RaDIATE collaboration meeting at Granada, 16th – 18th Sep. 2024

RaDIATE activities at J-PARC

Shunsuke Makimura
on behalf of J-PARC RaDIATE

R a D I A T E Collaboration

Radiation Damage In Accelerator Target Environments

J-PARC RaDIATE

J-PARC participated in RaDIATE collaboration in December 2017

- Beam window for T2K
- Target materials for pion/muon production
- DPA cross section measurements,,,

Mostly, thanks to US-JP collaboration

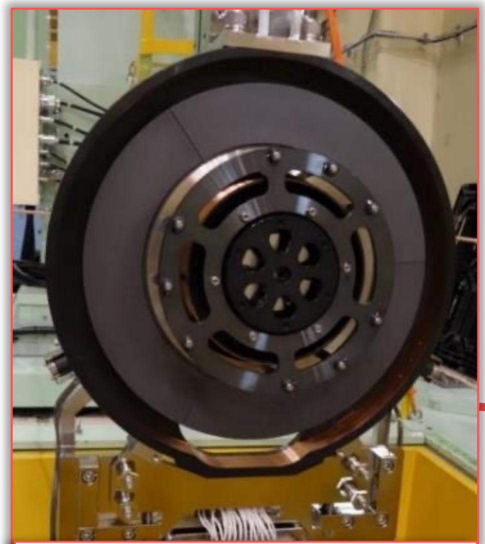
Transition from individual activities by volunteer-based members to J-PARC-wide mission led by the director of J-PARC Center

<u>J-PARC-wide activities:</u>

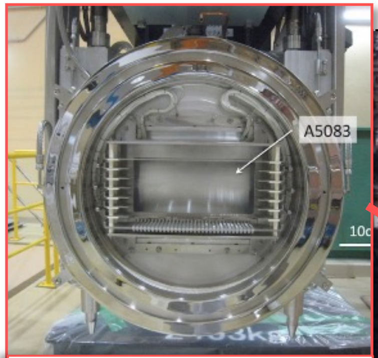
Irradiation damage studies in Targets, beam windows, and beam-intercepting components in the entire Experimental & Accelerator facilities.

- Not only the official RaDIATE activities but also any radiation damage studies
- Quarterly core-members meeting
- Some budget is allocated for the activities.

