

## Irradiation study of Ti-6Al-4V and Ti-6Al-4V-1B for FRIB beam dump:

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# Outline

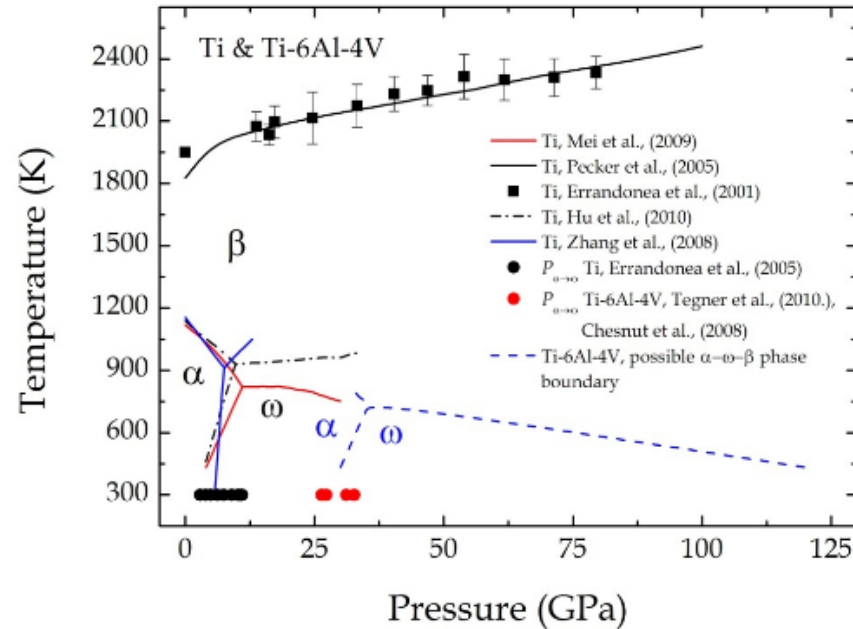
- Irradiation of samples with high energy heavy ions (NSCL-MSU)(Ca 40 @ 2000 MeV) and low energy heavy ions at CIMAP-France
- XRD and TEM observations( in collaboration with CIMAP)
- Surface characterization using SEM-EBSD
- Nano-indentation tests
- Vickers Hardness tests
- Insitu-tensile tests

# Irradiation experiments

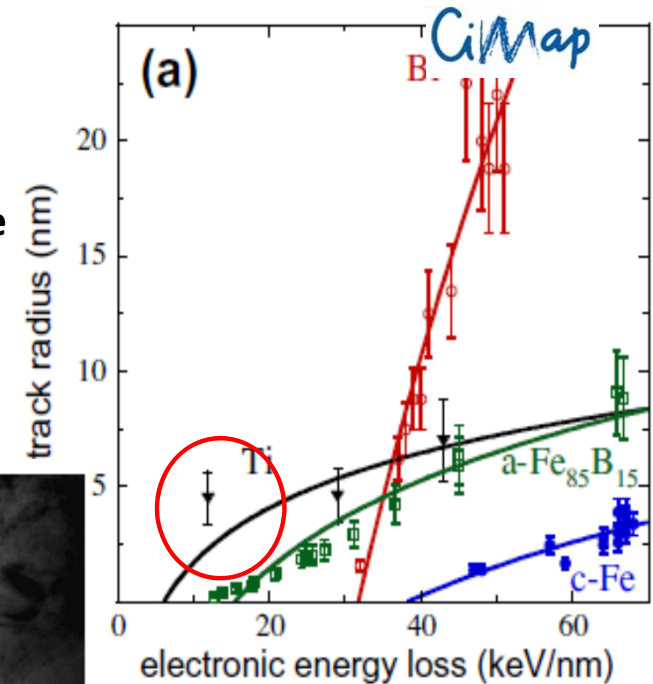
Facilities	Beam	Energy [MeV]	Range [ $\mu\text{m}$ ]	$S_e$ [keV/nm]	Fluence [ions/cm <sup>2</sup> ]	Max dpa in sample	Date	Number of samples	Type
IRRSUD	<sup>82</sup> Kr	25	4.73	9.9	5.10 <sup>11</sup> - 5.10 <sup>12</sup> - 2.10 <sup>14</sup>	0.6	Jul-13	6	Foils
IRRSUD	<sup>131</sup> Xe	92	8.5	19.7	2.10 <sup>11</sup>	0.001	Jul-13	2	Foils
IRRSUD	<sup>82</sup> Kr	45	6.43	13.1	5.10 <sup>11</sup> - 5.10 <sup>13</sup>	0.16	Jul-13	4	Foils
IRRSUD	<sup>82</sup> Kr	45	6.43	13.1	2.10 <sup>14</sup> 2.5.10 <sup>15</sup>	8	Oct-13	6	Foils
IRRSUD	<sup>36</sup> Ar	36	6.8	7.5	10 <sup>15</sup>	1.5	Dec-13	23	TEM and dogbone
IRRSUD	<sup>129</sup> Xe	92	8.5	19.7	If 3 10 <sup>14</sup> (~10h)	Estimated 1.7	Planned in June-2014		
NSCL	<sup>40</sup> Ca	2000	800	1.5	6 10 <sup>12</sup>	10 <sup>-5</sup>	Aug-13	1 x Ti64	Dogbone

# XRD and TEM observations( in collaboration with CIMAP)

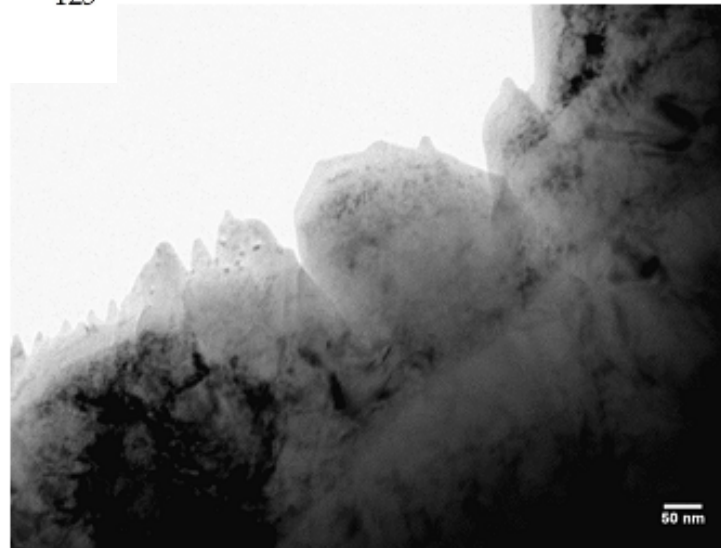
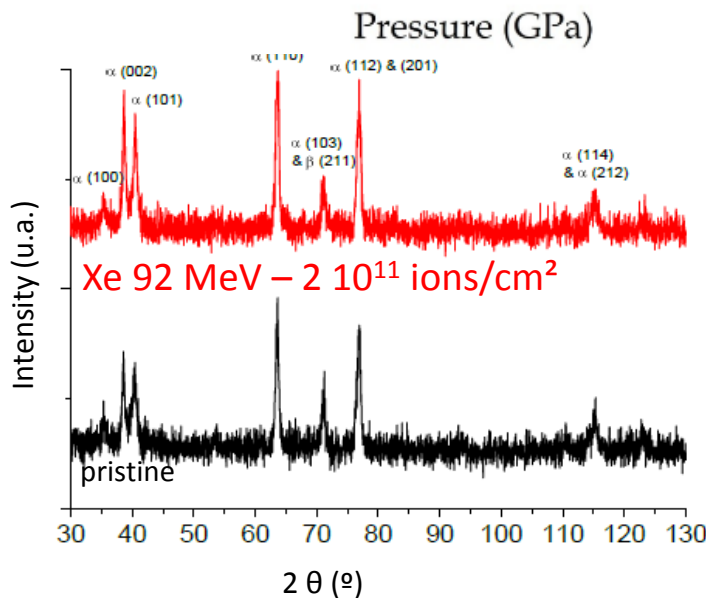
- No evidence of phase transformation or ion track formation in Ti-6Al-4V



**Ti-alloys are not sensitive to electronic excitation by swift heavy ions compared to pure Titanium**



M. Toulemonde et al./ NIMB 277 (212) 28-39



TEM image of a Ti-6Al-4V foil irradiated with Kr 45 MeV –  $5 \cdot 10^{13}$  ions/cm<sup>2</sup>

## Characterization of the microstructure and mechanical properties:

- Scanning electron microscopy (SEM) as well as electron backscatter diffraction (EBSD) were used to characterize the microstructure of the samples before and after irradiation.
- Nano-indentation , Vickers Hardness and in-situ tensile tests were used to investigate the change in the mechanical properties.

