Titanium Alloy Radiation Damage Tests

Aida Amroussia, PhD Candidate[1]
Pr. Carl J Boehlert[1]
Dr. Frederique Pellemoine[2]
Pr. Wolfgang Mittig[2]

[1]Michigan State University
[2]Facility For Rare Isotope Beams

May 19, 2015

MICHIGAN STATE UNIVERSITY

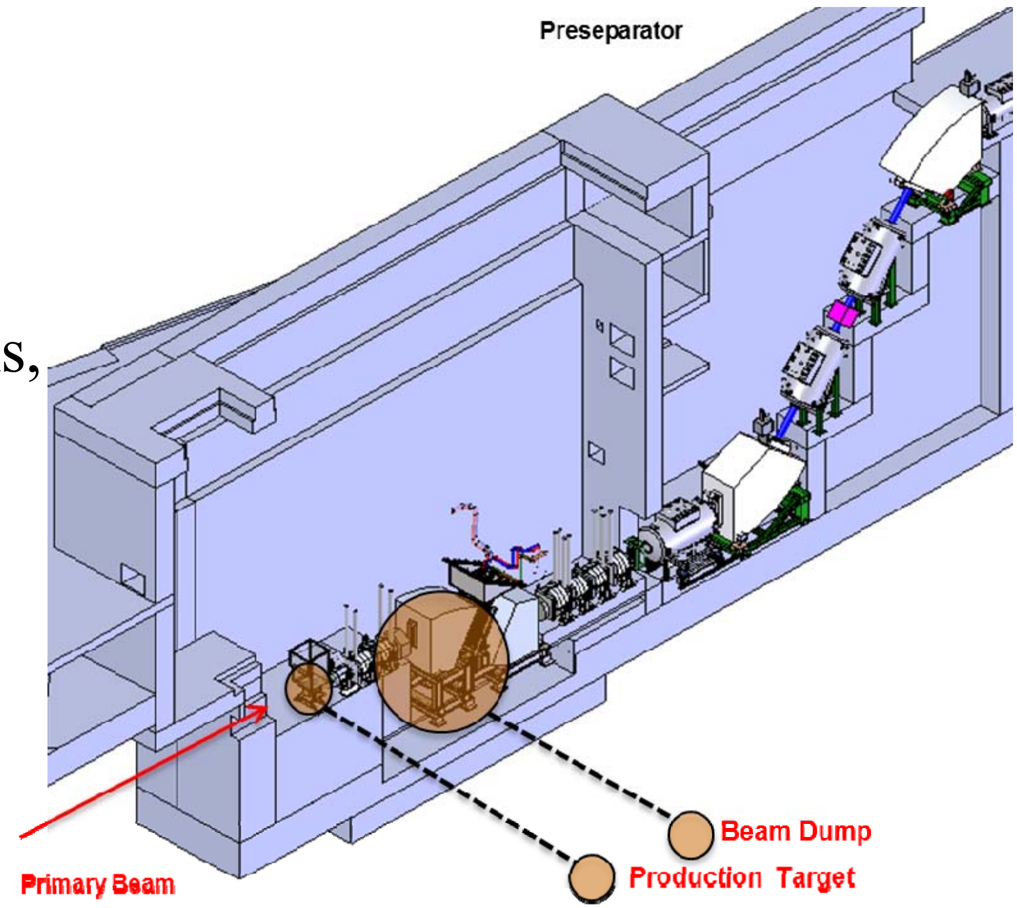
FACILITY FOR RARE ISOTOPE BEAMS

Outline

1. **Introduction**
2. **Microstructure characterization**
3. **Experimental Methods**
4. **Irradiation damage in Ti-6Al-4V: Literature review**
5. **Microstructure characterization**
6. **Hardness measurements**
7. **Conclusion**

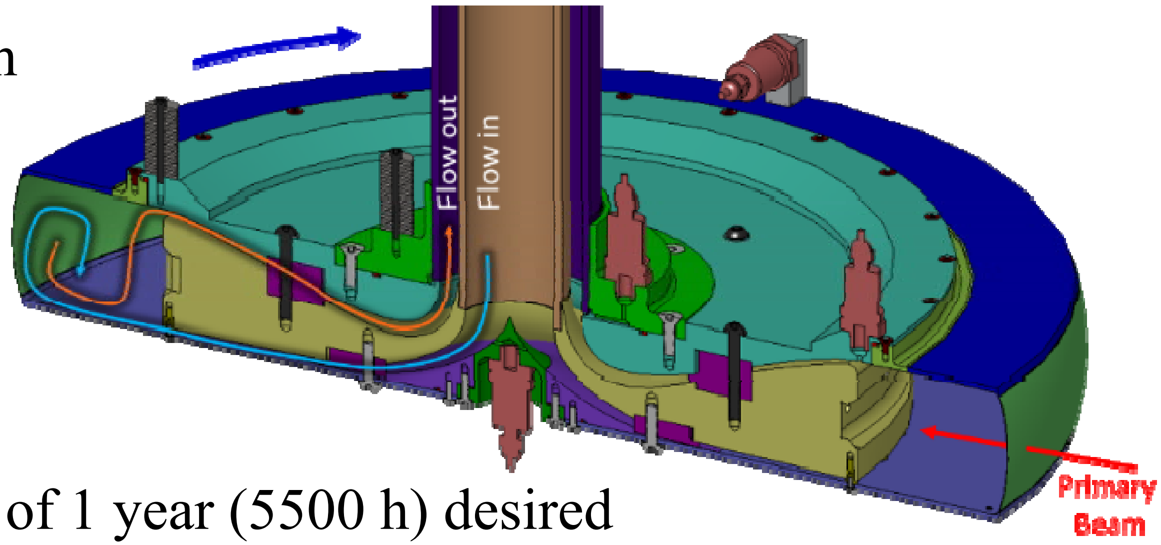
Facility for Rare Isotope Beams at Michigan State University

