Radiation Induced Effects in MoGr composites

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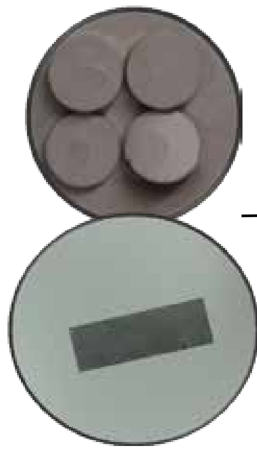
CERN

Overview

CERN

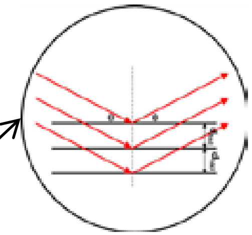
HL-LHC collimators

MoGR

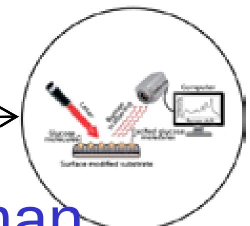


GSI

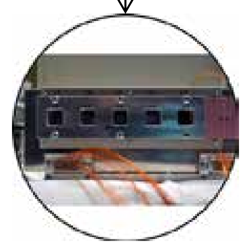
UNILAC



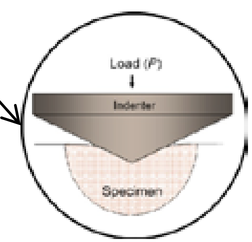
XRD



Raman Spectroscopy



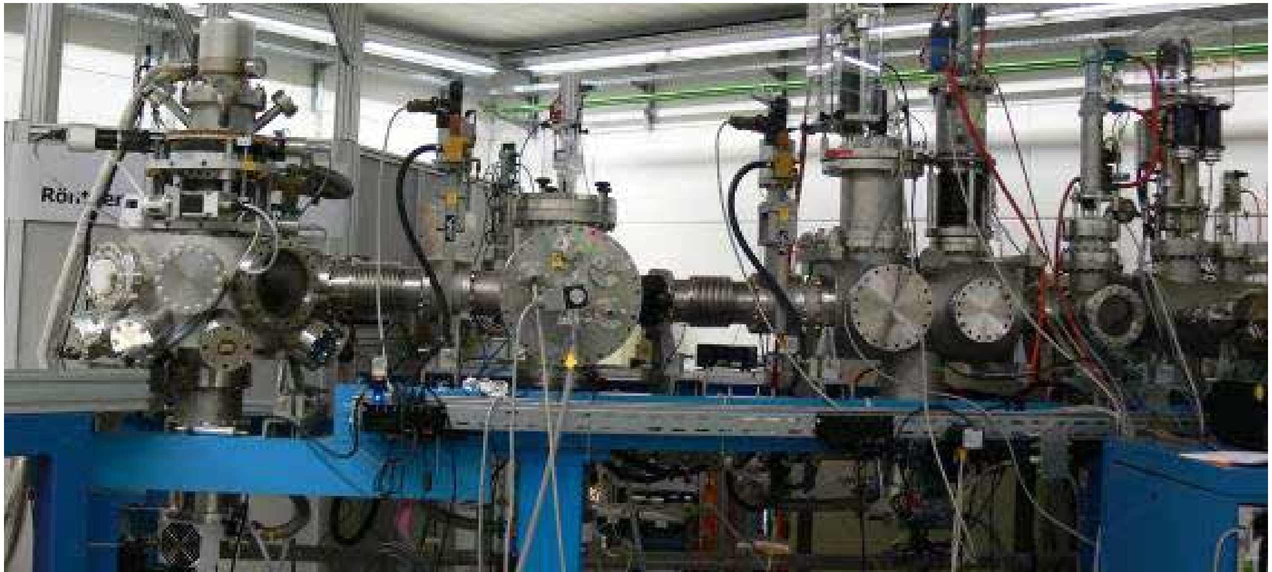
Resistivity Measurements



Nanoindentation

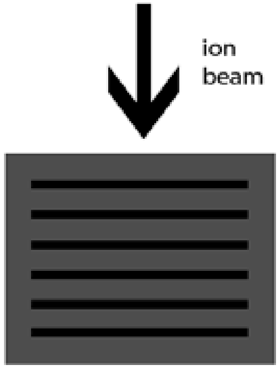
Ion irradiation at GSI

- U, 1.14 GeV, 0.5 ms, 0.6 Hz, $4x10^9$ ions/cm^2 s
- Bi, 1 GeV, 0.5 ms, 3.4 Hz, $1.2x10^9$ ions/cm^2 s
- Au, 945 MeV, 2ms, 40 Hz, $4x10^9$ ions/cm^2 s
- Ca, 236 MeV, 3ms, 40 Hz, $4x10^9$ ions/cm^2 s
- C, 71 MeV, 3ms, 32 Hz, $5x10^9$ ions/cm^2 s

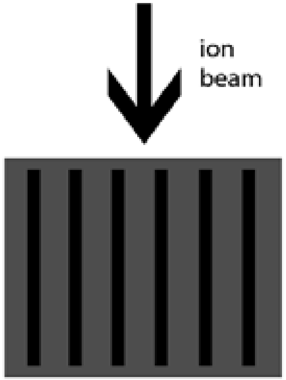


Molybdenum Carbide-Graphite Composites

- Molybdenum Carbide – Graphite – Carbon Fibers (MoGRCF)
- Inhomogeneous compound material
- Produced by hot pressing
- Anisotropic properties
- According to the basal planes orientation, the samples are categorized into in-plane and transversal direction
- Annealing applied before irradiation

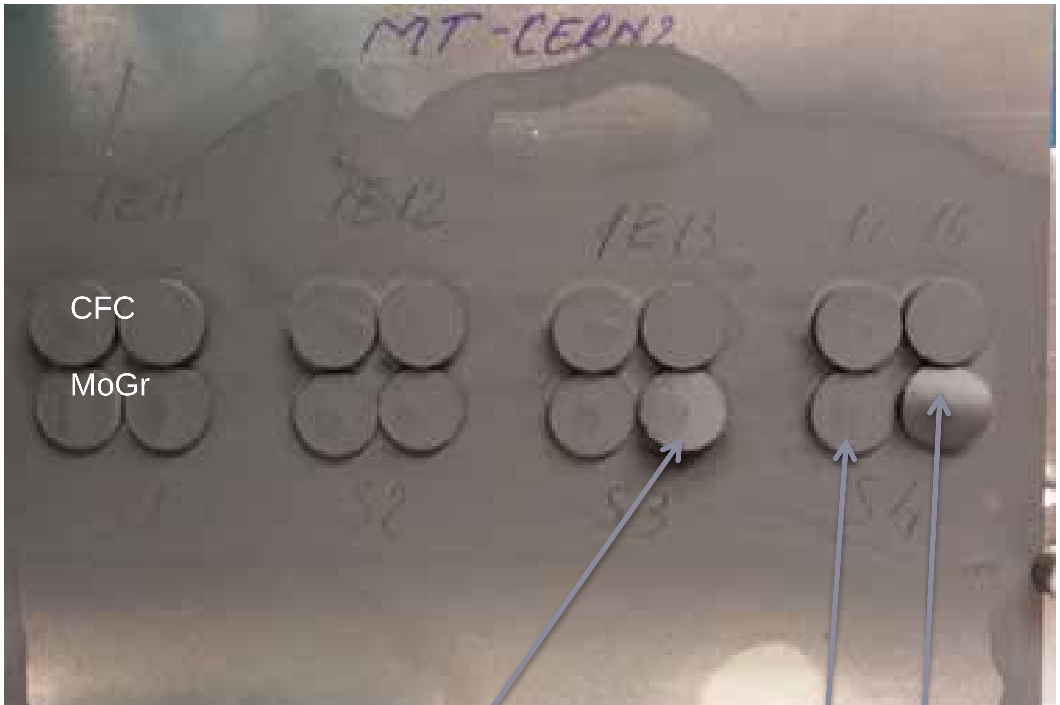


in-plane



transversal

Irradiation induced deformation in Mo-Gr



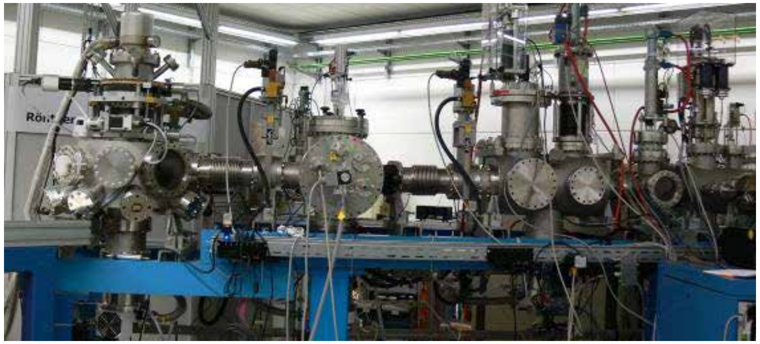
Not annealed

in plane

transverse

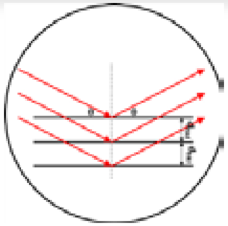
- Macroscopic bending was observed around 5×10^{12} U ions/cm²