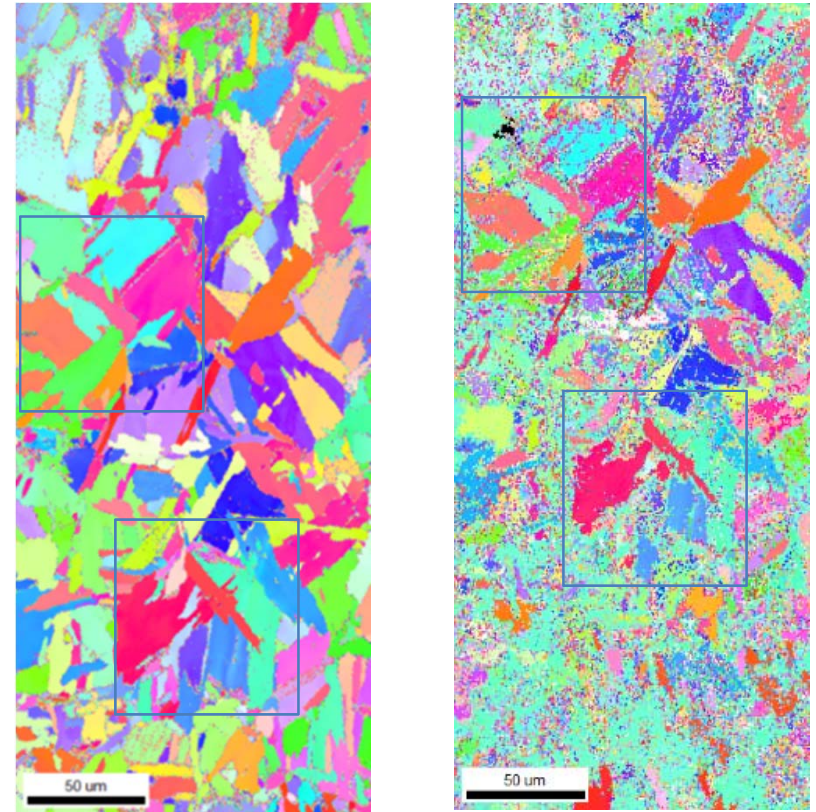
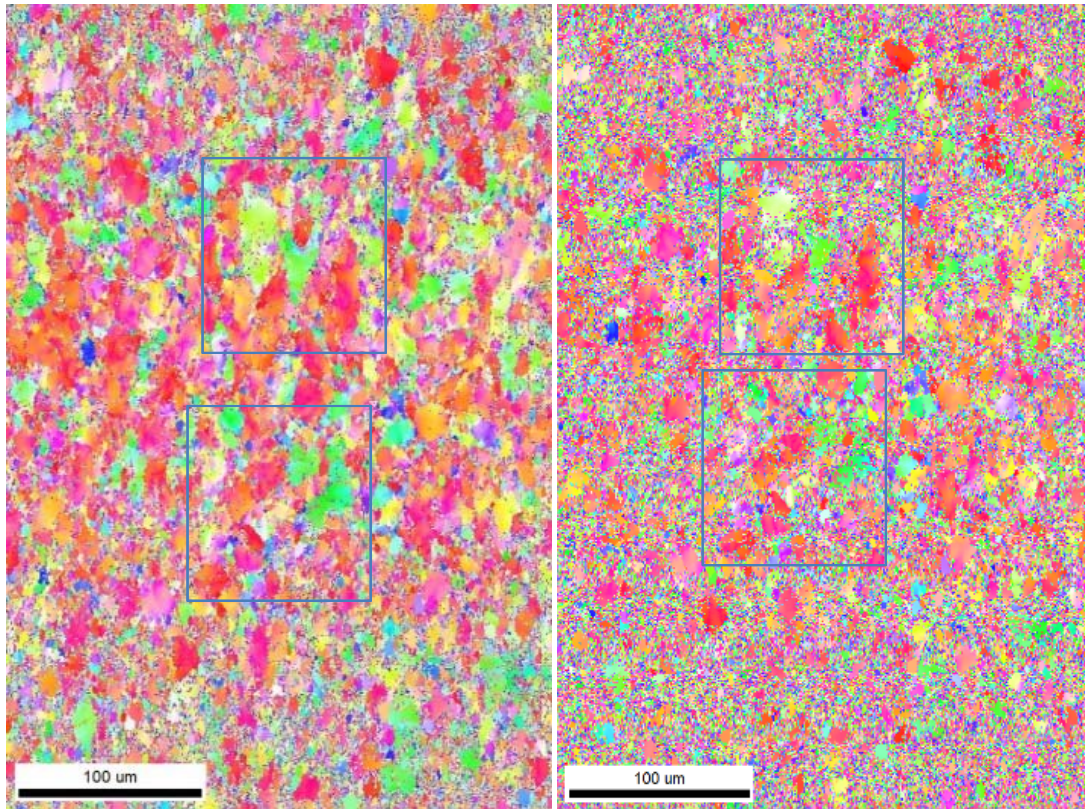


# Observations

- Deterioration of the quality of the EBSD scan after irradiation.

Ti-6Al-4V

Ti-6Al-4V-1B



IPF map **before** irradiation

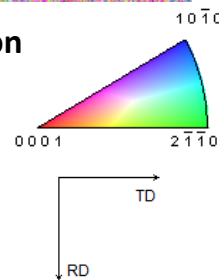
IPF map **after** irradiation

IPF map **before** irradiation

IPF map **after** irradiation

Ti-6Al-4V Irradiated at NSCL: Ca@2000MeV  
 T=20°C and a fluence of  $6 \cdot 10^{12}$  ions/cm<sup>2</sup> and  
 dpa at the surface of  $10^{-5}$  dpa

Ti-6Al-4V-1B Irradiated at CIMAP: Ar@36MeV  
 T=350°C and a fluence of  $10^{15}$  ions/cm and  
 dpa at the surface of 0.038 dpa





# SEM and EBSD characterization of the surface of the samples:

No change in the microstructure or the orientation of the grains at the surface.

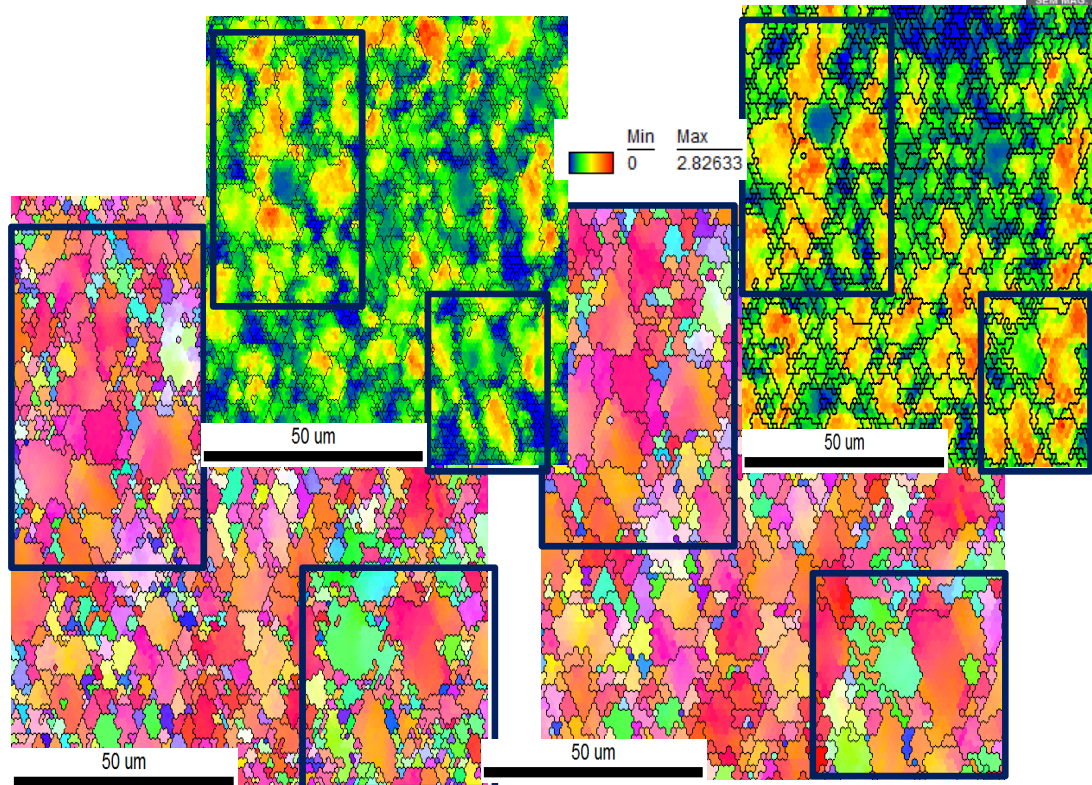
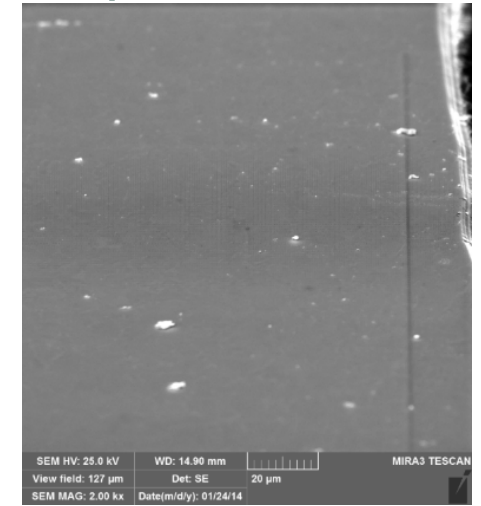
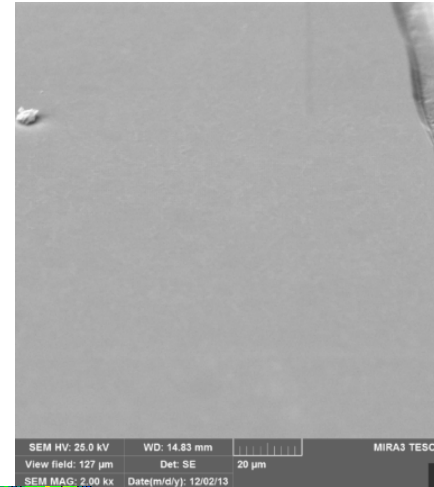
Irradiated at CIMAP: Ar@36MeV