- The FRIB at Michigan State University is a new generation accelerator with high power heavy ion beams.
- It will provide primary beams from O to U with an energy of 200 MeV/u for heavy ion beams, and higher energies for lighter beams.
- Beam Dump
    - Up to 325 kW



FRIB Beam Dump

- Water-filled rotating drum beam dump chosen for FRIB baseline



- FRIB conditions:
  - ➢Beam Dump lifetime of 1 year (5500 h) desired
  - ➢Estimated cumulative dpa after one year of use ~9 dpa with a fluence of 10^{15} ions.cm^{-2}
  - ➢Se from 0.08 keV/nm (with O beam) to 12.6 keV/nm (with U beam)

  - Ti-6Al-4V and Ti-6Al-4V-1B were chosen as candidate materials
  - The current study addresses the radiation damage challenge and focuses on understanding Swift Heavy Ion (SHI) effects on Ti-alloy that can limit beam dump lifetime

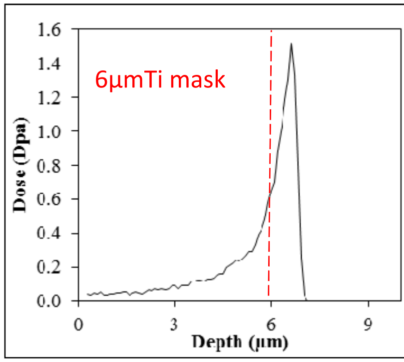
MICHIGAN STATE UNIVERSITY

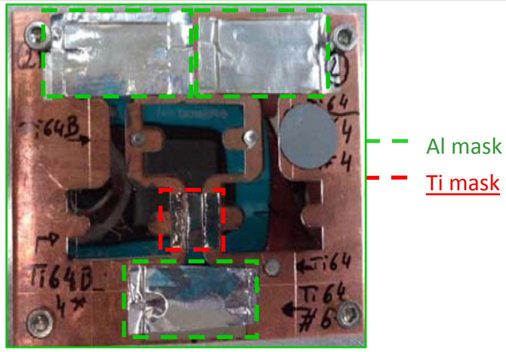
Irradiation set up

- Two main irradiation experiments with Ti-6Al-4V and Ti-6Al-4V-1B samples were performed at the IRRSUD beamline facility at the GANIL-CIMAP Laboratory, Caen France.
- The IRRSUD beam line was chosen due to comparable S_e values to the FRIB conditions (0.08 -13 $keV.nm^{-1}$) without the activation of the sample (> coulomb barrier)

Beam	Energy (MeV/u)	Ranges (µm)	S_e ($keV.nm^{-1}$)	Temperature (^oC)	Fluence ($ions.cm^{-2}$)
^{36}Ar	1	6.8	7.5	25 - 350	10^{15}
^{131}Xe	1.4	8.5	19.7	25 - 350	$2\text{-}7.\ 10^{14}$

The SRIM-2013 calculation of the dose in a Ti-6Al-4V sample for the ^{36}Ar @36 MeV beam with a fluence of 10^{15} $ions.cm^{-2}$





MICHIGAN STATE UNIVERSITY

Irradiation set up

Ti-alloys irradiations at CIMAP and NSCL

Facilities	Beam	Energy [MeV]	Range [µm]	S_e [keV/nm]	Fluence [ions/cm²]	Max dpa in sample	Date	Number of samples	Type
IRRSUD	^{82}Kr	25	4.73	9.9	5.10^{11}- 5.10^{12}- 2.10^{14}	0.6	Jul-2013	6	Foils
	^{131}Xe	92	8.5	19.7	2.10^{11}	0.001	Jul-2013	2	Foils
	^{82}Kr	45	6.43	13.1	5.10^{11}- 5.10^{13}	0.16	Jul-2013	4	Foils
	^{82}Kr	45	6.43	13.1	2.10^{14} $2.5.10^{15}$	8	Oct-2013	6	Foils
	^{36}Ar	36	6.8	7.5	10^{15}	1.5	Dec-2013	23	TEM and dogbone
	^{131}Xe	92	8.5	19.7	$2\ 10^{14}$ $7\ 10^{14}$	3.5	June-2014	6	Dogbone
NSCL	^{40}Ca	2000	800	1.5	$6\ 10^{12}$	10^{-5}	Aug-2013	1 x Ti64	Dogbone

