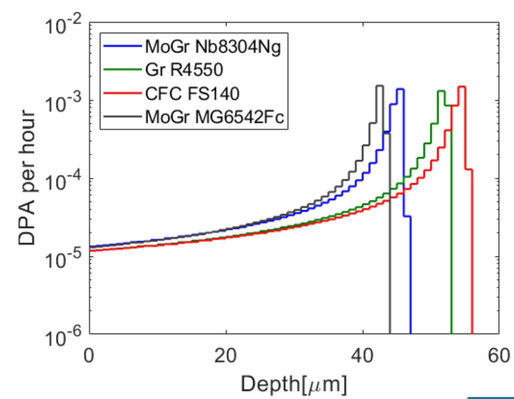
Peak dpa seen with DC 2 order of magnitude higher than RF



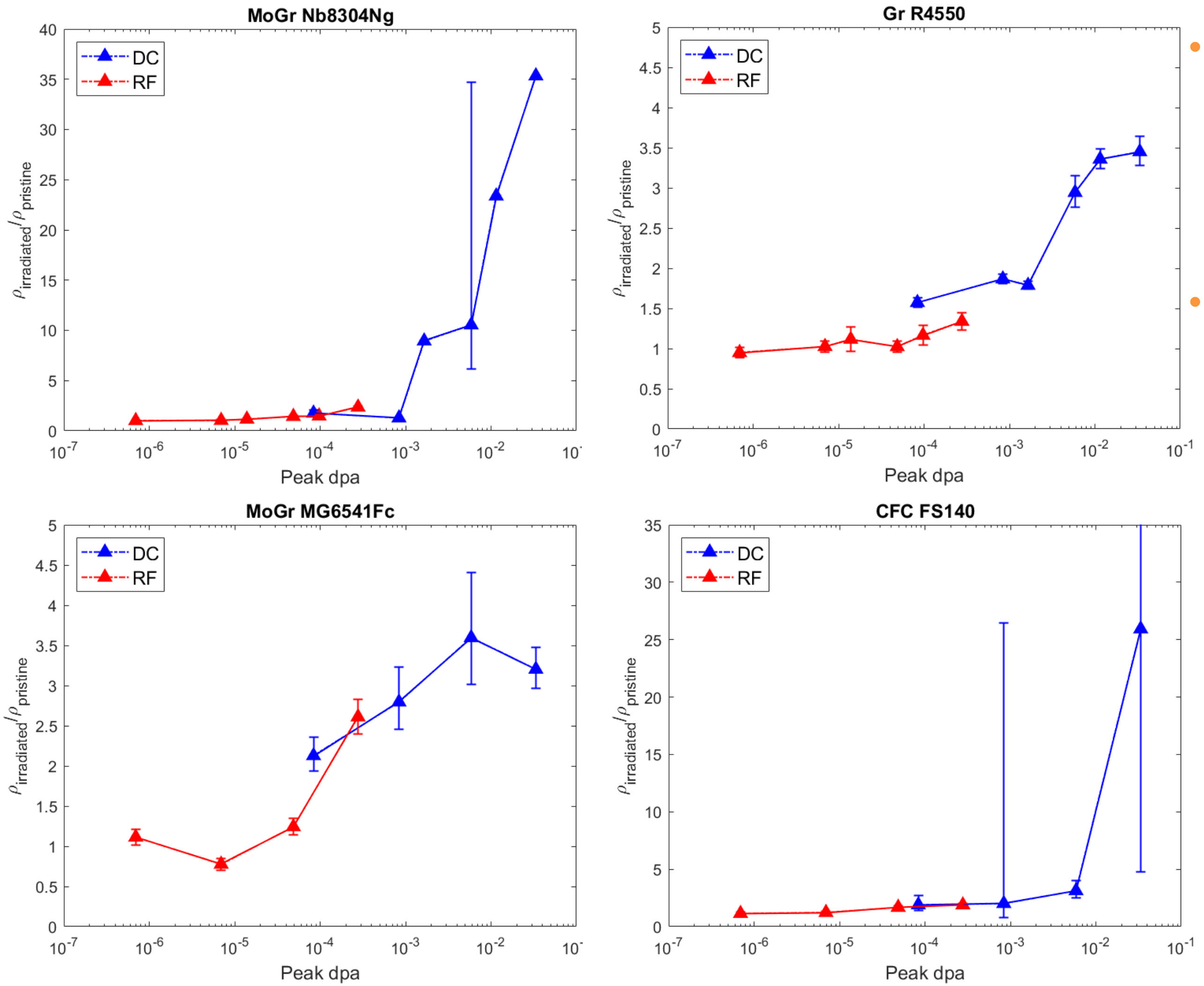


Fluences [ions/cm2]	dpa peak RF	dpa peak DC
$1 \cdot 10^{12}$	$\sim 7 \cdot 10^{-7}$	$\sim 8 \cdot 10^{--5}$
$1 \cdot 10^{13}$	$\sim 7 \cdot 10^{-6}$	$\sim 8 \cdot 10^{-4}$
$2 \cdot 10^{13}$	$\sim 1 \cdot 10^{-5}$	$\sim 1 \cdot 10^{-3}$
$7 \cdot 10^{13}$	$\sim 5 \cdot 10^{-5}$	$\sim 6 \cdot 10^{-3}$
$1.4 \cdot 10^{14}$	$\sim 1 \cdot 10^{-4}$	$\sim 1 \cdot 10^{-2}$
$4 \cdot 10^{14}$	$\sim 3 \cdot 10^{-4}$	$\sim 3 \cdot 10^{-2}$

- At lower fluences DC/RF similar values and trend
- At higher fluences:
  - Less sensitivity for higher resistivity of the irradiated layer
  - Non-linear phenomena related to clustering of defects→ peak dpa may be relevant