T= 350 °C

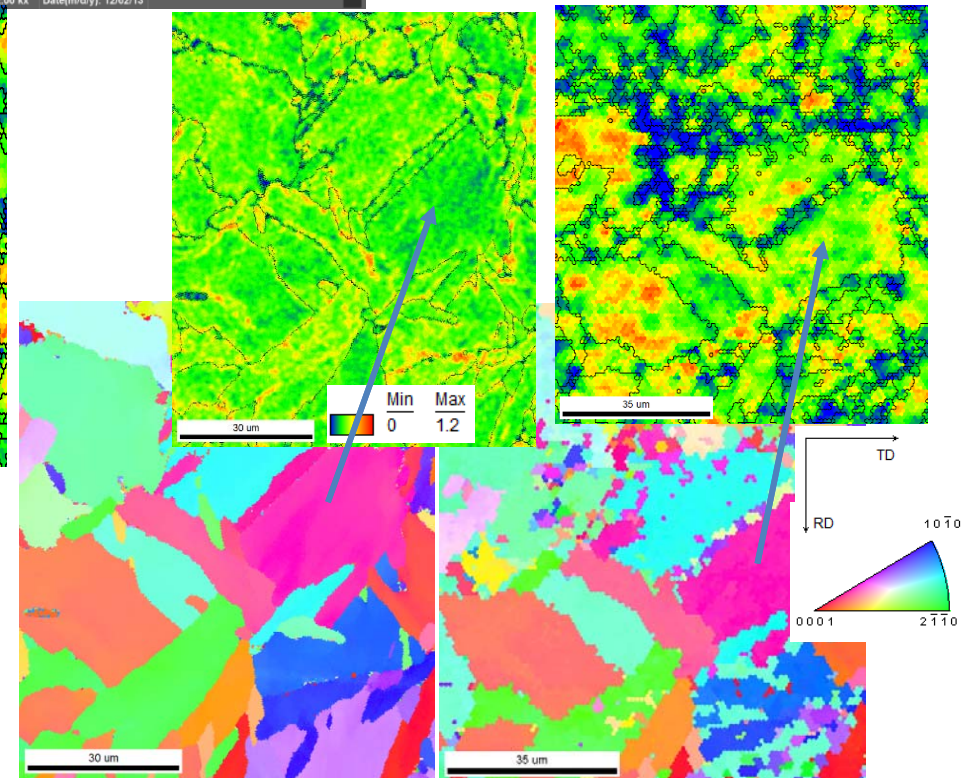
Fluence =  $10^{15}$  ions.cm<sup>-2</sup>

Dose at the surface= 0.038dpa

SEM image of the EBSD area before (a)  
and after (b) irradiation for Ti-6Al-4V



IPF and local average misorientation maps the grains before and after irradiation of Ti-6Al-4V



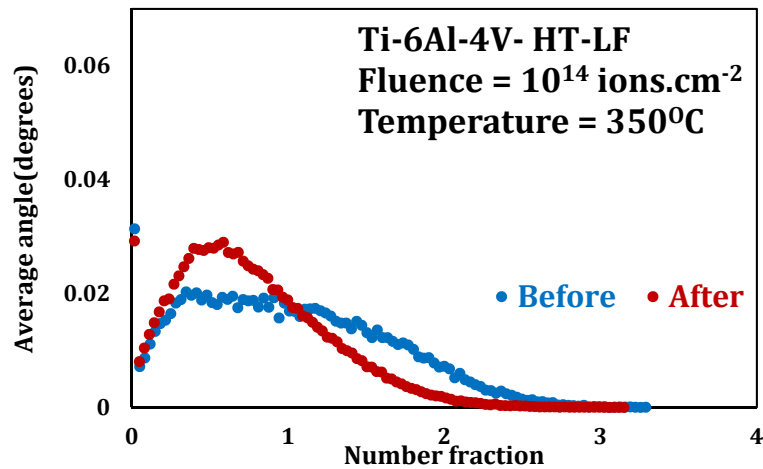
IPF and local average misorientation maps the grains before and after irradiation of Ti-6Al-4V-1B

# Local Average misorientation charts

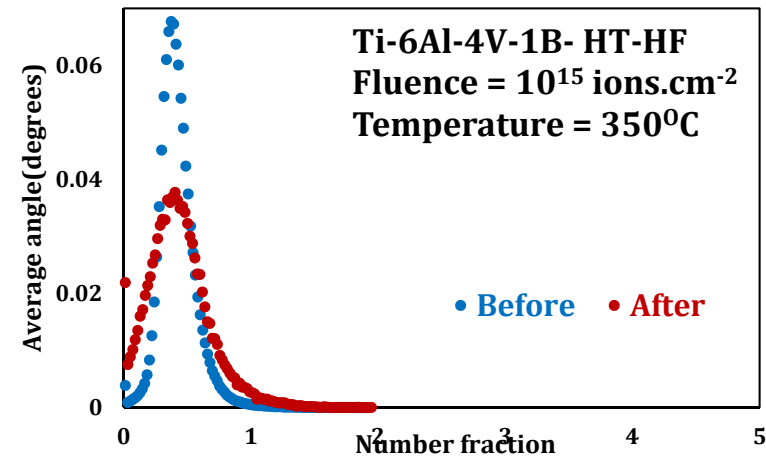
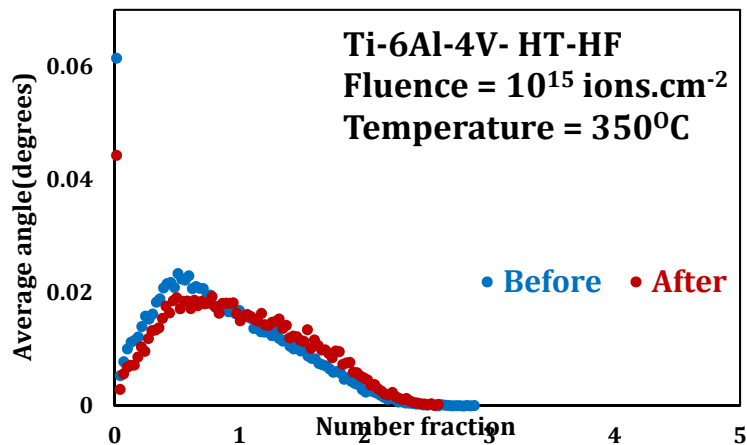
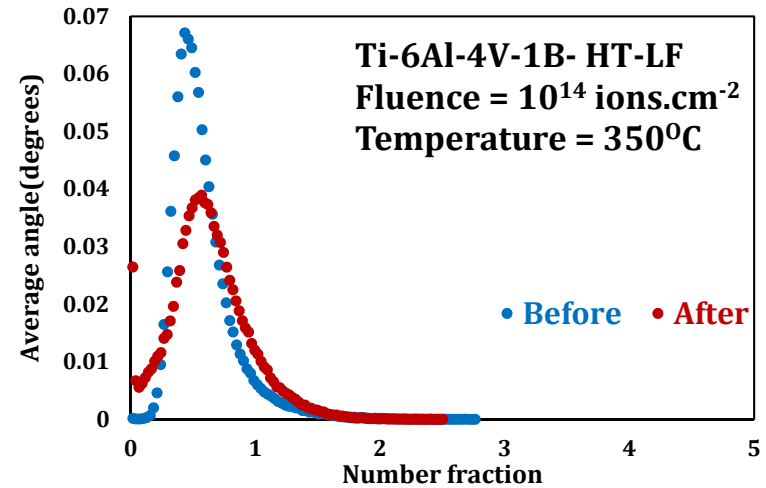
Low energy irradiation: Ar36@36MeV

Comparison between Ti-6Al-4V and Ti-6Al-4V-1B only alpha phase

## Ti-6Al-4V



## Ti-6Al-4V-1B

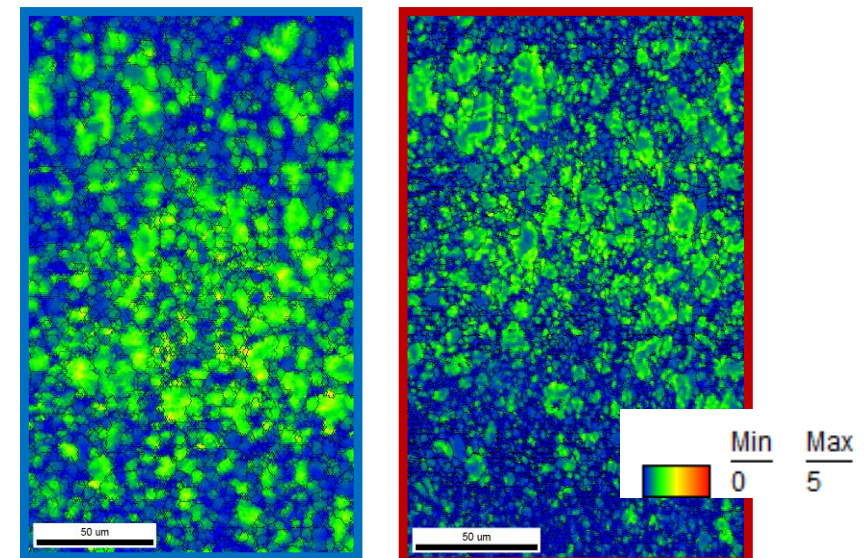
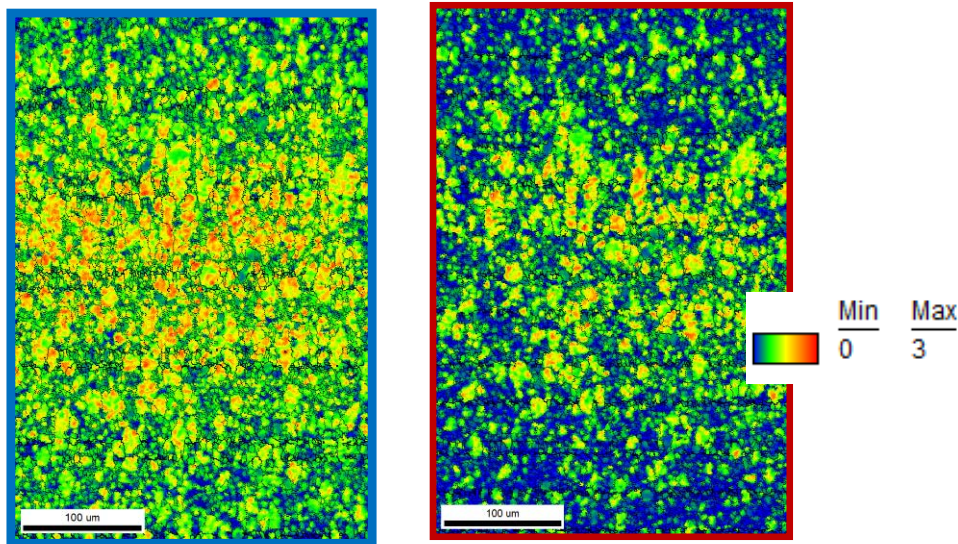
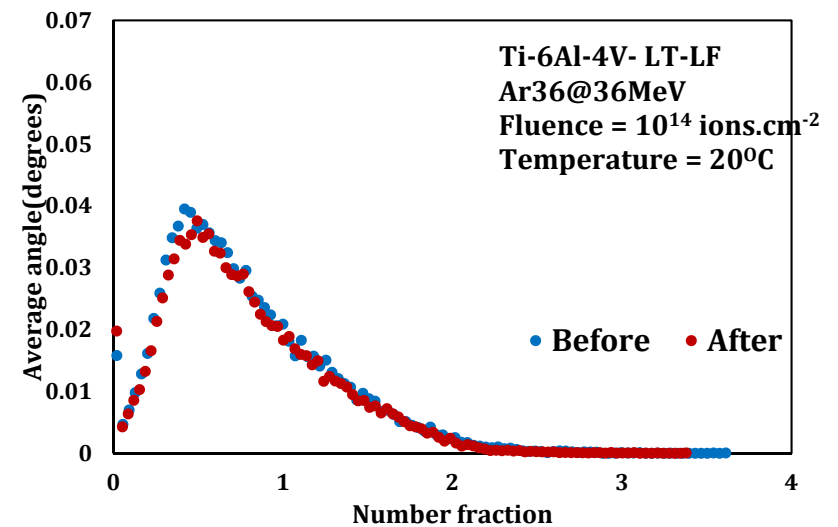
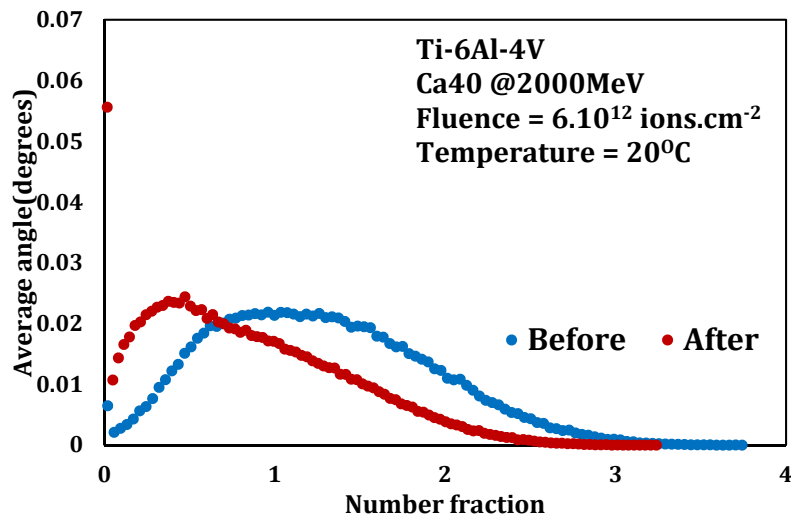


