



# Material studies and analysis in support of FRIB beam dump construction

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# Outline

- FRIB beam dump context and challenges
- Thermo-mechanical material studies
  - Simulations in support of the design
  - (Prototyping and electron beam test)
- Radiation damage studies
  - Low energy heavy ion irradiation
  - Intermediate energy heavy ion irradiation
  - High energy study
  - Material microstructure study
    - » In-situ TEM study in different microstructure
    - » Low energy heavy ion irradiation
- Conclusion

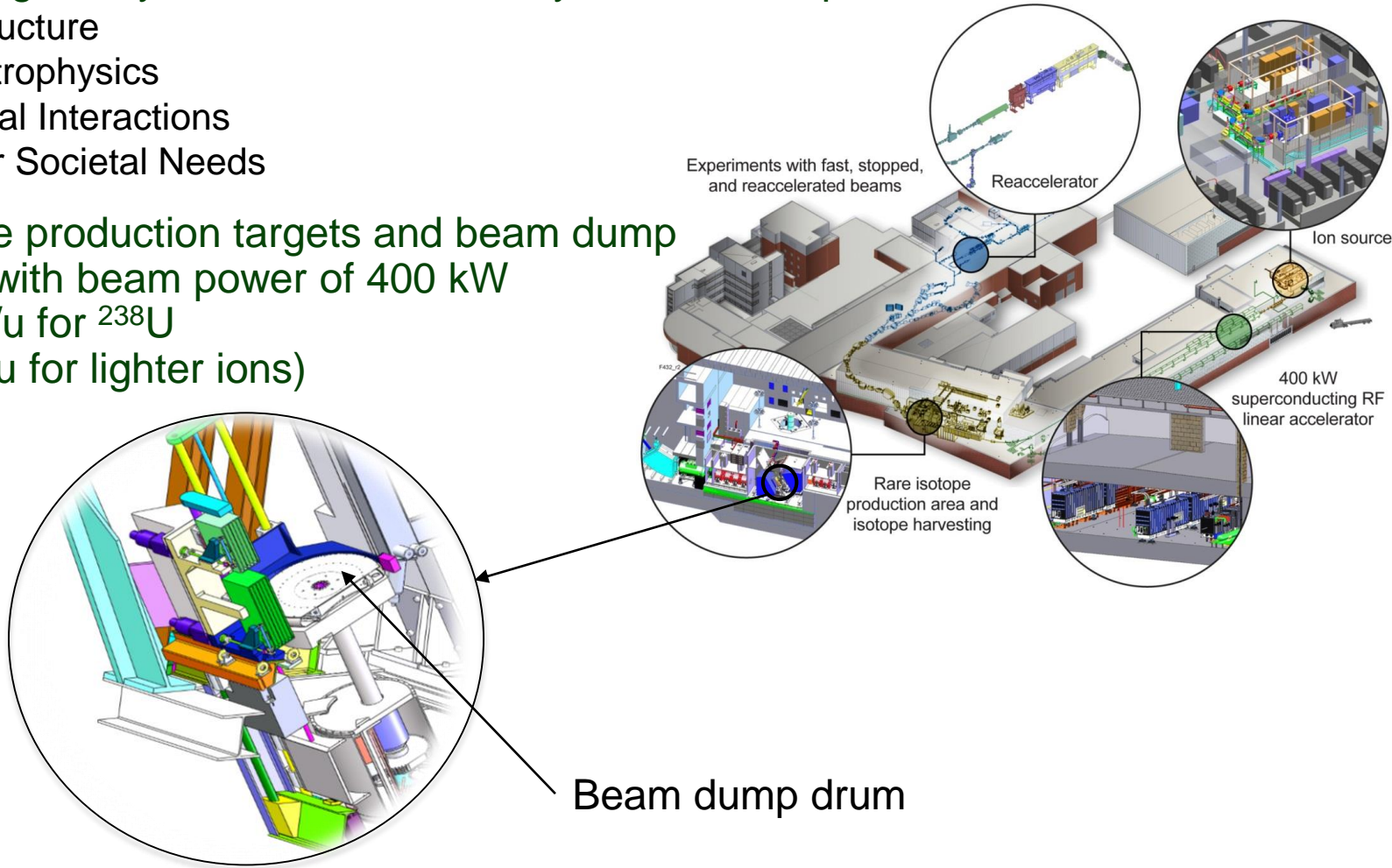
W. Mittig

# Facility for Rare Isotope Beams

- World-leading heavy ion accelerator facility for rare isotope science

- Nuclear Structure
- Nuclear Astrophysics
- Fundamental Interactions
- Isotopes for Societal Needs

- Rare isotope production targets and beam dump compatible with beam power of 400 kW at 200 MeV/u for  $^{238}\text{U}$  (>200 MeV/u for lighter ions)



# Primary Beam Dump

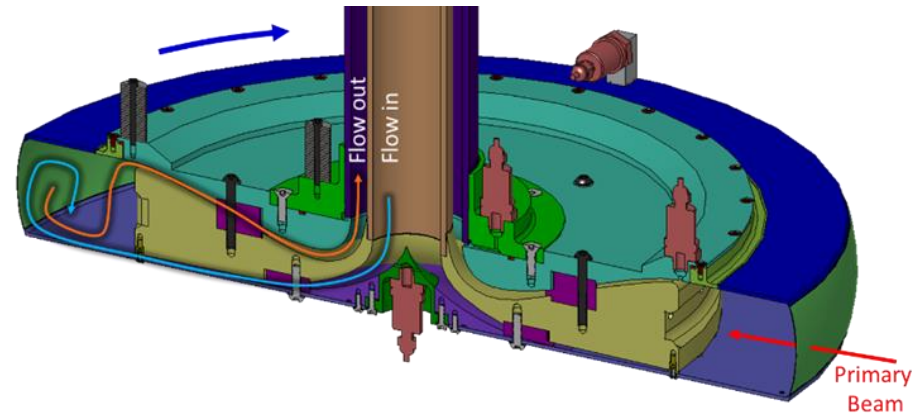
## Water-filled Rotating Drum Concept

- Beam Dump requirements
  - High power capability up to 325 kW
  - 1 year (5500 h) lifetime desirable
    - » fluence  $\sim 10^{18}$  ion/cm<sup>2</sup>
    - » dpa (U beam)  $\sim 7$  (dpa/rate  $\sim 4 \cdot 10^{-7}$  dpa/s)
  - Remote replacement and maintenance