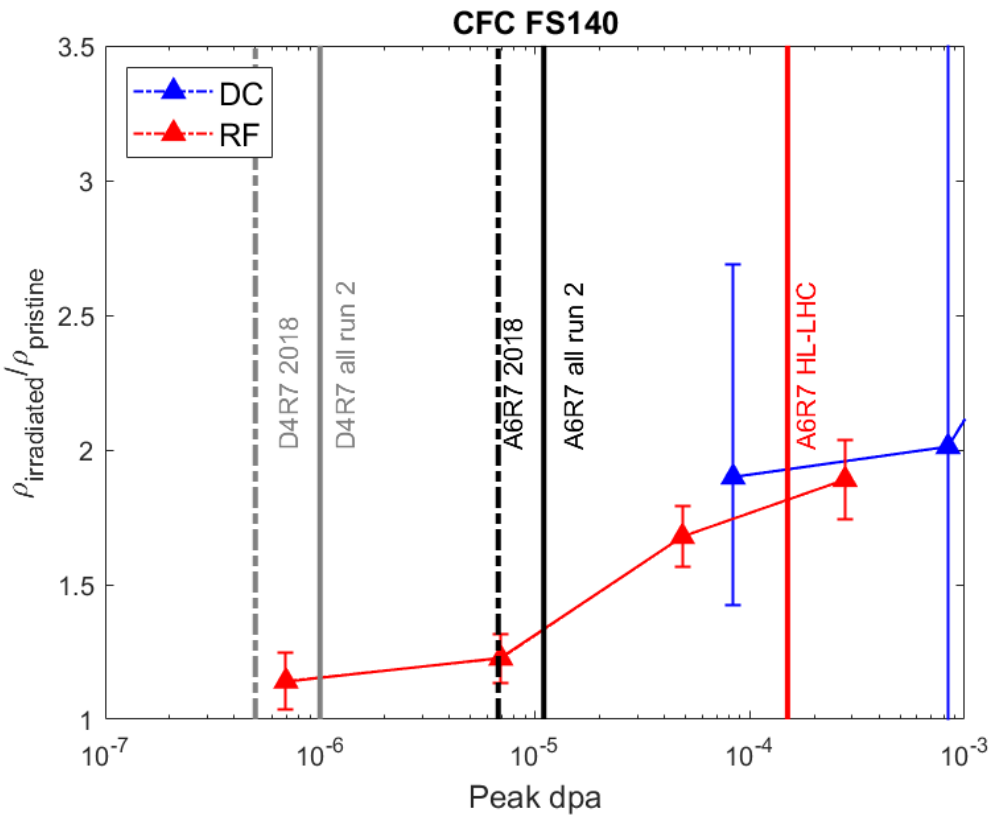


Resistivity vs peak dpa

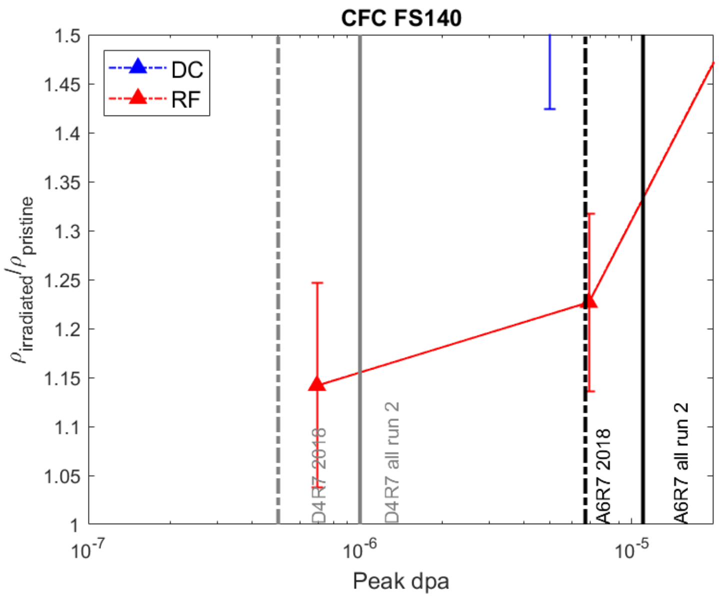


- Trend more similar, important role of peak dpa→ more stable