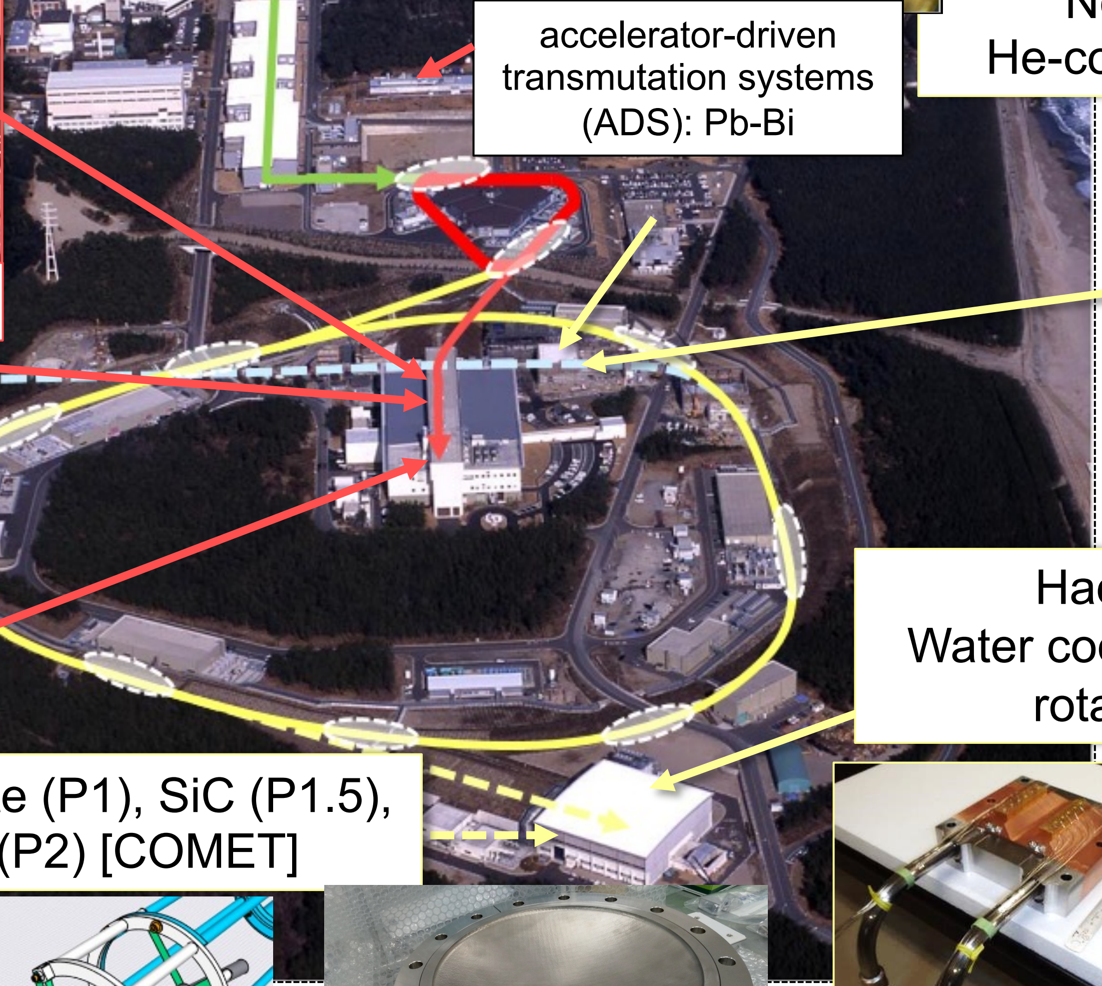
Targets & Beam windows at J-PARC



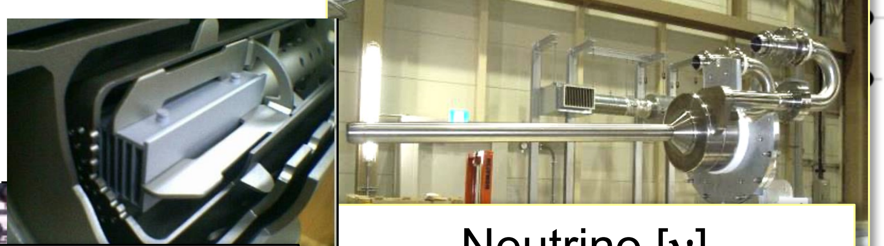
Muon [MLF-MUSE]
Rotating Graphite



Al A5083 BW

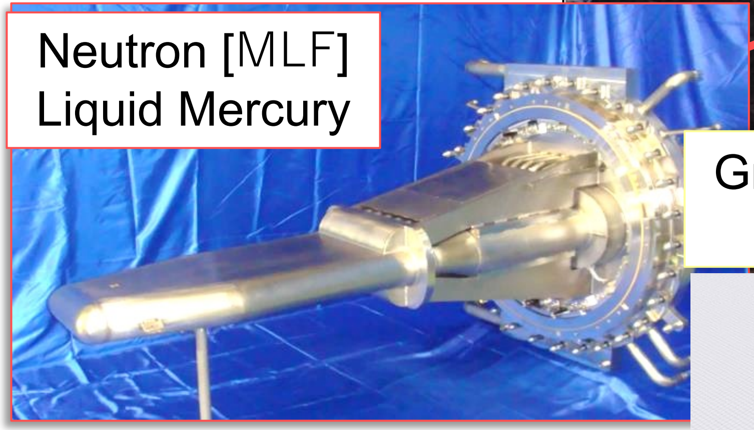


accelerator-driven transmutation systems (ADS): Pb-Bi



Neutrino [ν]