FRIB conditions

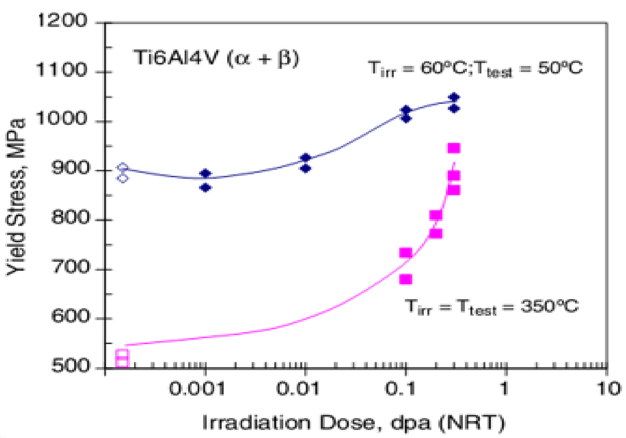
- Estimated cumulative dpa after one year of use ~9 dpa with a fluence of 10^{15} ions/cm²
- Se from 0.08 keV/nm (with O beam) and 12.6 keV/nm (with U beam)

Irradiation damage in Ti-6Al-4V

Effect of dose and temperature on the microstructure of neutron irradiated Ti-6A-4V (Tähtinen *et al.* , Sastry *et al.*, Peterson)

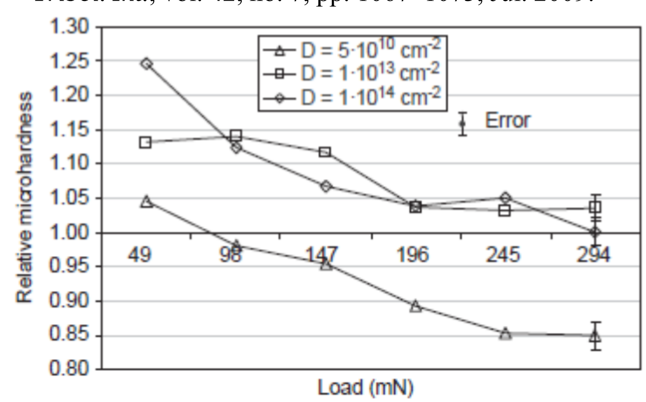
Temperature and dose level	Microstructure change observations
50ºC , 0.3 dpa	A high concentration of uniformly distributed defect clusters in the α-phase
350º C, 0.3 dpa	Dislocation loops Vanadium precipitates
450ºC, Dose 2.1 and 32 dpa	Dislocation loops β-phase precipitates in α phase
550ºC 32 dpa	Extensive void formation Coarse β-precipitates

P. Budzynski, V. A. Skuratov, and T. Kochanski, "Mechanical properties of the alloy Ti–6Al–4V irradiated with swift Kr ion," *Tribol. Int.*, vol. 42, no. 7, pp. 1067–1073, Jul. 2009.



Dose dependence of yield strength of Ti-6Al-4V irradiated with neutrons

Different hardening mechanisms operate at 50ºC than at 350ºC.



Relative micro-hardness in Ti-6Al-4V irradiated with swift 250Mev Kr^{+26} at different fluences

MICHIGAN STATE UNIVERSITY

Tähtinen et al. / *Journal of Nuclear Materials*, 367-370 (2007), 627–632
Sastry et al / Fourth International Conference on Titanium, Kyoto, Japan, 1980, vol. 1, p. p. 651.
D.T. Peterson, / *Effects of Radiation on Materials: 11th International Symposium*, Philadelphia, PA, 1982, p. p. 260.