defects, more impact on resistivity
- IMPORTANT to consider both average and peak dpa.

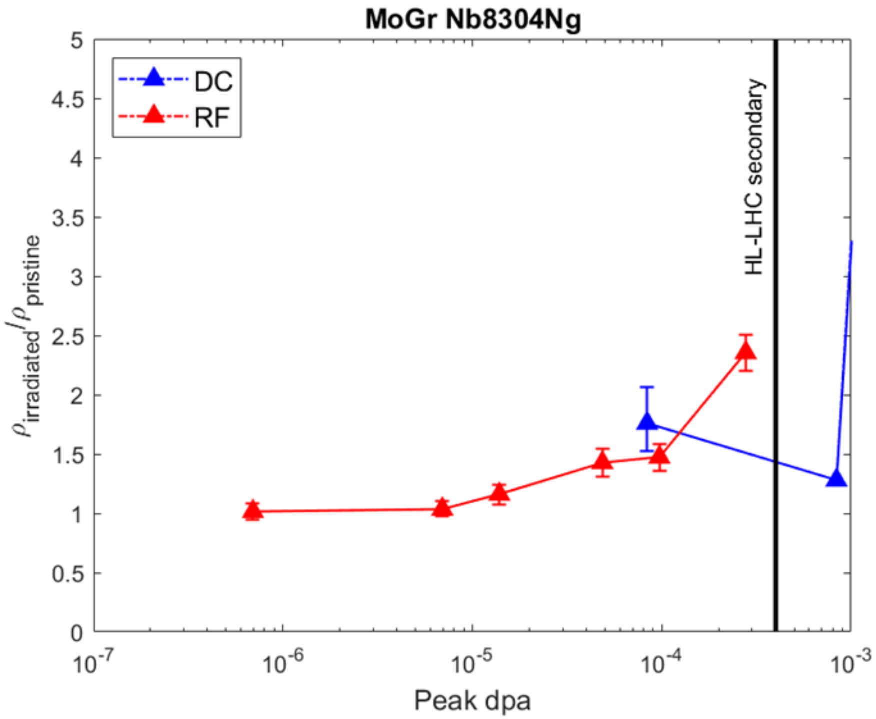
Comparison with expected value in the LHC