He-cooled graphite

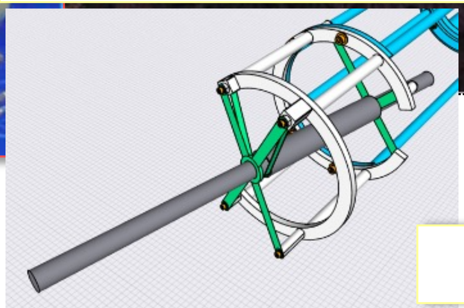
Ti-6Al-4V BW

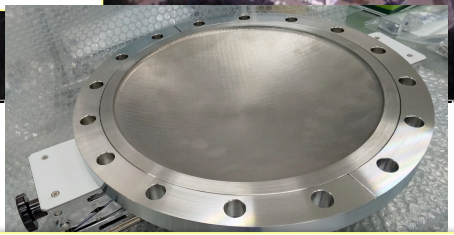


Neutron [MLF]
Liquid Mercury

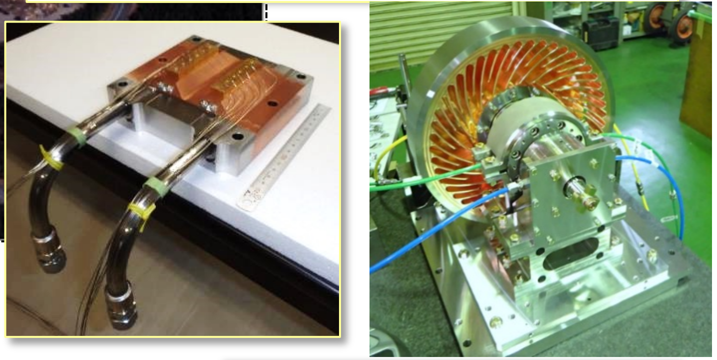
Hadron [HEF]
Water cooled gold/ high-Z rotating target

Graphite (P1), SiC (P1.5), W (P2) [COMET]