- Water-filled rotating drum concept chosen for FRIB baseline
  - Using water to stop the primary beam and absorb beam power

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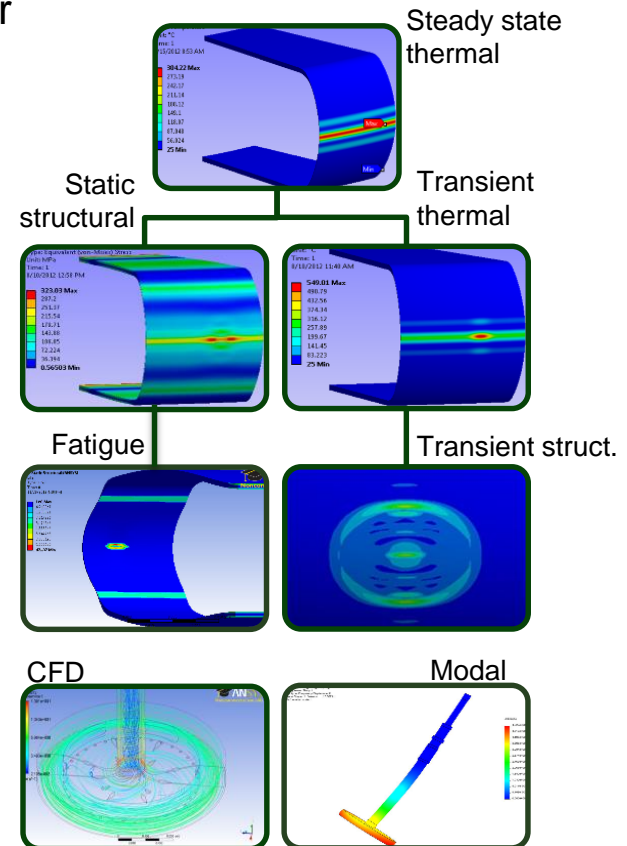
- Design parameters
  - Ti-alloy shell thickness 0.5 mm to minimize power deposition in shell
  - 600 rpm and 70 cm diameter to limit maximum temperature and amplitude of temperature changes
  - 60 gpm water flow to provide cooling and gas bubble removal
  - 8 bar pressure inside the drum increases water boiling point to 150°C
- Ti-6Al-4V was chosen as candidate material for the beam dump shell



# Challenges Overview

- **Extreme conditions due to heavy ion beams**
  - Energy loss of U beam at 156 MeV/u in Ti-alloy shell is 4 order of magnitude higher compare to proton beam at 1 GeV
- **Challenges addressed in simulation**
  - High power – up to 60 kW in the shell
    - » Thermal stress
    - » Water near the boiling point limits max. temperature of the shell
    - » Sufficient wall heat transfer required
  - Rotating drum: 600 rpm
    - » Temperature variation
      - Fatigue, Stress wave through the drum shell
    - » Elevated mechanical stress due to internal pressure
    - » Vibration and mechanical resonances
- **Water**
  - Corrosion, Cavitation
- **Swift heavy ions**
  - Radiation damage in material
  - Sputtering
  - Radiolysis (gas production)

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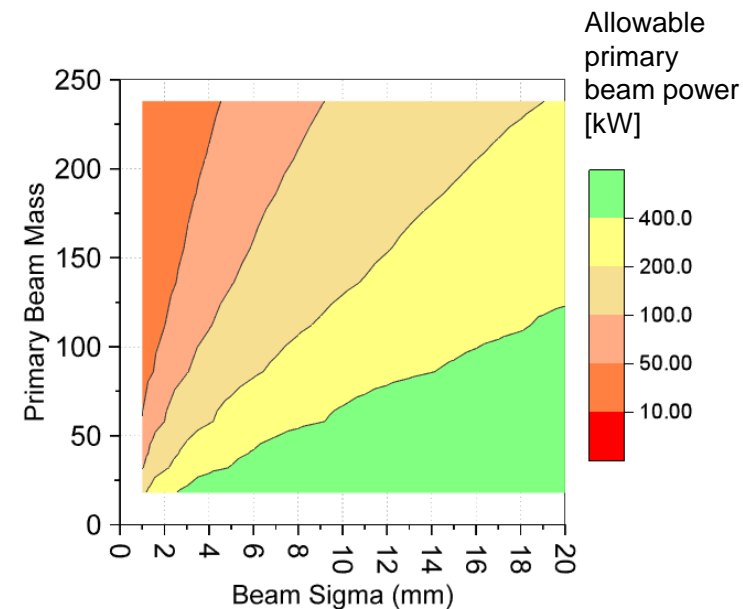
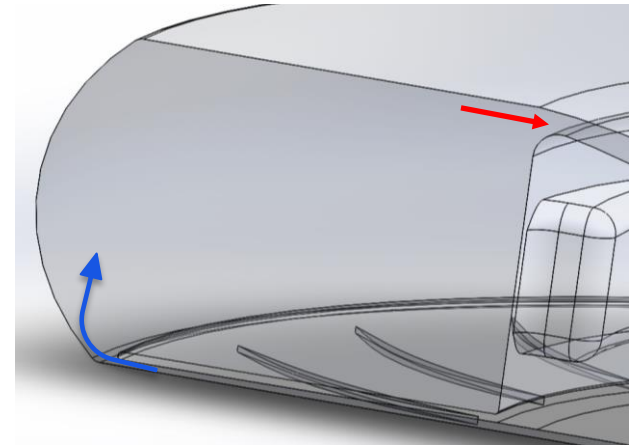
# Beam Dump for First Years of Operation