- RF investigates low-dpa area, useful to compare with LHC data
- Discussed *@ ColUSM 127: https://indico.cern.ch/event/921426/*

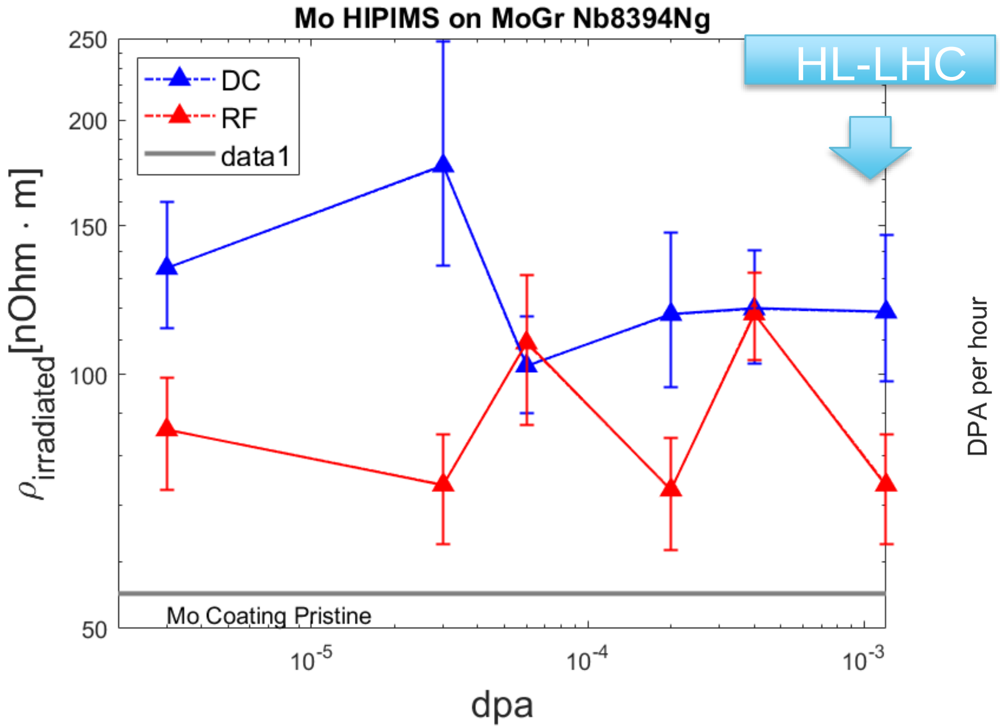


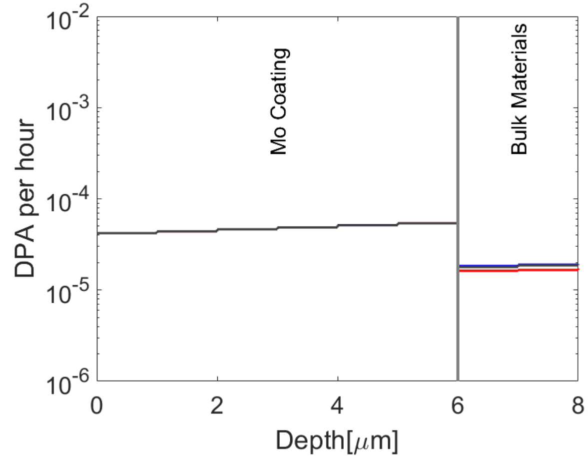
Comparison with expected value in the HL-LHC



- DC investigate higher peak dpa levels, possible to reach dpa level of secondary collimators

Comparison materials-Coating





- Dpa uniform in the coating, not needed to distinguish average and peak
- No clear trend of increase of conductivity as a function of the dpa.
- RF measured only after irradiation
- Both methods indicate a maximum resistivity increase of a factor 2