Unexpected changes in the local average misorientation for Ti-6Al-4V-1B



# Local Average misorientation charts

Comparison between high energy and low energy irradiation



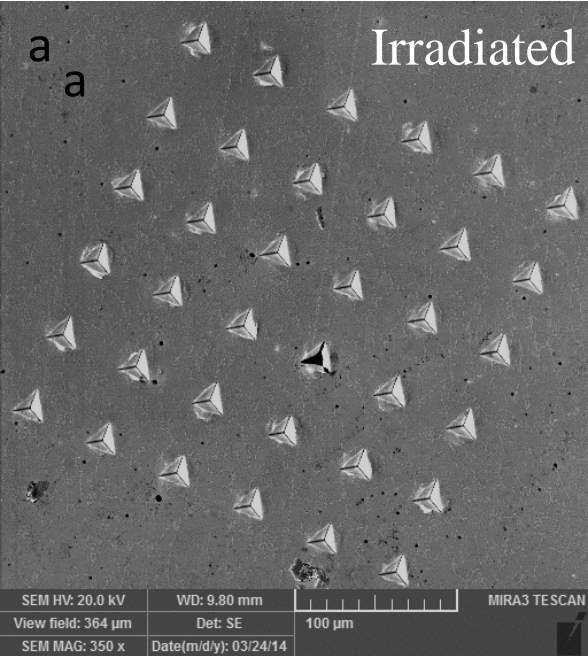
At high energy irradiation, the local average misorientation are more affected in Ti-6Al-4V

# Mechanical testing: Nano-indentation

Obtain the properties of the materials in depth.

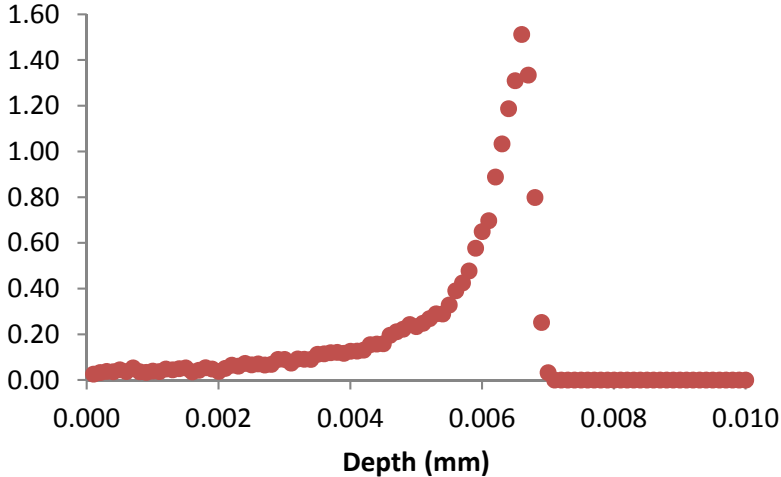
Parameters:

- Berkovich tip
- Strain rate :  $0.05s^{-1}$
- Poisson ratio=0.33
- Distance between indents:  $50\mu m$

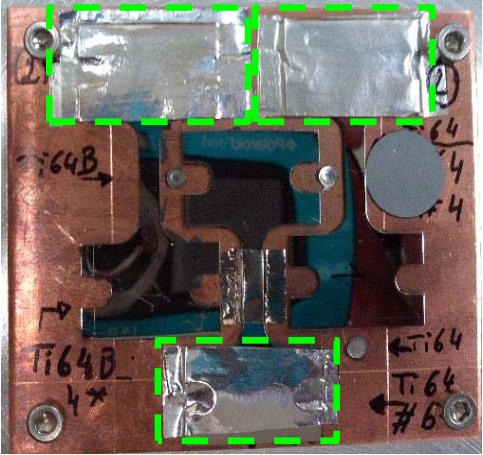


SEM image of nanoindentations matrix in Ti-6Al-4V irradiated at  $T= 20^{\circ}C$  and a fluence of  $10^{15} \text{ ions.cm}^{-2}$

dpa for  $\Phi=1e15 \text{ ion/cm}^2$   
Ar 36 @ 36 MeV



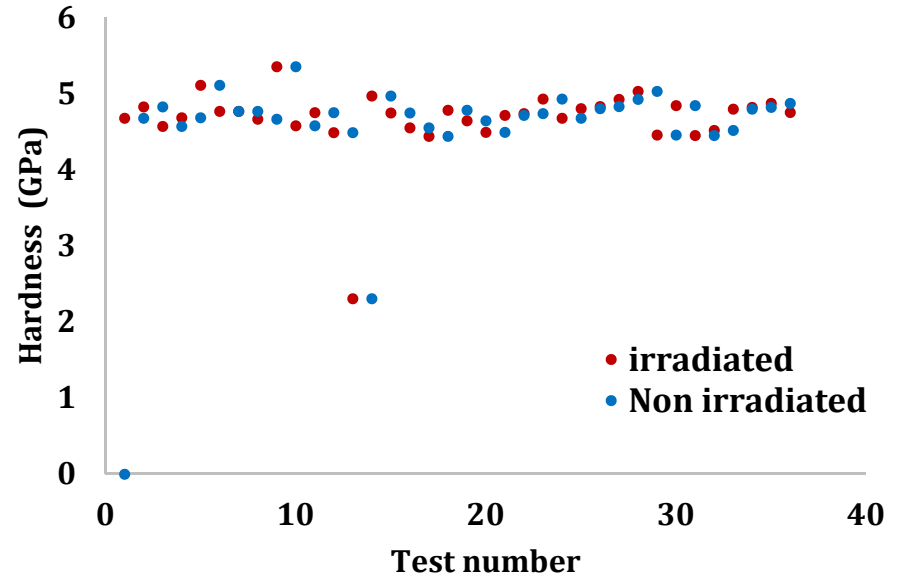
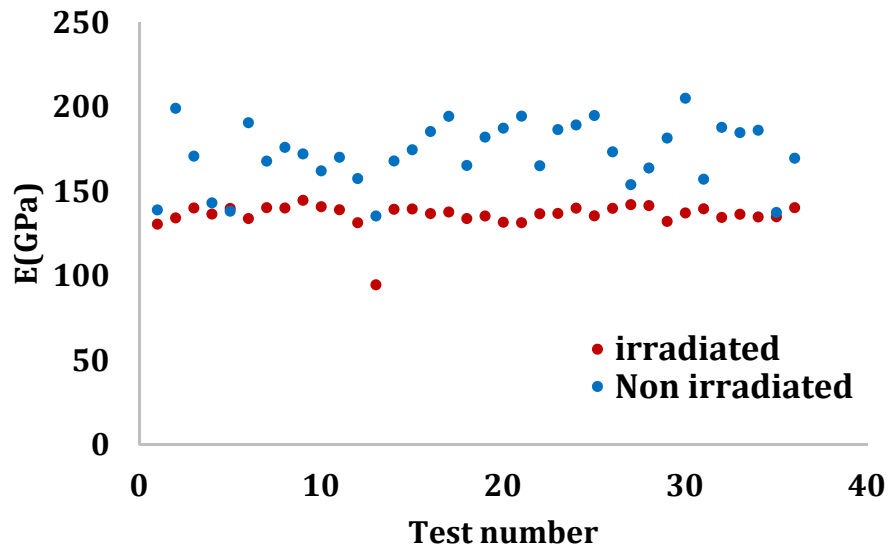
Al mask



In all samples one grip was masque in order to compare irradiated to not irradiated properties in the same sample

# Nano-indentation results

Ti-6Al-4V  
Ar36 @36MeV  
T= 20 °C  
Fluence =  $10^{15}$  Ions.cm<sup>-2</sup>  
Dose= 0.038dpa