## Robust Solution (Single Shell Drum)

- Go forward with robust single shell beam dump
  - Single shell geometry with single-phase fluid flow
  - Maximum operation temperature limited by water boiling point
  - Full power (325 kW) for light beams, up to 100 kW for the heaviest  $^{238}\text{U}$  beam
  - Maximum tolerable heat flux of  $0.35 \text{ MW/m}^2$  based on conservative assumptions
  - Get operational experience during first years, assess heat removal from drum shell, learn about material behavior under heavy-ion irradiation at FRIB beam conditions, corrosion effects

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- Continue to perform studies on heat removal and material behavior



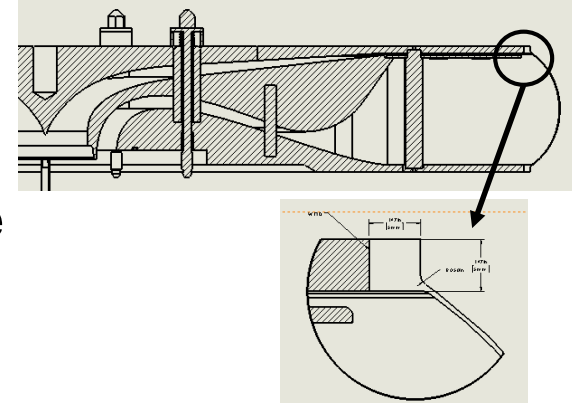


# Simulation to support the design

## Single Shell Drum Design Optimized

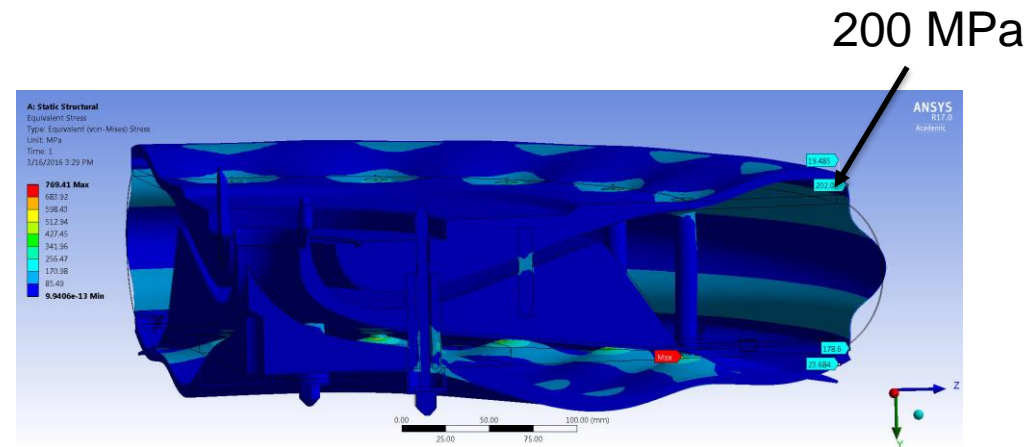
### ■ Mechanical stress in the shell due to water pressure minimized

- Increased thickness of the top end plate at the channel area to reduce the stress and deformation
- Shell profile optimized to withstand high stress level
- Minimum safety factor of  $\sim 4$  at 8 bar water pressure near the shell



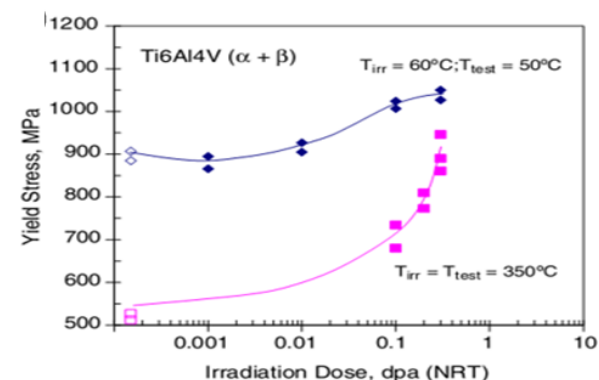
### ■ Thermo-mechanical stress

- Significantly lower than that induced by pressure ( $\sim 80$  MPa)
- Minimum safety factor of 3 for 8 bar pressure when beam close to the top and bottom plate



# Radiation Damage in Material

- Irradiation induces material changes that could decrease the Beam Dump performance
  - Thermo-mechanical properties (thermal conductivity, hardening, embrittlement), Electronic properties (resistivity), Structural properties (microstructure and dimensional changes), Sputtering
  - Combined effects
    - » Thermal and radiation enhanced creep, reduction of creep life
    - » Corrosion combined with radiolysis
    - » Gas production enhanced embrittlement
- Data exists for neutron and proton irradiation but no data were found for heavy ion irradiation in Ti-alloys
  - Heavy ion induced radiation damage depends on two parameters
    - » Sn: nuclear energy loss (ballistic effect)
    - » Se: electronic energy loss (phase transformation, track formation and structural modification such as amorphization)
- No heavy ion beam facility exists that allows us to test all extreme conditions combined together
- Perform studies that combine some material challenges using existing facilities
  - Electron beams, neutron beams, Swift Heavy Ion (SHI) beams
  - Radiation damage, corrosion, creep