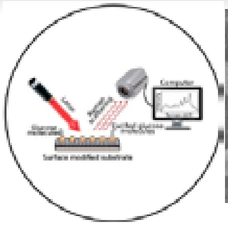
Irradiation of novel composites at GSI

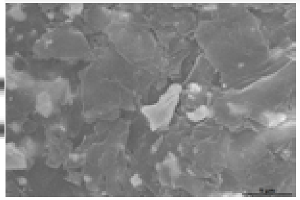


Ion irradiation: C – U
Energies: 70 MeV – 1 GeV
Fluence: up to $1x10^{14}$ i/cm2

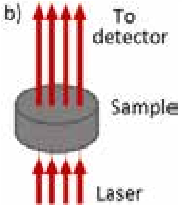
Structural characterization:
XRD, Raman spectroscopy, SEM

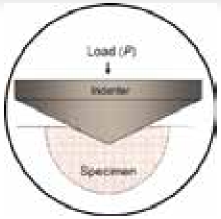


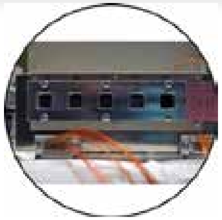




Functional properties degradation:
thermal ,mechanical, electrical







Material optimization

Overview of irradiation tests on MoGr

BNL (p^+, n) | GSI (^{238}U, ^{209}Bi, ^{197}Au, ^{152}Sm, Ca, C ions) | BNL (p^+, n)

mid 2013 — Feb. 2014 — July 2014 — mid 2015 — 2016

3th generation

MG 1110-E
($3.7\ g/cm^3$)
NOT fully molten
20%v Mo
40%v C-fibers
(long+short)

4th generation

MG 3110-P
($2.67\ g/cm^3$)
Fully molten
20%v Mo
40%v C-fibers
(long+short)

5th generation

MG 5200-S
($2.65\ g/cm^3$)
Fully molten
7.2%v Mo
46.4%v C-fibers
(long+short)
2 annealing cycles:
- 1150°C
- 1300°C

6th generation

MG 6400-U
($2.63\ g/cm^3$)
Fully molten
4.5%v Mo
NO C-fibers
Annealing 1800˚C

MG 6541-Aa
($2.5\ g/cm^3$)
Fully molten
4.5%v Mo
0. %v Ti
5%v C-fibers
(short only)
Annealing 1900°C

MG 6530-Aa
($2.5\ g/cm^3$)
Fully molten
4.5%v Mo
5%v C-fibers (long only)
Annealing 1900°C

7th generation

MG 6403-Aa
(g/cm^3)
Fully molten
4.5%v Mo
%v Ti
NO C-fibers

E. Quaranta, CERN

6th generation – high temperature annealed MoGr, Ti addition

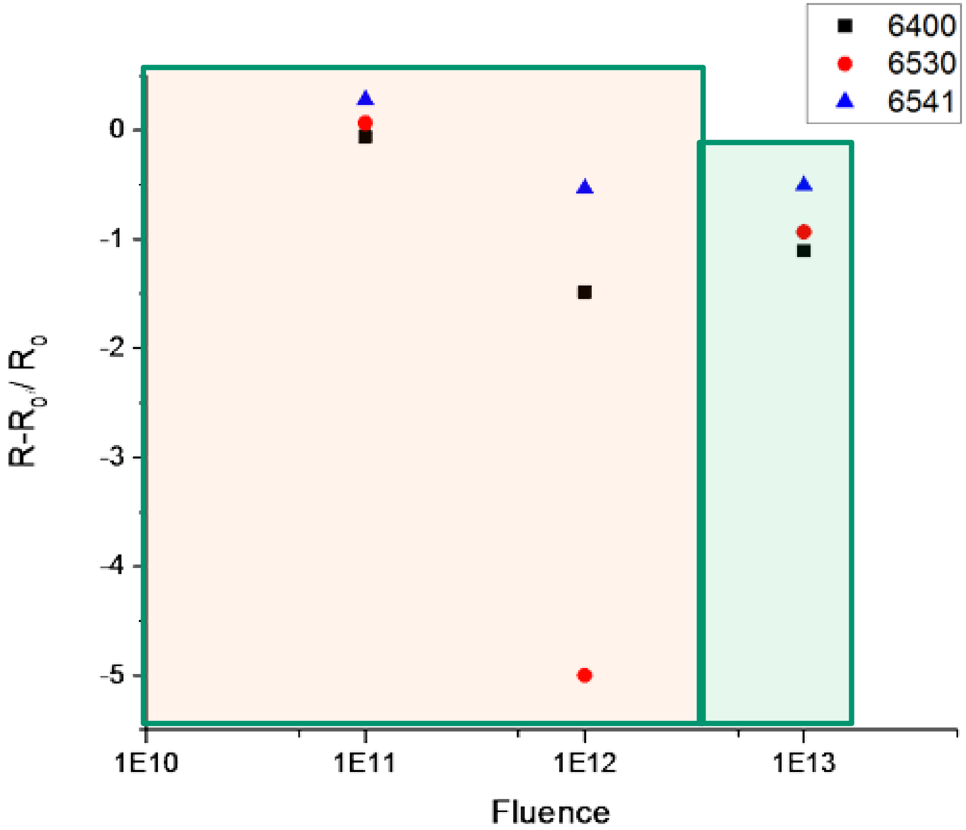
MG 6400-U, MG 6530-AA AND MG 6541-AA

MG 6400-U, MG 6530-AA, MG 6541-AA VS MG5220S

Optimization of MoGr composition as respect to irradiation induced deformation

stress accumulation and deformation

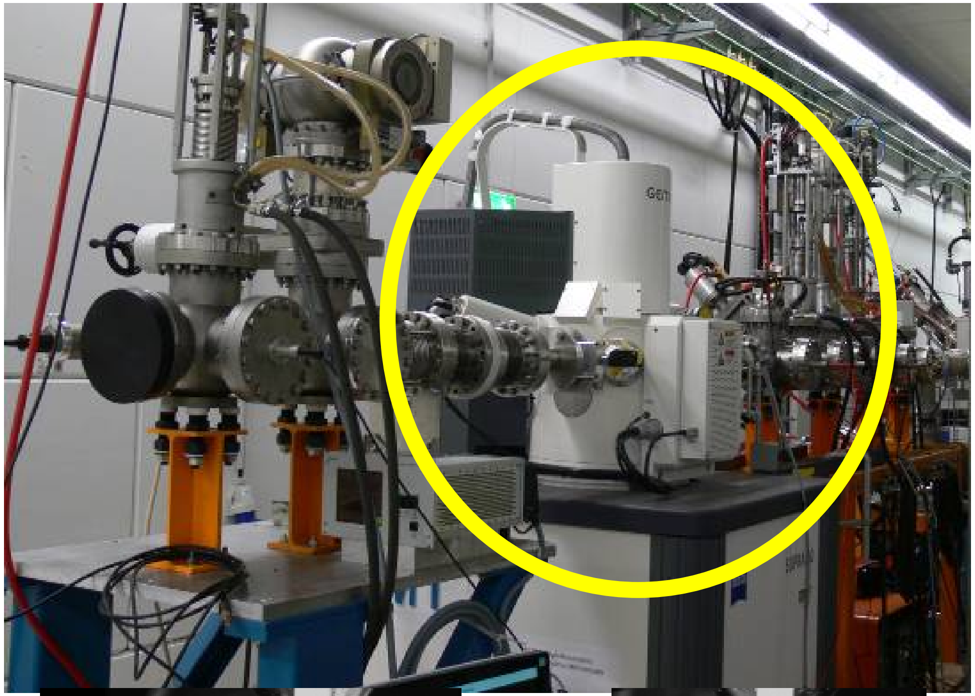
stress release by crack formation and creep - graphite matrix