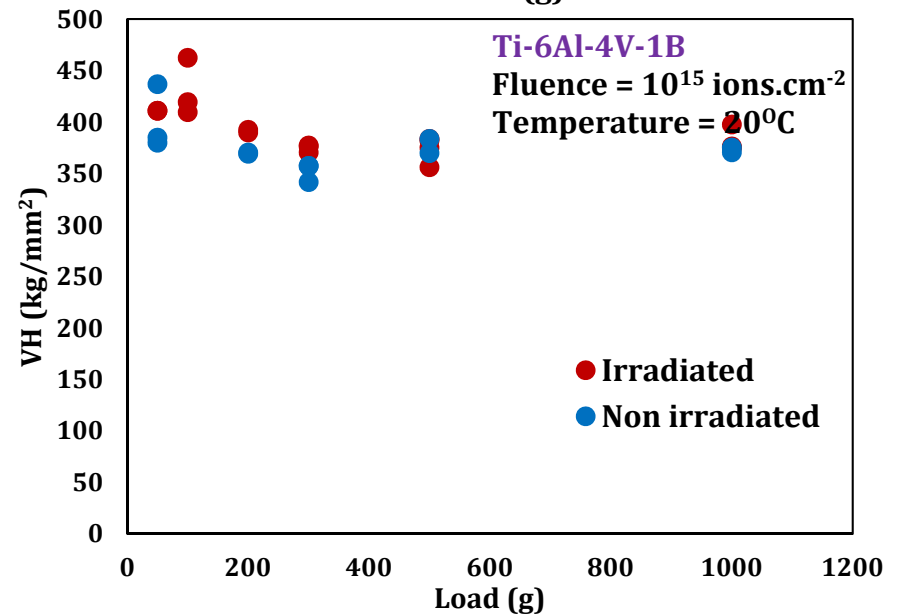
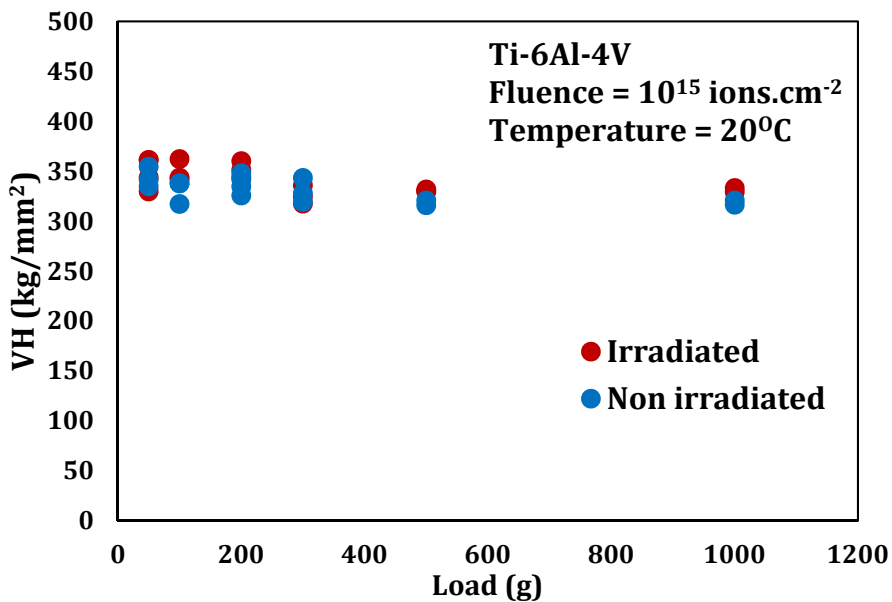
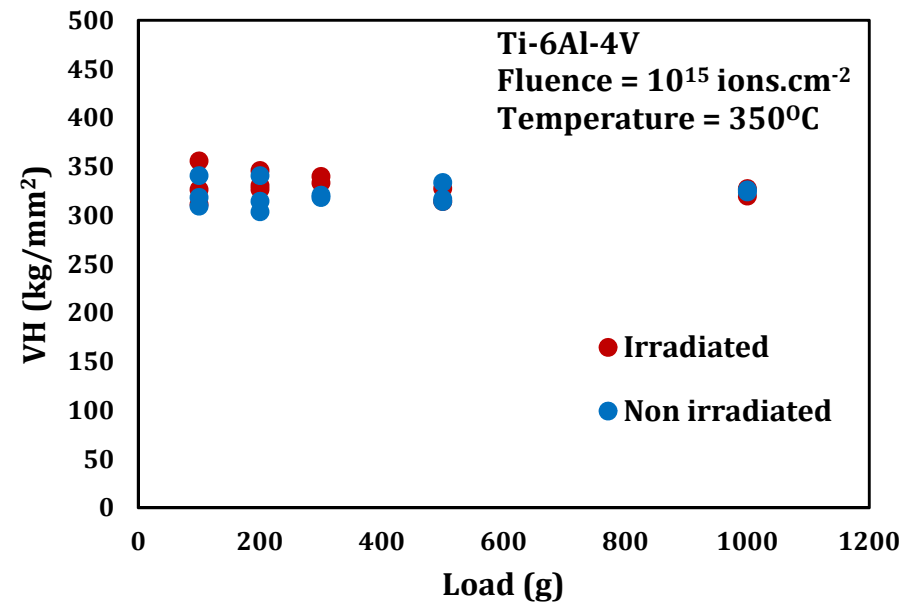
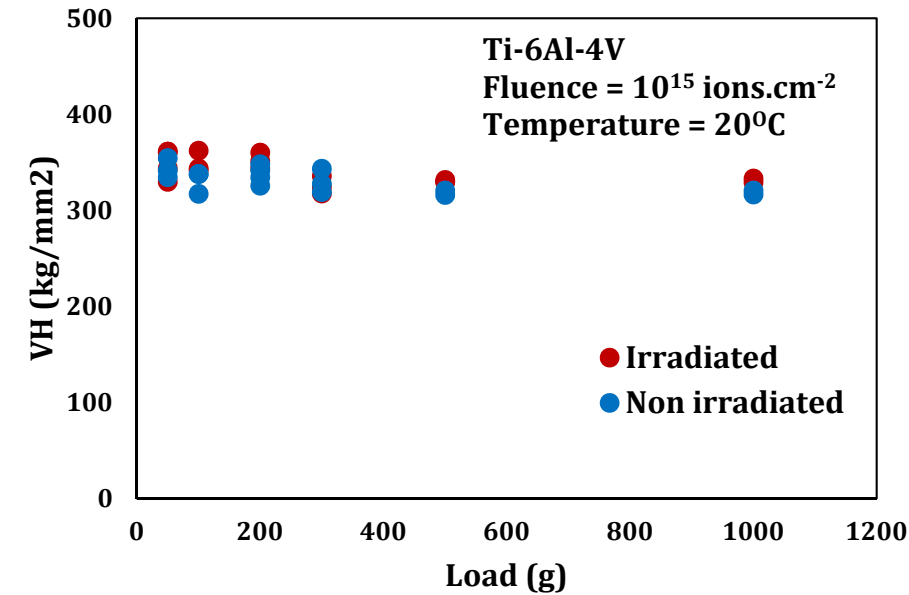


No change in hardness was observed  
Decrease in the elastic modulus after irradiation

# Vickers Hardness tests:

Ar @36 MeV

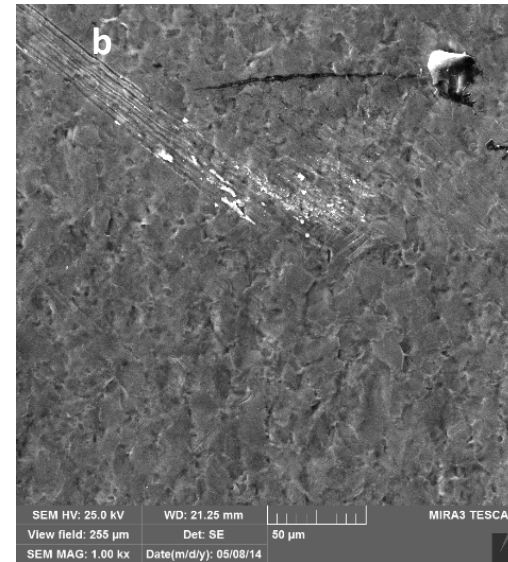
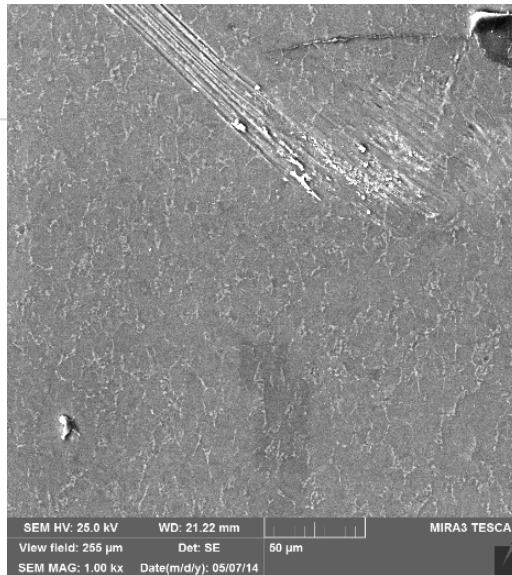
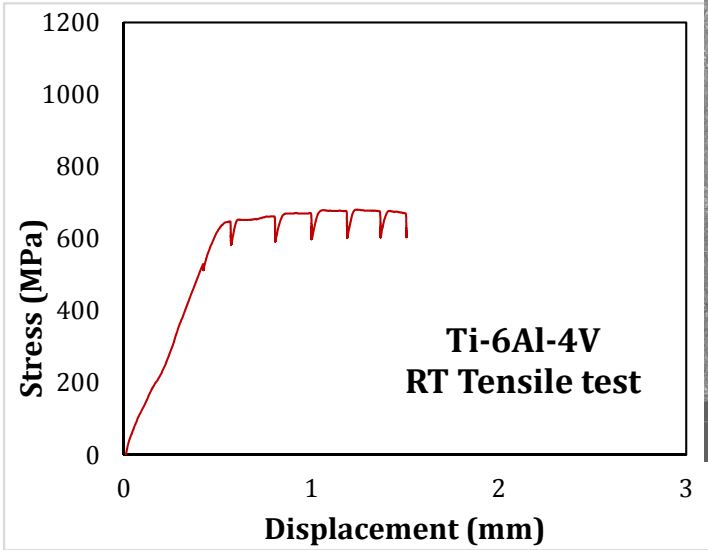
## No change in hardness