3D-printed Ti-6Al-4V BW



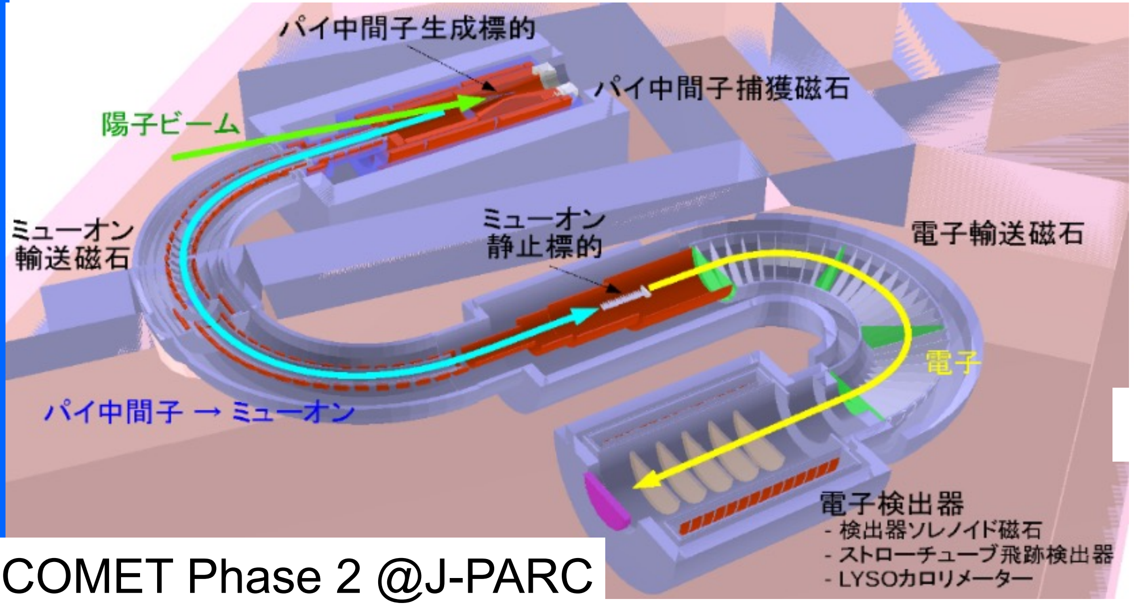
Beryllium BW

Outline

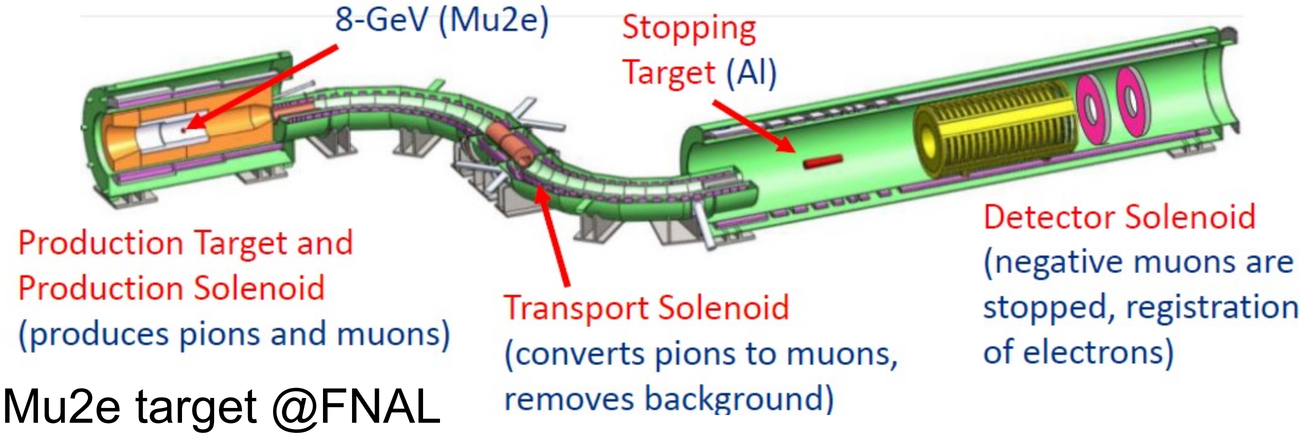
1. Tungsten alloy for muon target
2. Titanium alloy for neutrino beam window
3. SS 316L for neutron target
4. Superconductor for magnet
5. Summary

1. Tungsten alloy for muon target

Tungsten is expected as target materials



COMET Phase 2 @J-PARC



Mu2e target @FNAL

- Higher density: smaller spatial volume of pion
- High melting point

	Mu2e / Early-stage COMET P2	COMET P2 / Upgrade Mu2e
Proton beam	8 GeV, 8 kW	8 GeV, 56 kW
Target material	Rad. cooling tungsten	Water cooling tungsten
Target thickness	160 mm	160 mm
Time structure	0.4 s. extra. in 1.2/1.4 s.	(0.5 s. extra. in 2.5 s.)

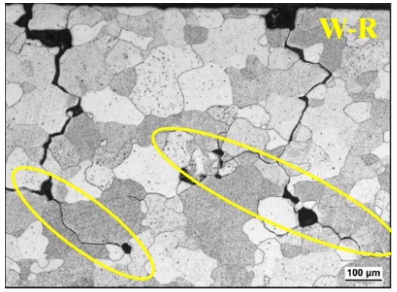
⇨

W target in future physics
- J-PARC MLF 2nd Target S
- J-PARC Hadron target
- ORNL 2nd Target S
- ESS Neutron source
- Anti-p+ target at CERN
- Positron source at ILC
- Muon collider etc.