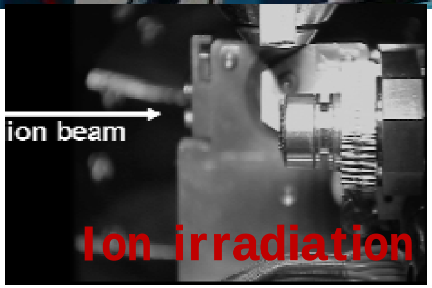


- MG6541 (short fibers, Ti addition) smallest deformation

In situ HRSEM at M-branch, GSI

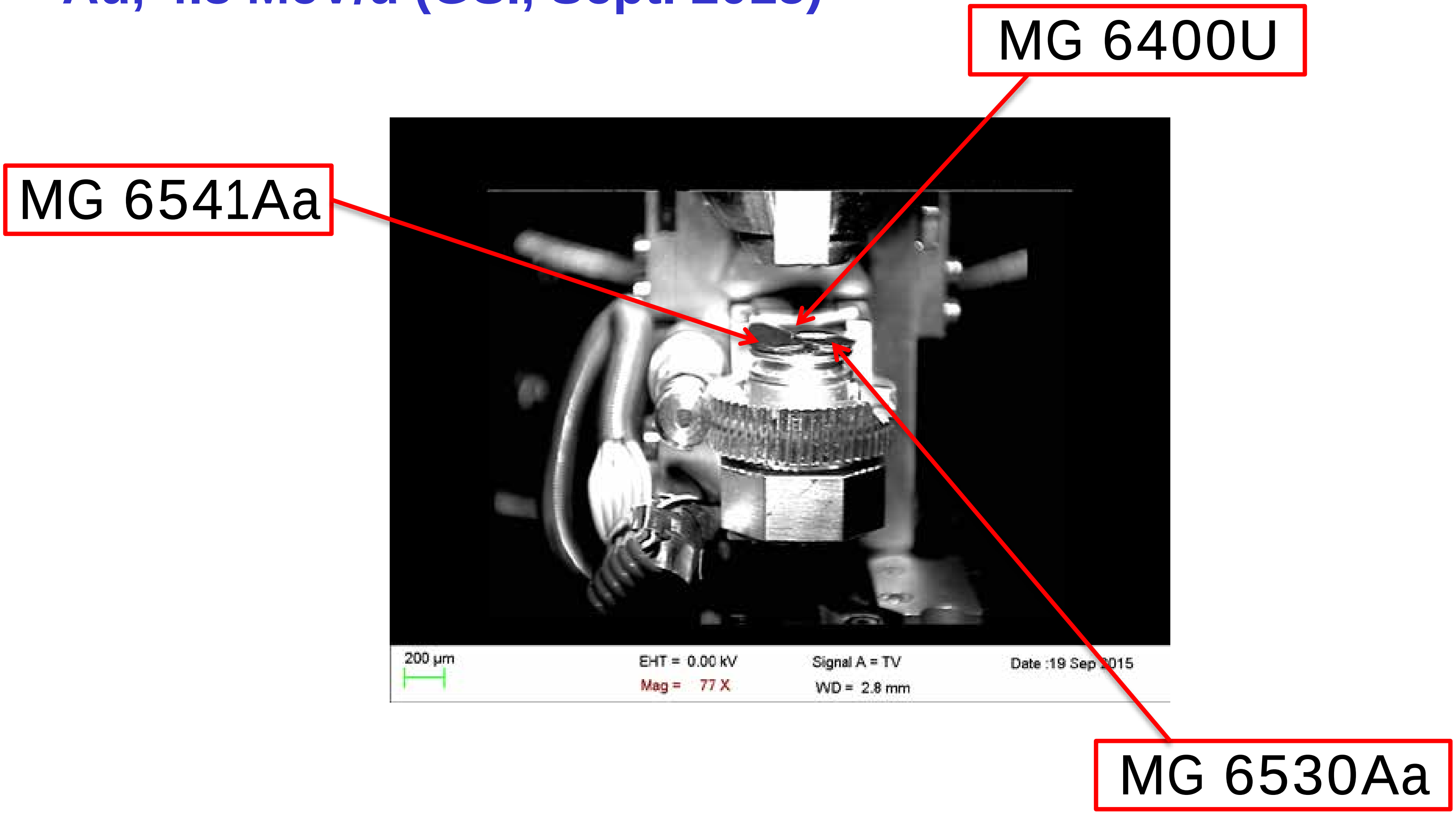
Au, 4.8 MeV/u (GSI, Sept. 2015)

