Graphite and Titanium Alloy Radiation Damage Tests

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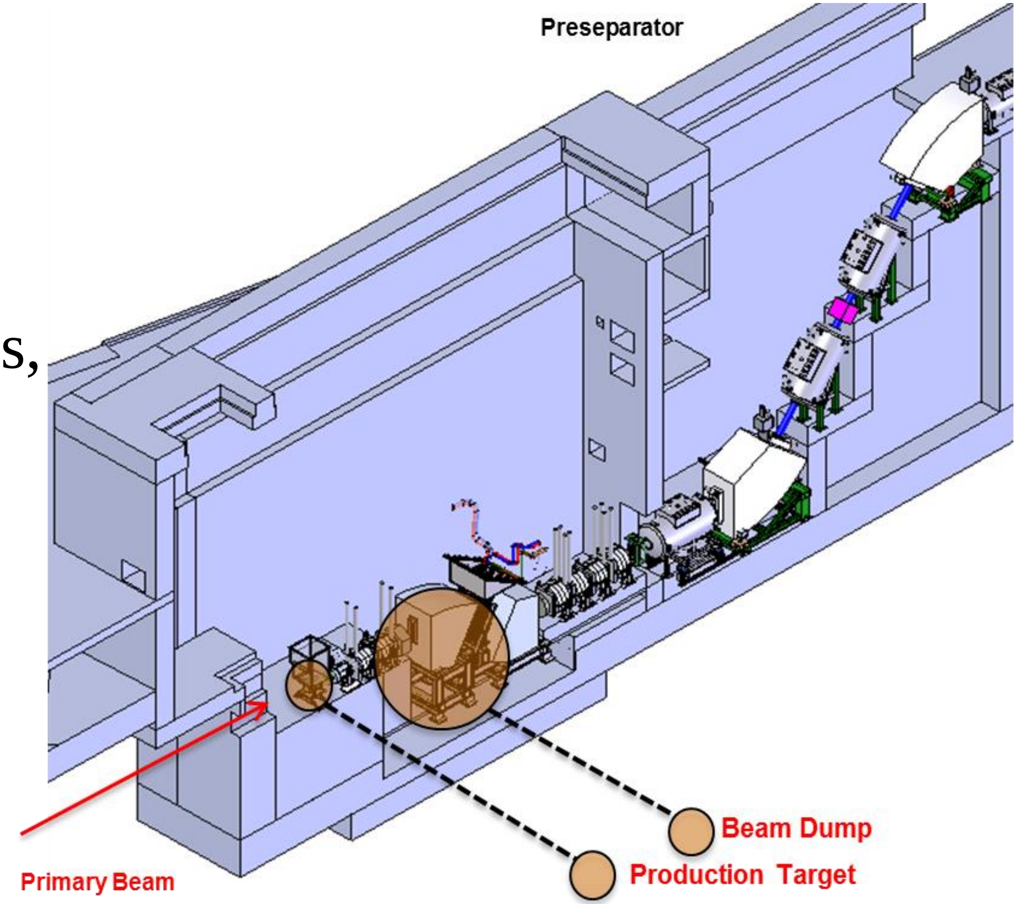
Outline

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**

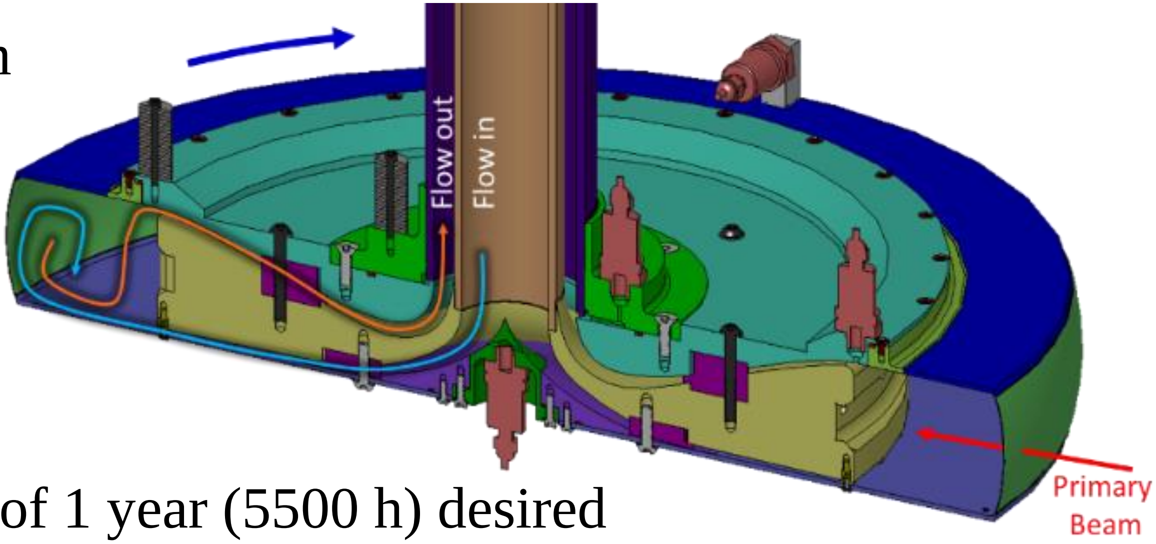
Facility for Rare Isotope Beams at Michigan State University

- 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)

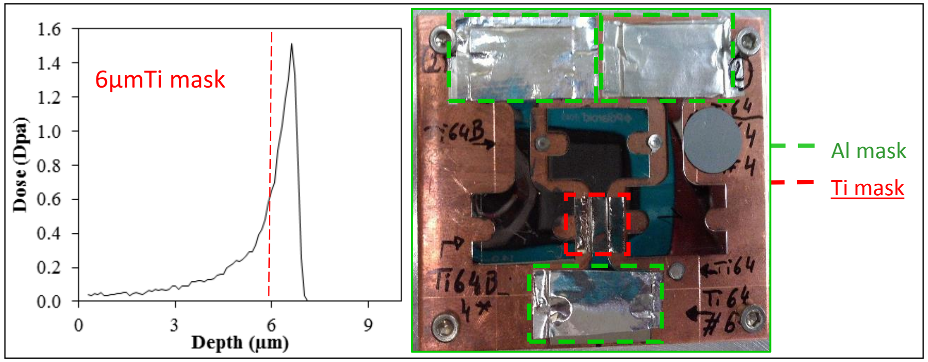
  - Ti-6Al-4V and Ti-6Al-4V-1B were chosen as candidate materials
  - The current study addresses the radiation damage challenge and focuses on understanding Swift Heavy Ion (SHI) effects on Ti-alloy that can limit beam dump lifetime

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 (°C)	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	2-7. 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}

Irradiation set up

Ti-alloys irradiations at CIMAP and NSCL

Facilities	Beam	Energy [MeV]	Range [µm]	S_e [keV/nm]	Fluence [ions/cm^2]	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

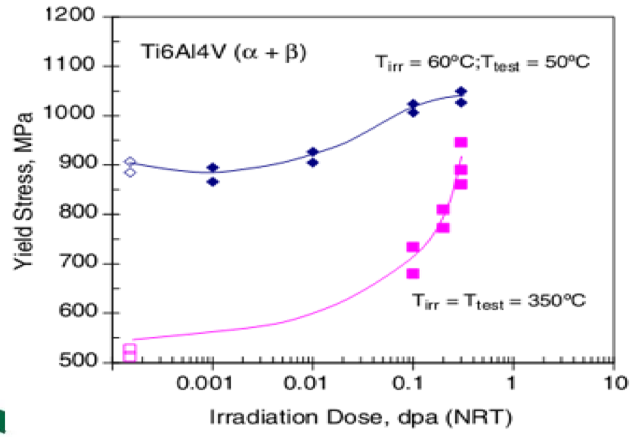
FRIB conditions

- Estimated cumulative dpa after one year of use ~9 dpa with a fluence of 10^{15} ions/cm^2
- 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)

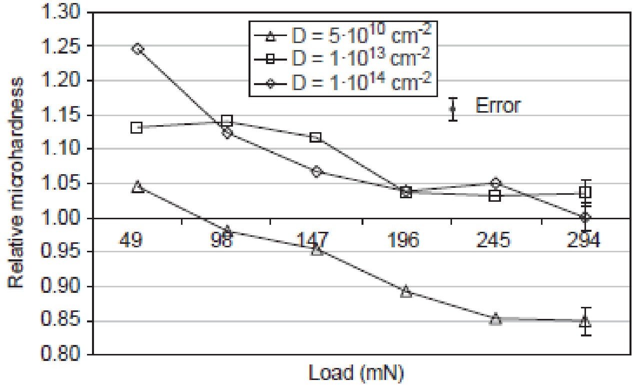
Temperature and dose level	Microstructure change observations
50°C , 0.3 dpa	A high concentration of uniformly distributed defect clusters in the α-phase
350° C, 0.3 dpa	Dislocation loops Vanadium precipitates
450°C, Dose 2.1 and 32 dpa	Dislocation loops β-phase precipitates in α phase
550°C 32 dpa	Extensive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



Different hardening mechanisms operate at 50°C than at 350°C.