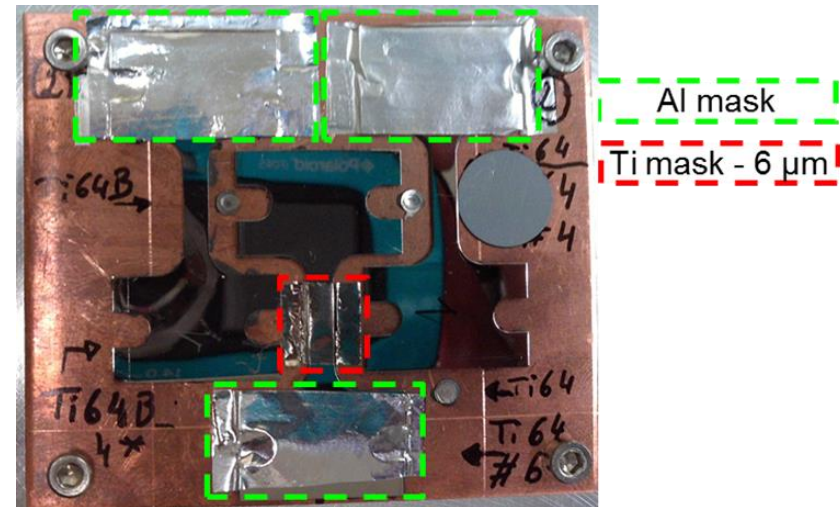
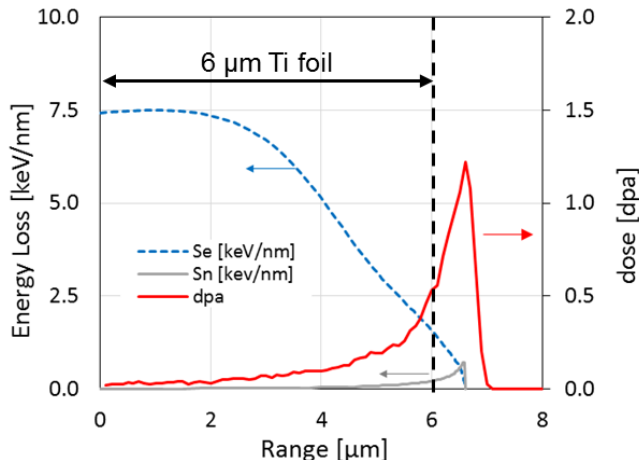
Dose dependence of yield strength of Ti-6Al-4V irradiated with neutrons [1]



# Material Study with Heavy Ion Irradiation at Low Energy [1]

- Samples irradiated at IRRSUD (GANIL-CIMAP) up to a fluence of  $10^{15}$  ions/cm<sup>2</sup> with
  - <sup>36</sup>Ar beam @ 36 MeV
    - » Se = 7.5 keV/nm – Sn = 0.015 keV/nm
  - <sup>131</sup>Xe beam @ 92 MeV
    - » Se = 20 keV/nm – Sn = 0.15 keV/nm
- FRIB conditions
  - Se from 0.08 keV/nm (with O beam) to 12.6 keV/nm (with U beam)
  - Sn from  $2.5 \cdot 10^{-5}$  keV/nm (with O beam) to  $4 \cdot 10^{-3}$  keV/nm (with U beam)
  - ~7 dpa after one year of operation with U beam

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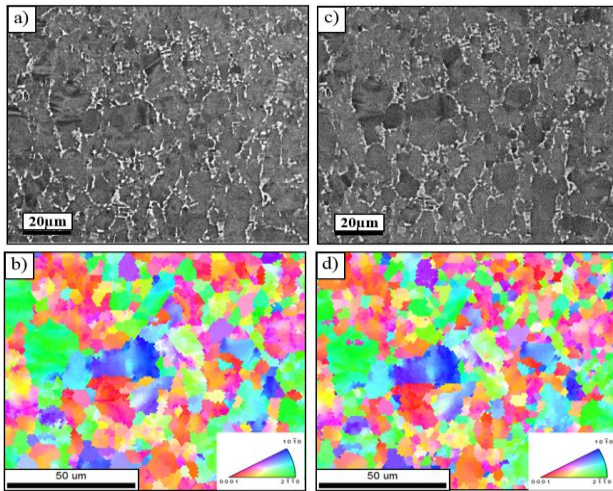


The SRIM-2013 calculation of the dose in a Ti-6Al-4V sample for the <sup>36</sup>Ar @ 36 MeV beam with a fluence of  $10^{15}$  ions.cm<sup>-2</sup>

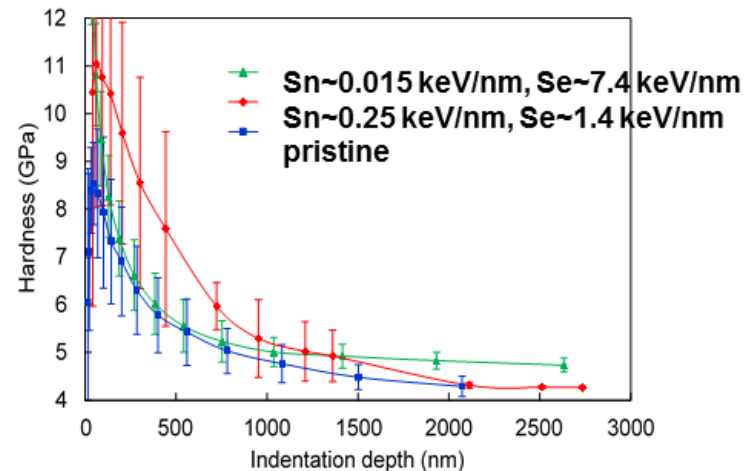
# Material Study with Heavy Ion Irradiation at Low Energy [2]

- X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Electron Backscatter Diffraction (EBSD) analysis, Nano-indentation, Vickers test done in collaboration with GANIL-CIMAP and MSU-CHEMS (MatX Strategic Partnership)
  - No sign of track formation due to electronic excitations for any of the experimental conditions used (ions, species, energy or fluence)
  - No change in the microstructure, the crystallographic orientation or in hardness was observed.

CiMap



BSE images and IPF maps before (a), (b) and after irradiation at the same area (c), (d) in the Ti-6Al-4V sample I-T3 irradiated with the 92 MeV  $^{131}\text{Xe}$  beam at  $T = 25^\circ\text{C}$  and a fluence of  $2 \cdot 10^{14} \text{ ions.cm}^{-2}$