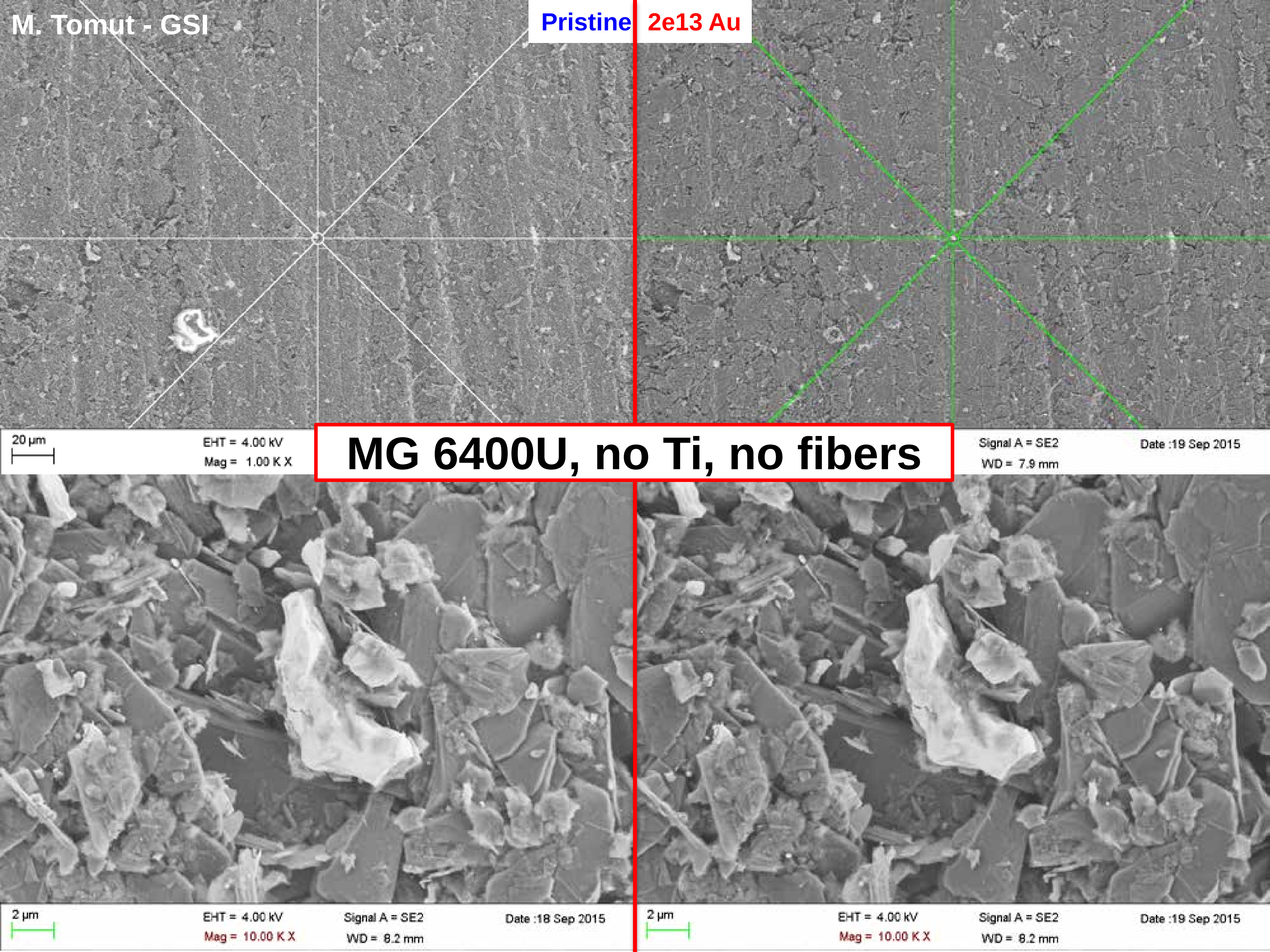




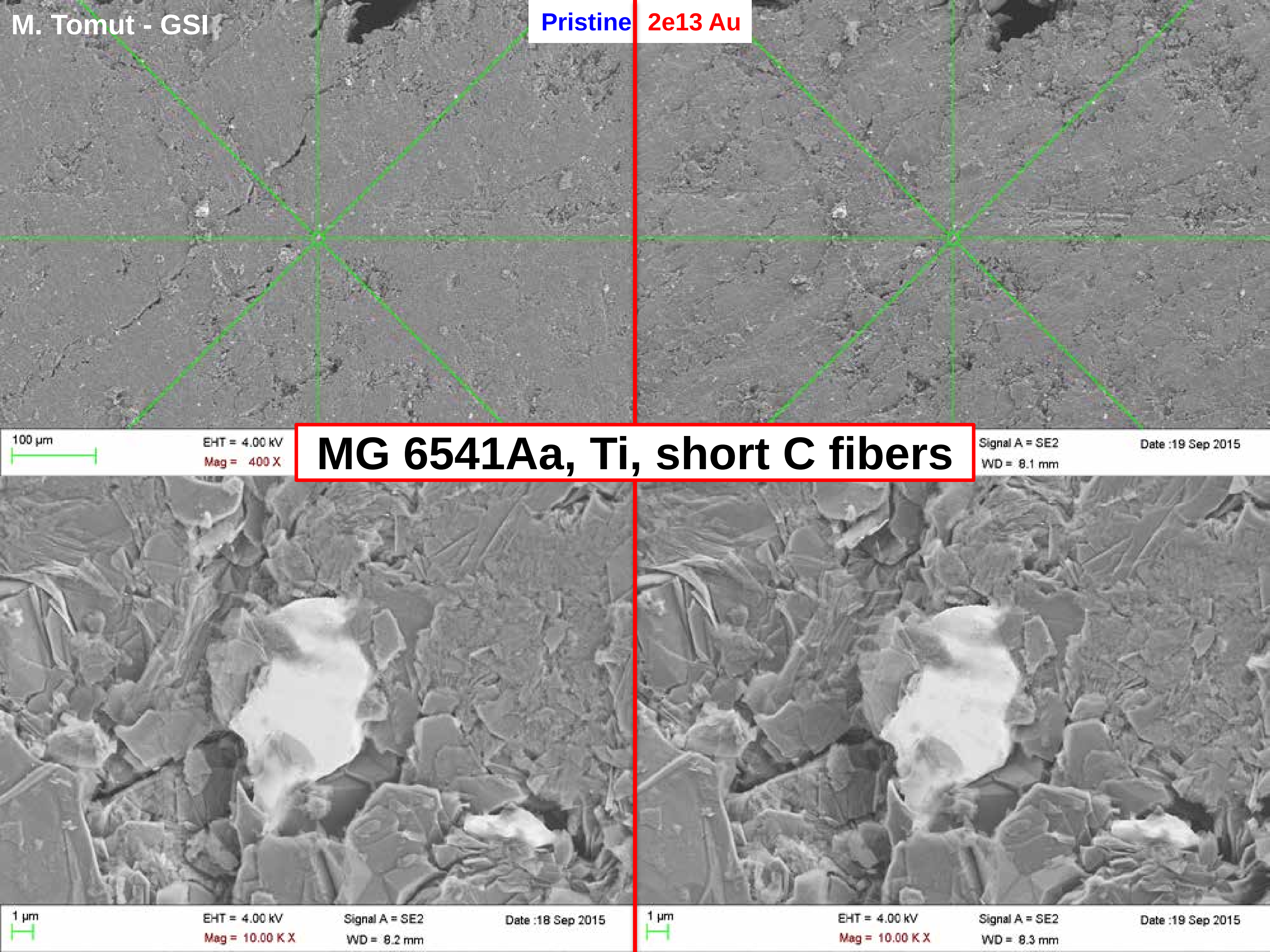
In situ SEM monitoring of microstructural changes during ion irradiation of MoGR

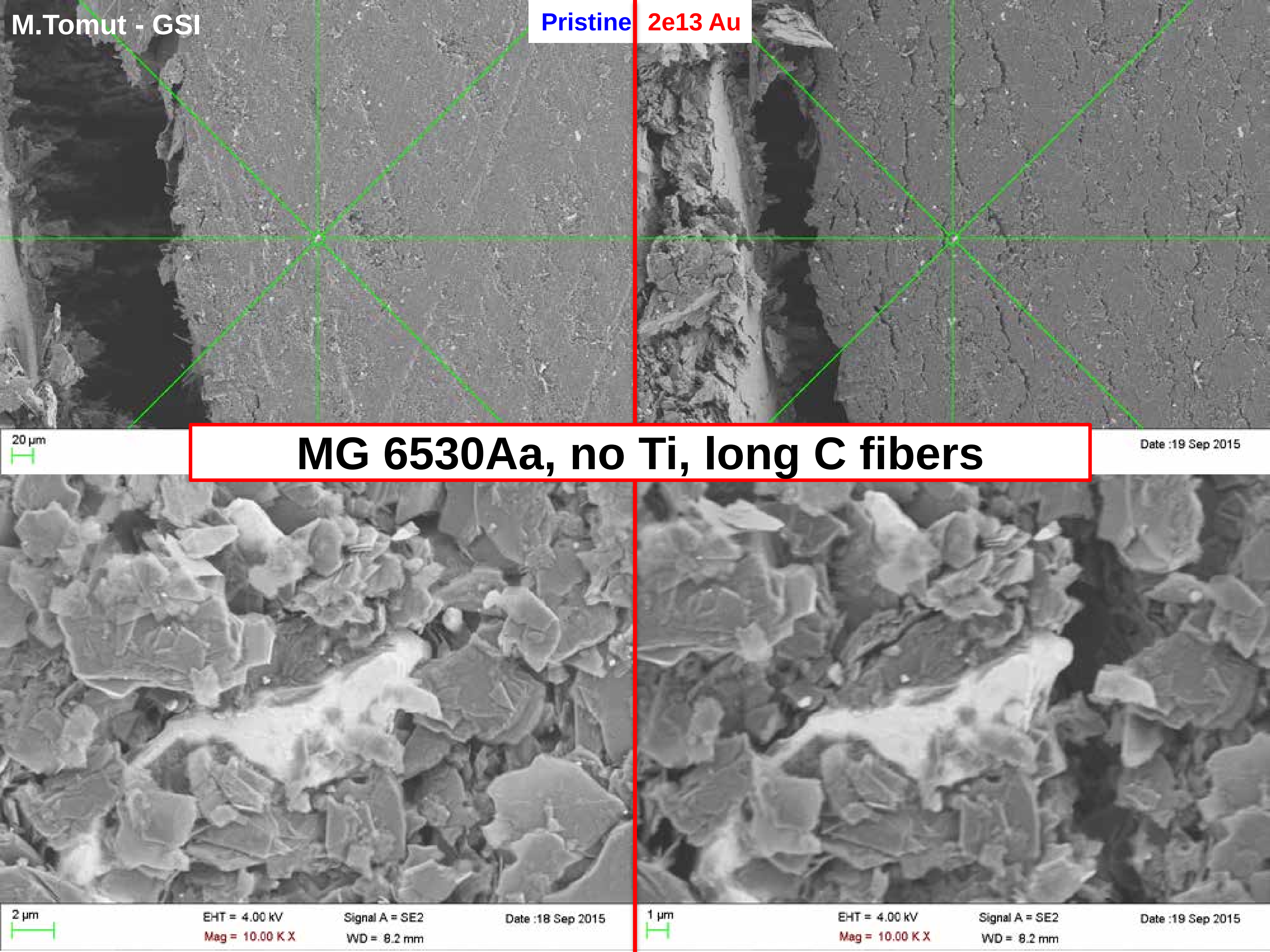
Au, 4.8 MeV/u (GSI, Sept. 2015)




M. Tomut - GSI
Pristine
2e13 Au
MG 6400U, no Ti, no fibers
20 µm
EHT = 4.00 kV
Mag = 1.00 K X
Signal A = SE2
WD = 7.9 mm
Date :19 Sep 2015
2 µm
EHT = 4.00 kV
Mag = 10.00 K X
Signal A = SE2
WD = 8.2 mm
Date :18 Sep 2015
2 µm
EHT = 4.00 kV
Mag = 10.00 K X
Signal A = SE2
WD = 8.2 mm
Date :19 Sep 2015