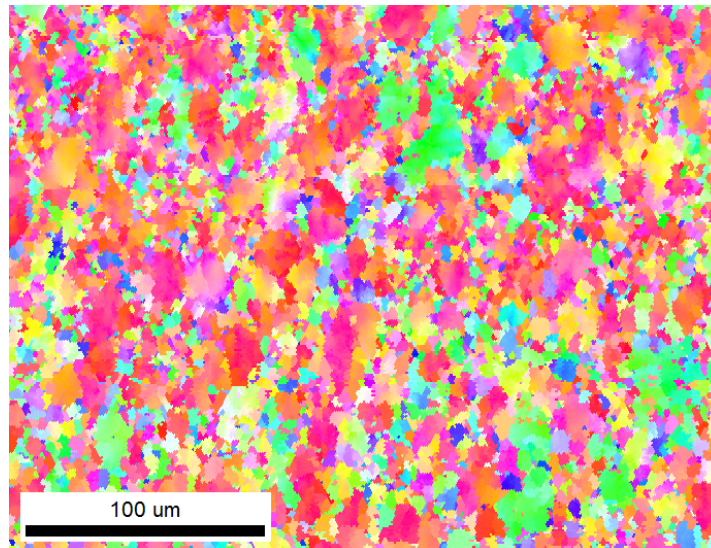
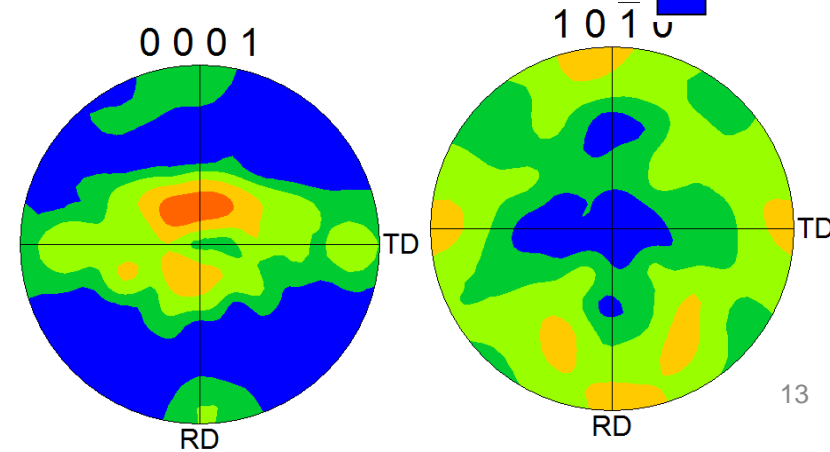
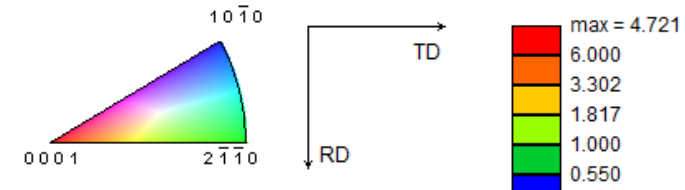


# In-situ Tensile tests: Preliminary results

**Ion beam : Ca 40 @2000MeV**  
**T= 20°C**  
**Fluence =  $6.10^{12}$  ions.cm<sup>-2</sup>**  
**Max dpa=  $10^{-5}$  dpa**



**SEM images of  
the same area  
(a) before the  
tensile test  
and (b) at 13%  
strain**

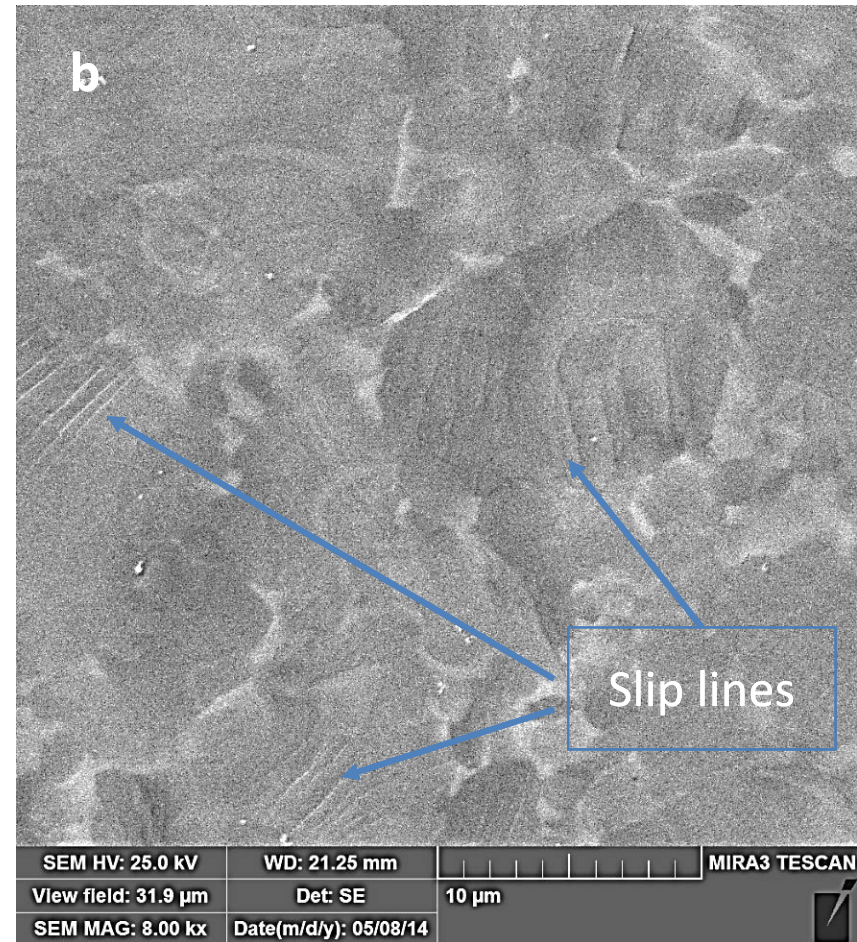
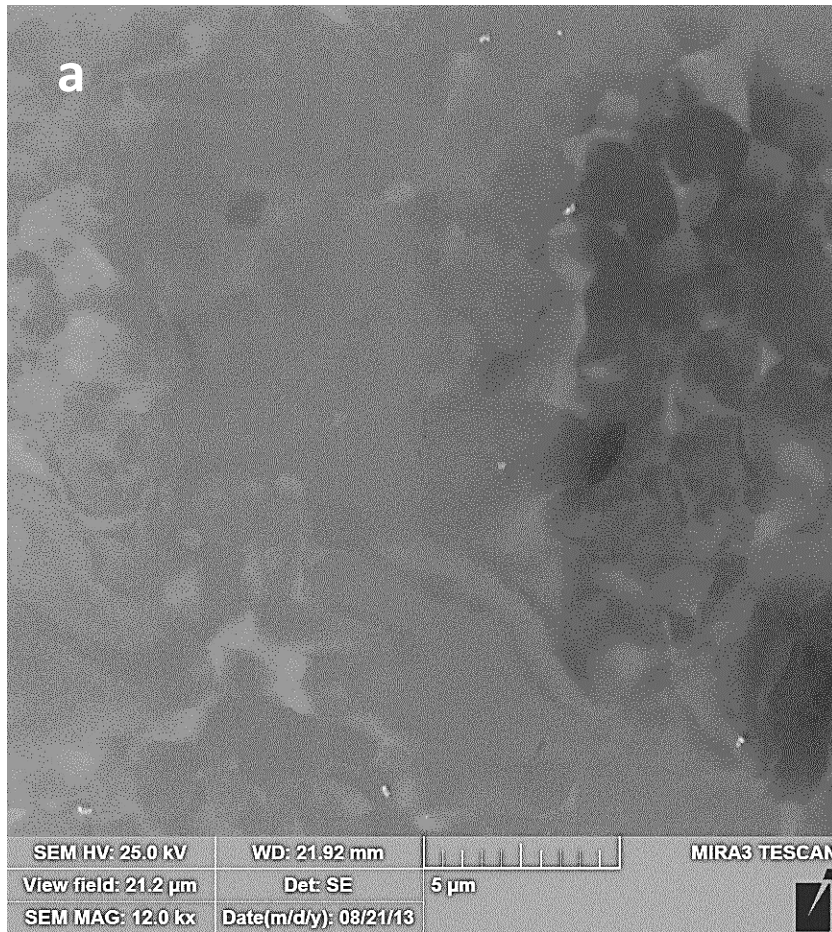


**IPF map , {0001} and {10-10}  
pole figures of the α phase  
in the same area before  
irradiation**



# Evolution of the microstructure during the test

Ion beam : Ca 40 @2000MeV  
T= 20°C  
Fluence =  $6.10^{12}$  ions.cm<sup>-2</sup>  
Max dpa=  $10^{-5}$  dpa