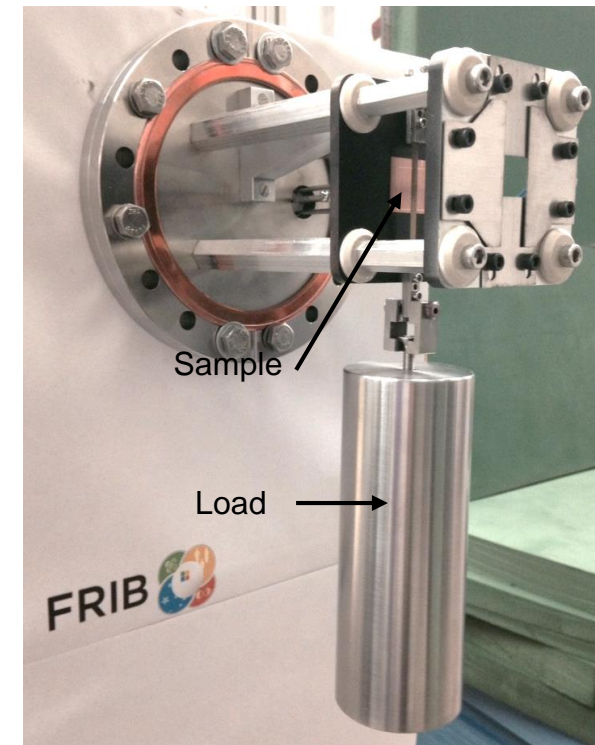
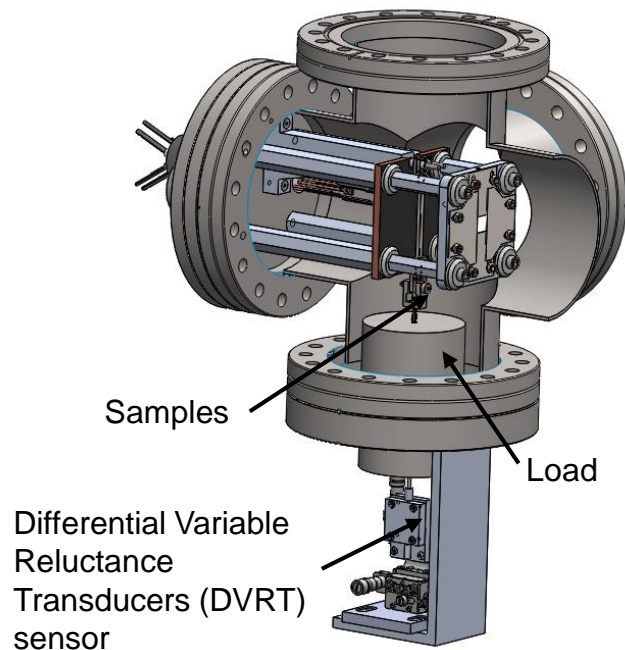


Nanoindentation Hardness of Ti-alloys, pristine and irradiated with  $^{36}\text{Ar}$  @ 36 MeV –  $T_{\text{irr}} = 350^\circ\text{C}$  – fluence of  $10^{15} \text{ ions/cm}^2$

A. Amroussia et al., NIMB 365 (2015) 515-521

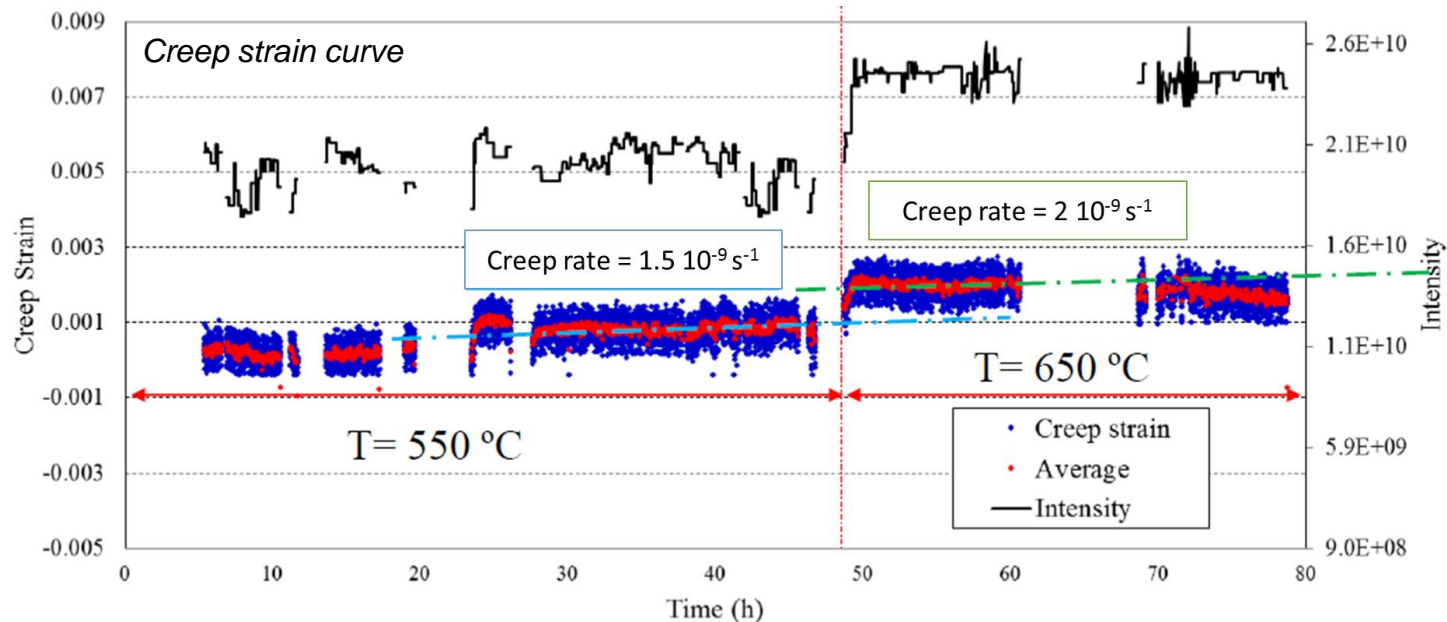
# Material Study with Heavy Ion Irradiation at Intermediate Energy [1]

- Irradiation creep test was performed in June 2015 at GANIL-France
  - Dose rate with  $^{36}\text{S}^{15+}$  at 12 MeV/u was representative for intermediate mass beams at FRIB
  - Samples were irradiated with a fluence up to  $4.6 \cdot 10^{15}$  ions/cm<sup>2</sup>



# Material Study with Heavy Ion Irradiation at Intermediate Energy [2]

- Preliminary results, including irradiation-enhanced creep and thermal creep, show that a low value of creep was observed and are similar to “pure thermal” creep