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

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.



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

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.

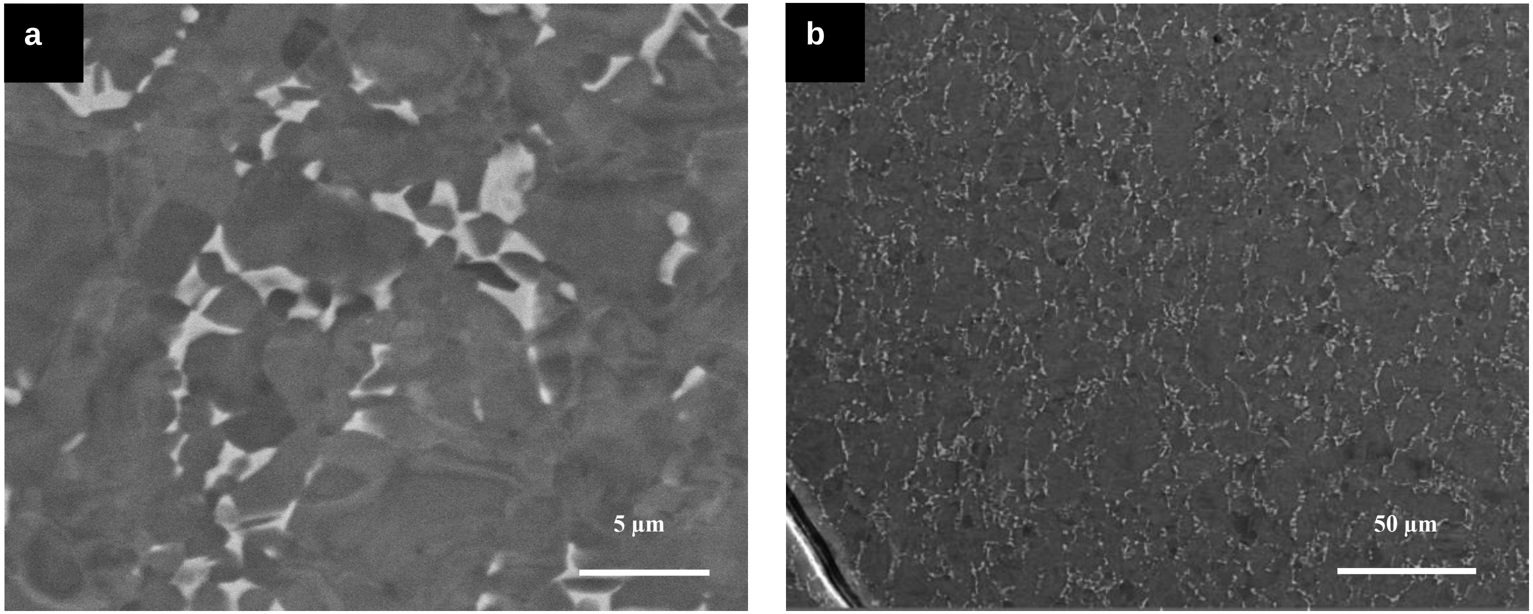
Microstructure of the as-received materials

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.



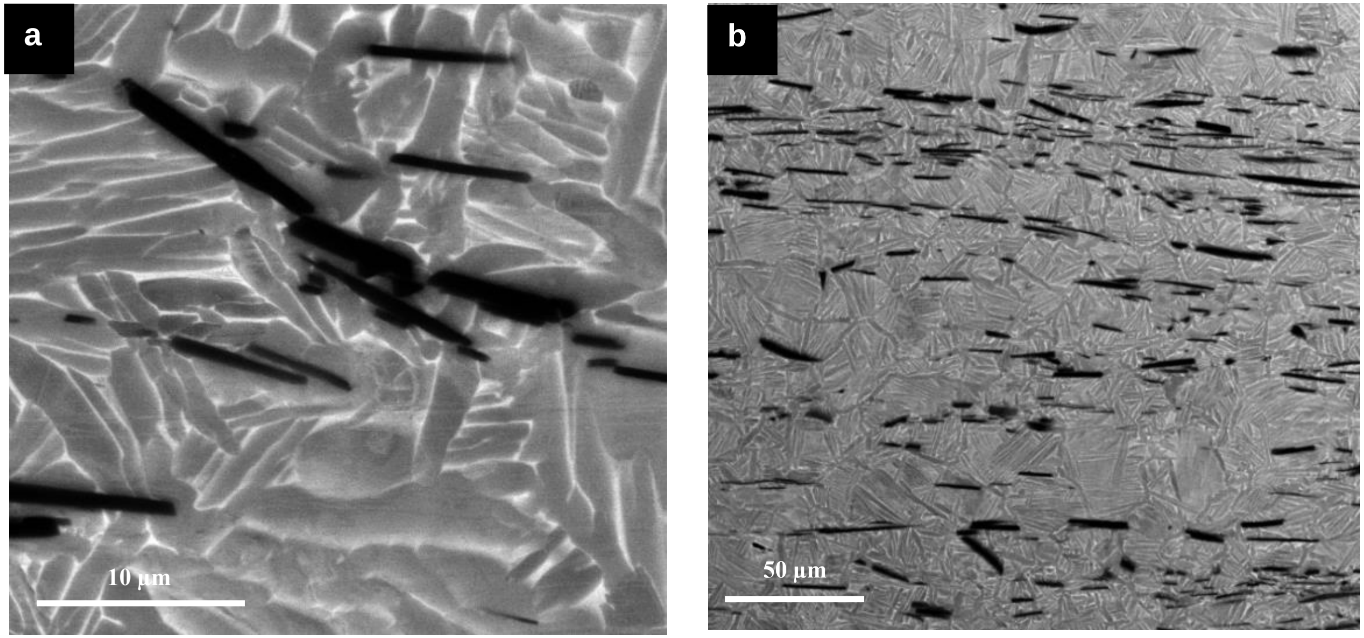
BSE images of the initial microstructure of Ti-6Al-4V (a) higher and (b) lower magnification

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%.

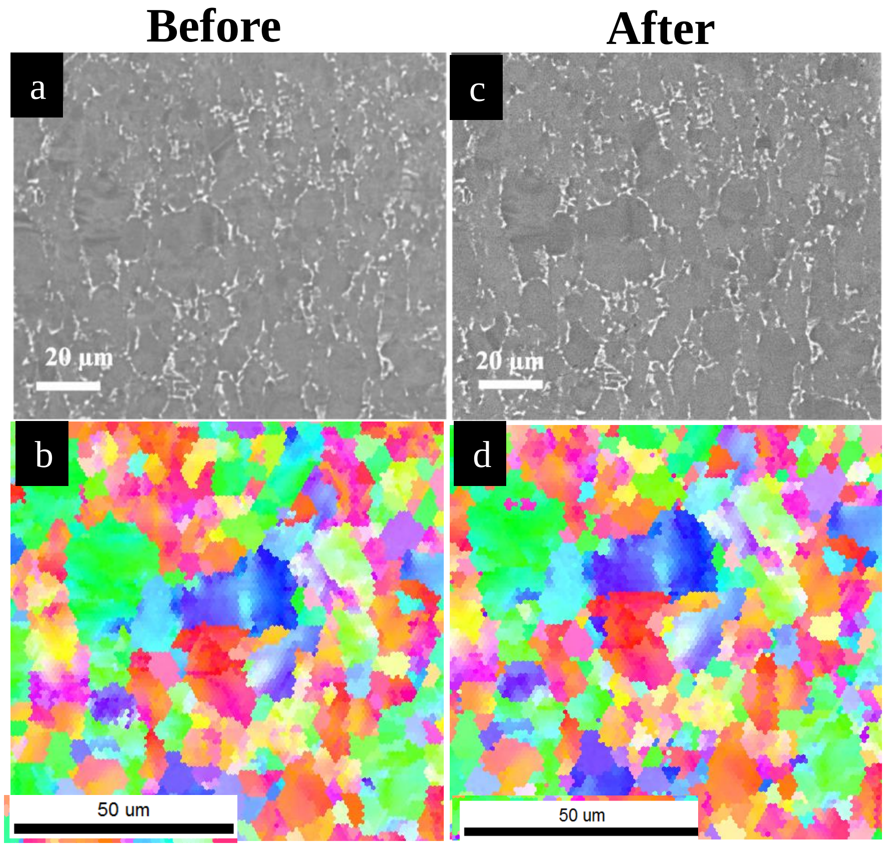
The β-phase volume percent was ~ 15 vol.% while the TiB phase volume percent was ~5.9 vol.%.(Chen *et al.)*