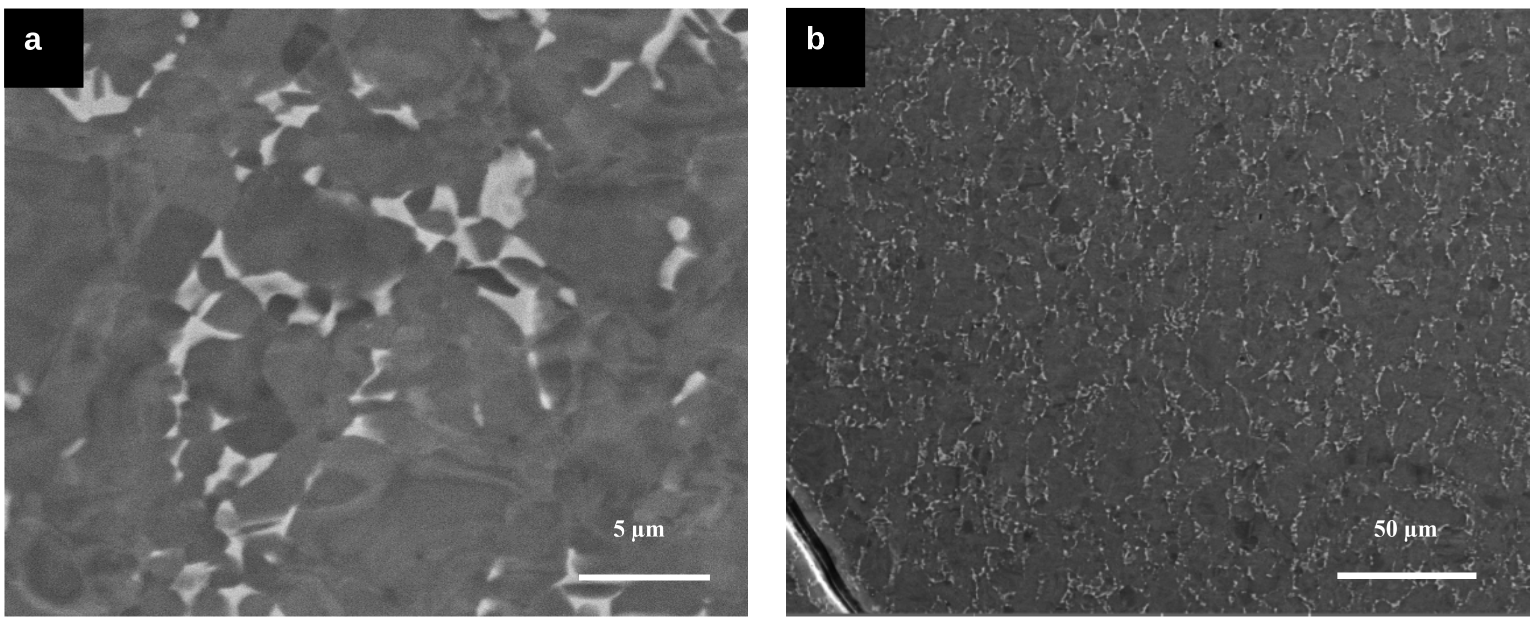
Microstructure of the as-received materials

Ti-6Al-4V:

Lenticular α-phase with mostly an intergranular β-phase. Intra-granular β-phase was also observed.

The volume fraction of the β-phase was ~6.6 vol.% and the α-phase ~ 93.4 vol.%.

The grain size of the α-phase ranged between 5 ~ 20 µm.



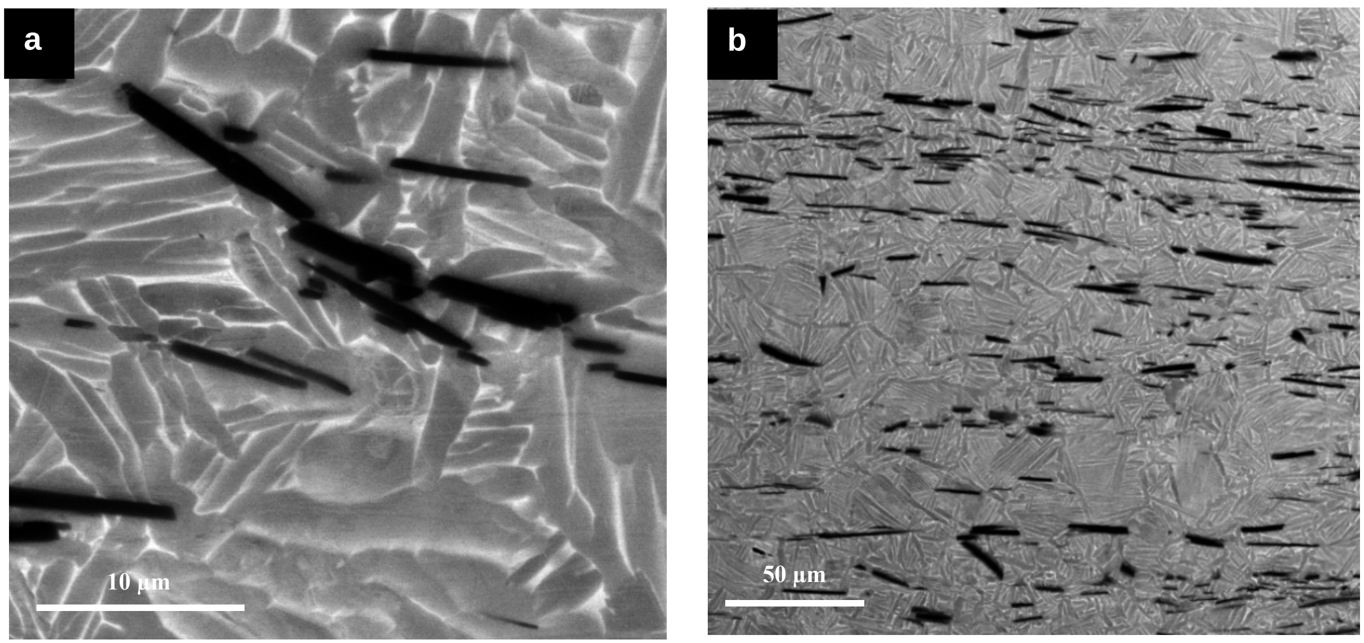
BSE images of the initial microstructure of Ti-6Al-4V (a) higher and (b) lower magnification

Microstructure of the as-received materials

Ti-6Al-4V-1B

The microstructure contained both an equiaxed (7.4µm) and lenticular α-phase; total volume percent α-phase was ~ 79%.