COMET-Mu2e collaboration has been launched.

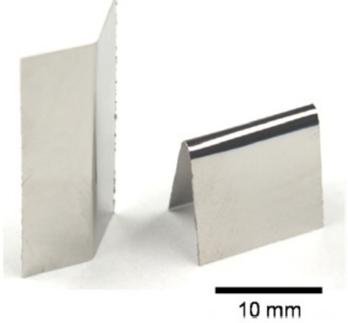
Recrystallization embrittlement & Irradiation embrittlement

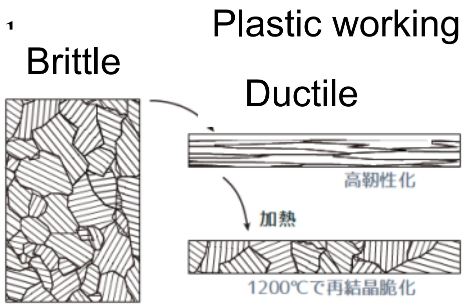
- ✓Tungsten is brittle, because grain boundary is weak.
- ✓Brittleness is improved by heavy plastic working.
- ✓Revert to the brittle material at recrystallization temperature (1200 ˚C at Pure W)



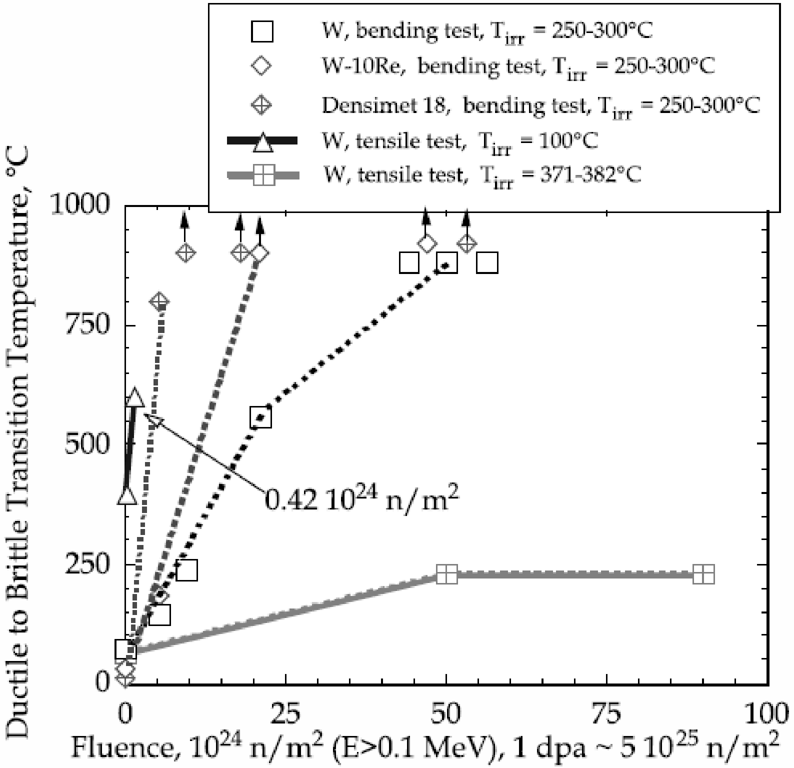
G. Pintsuk et al.