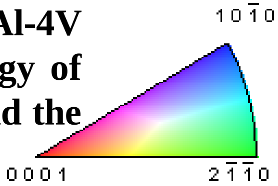
BSE images of the initial microstructure of Ti-6Al-4V-1B (a) higher and (b) lower magnification

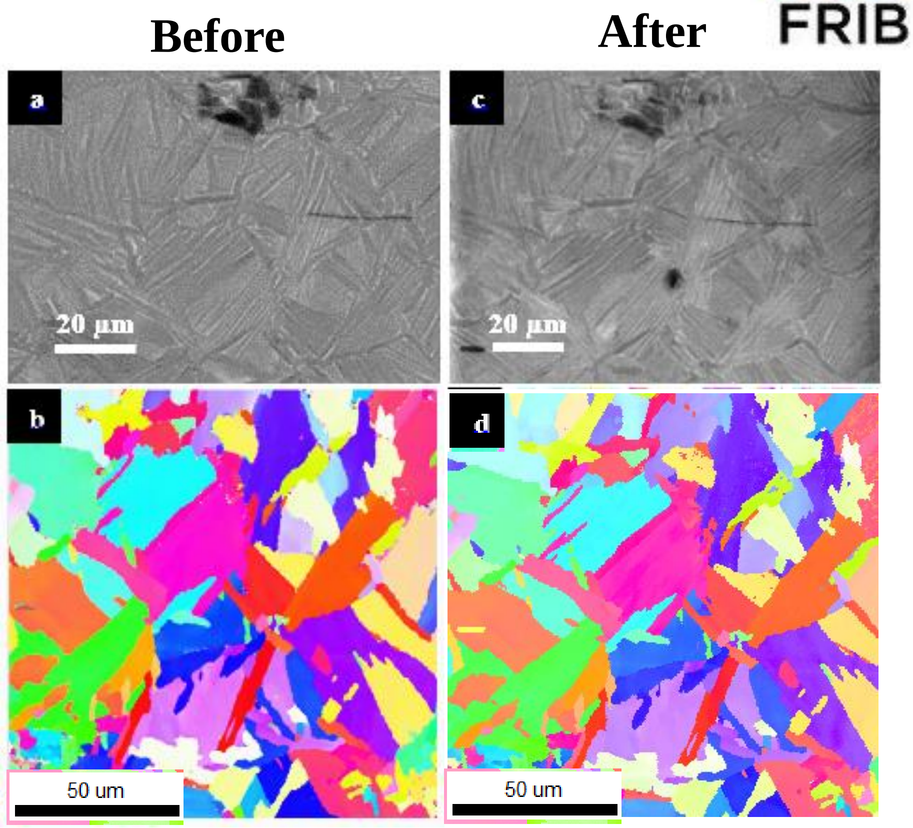
W. Chen et al / *Key Eng. Mater.*, vol. 436, pp. 195–203, May 2010.

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.**

Hardness measurements

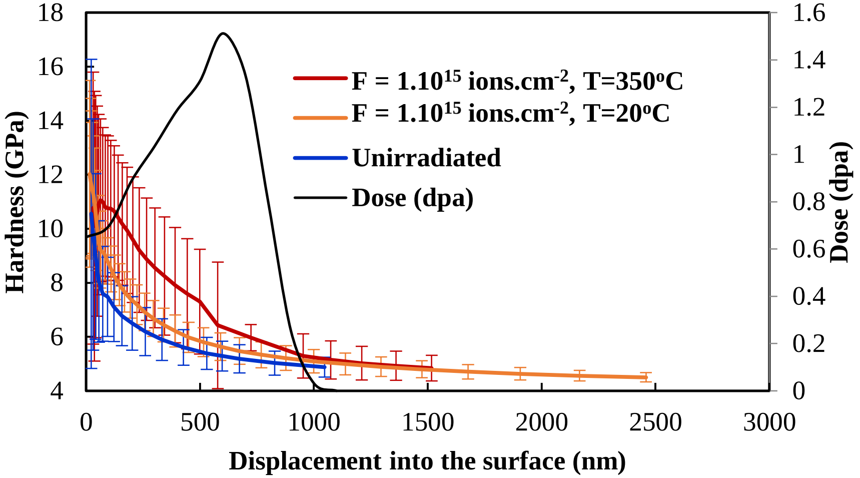
Nano-indentation

Obtain the properties of the materials in depth.

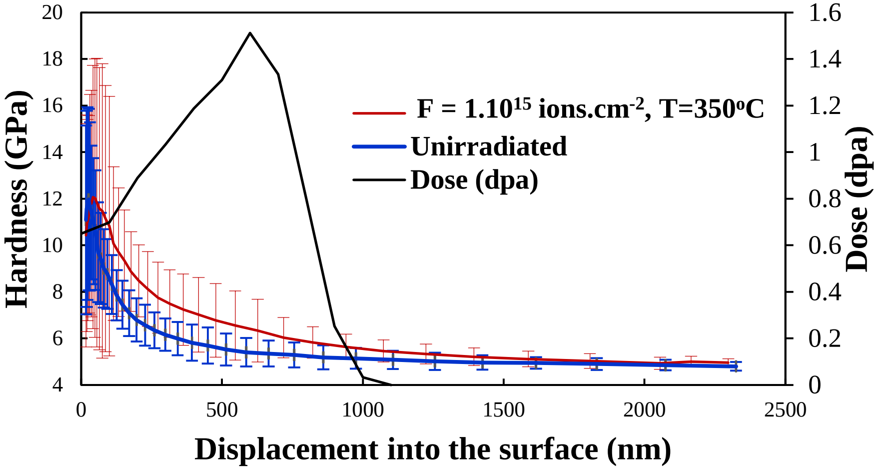
Parameters:

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

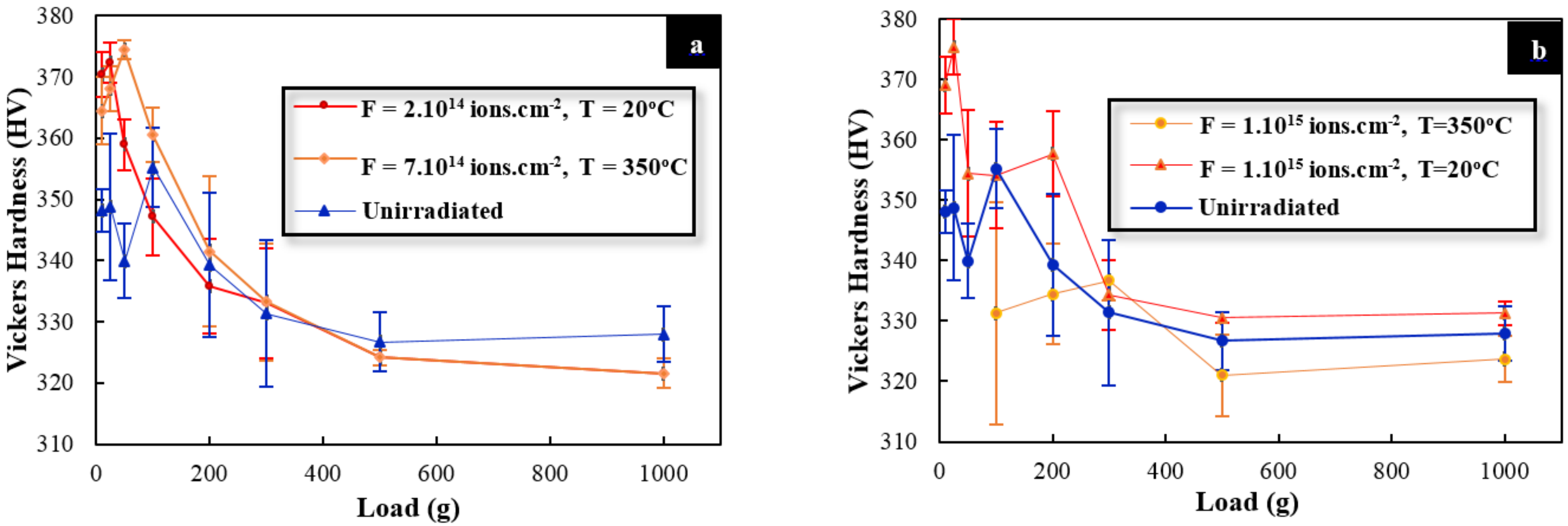
Boron addition to Ti-6Al-4V did not change its irradiation resistance