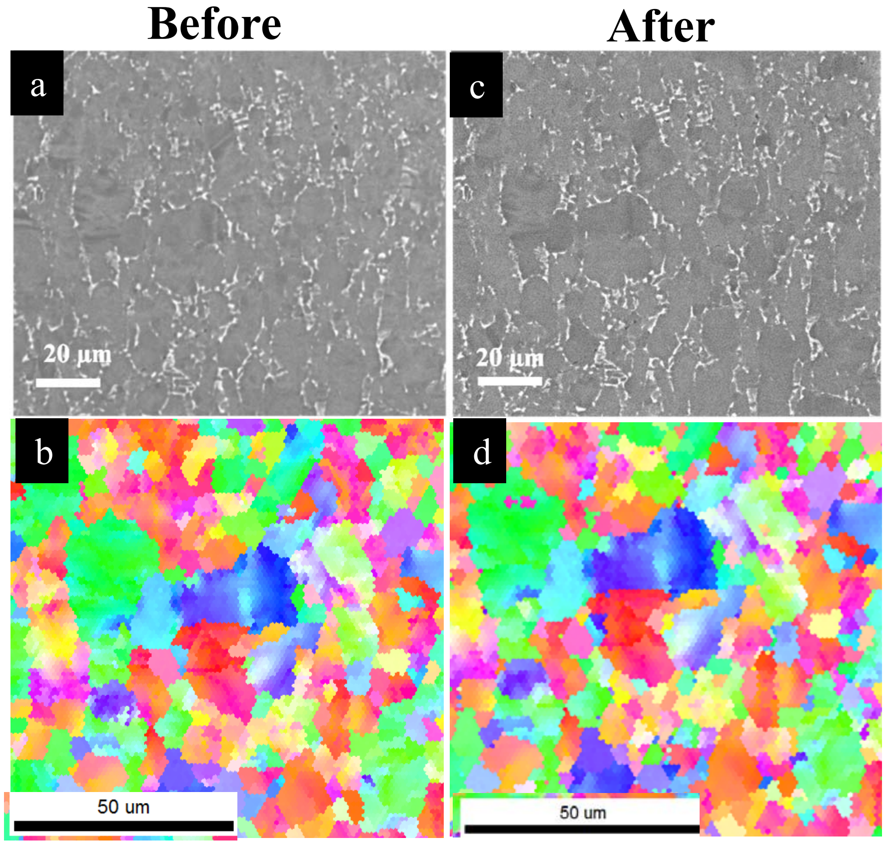
The β-phase volume percent was ~ 15 vol.% while the TiB phase volume percent was ~5.9 vol.%.(Chen *et al.)*



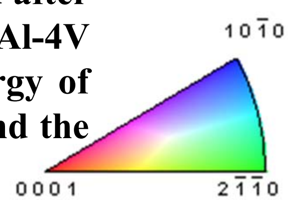
BSE images of the initial microstructure of Ti-6Al-4V-1B (a) higher and (b) lower magnification

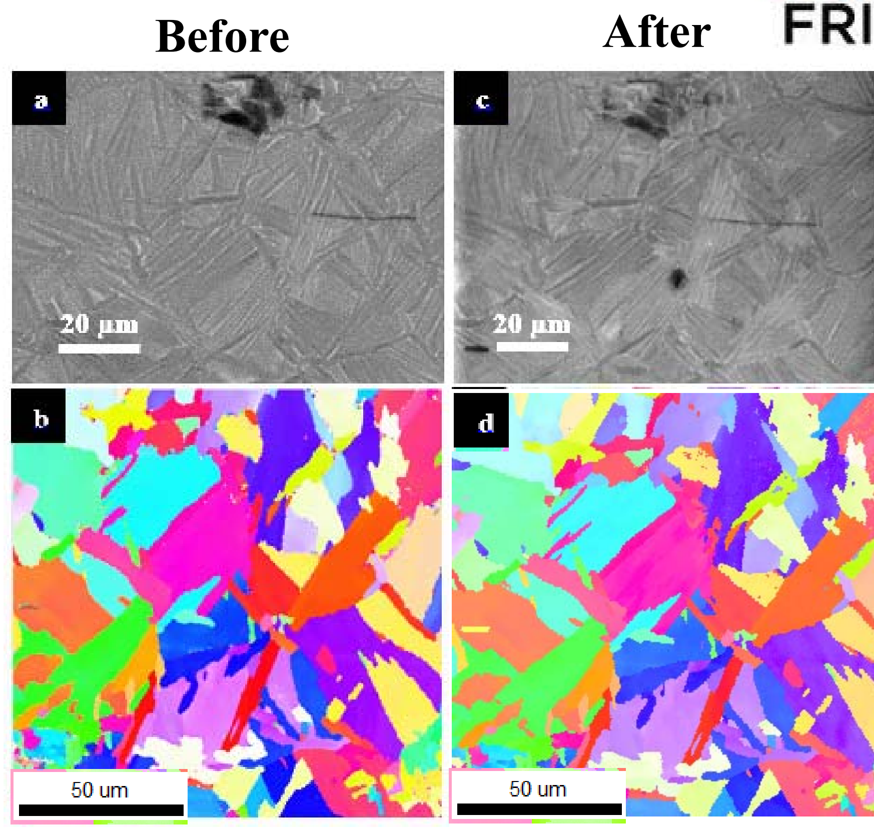
W. Chen et al / *Key Eng. Mater.*, vol. 436, pp. 195–203, May 2010.