Conclusions

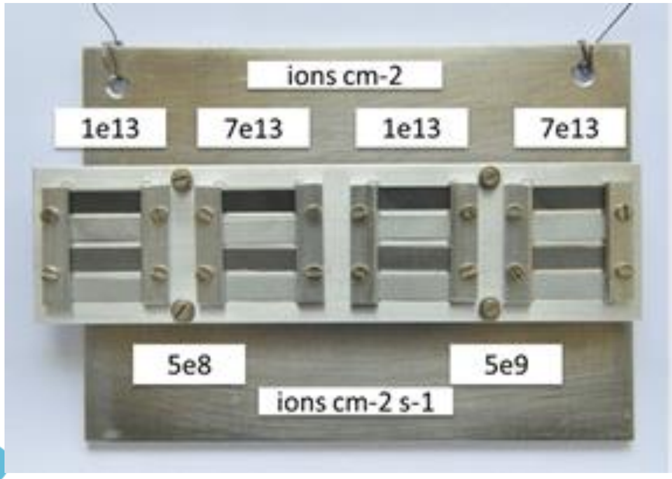
- The two techniques investigate different dpa levels:
  - DC measurement overestimates the increase of resistivity in terms of average dpa→ the peak dpa must be taken into account
  - DC and RF are complementary techniques for measuring as a function of peak dpa: the investigated dpa ranges covers both LHC and HL-LHC expected dpa levels
- RF measurement confirm that there is no strong dependence of coating resistivity increase as a function of the dpa
- Both techniques indicate a maximum resistivity increase of Mo coating of a factor 2

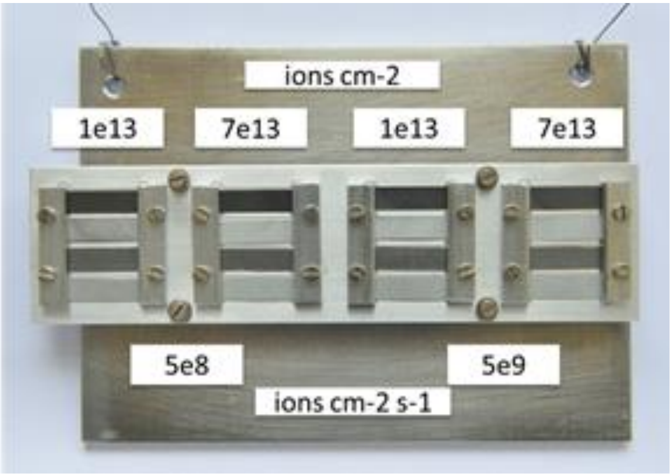
Future irradiation campaign

- **CFC collimator** coming from Run II DC measurements planned for this year

- **Ion irradiation** foreseen in 2020, postponed to March,2021
- 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?)

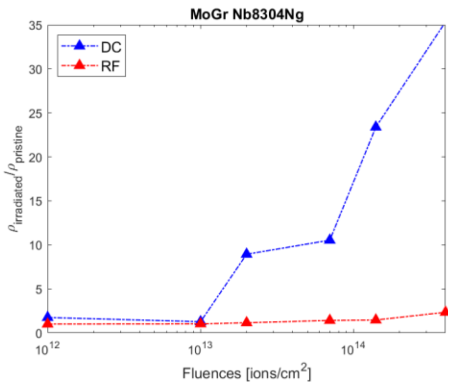
- **Proton irradiation** at BNL-BLIP
- DCMS Mo coating on MoGr and CFC→ irradiated in 2018, capsule analysis planned in spring 2021
- HIPIMS Mo coating on MoGr and Gr→ characterized at CERN, irradiation pending (*see N. Solieri et al., Update on BLIP irradiation tests and RaDIATE activities, Tech. Rep. ARIES WP17 (PowerMat) Annual Meeting (CERN, Geneva, Switzerland, 2020*)