- ➢ 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

Hardness measurements

Vickers Hardness

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



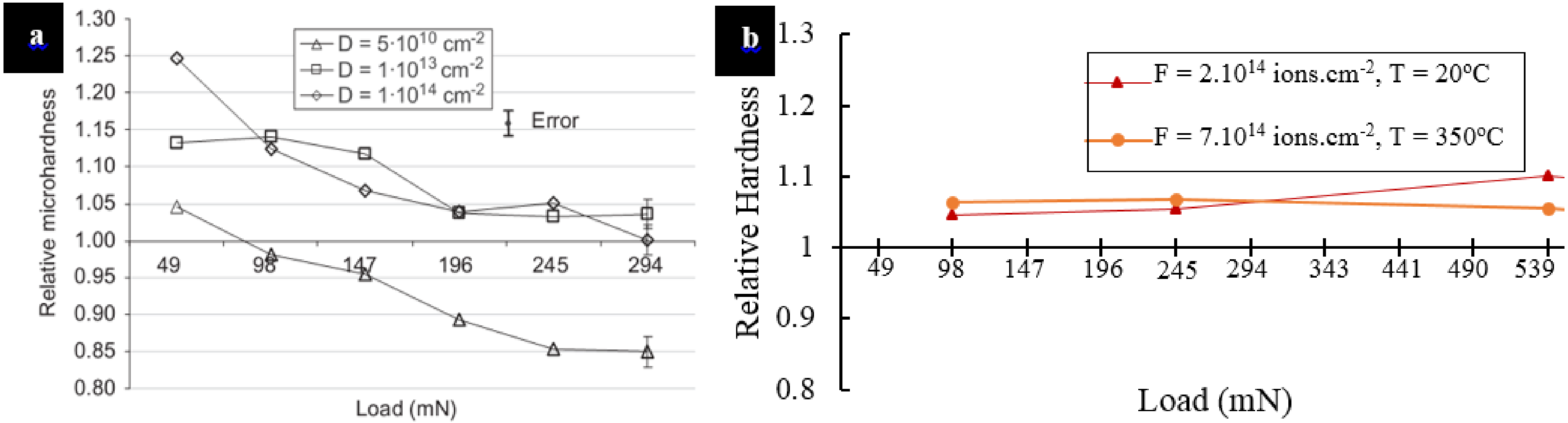
Vickers Hardness measurements for Ti-6Al-4V irradiated with: a) ^{131}Xe @ 92 MeV and b) ^{36}Ar @ 36 MeV

- 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)

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

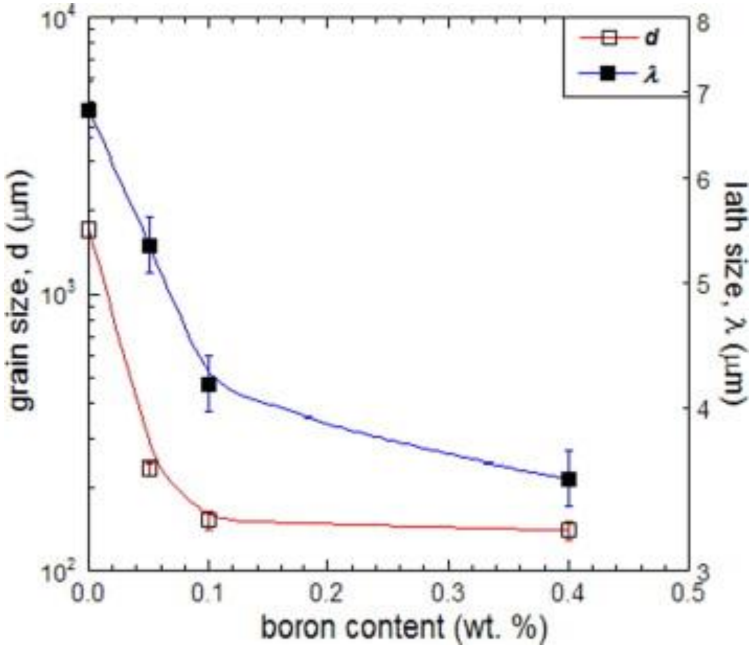


Relative micro-hardness of the Ti–6Al–4V alloy as a function of applied load for Ti-6Al-4V irradiated with: a) Kr^{+26}@350 MeV (Budzynski et al. 2009) and b) ^{131}Xe @ 92 MeV.

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.

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

Radiation Damage and Annealing in Graphite:
Ways to Improve the Lifetime of Targets

Frederique Pellemoine

Wolfgang Mittig

This material is based upon work supported by the U.S. Department of Energy Office of Science under Cooperative Agreement DE-SC0000661, the State of Michigan and Michigan State University. Michigan State University designs and establishes FRIB as a DOE Office of Science National User Facility in support of the mission of the Office of Nuclear Physics.

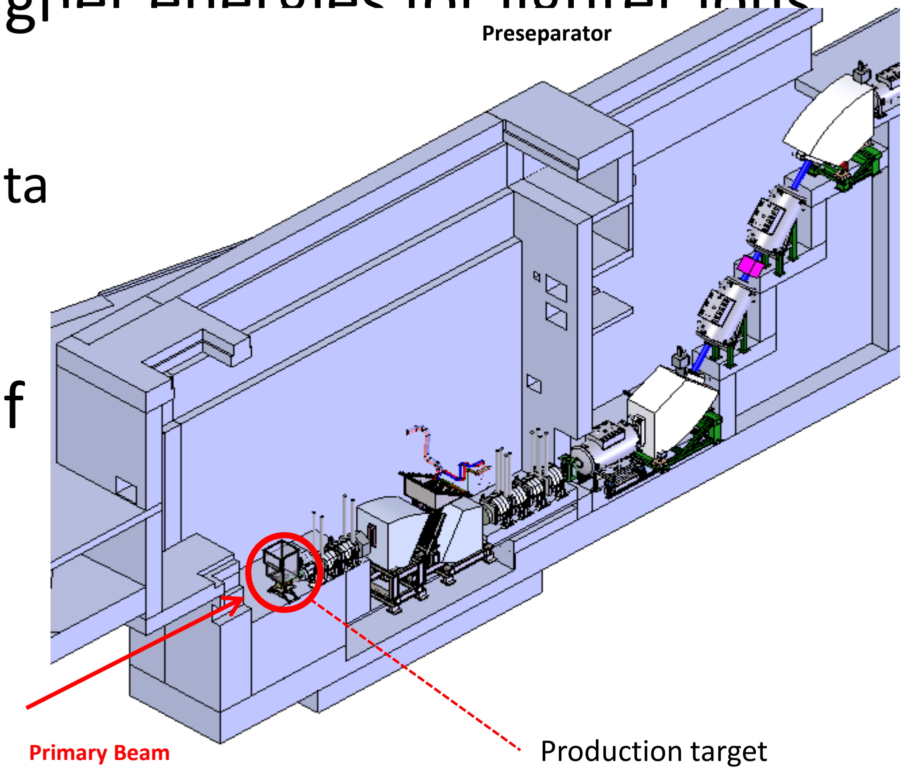
Outline

- FRIB High-power production targets
  - Design and challenges
  - Irradiation and annealing studies of graphite
    - Temperature effect
- NSCL-FRIB stripper
  - Challenges
  - Irradiation and annealing studies of graphite
    - Temperature effects
- Conclusions