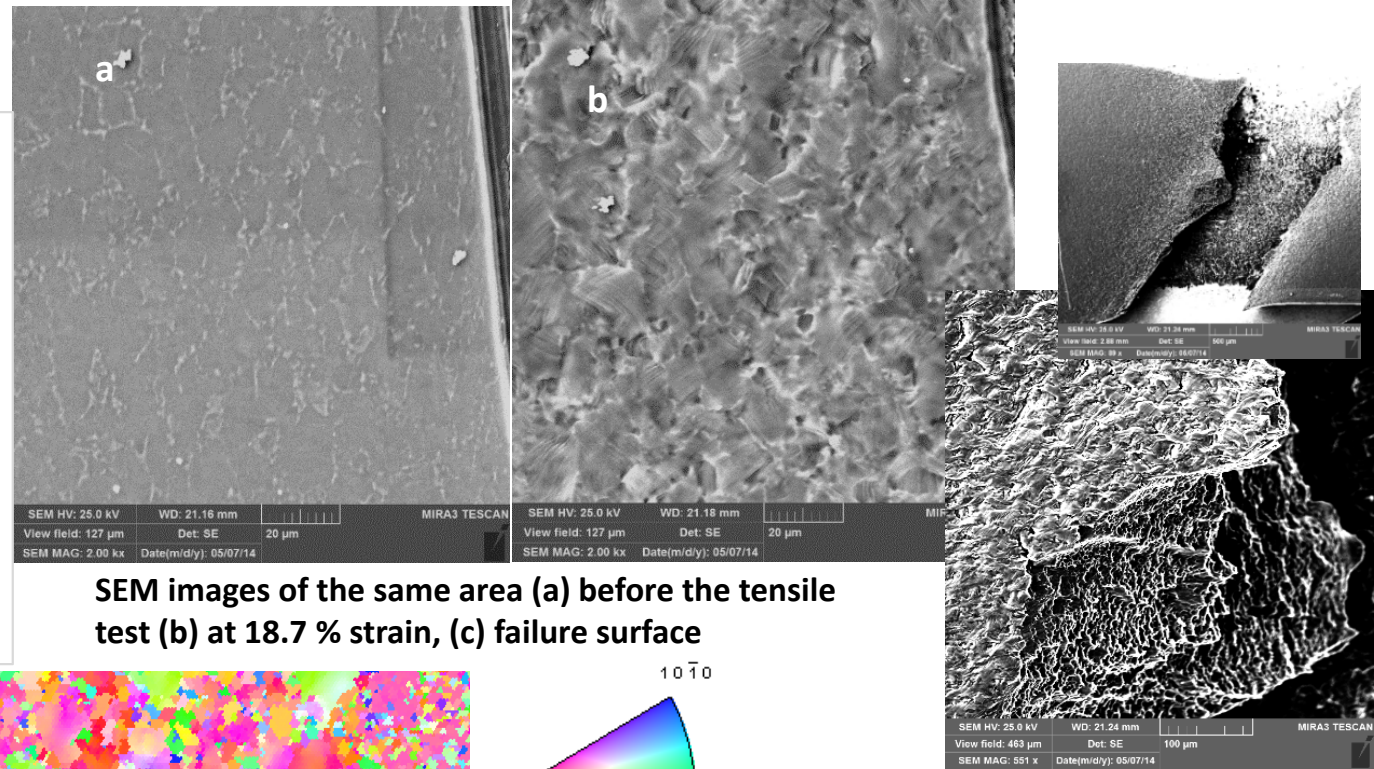
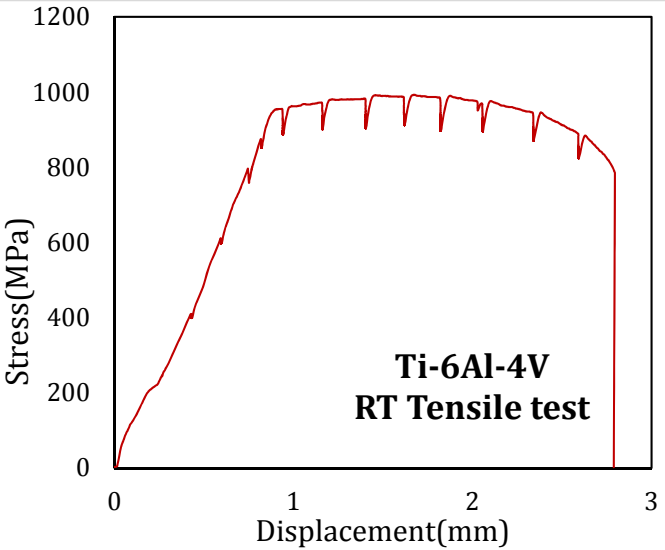


Microstructure of irradiated Ti-6Al-4V (a) before the tensile test and (b) at 5.27% strain



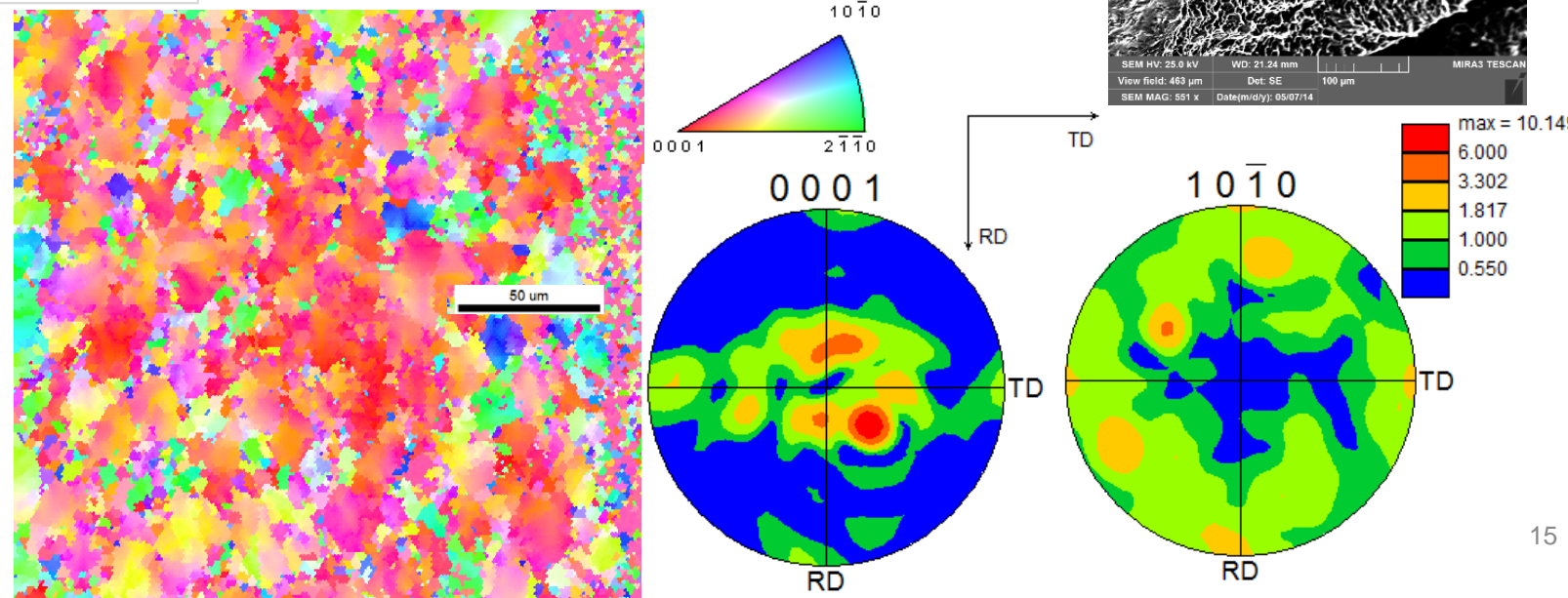
# In-situ Tensile tests: Preliminary results

**Ion beam : Ar@36MeV**  
**T= 350°C**  
**Fluence =  $10^{15}$  ions.cm<sup>-2</sup>**  
**Max dpa= 1.5 dpa**



SEM images of the same area (a) before the tensile test (b) at 18.7 % strain, (c) failure surface