MG 6541Aa, Ti, short C fibers



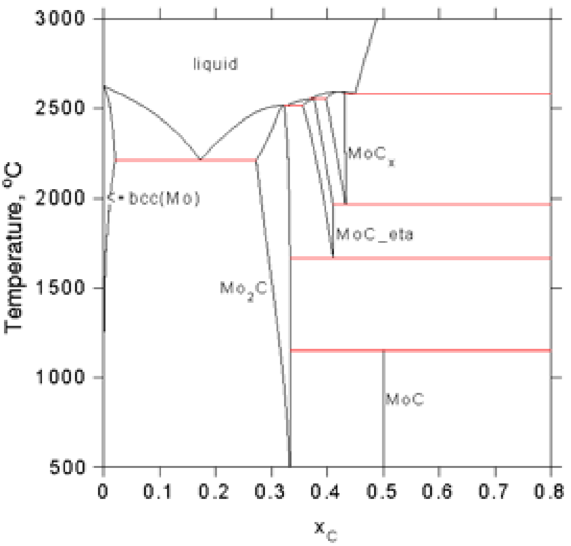


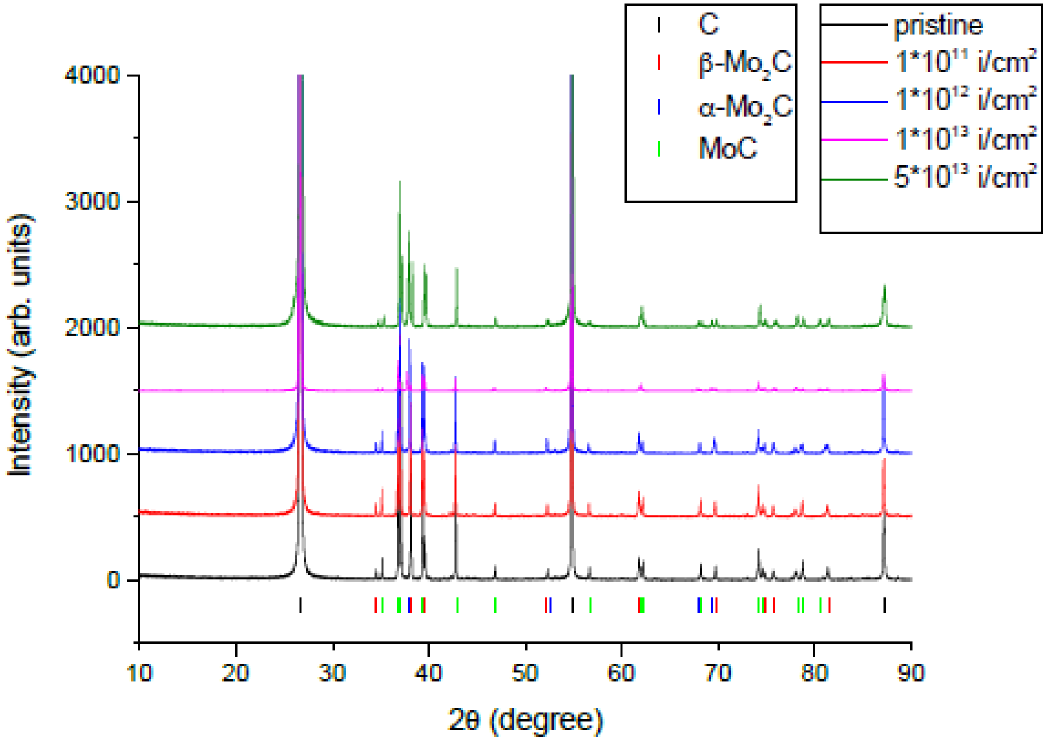
M.Tomut - GSI
Pristine
2e13 Au
20 µm
MG 6530Aa, no Ti, long C fibers
Date :19 Sep 2015
2 µm
EHT = 4.00 kV
Mag = 10.00 K X
Signal A = SE2
WD = 8.2 mm
Date :18 Sep 2015
1 µm
EHT = 4.00 kV
Mag = 10.00 K X
Signal A = SE2
WD = 8.2 mm
Date :19 Sep 2015