Microstructure Characterization



BSE images and IPF maps before (a,b) and after irradiation at the same area (c,d)in a Ti-6Al-4V sample irradiated with ^{131}Xe with an energy of 92 MeV. The fluence was 2.10^{14} ions.cm^{-2} and the temperature 25^{o}C





BSE images and IPF maps before (a,b) and after irradiation at the same area (c,d)in a Ti-6Al-4V-1B sample irradiated with ^{36}Ar with an energy of 36 MeV. The fluence was 1.10^{15} ions.cm^{-2} and the temperature 350^{o}C

- No change in microstructure or grain orientation at the surface.

FRIB

MICHIGAN STATE UNIVERSITY

Hardness measurements

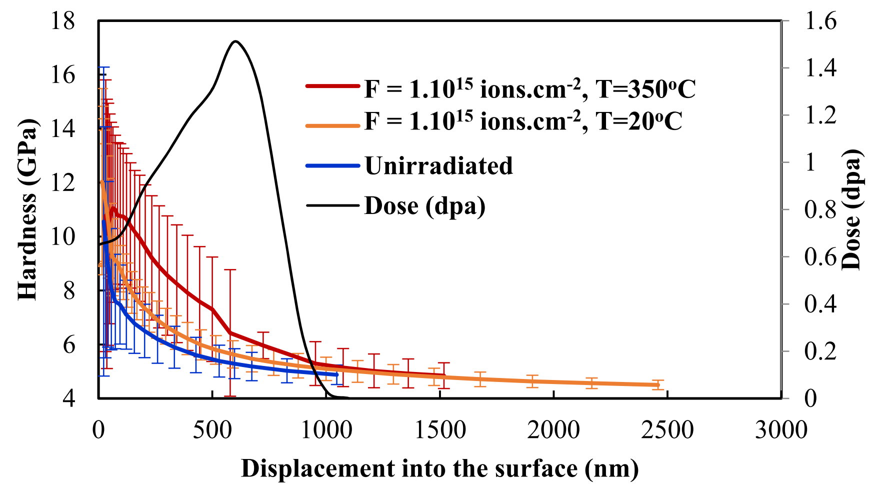
Nano-indentation

Obtain the properties of the materials in depth.

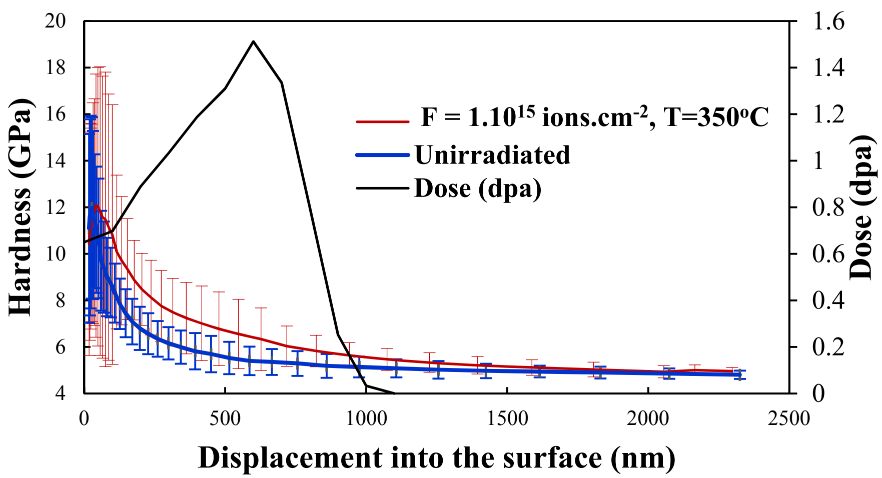
Parameters:

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

Ti-6Al-4V



Ti-6Al-4V-1B



Nano-indentation results for Ti-6Al-4V and Ti-6Al-4V-1B irradiated with ^{36}Ar @36 MeV at fluence of 1.10^{15} ions.cm^{-2}with the CP –Ti foil on the surface.