High-Power Production Target

Scope and Technical Requirements

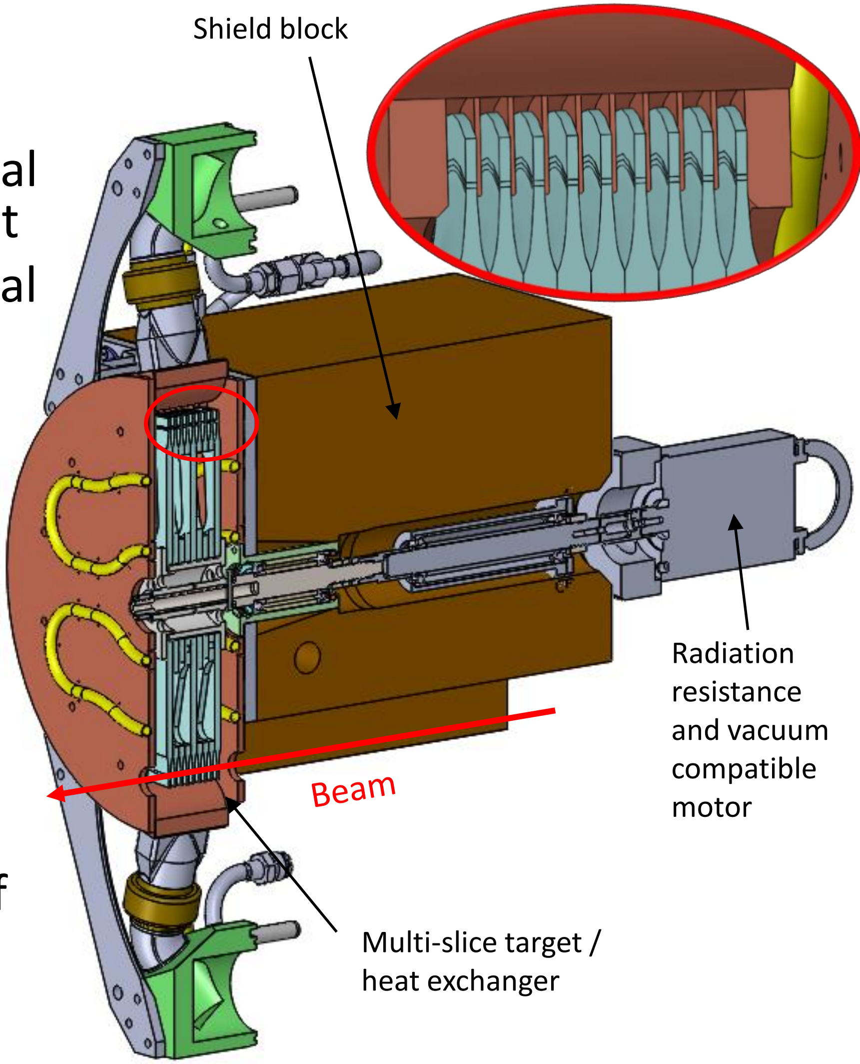
- In-flight rare isotope beam production with beam power of 400 kW at 200 MeV/u for ^{238}U and higher energies for lighter ions
- High power capability
  - Up to 100 kW in a ~ 0.3 - 8 g/cm² ta isotope production via projectile fragmentation and fission
- Required high resolving power of fragment separator
  - 1 mm diameter beam spot
  - Maximum extension of 50 mm in beam direction
- Target lifetime of 2 weeks to meet experimental program requirements



FRIB Production Target

Rotating Multi-slice Graphite Target Design

- Rotating multi-slice graphite target chosen for FRIB baseline
  - Increased radiating area and reduced total power per slice by using multi-slice target
  - Use graphite as high temperature material
  - Radiation cooling
- Design parameters
  - Optimum target thickness is ~ ⅓ of ion range
    - 0.15 mm to several mm
  - Maximum extension of 50 mm in beam direction including slice thickness and cooling fins to meet optics requirements
  - 5000 rpm and 30 cm diameter to limit maximum temperature and amplitude of temperature changes



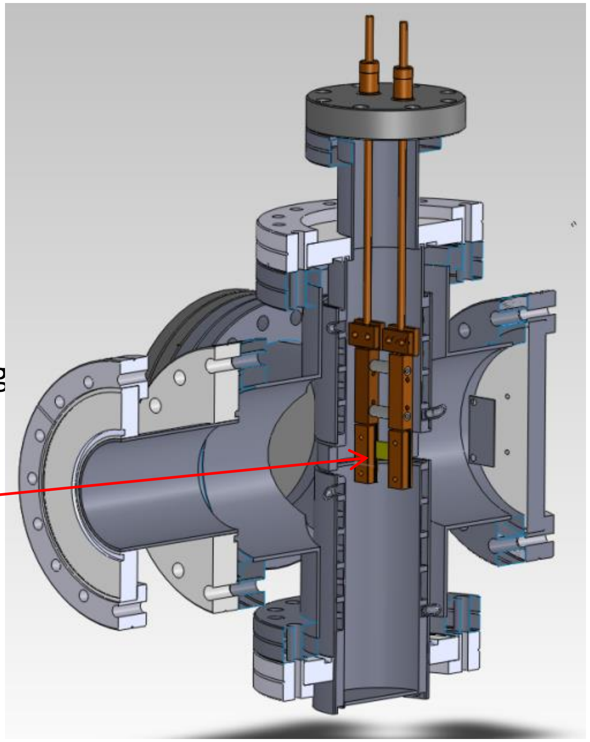
FRIB Production Target

Challenges

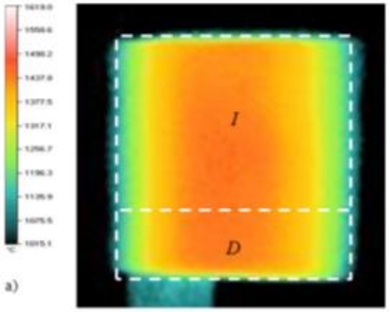
- Thermo-mechanical challenges
  - High power density: ~ 20 - 60 MW/cm³
    - High temperature: ~ 1900 ºC: Evaporation of graphite, stress
  - Rotating target
    - Temperature variation: Fatigue, Stress waves through target
- Swift Heavy Ion (SHI) effects on graphite
  - Radiation damage induce material changes
    - Property changes: thermal conductivity, tensile and flexural strength, electrical resistivity, microstructure and dimensional changes, …
  - Swift heavy ions (SHI) damage not well-known
  - $5 \cdot 10^{13}$ U ions/s at 203 MeV/u may limit target lifetime
    - Fluence of ~$9.4 \cdot 10^{18}$ ions/cm² and 10 dpa estimated for 2 weeks of operation
- Similar challenges at
  - Facility for Antiproton and Ion Research (FAIR) at GSI
  - Radioactive Ion Beam Factory (RIBF) at RIKEN

Irradiation Test at UNILAC at GSI/Darmstadt

- Polycrystalline isotropic graphite
    - 2 Grades MERSEN 2360 (5 µm) / 2320 (13 µm)
- Irradiation test at UNILAC at GSI/Darmstadt
    - Au-beam 8.6 MeV/u
        - Up to $5.6 \cdot 10^{10}$ ions/cm²·s and fluence up to 10^{15} ions/cm²
            - Equivalent to a fluence of 10^{18} ions/cm² for FRIB beam energ or 2 days of operation
        - Electronic energy loss ≈ 20 keV/nm
    - Ohmic heating (up to 35 A, 250 W) of samples to different temperature during irradiation

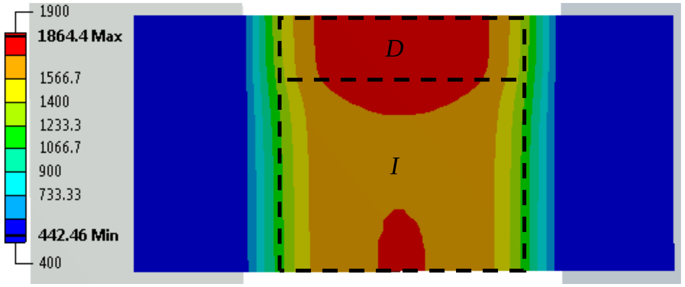


I = 35 A

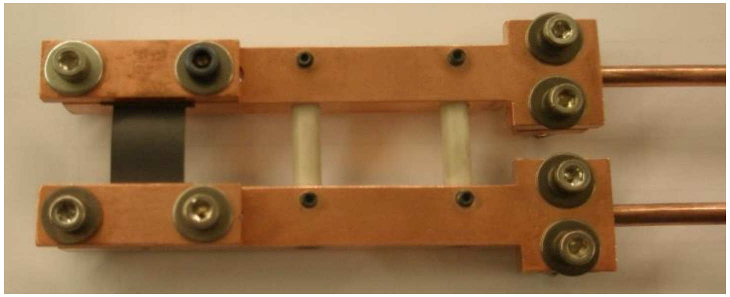


T_{max} = 1480 (± 30 ºC)

I = 35 A + beam



T_{max} = 1635ºC



Radiation Damage Studies in Graphite [1]