J.Reiser et al. JNM, 423 (2012)





Recrystallization embrittlement

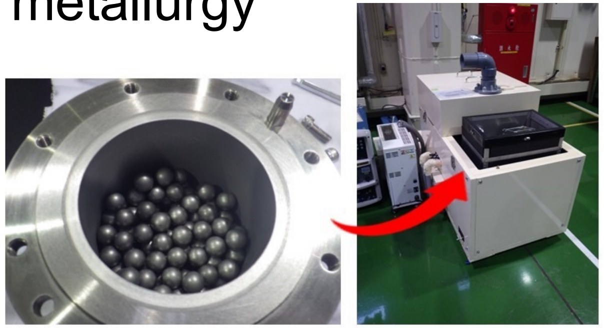


Irradiation embrittlement

To overcome recrystallization & irradiation embrittlement, TFGR-W, based on powder metallurgy, has been developed under academia-industrial collaboration in J-PARC.

Toughened Fine-Grained Recrystallized Tungsten (TFGR-W-TiC)

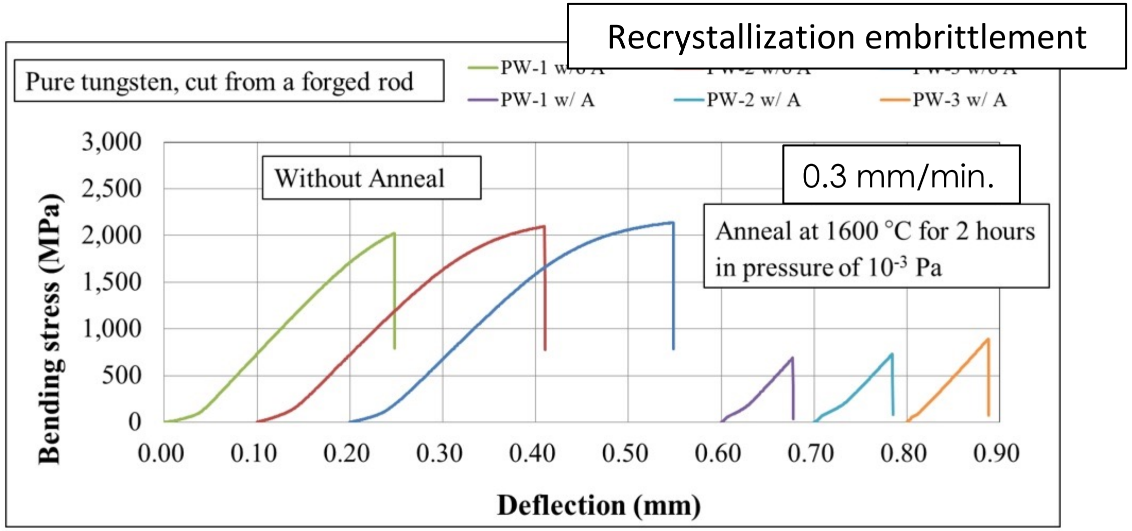
- ✓ Equiaxed and Fine-grained
- ✓ Grain boundaries are reinforced by titanium carbide through grain boundary sliding
- ✓ Manufactured by powder metallurgy