X-RAY DIFFRACTION

Ion-induced Structural Changes – XRD

- Hexagonal Mo_2C
- Monoclinic MoC
- Hexagonal graphite

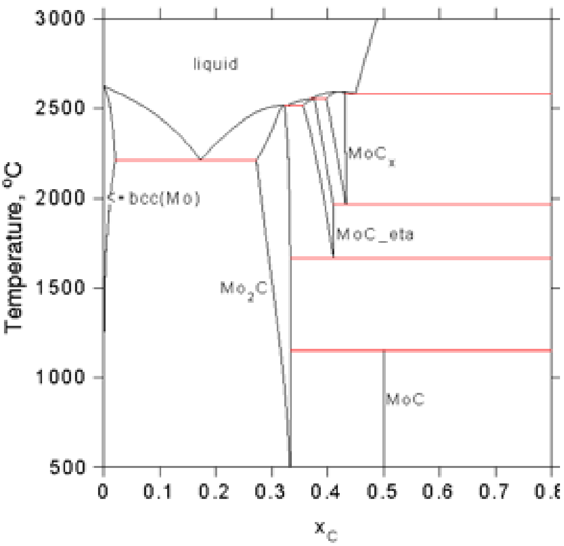


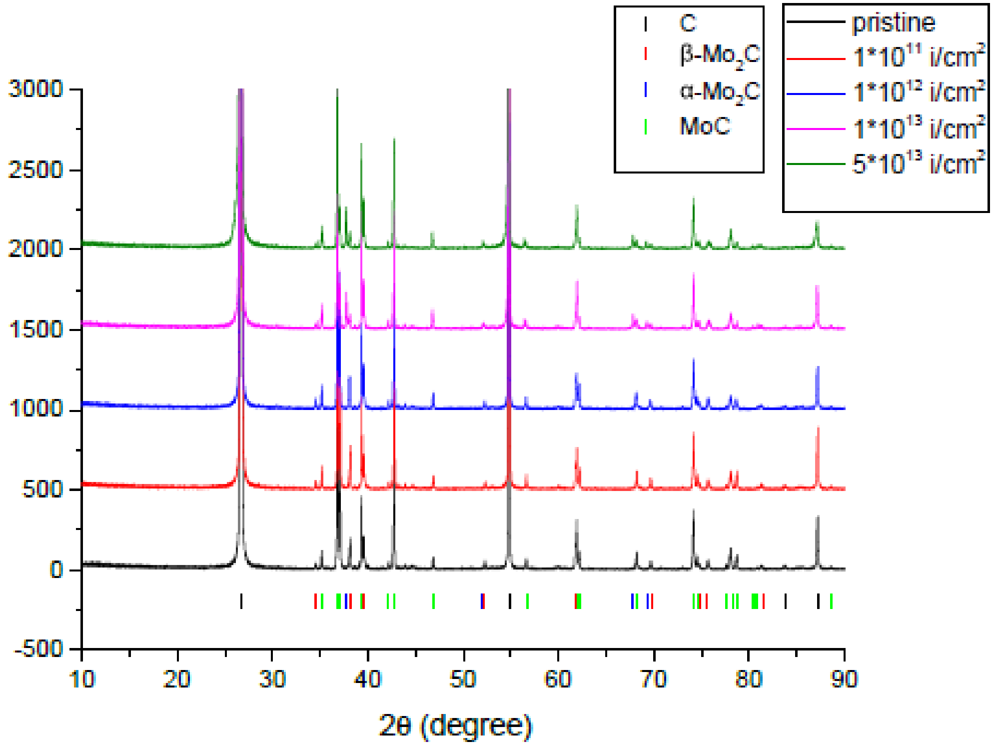


MG6530

Ion-induced Structural Changes – XRD

- Hexagonal Mo_2C
- Monoclinic MoC
- Hexagonal graphite

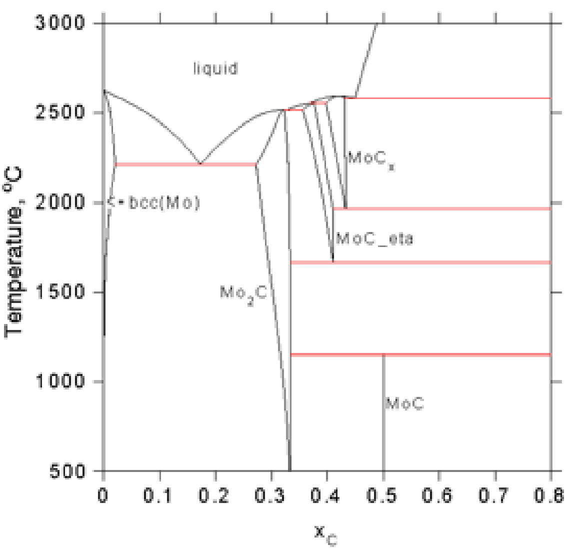


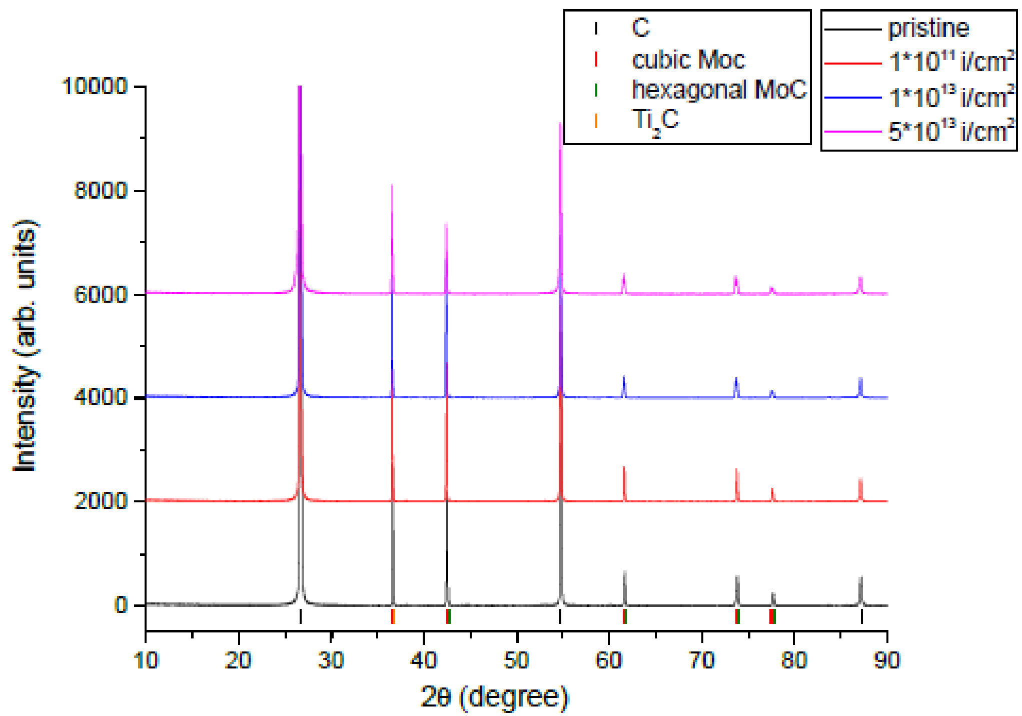


MG6400

Ion-induced Structural Changes – XRD

- Hexagonal Mo_2C
- Monoclinic MoC
- Hexagonal graphite

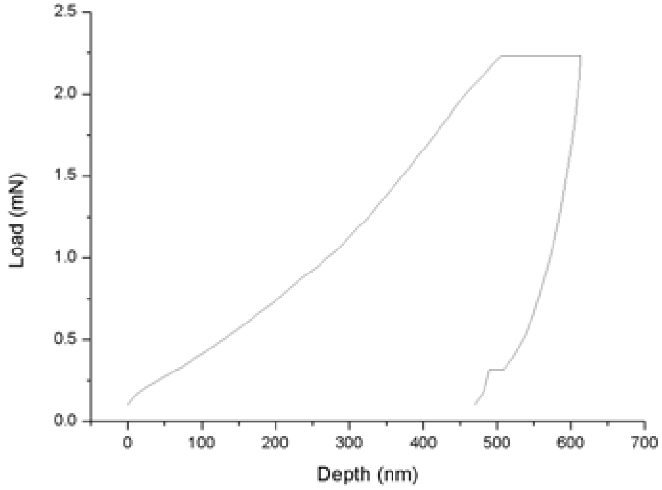




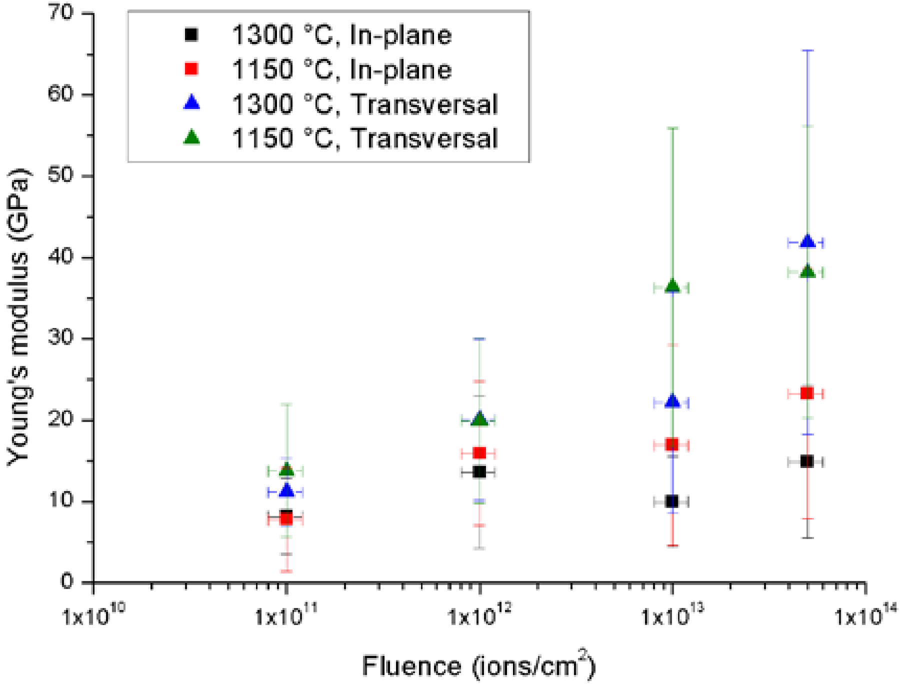
MG6541

NANOINDENTATION

Ion-induced hardening - old MoGr grades

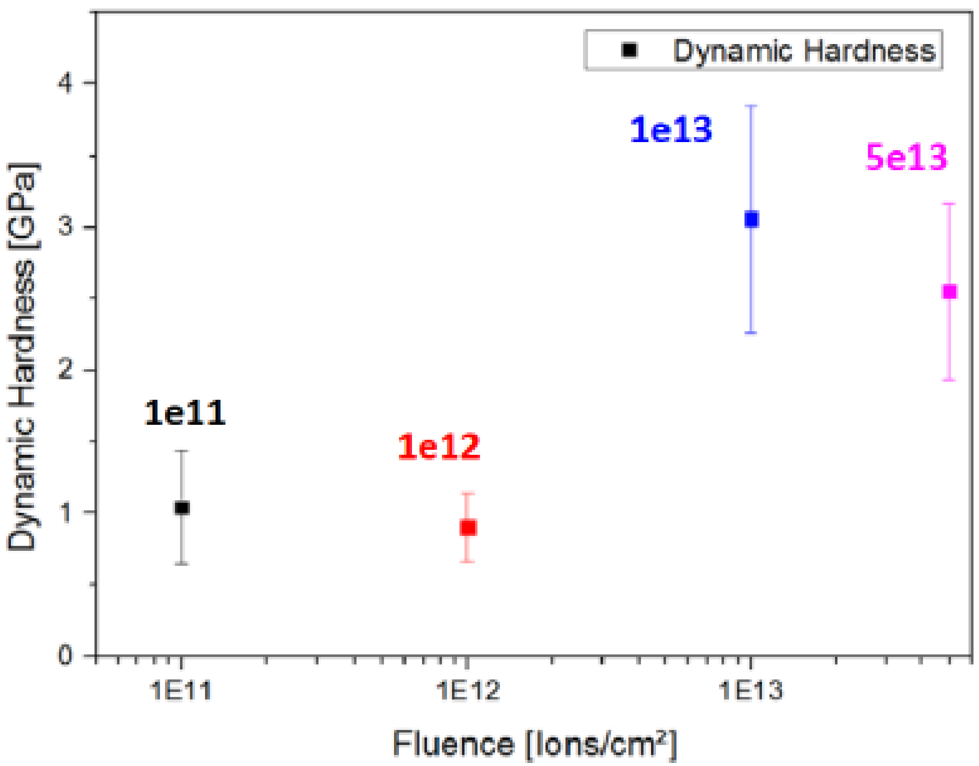


- Load/Depth controlled
- Maximum depth: 500 nm
- Maximum load: 50 mN
- Berkovich tip
- Mapping with 100 indentations



- Young's modulus increases with dose
- Dependency on pre-treatment
- Anisotropic behavior

Impact response of irradiated Mo-Gr composites



Irradiation:

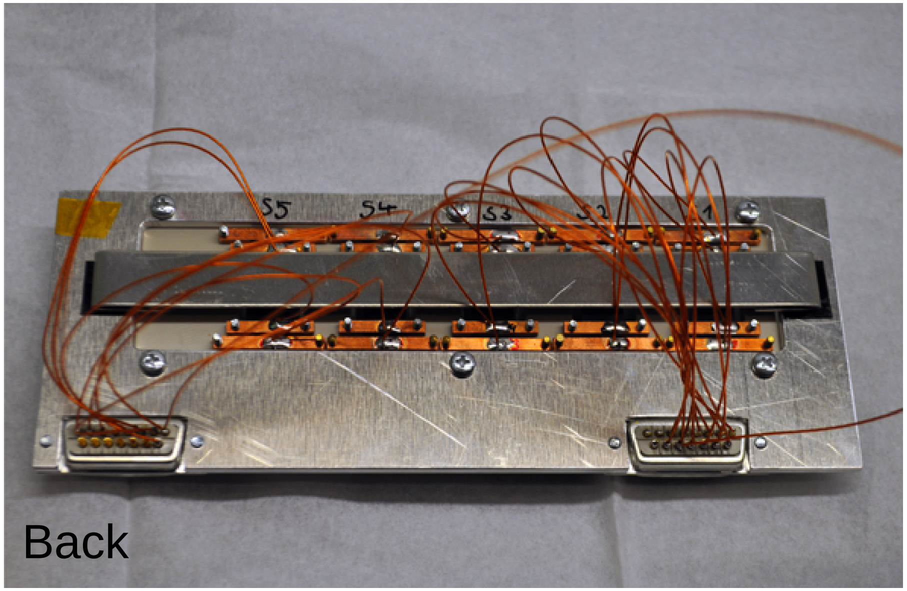
Ion: Bi 27+
Energy: 4.8 MeV/u
Flux: 2e9 ions/cm²s
Frequency: 2 Hz
Pulse length: 0.3 ms