- Challenge to obtain creep rate at lower temperature
  - » Sample heated by the beam deposition
  - » No active cooling possible without disturbing creep measurement

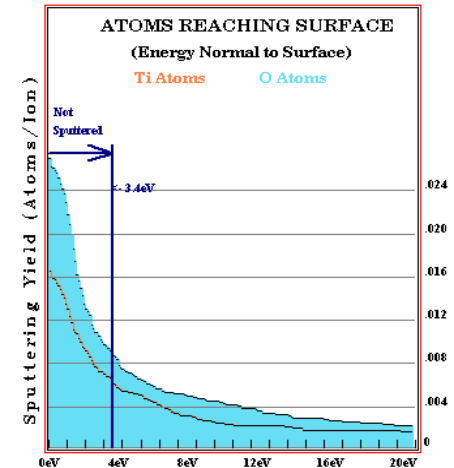
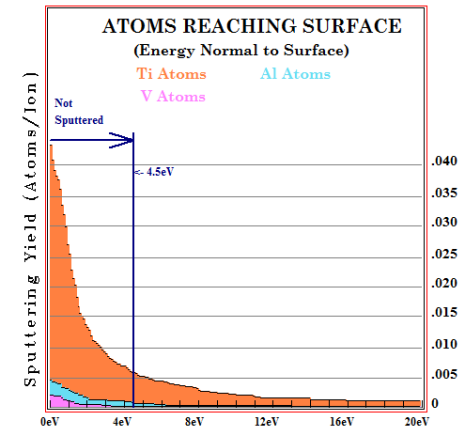


# Material Study with High Energy Heavy Ions

## Sputtering Effect and Gas Production Negligible

- Estimated earlier in the project for conditions more severe than present in FRIB dump (collaboration with GANIL-CIMAP, M. Toulemonde)
  - Electronic sputtering is negligible
    - » Rate with U beam and drum shell at 600 °C. (<200 °C expected in reality)
      - Ti ~  $1 \cdot 10^{-4}$  sputtered atoms / incident ions
      - TiO<sub>2</sub> ~  $1.5 \cdot 10^{-2}$  sputtered atoms / incident ions
      - » Ti ~ 0.03 nm, TiO<sub>2</sub> ~ 5 nm
  - Nuclear sputtering estimated by SRIM code is negligible
    - » Ti ~ 2 nm, TiO<sub>2</sub> ~ 4.7 nm
- Calculated concentrations of the produced gases in the shell (H, He and tritium) during one year of operation are too small to significantly alter the Ti-6Al-4V properties
  - H ~ 23 appm, He ~ 8 appm and Tritium ~ 1.2 appm with PHITS code
    - » Factor 2 less in gas production estimated with MARS15 code

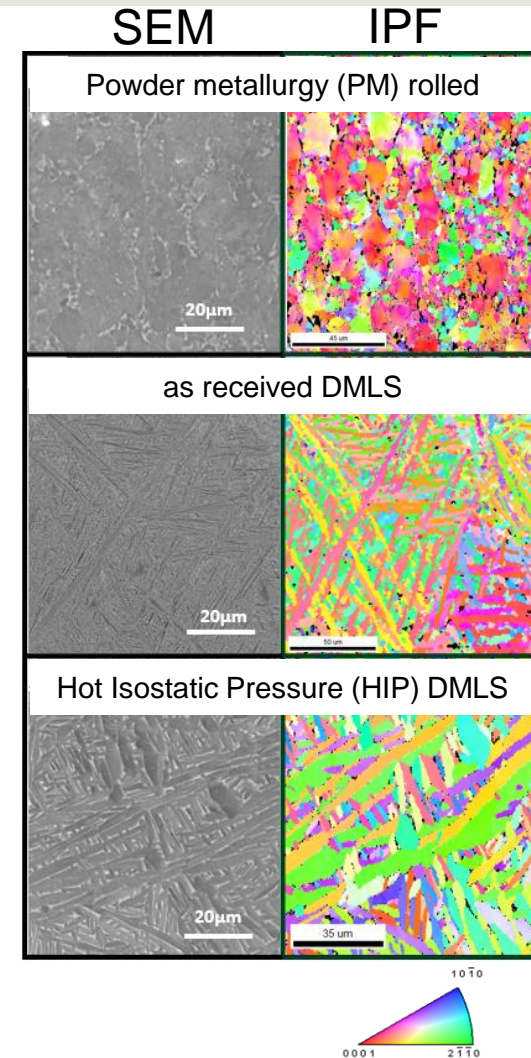
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# 3D Printed Material for Beam Dump Drum

## Which impact on material properties/behavior?

- 3D printing (with Direct Metal Laser Sintered DMLS process) likely fabrication process for FRIB beam dump shell
  - Microstructure of Ti-6Al-4V alloys may change due to fabrication process
    - » Characterization of different microstructures (Powder Metallurgy (PM) rolled, 3D printed (As received DMLS), 3D printed Hot Isostatic Pressure (HIP) (HIP-ed DMLS) ongoing
    - » 3D printed sample analysis shows lamellar microstructure
- The HIP (Hot Isostatic Pressure) was performed at a temperature of 900°C at 102 MPa for 2 hours
- The powder used for DMLS samples is Grade 5 Ti-6Al-4V





# In-situ TEM with Heavy Ion Beam

- In-situ TEM irradiation was performed at the Intermediate Voltage Electron Microscopy (IVEM) - Tandem Facility at Argonne National Laboratory
- $\text{Kr}^{2+}$  at 1 MeV at 350°C