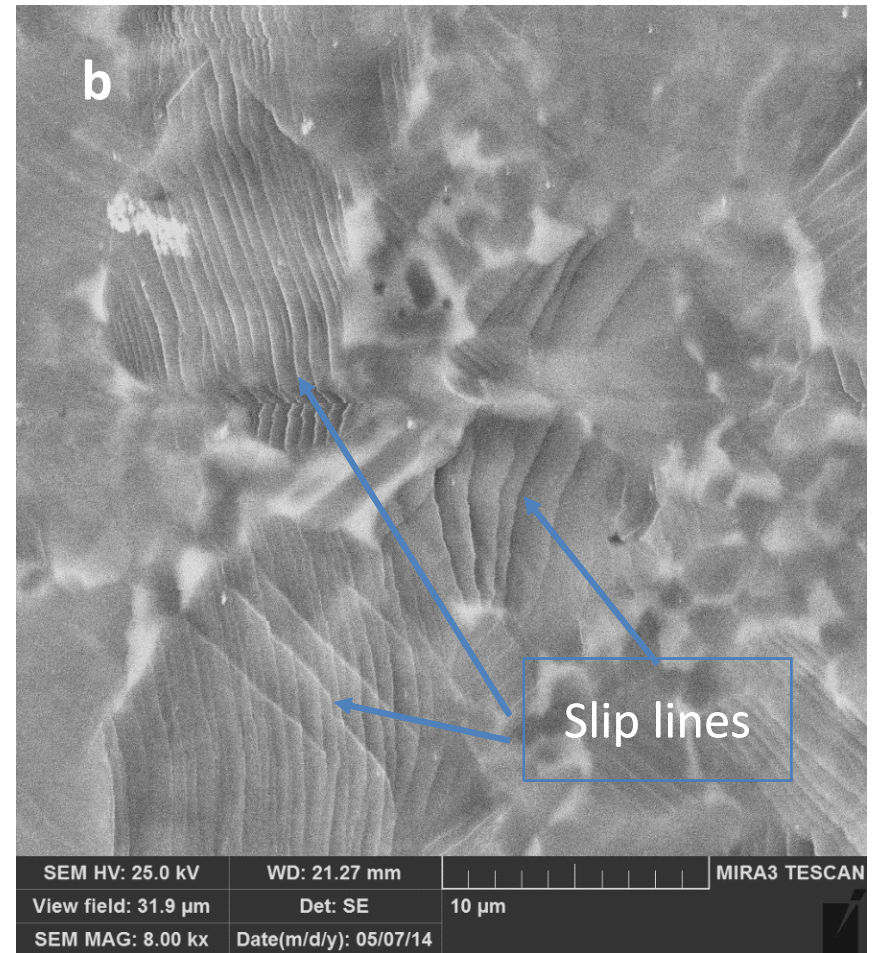
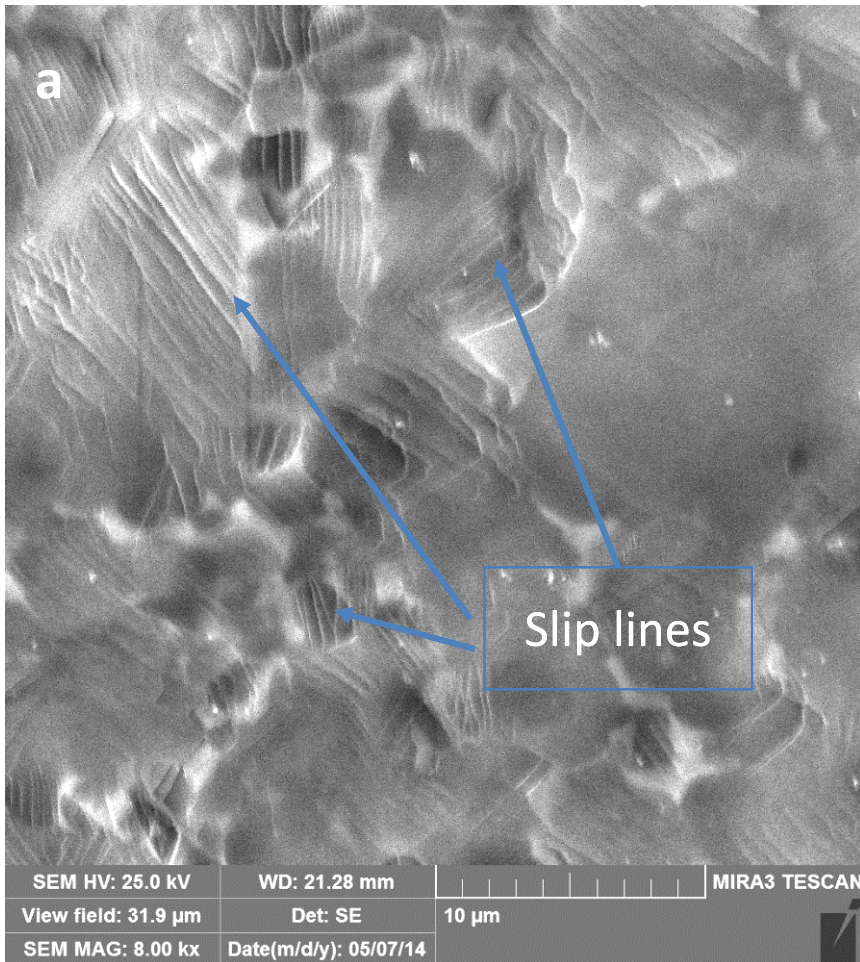
IPF map , {0001} and {10-10} pole figures of the  $\alpha$  phase in the same area before irradiation





# Evolution of the microstructure during the test

Ion beam : Ar@36MeV  
T= 350 °C  
Fluence =  $10^{15}$  ions.cm<sup>-2</sup>  
Max dpa= 1.5 dpa

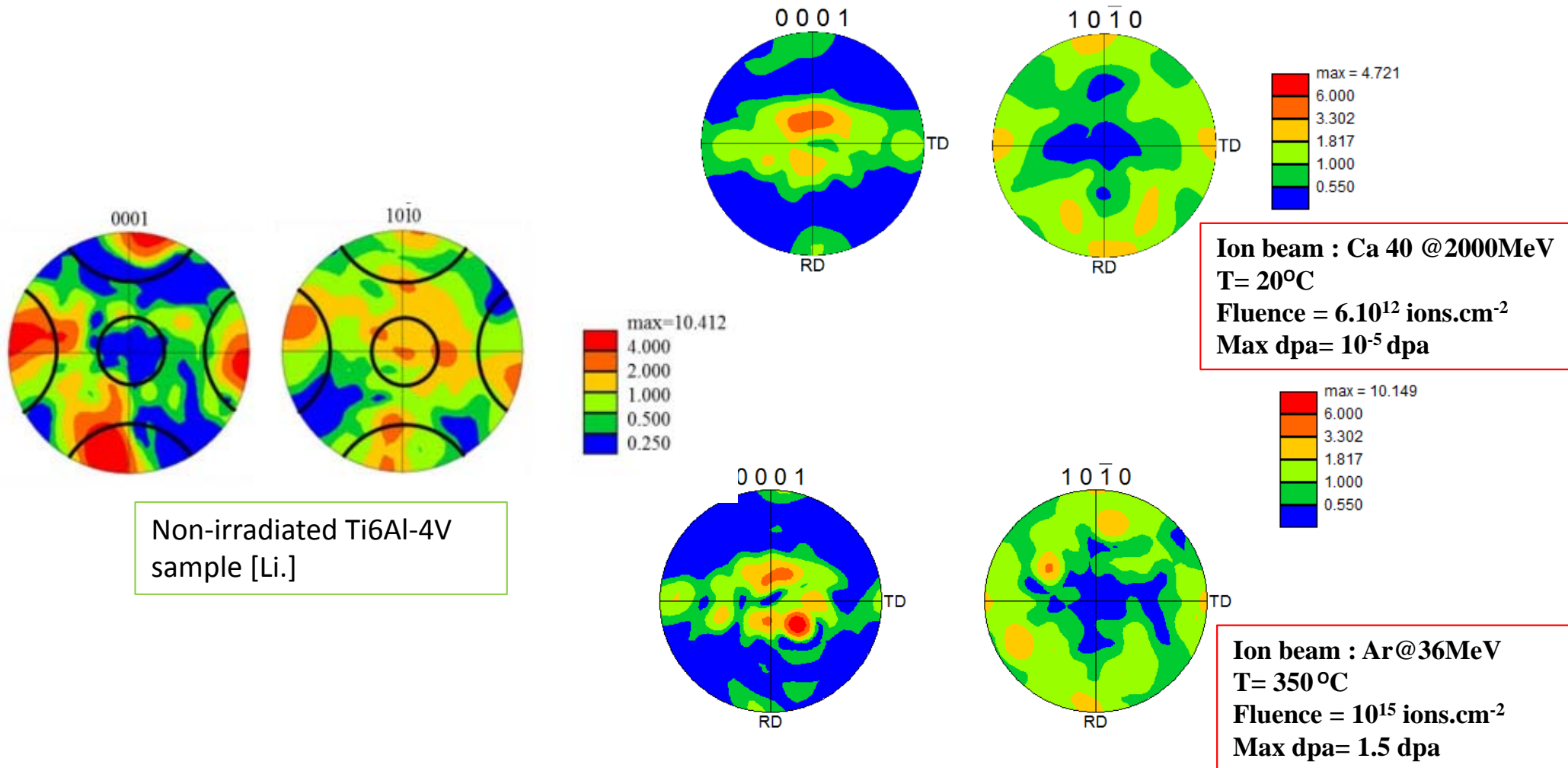


Microstructure of irradiated Ti-6Al-4V (a) at 6.77% strain and (b) at 9.38% strain

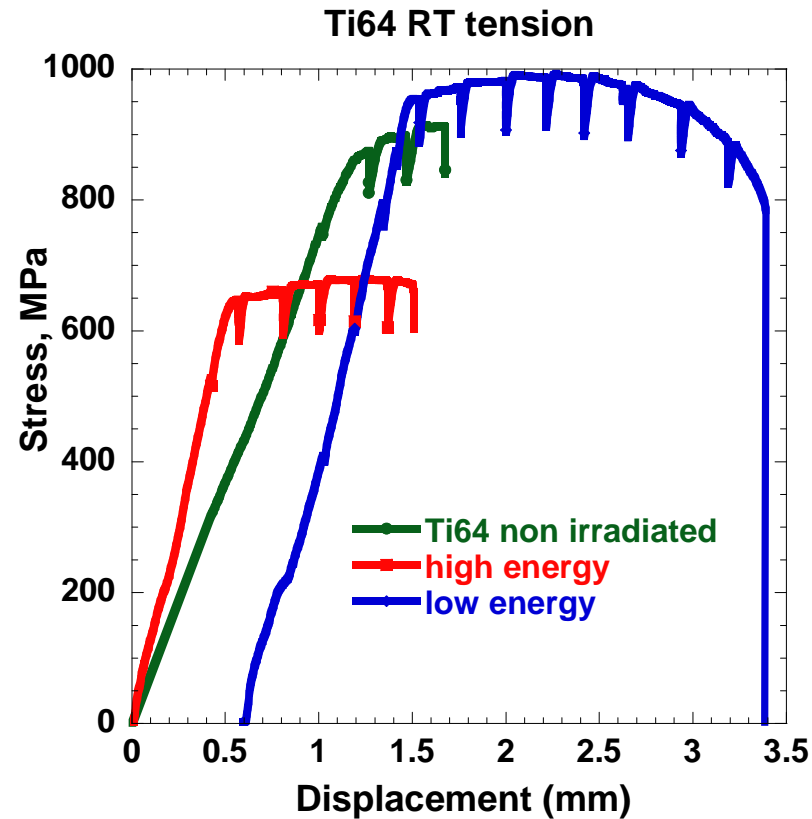


# In-situ Tensile tests: Preliminary results

Texture of the tested Ti-6Al-4V



# In-situ Tensile tests: Comparison with other Ti64 RT tension Test



For low energy irradiation no change in the mechanical properties ( the damage is only on the surface (7 microns).

For high energy irradiation, even at low doses, we observed a significant decrease in the UTS.



More tests are required

# Conclusion

- Ongoing analyses:
  - Nano-indentation tests on the cross sections of the samples will allow extraction of hardness and Young modulus for different dpa doses.
  - In-situ tensile tests: Comparison between non irradiated and irradiated Ti-alloys and slip trace analysis
- Future analyses
  - New EBSD analyses planned after electro-polishing the samples
  - Swelling measurements on each samples
  - Possibility to use FIB (Focused Ion Beams) to study the damage in the depth of the sample for TEM, SEM/EBSD analyses
  - In-situ irradiation and creep test