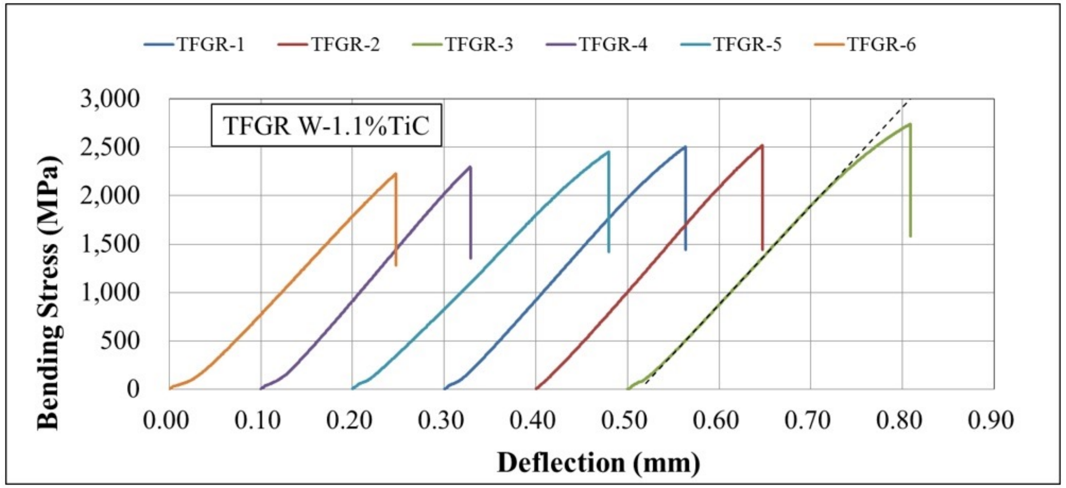


Mechanical alloying process

Then, sintered in Spark Plasma Sintering.

Mater. Sci. Forum, Spallation Materials Technology, 1024(2021)103-109





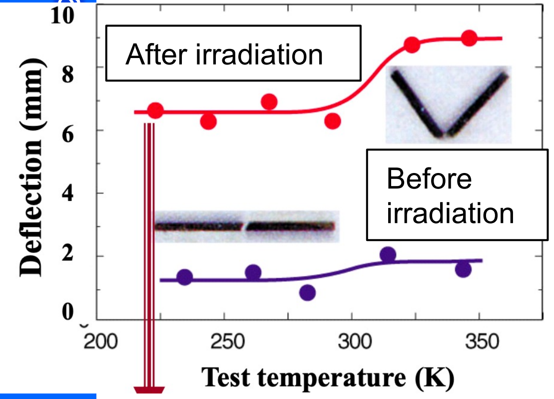
Overcoming of recrystallization embrittlement

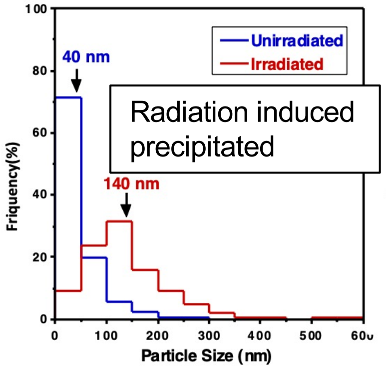
Then, irradiation resistance ?

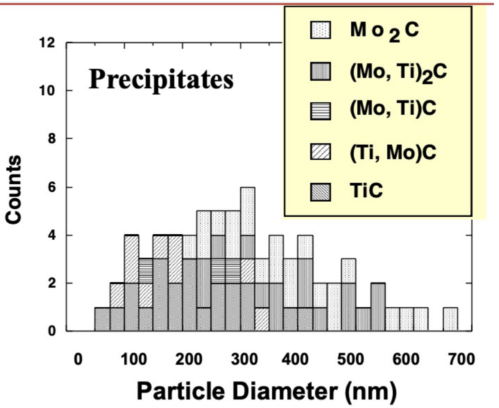
- ✓ Irradiation results has not been obtained sufficiently yet.
- ✓ But the sign of high irradiation resistance exists.
- ✓ High sink-site with fine-grained and semi-coherent grain-boundaries between W and TiC