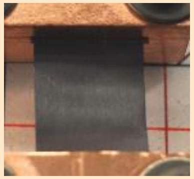
Annealing of Damage at High Temperature (> 1300ºC)

1 A - 350°C
10^{14} cm^{-2}

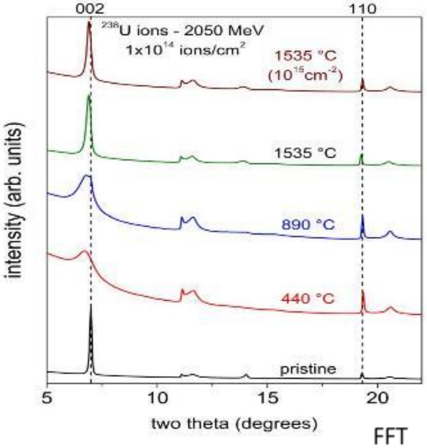
11 A - 750°C
10^{14} cm^{-2}

25 A - 1205°C
10^{14} cm^{-2}

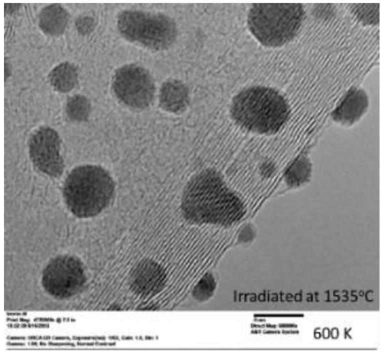
35 A - 1635°C
10^{15} cm^{-2}



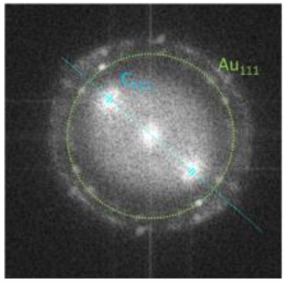
X-Ray Diffraction analyses

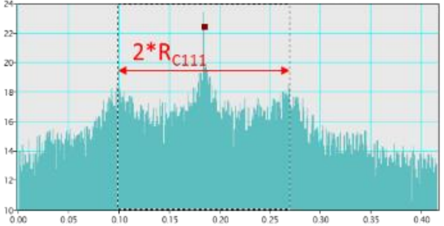


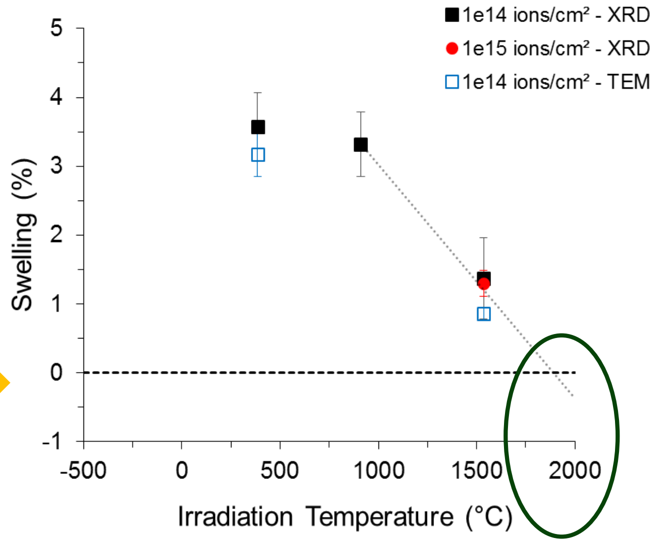
TEM analyses



FFT





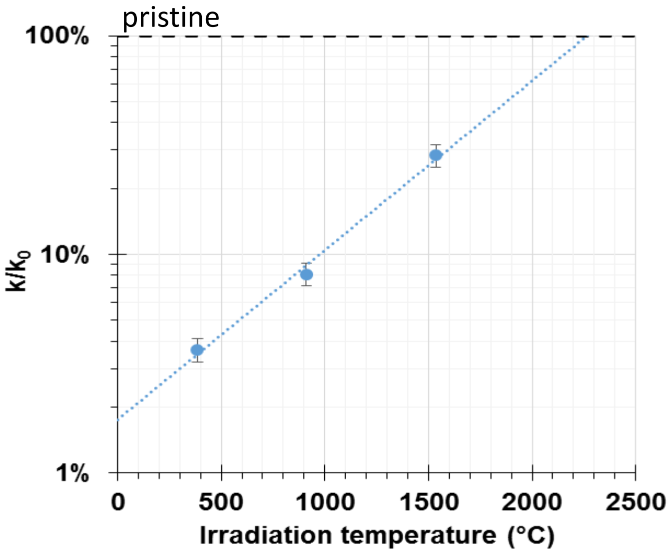


Swelling is completely recovered at 1900ºC

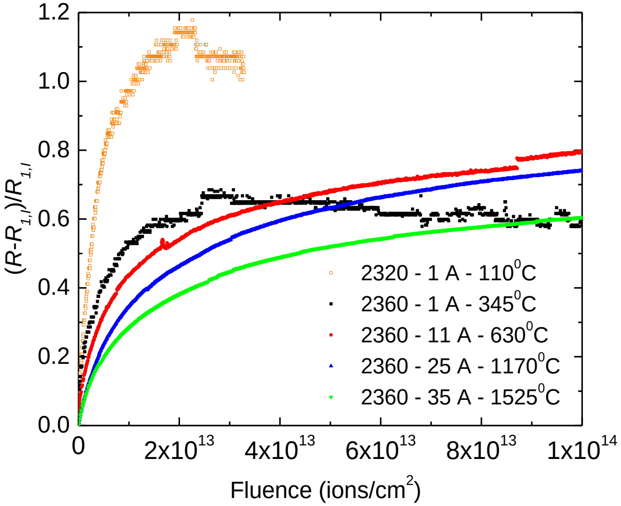
Radiation Damage Studies in Graphite [2]

Annealing of Damage at High Temperature (> 1300ºC)

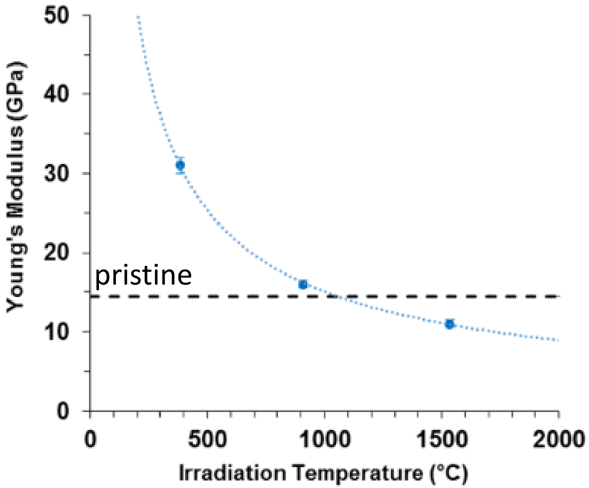
Thermal conductivity change of irradiated graphite samples - ^{197}Au fluence 10^{14} ions/cm^2



Electrical resistance change of irradiated graphite samples - ^{197}Au



Young's Modulus of irradiated graphite samples - ^{197}Au fluence 10^{14} ions/cm^2