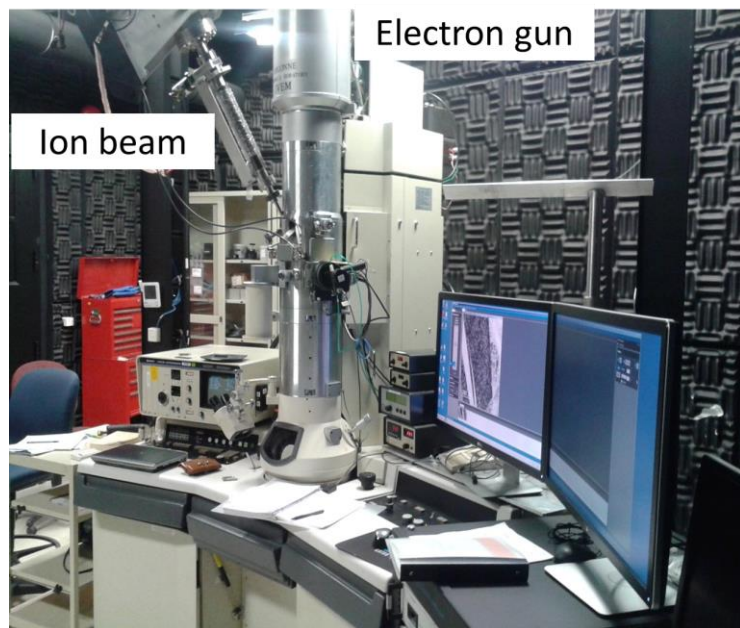
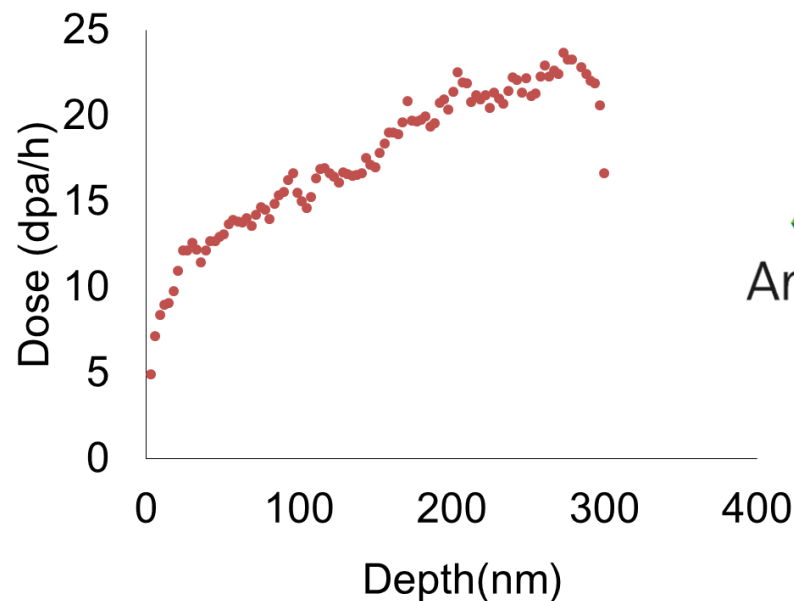


Image of the IVEM-Tandem facility at ANL.

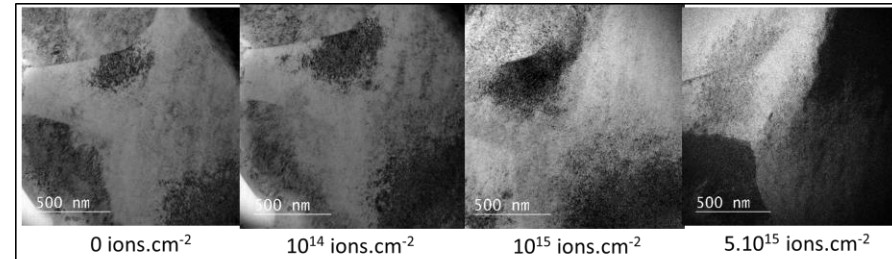


Dose rate Vs depth of Kr ions in a Ti-6Al-4V target  
SRIM-2013 calculation

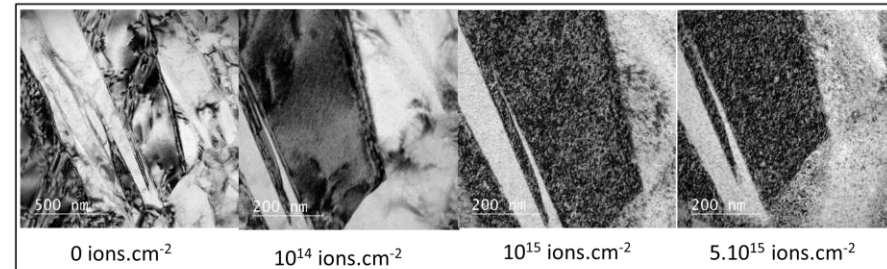


# In-situ TEM study with Low Energy Heavy Ion Irradiation

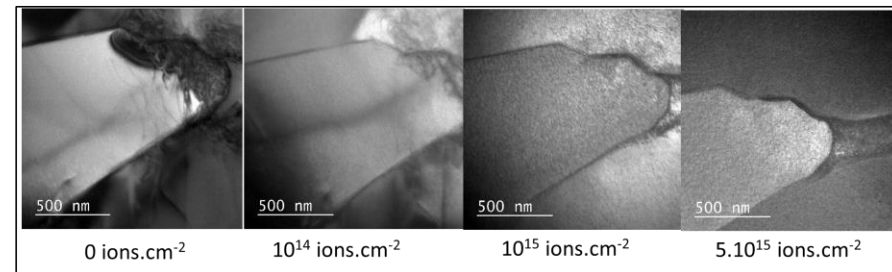
- Fluence up to  $10^{16}$  ions/cm<sup>2</sup> (~ 28 dpa) at ~14 dpa/h
- A preliminary analysis of the defect accumulation with in-situ TEM irradiation shows an accumulation of fine defects without recombination
  - In all irradiated samples, we observed fine nanometer size black spots indicative of defects formed due to radiation damage.
  - Initial dislocations and some features such as sub-grains observed in the un-irradiated microstructure disappeared after irradiation.
  - The PM-rolled sample irradiated up to a fluence of  $10^{16}$  ions/cm<sup>2</sup> (~ 28 dpa) was bent heavily after irradiation. The HIP-ed sample irradiated to the same fluence showed a better irradiation resistance.