Sampling time: 0.5 s
Impact load: 2mN
Impact distance: 15000 nm

ONLINE RESISTIVITY DEGRADATION MEASUREMENTS

Online Resistivity Measurements Set-up M-branch, UNILAC GSI

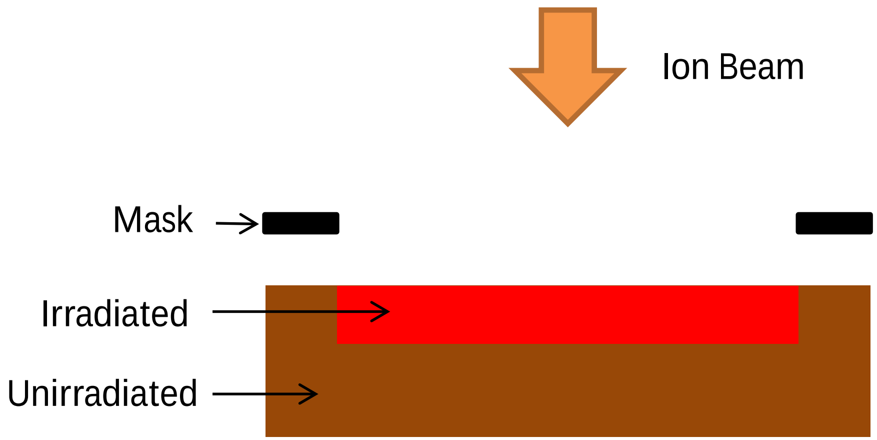

Front


Back

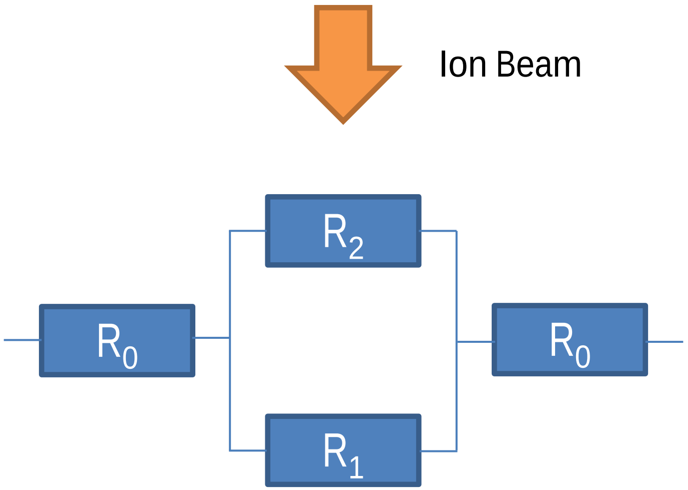
- Five samples in series irradiated one by one while measuring the voltage drop across the sample with a 4-probe setup.
- Investigation of MoGR with long carbon fibers (3 mm), short carbon fibers (300 µm) and without fibers.

Ion-induced Electrical Properties Changes – Equivalent electrical circuit

(a)



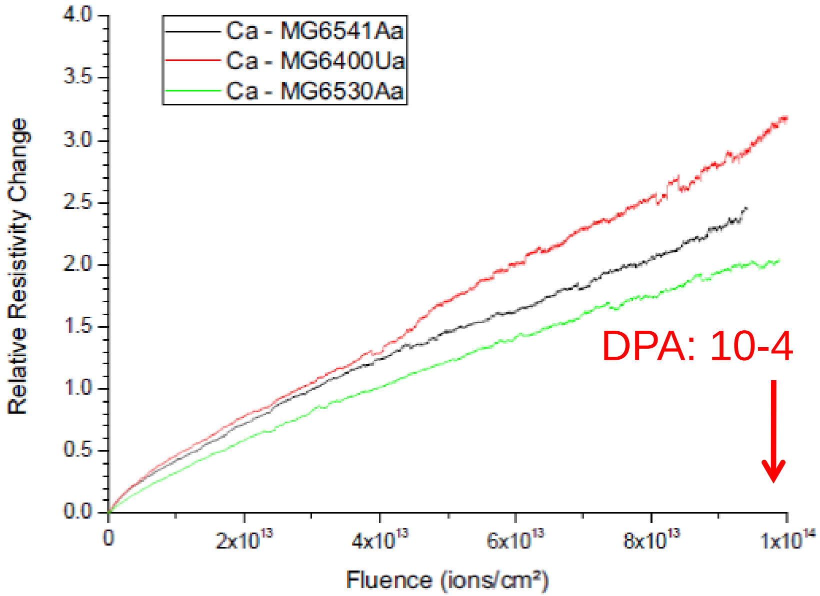
(b)

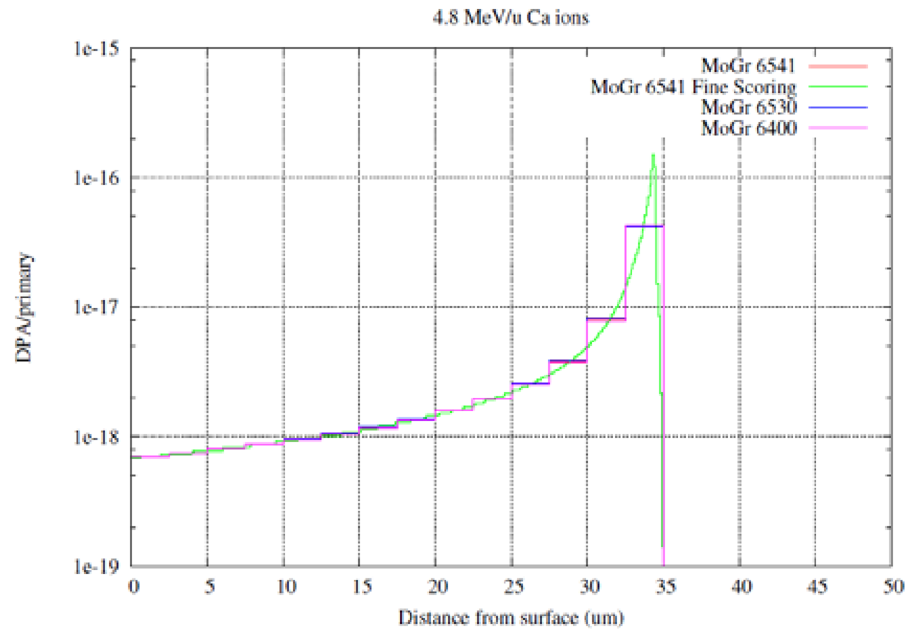


$$R_2 = \frac{R_{tot}R_1 - 2R_0R_1}{R_1 - R_{tot} + 2R_0}$$

Online study of electrical resistivity degradation for isotropic graphite – heavy ions

Ca ions to simulate damage induced by secondary particles and recoil atoms in collimator jaw





Fluka, DPA calculations - Eleftherios Skordis

- relative change of average resistivity for the whole ion range ~ 35 µm)

MG6530 (long fibers, no Ti) < MG6541 (short fibers, Ti) < MG6400 (no fibers, no Ti)

Summary

- Macroscopical bending was observed to have a maximum around 5×10^{12} ions/cm^2; at higher fluences is relaxed by crack formation and creep
- With increasing fluence, the disorder in the graphite matrix and the carbon fibers increased. The defect density increased while the (graphitic) crystallite size decreased
- Hardness and Young's modulus increased with accumulated dose. Samples annealed at higher temperatures show less degradation
- Carbon fibers reduce material's resistivity degradation, Ti as well,
  - a compromise between deformation and resistivity degradation mitigation needs to be found
- Further optimization of the material to avoid swelling and deformation - Post-production annealing heat treatments decrease irradiation-induced deformations à T~1900°C