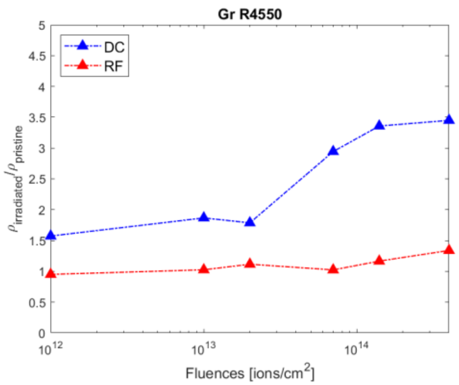


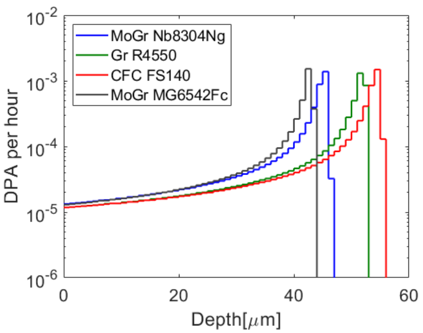


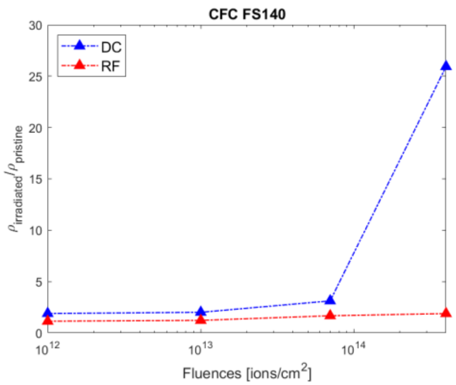
Thank you for your attention!

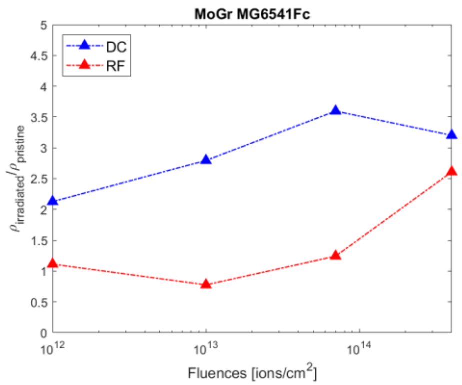
Resistivity vs fluence

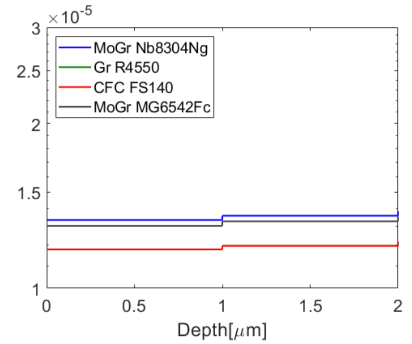






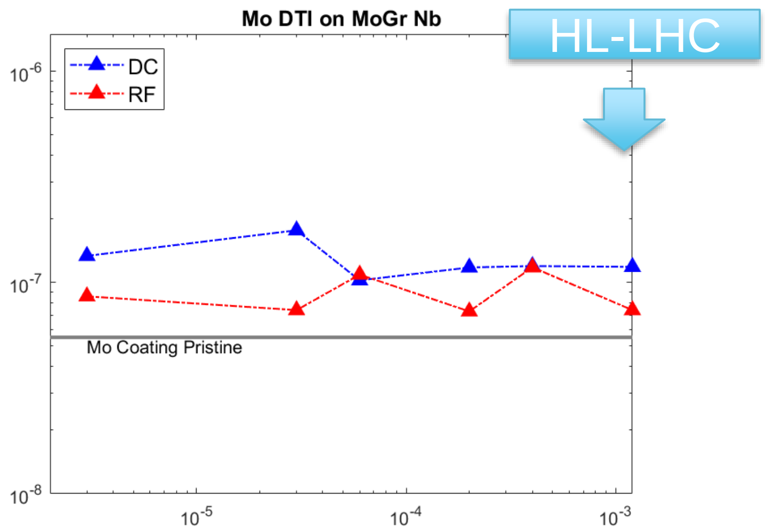






- Comparison in terms of fluences not meaningful because dpa rate changes as a function of depth:
  - DC is measuring all the irradiated layer
  - RF is measuring only 1um (dpa<<)

Coating



- No clear trend of increase of conductivity as a function of the dpa.
- RF measured only after irradiation
- Both methods indicate a maximum resistivity increase of a factor 2