Boron addition to Ti-6Al-4V did not change its irradiation resistance

➢ A slight increase in hardness observed for the sample irradiated with a higher fluence (1.10^{15} ions.cm^{-2}) and lower temperature (T = 350°C) for the higher doses

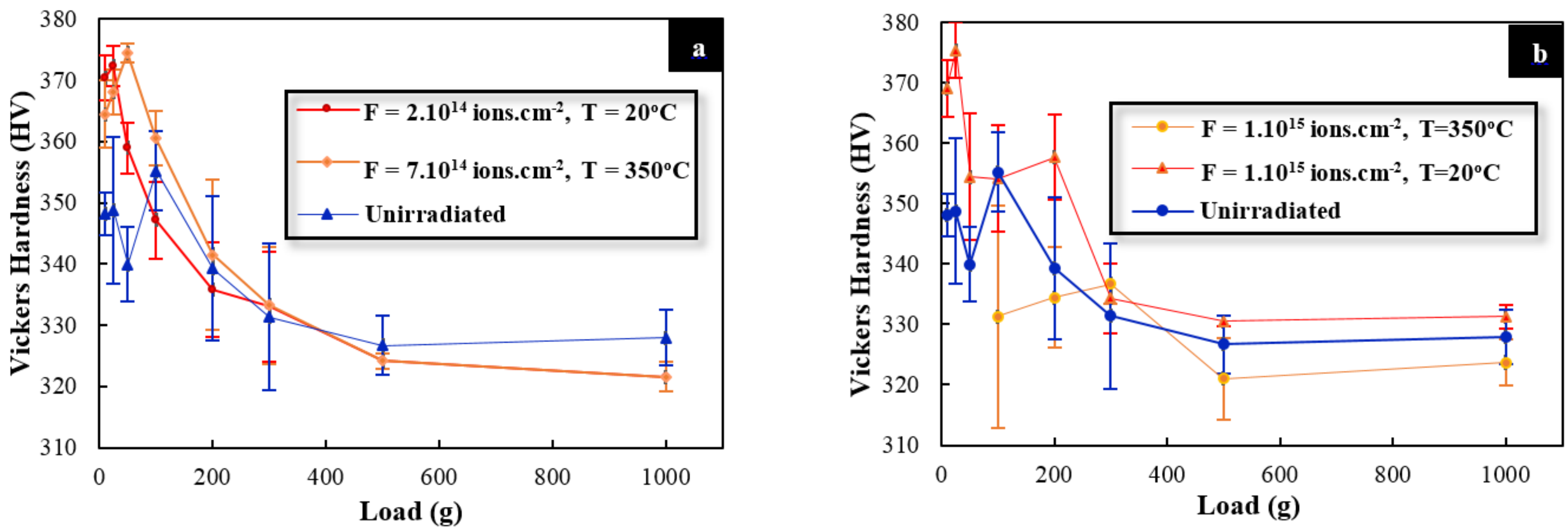
MICHIGAN STATE UNIVERSITY

FRIB

Hardness measurements

Vickers Hardness

- Vickers hardness was performed on 4 irradiated Ti-6Al-4V samples.



Vickers Hardness measurements for Ti-6Al-4V irradiated with: a) ^{131}Xe @ 92 MeV and b) ^{36}Ar @ 36 MeV

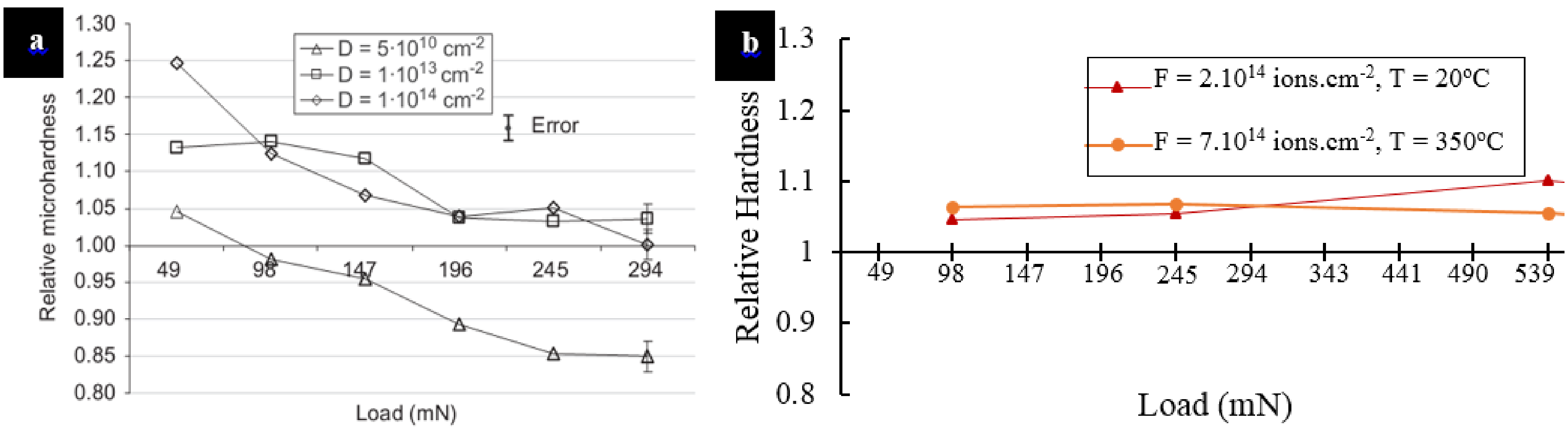
- The large scatter is due to the presence of two phases in the material
- A slight increase in hardness was observed for the sample irradiated with a higher fluence at lower loads (< 50g) (depth~ 1.6µm)