Coming beamtime GSI

- UNILAC

- heavy duty cycle Au
  - 02.05-04.05
  - 12.06-13.06
  - 21.06-23.06

4.8 MeV/u, 5e9 ions/cm2 s, “25 Hz, 5 ms pulse lengths.

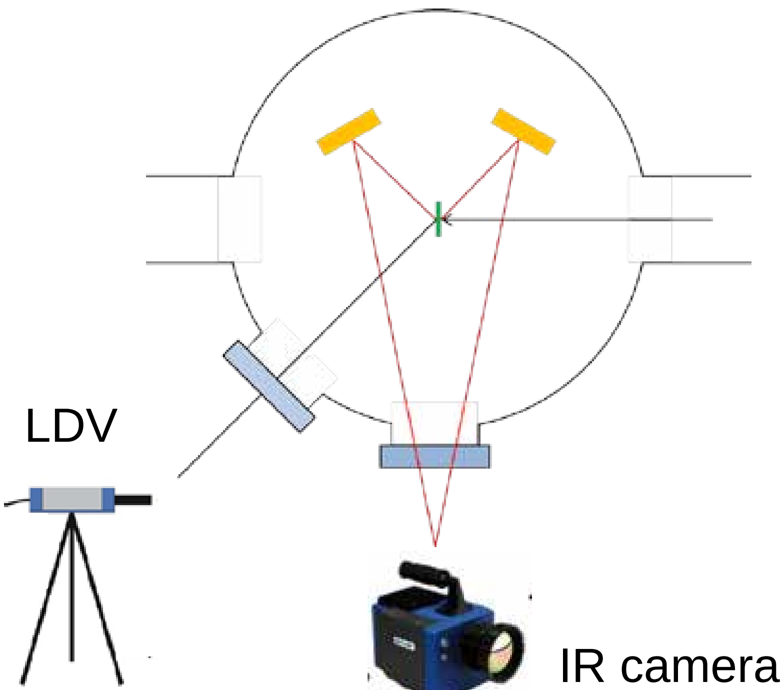
- low duty cycle U
  - 20.05-25.05

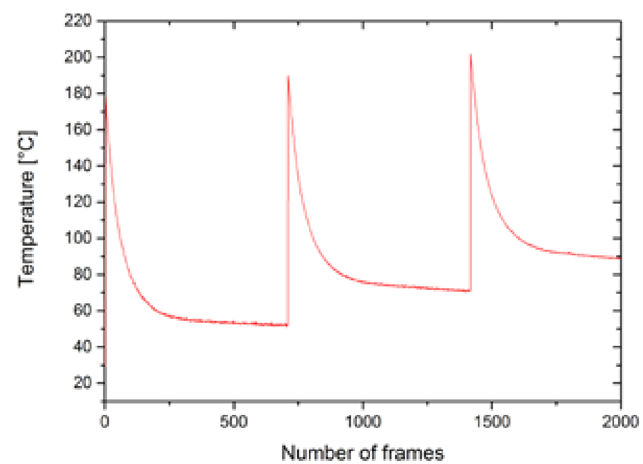
4.8 MeV/u, up to 1e10 ions/cm2 pulse, 0.2 Hz

Dynamic response in context of radiation damage

Experimental details
Irradiation parameters & set-up

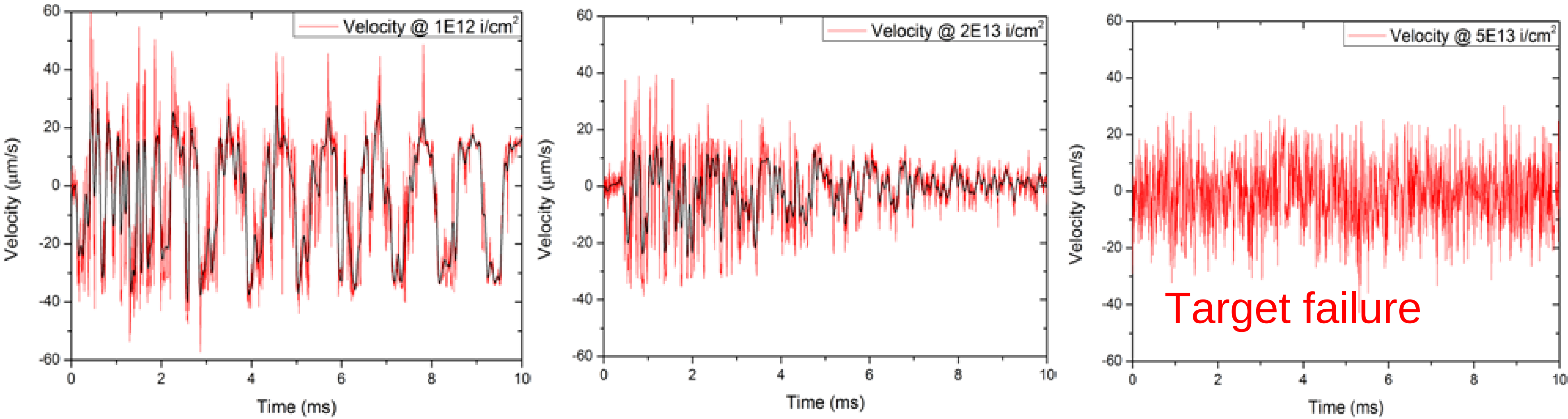
- Beam parameters
  - 4.8 MeV/u U^{28+};; 0.15 ms pulse length; 1 Hz
  - Up to 1.5×10^{10} i/cm^2 per pulse
  - Up to fluences of 1×10^{14} i/cm^2





Oscillation monitoring as a function of fluence

Polycrystalline isotropic graphite 140 µm

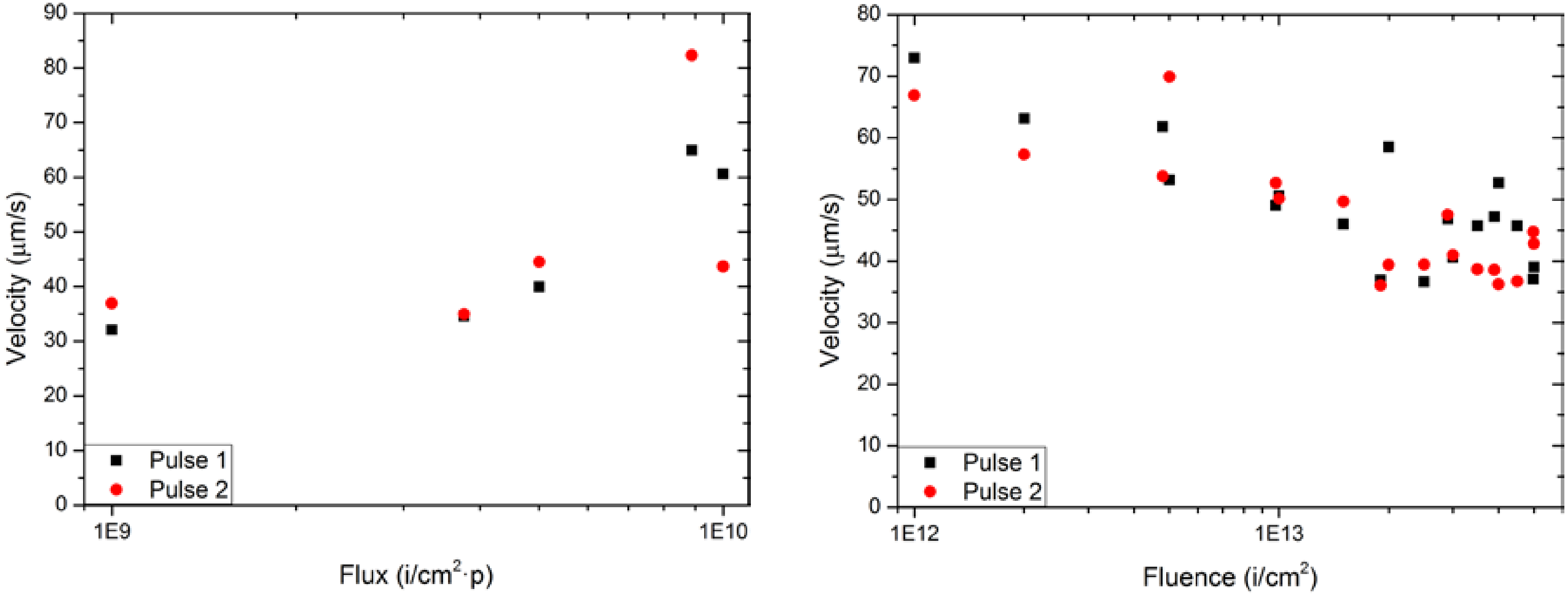


With radiation damage accumulation:

– Additional reflection at irradiated / non-irradiated interface

- increase of frecquency
- increase of damping
- decrease of velocity

Measured maximum velocities - LDV



- Decrease of maximum velocity with accumulated dose:
  Radiation damage:
  – density reduction in beam spot
  – internal friction
  – plastic deformation processes


Thank you for your attention!