Observation of defect accumulation in PM rolled Ti-6Al-4V for different fluences



Observation of defect accumulation in as-received DMLS Ti-6Al-4V for different fluences

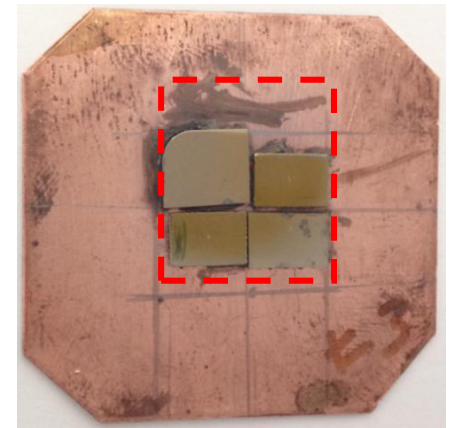
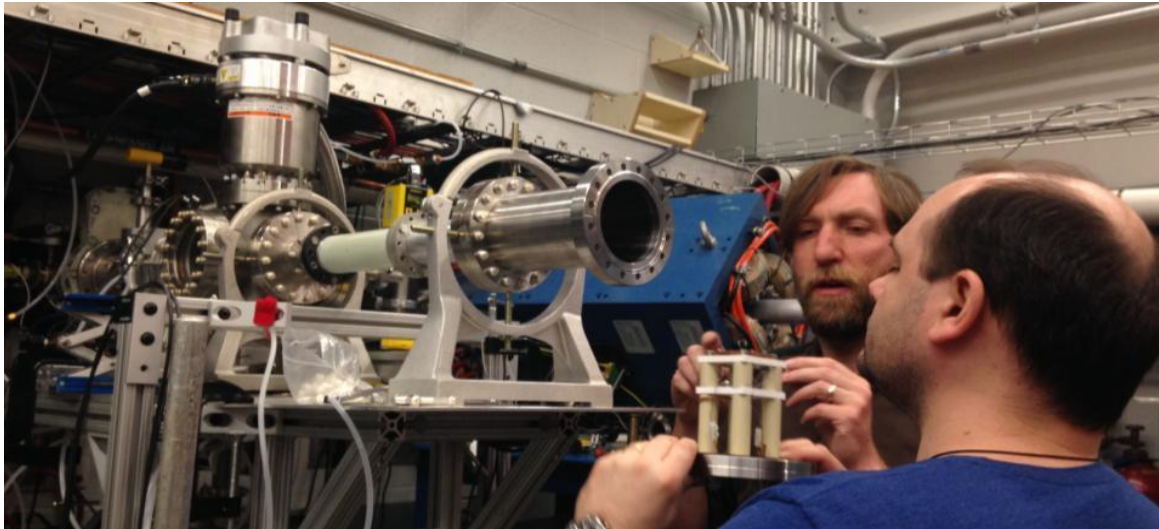


Observation of defect accumulation in DMLS-HIPed Ti-6Al-4V for different fluences

# Support for Beam Dump Drum Design

## Low Energy Heavy Ion

- Low energy heavy ion irradiation at University Notre Dame to correlate mechanical properties of Ti-alloys with radiation damages observed during in-situ study at Argonne
  - 4 different microstructures of Ti-alloy were irradiated under 3 irradiation conditions in May 2016
    - » at room temperature and at 350°C
    - » Up to a fluence of  $5 \cdot 10^{16}$  ions/cm<sup>2</sup> (or 14 dpa)
    - » Two different dose rates ( $\sim 14$  dpa/h and 0.8 dpa/h)

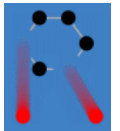


350°C,  
dose  $\sim 14$  dpa, 12 dpa/h



# Future work on Ti-6Al-4V

- Post-irradiation characterization of TEM samples to identify defect (precipitates, dislocation loops) is planned at Argonne
- New in-situ TEM
  - at room temperature planned at Argonne next week (September 2016)
  - at 150°C approved at Orsay-JaNNUS (France) for test in 2017
- Low energy heavy ion irradiation at 150°C was approved at GANIL-CIMAP
- Correlation with mechanical behavior with irradiation of samples for nano-indentation testing at Notre Dame University (room temperature and 350°C)
- Proton irradiation of Ti-6Al-4V with BLIP facility (BNL) samples under preparation with RaDIATE collaboration (February 2017)
- Corrosion effect will be investigated in collaboration with University of Michigan
- Mechanical tests at MSU on 3D printed Ti-6Al-4V to study orientation effect



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# Conclusion

- Beam Dump faces various thermo-physical-mechanical and chemical challenges
  - Some effects may be enhanced by the presence of the other
    - » corrosion in presence of radiation, stress limit change in the presence of radiation
- Up to now no facility exists to study the impact from all effects combined together
  - The changes of material have to be studied case-by-case experimentally
- Ti-6Al-4V confirmed as suitable material choice for the beam dump drum
  - No radiation damage observed from several studies promises good radiation resistance of this alloy and gives confidence in lifetime
  - Defect accumulation analysis ongoing

# Acknowledgements

- **GANIL-CIMAP**
  - F. Durantel, C. Grygiel, I. Monnet, F. Moisy, M. Toulemonde
- **University of Michigan**
  - G. Was
- **Argonne National Laboratory**
  - P. Baldo, M. Kirk, M. Li, E. Ryan
- **Notre Dame University**
  - T. Ahn, D. Robertson, E. Stech
- **MSU Department of Chemical Engineering and Material Science**
  - A. Amroussia, C. Boehlert
- **FRIB**
  - M. Avilov, P. Ferrante, J. Kramer, W. Mittig, H. Patel, B. Peruski, B. Phillips, M. Schein

This work was partially supported by the U.S. Department of Energy, Office of Science under Cooperative Agreement DE-SC0000661. This work was also supported by Michigan State University under the Strategic Partnership Grant "FRIB - Materials in Extreme Environments".

The in-situ TEM irradiation was supported by the U.S. Department of Energy, Office of Nuclear Energy under DOE Idaho Operations Office Contract DE-AC07-051D14517 as part of NSUF experiment.



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# Thank you for your attention



*FRIB construction area – August 29th 2016*



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