MICHIGAN STATE UNIVERSITY

Hardness measurements

Vickers Hardness

- The lower irradiation damage observed in our investigated Ti-6Al-4V samples compared to results reported by Budzynski et al. (2009) could be explained by
  - The difference in microstructure: larger grains (~100μm)
  - The gs was 5-20μm in our material and gbs act as sinks for radiation-induced-effects



Relative micro-hardness of the Ti–6Al–4V alloy as a function of applied load for Ti-6Al-4V irradiated with: a) Kr^{+26}@350 MeV (Budzynski et al. 2009) and b) ^{131}Xe @ 92 MeV.

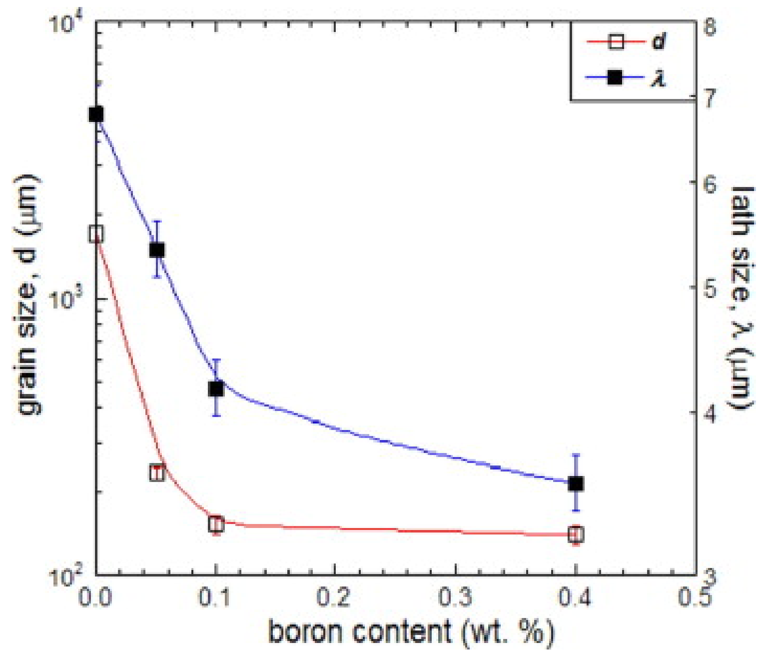
P. Budzynski, V. A. Skuratov, and T. Kochanski, "Mechanical properties of the alloy Ti–6Al–4V irradiated with swift Kr ion," *Tribol. Int.*, vol. 42, no. 7, pp. 1067–1073, Jul. 2009.

MICHIGAN STATE UNIVERSITY

FRIB

Discussion

- ➢Effect of the microstructure in the irradiation resistance of this Ti-alloy.
- ➢Effect of the small grains (5-20μm)
- ➢Boron addition causes grain refinement
- ➢Thermomechanical processing can improve its properties



Variation of prior β grain size, *d*, and the α lath size, *λ*, in Ti64 with wt.% B addition (Sen *et al.*)

Sen *et al*. Acta Materialia, Volume 55, Issue 15, September 2007, Pages 4983-4993,

Conclusion

- The analyzed hardness and nano-indentation suggest a higher irradiation damage resistivity in the two studied Ti-alloys than reported in literature for Ti-6Al-4V.

- Slight differences in the microstructure caused by the thermomechanical processing may be responsible for this difference.

- 1% boron addition to Ti-6Al-4V didn't degrade the radiation resistance

- Ongoing and Future work:
  - Irradiation creep test
  - In-situ tensile tests and slip trace analysis: Deformation mechanisms
  - X-ray diffraction: Investigate phase transformation
  - Effect of the microstructure on the irradiation damage in Ti-alloys

Acknowledgements

- MSU Department of Chemical Engineering and Material Science

- FRIB
  - Mikhail Avilov
  - Reginald Ronningen

- GANIL-CIMAP
  - Florent Durantel
  - Clara Grygiel
  - Isabelle Monnet
  - Florent Moisy
  - Marcel Toulemonde

Thank you for your attention