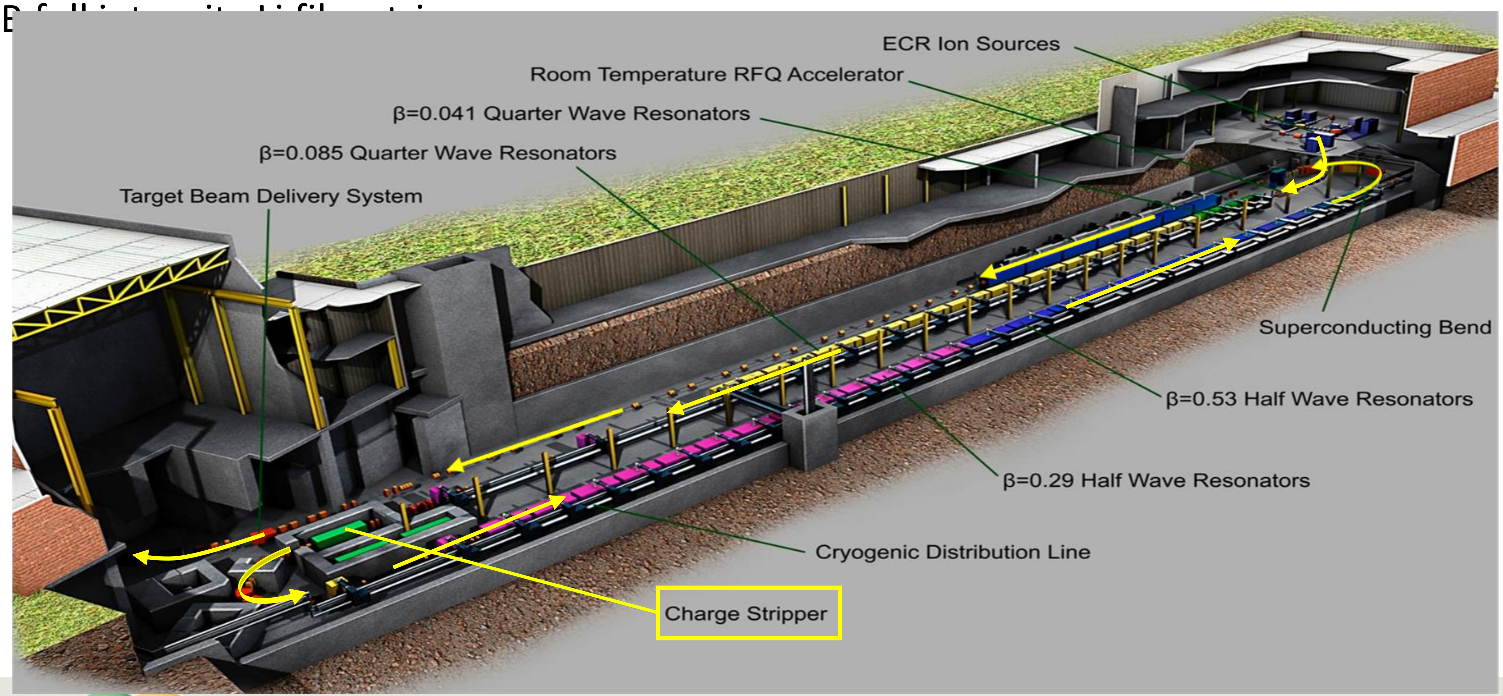


- Annealing at high temperature confirmed

NSCL-FRIB Strippers

Challenges

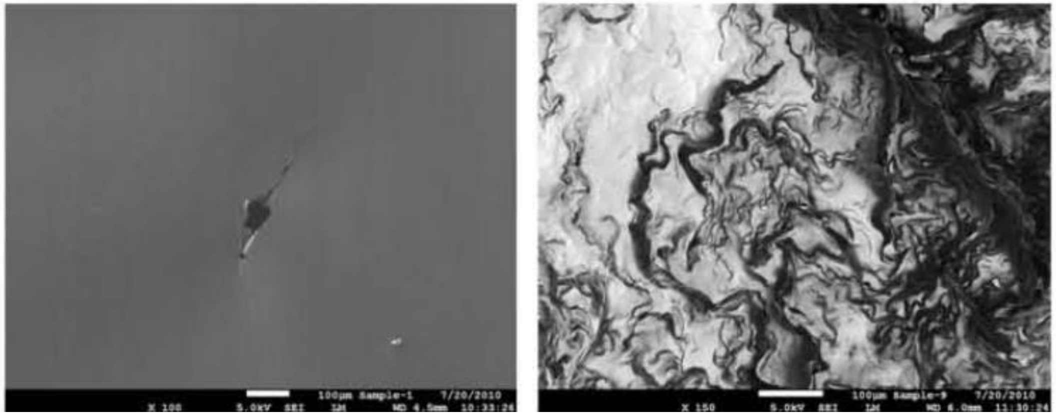
- It is known that thin foils (stripper) used in accelerator suffer a quick degradation due to radiation damage such as swelling and thermo-mechanical changes
  - Limits the lifetime of few hours
- How can we improve the lifetime?
  - Annealing at high temperature
  - Influence of nano-structure on annealing
  - FRIB
  - ….



NSCL-FRIB Strippers

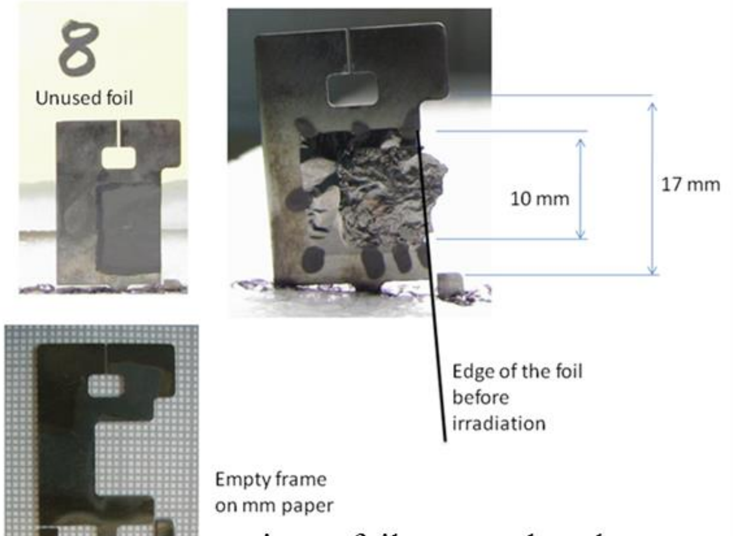
Radiation Damage

- Recent tests at NSCL have shown quick deterioration of graphite foils under heavy ion bombardment due to thermal and mechanical stresses and radiation damage
  - Carbon irradiated with Pb beam @ 8



SEM photographs of unused carbon foil (left) showing a small pinhole for illustration and a foil exposed to 8.1 MeV/u Pb beam

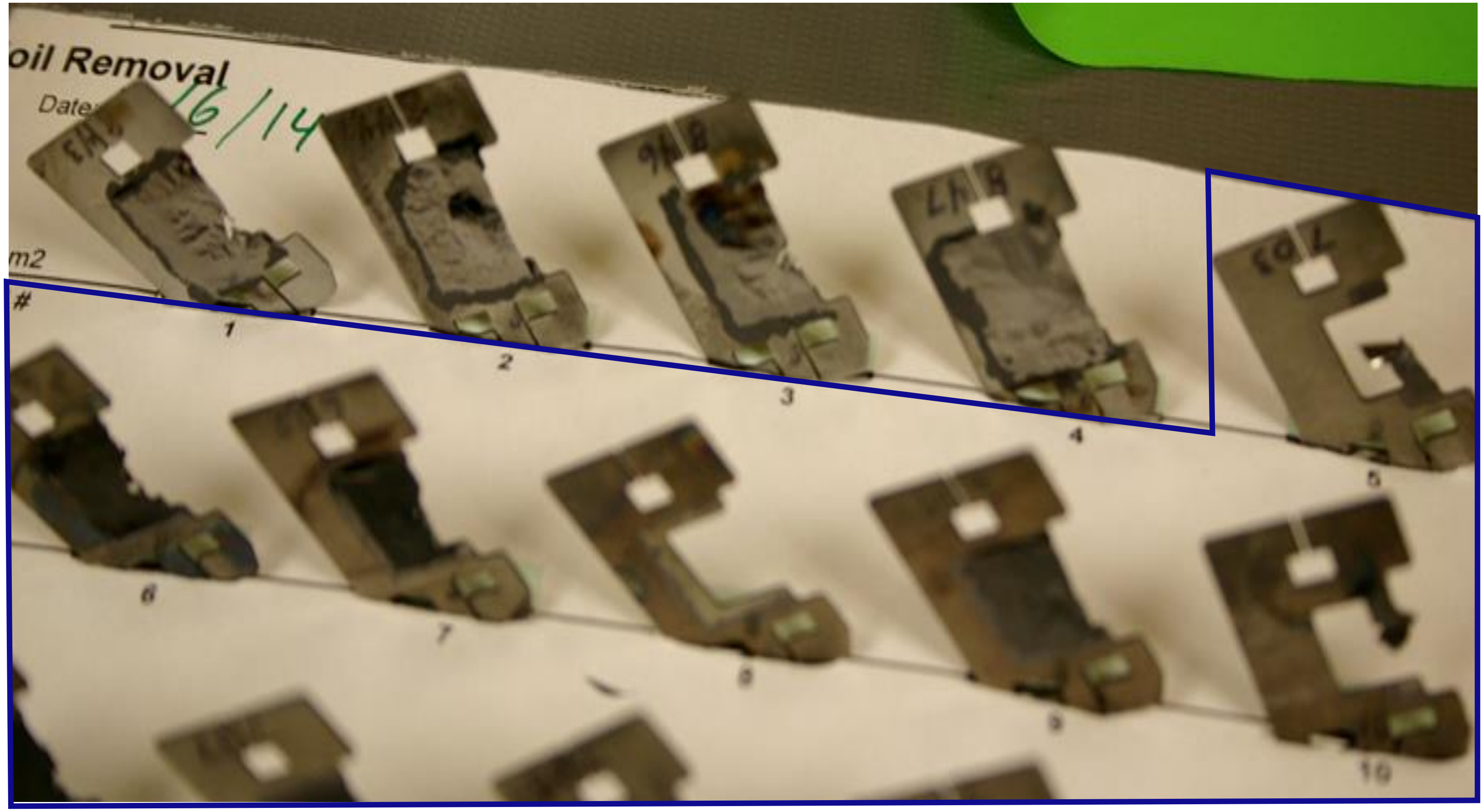
F. Marti et al., "A carbon foil stripper for FRIB", TUP 106, Proceedings of Linear Accelerator Conference LINAC2010, Tsukuba, Japan, TUP105, 2010.



stripper foils as used at the NSCL CCF. Courtesy of F.Marti

NSCL-FRIB Strippers

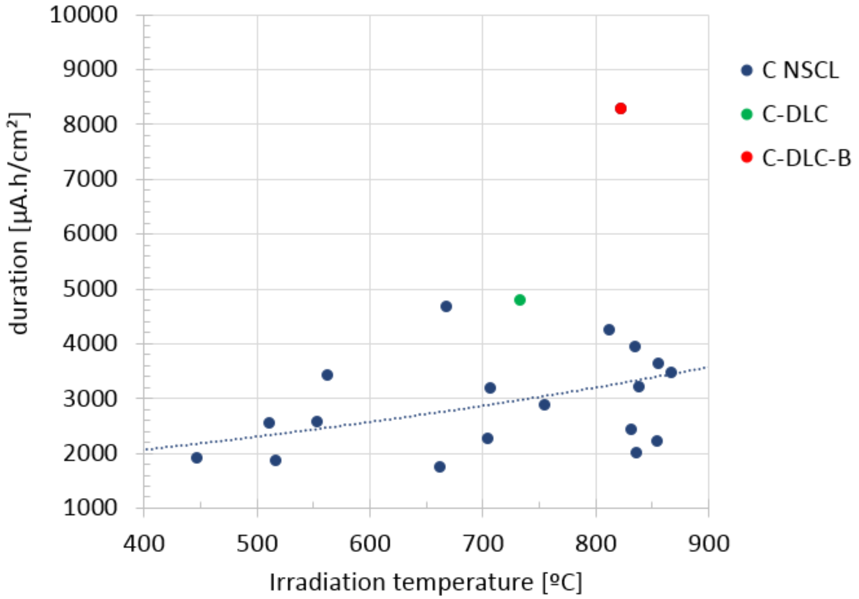
Irradiated Strippers at NSCL



Current carbon strippers used at NSCL

Improvement of the lifetime

- Previous studies [3] showed annealing effects of radiation damage at high temperature. The heating by the beam was evaluated to produce temperatures of up ~900 ºC. A clear tendency of increased lifetime with irradiation temperature was observed.
- The lifetime of the 10 multilayer foil C-DLC-B was significantly higher (factor 3) than the standard C-NSCL foils. The 10 multilayer foil C-DLC was somewhat superior (about a factor 2) as compared to the standard foils.



Lifetime time (µA·h/cm²) as a function of the irradiation temperature and the microstructure of graphite stripper foils.

[3] S. Fernandes et al., "In-Situ Electric Resistance Measurements and Annealing Effects of Graphite Exposed to Swift Heavy Ions", Nucl. Instrum. Methods Phys. Res. B 314 (2013) 125-129.

Summary and Conclusions

- Heavy-ion irradiation tests and annealing studies performed in the context of high-power target and strippers for high intensity accelerator were performed
- High temperature annealing of heavy-ion induced radiation damage observed in production target
  - First experiment of this kind
  - Confirmed by several analysis
- Graphite as a material for FRIB beam production targets promises sufficient lifetime
- High temperature annealing of heavy-ion induced radiation damage observed in NSCL strippers

Acknowledgements

- FRIB/NSCL Michigan State University
  - Mikhail Avilov, Saul Beceiro Novo, Sandrina Fernandes, Felix Marti, Mike Schein, Andreas Stolz, Genevieve West
- GSI Helmholtzzentrum für Schwerionenforschung GmbH
  - Markus Bender, Markus Krause, Daniel Severin, Marilena Tomut, Christina Trautmann
- University of Michigan
  - Rodney Ewing, Maik Lang, Weixing Li

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Thank you for your attention

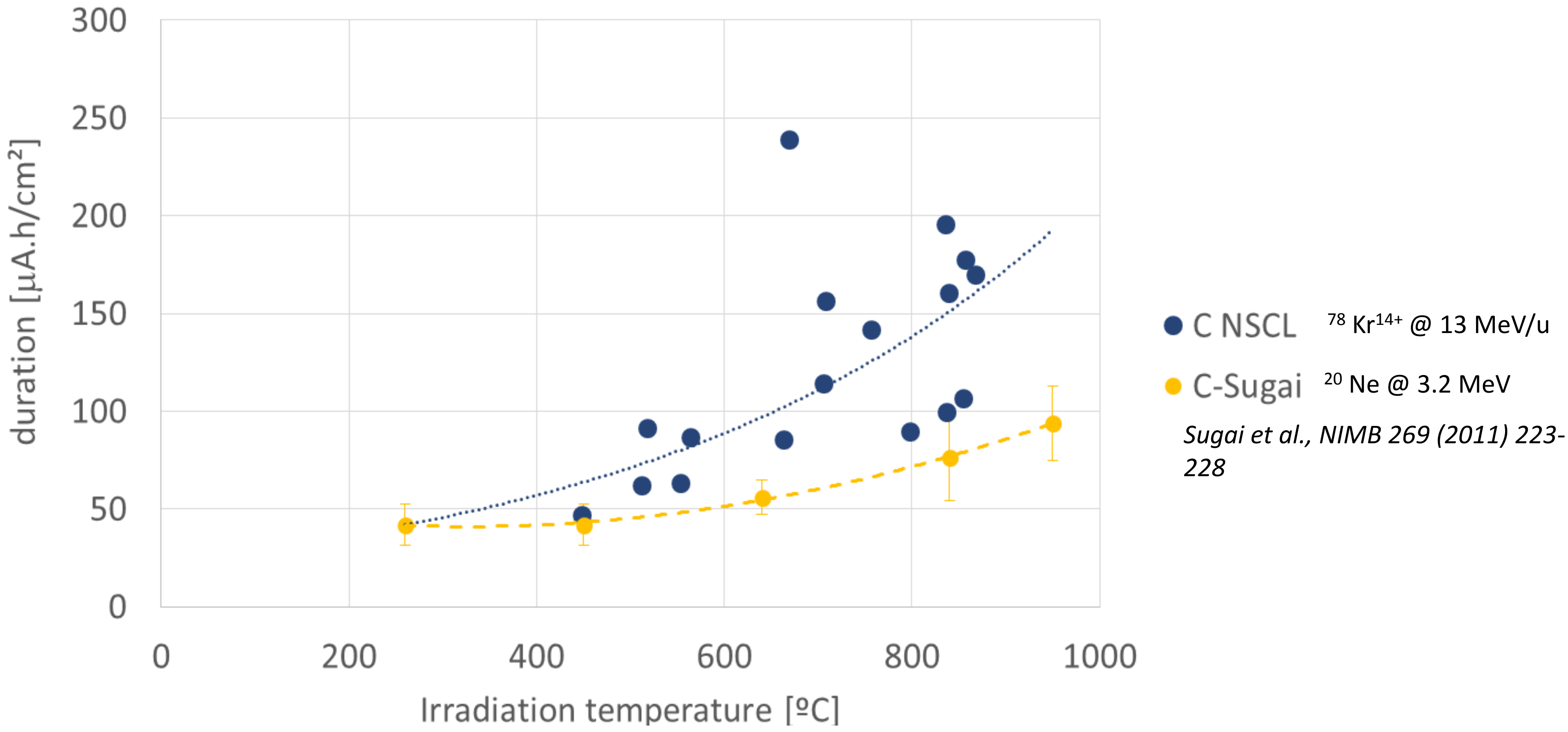
FRIB construction area – October 27 2014

NSCL-FRIB Strippers

Lifetime measurement at NSCL

- Effect of the temperature on lifetime improvement observed at NSCL

Preliminary results



Sugai et al., NIMB 269 (2011) 223-228