Mo-1.0%TiC: Radiation Induced Ductilization,

Y. Kitsunai et al., JNM, 239, (1996)
Neutron irradiation: 573-773 K, 0.1 dpa







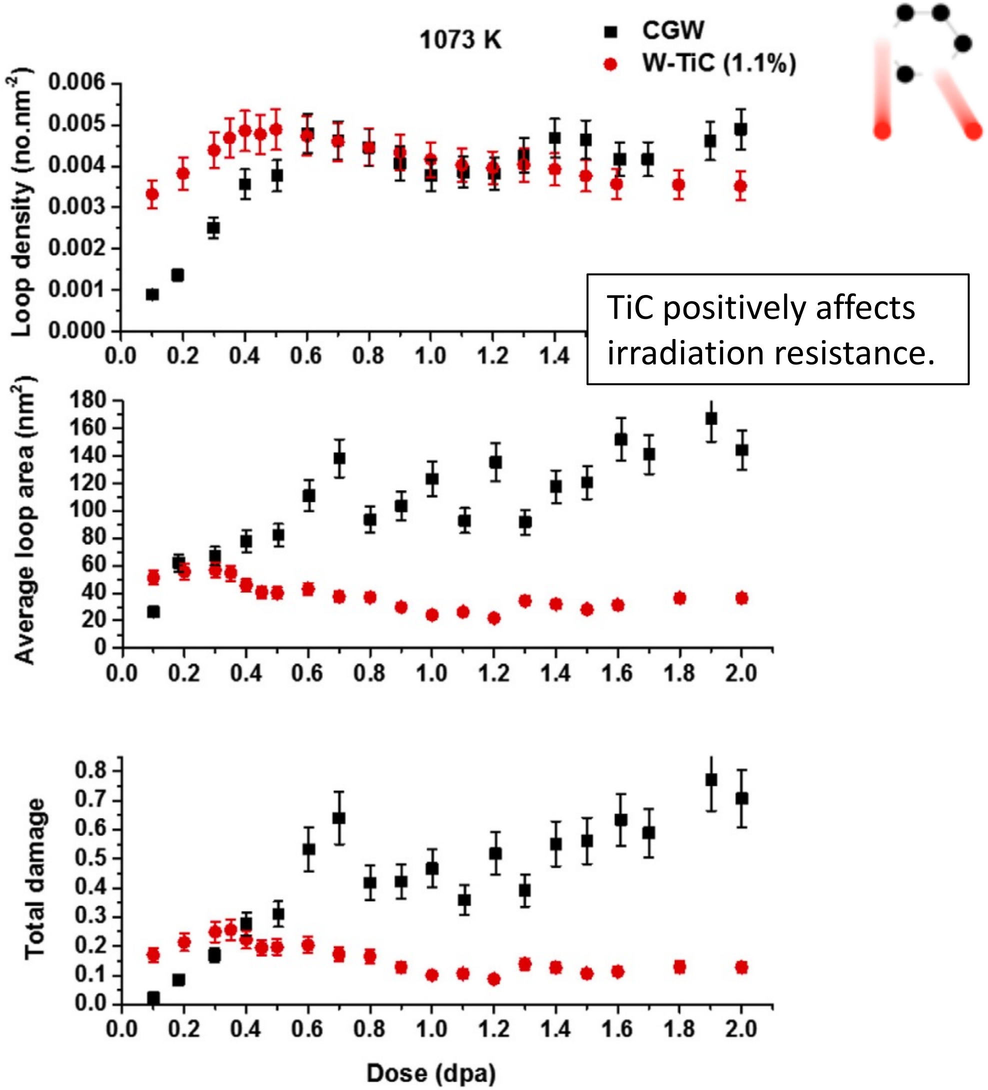


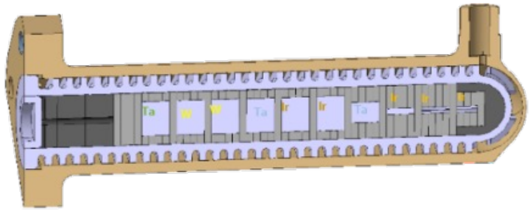
Fig. 11. Comparisons of average areal loop density, loop area, and total damage (loop density × loop area) as a function of dpa for the *in-situ* 1 MeV Kr^{+2} irradiated CGW and W-TiC (1.1%) sample at 1073 K. Error bars are included to reflect errors in quantifying the same area several times.

"In-situ irradiation tolerance investigation of high strength ultrafine tungsten-titanium carbide alloy", LANL group.
O. El-Atwani et al., Acta Materialia 164 (2019) 547e559

RaDIATE

Radiation Damage In Accelerator Target Environments

HiRadMat Experiments HRMT48 and HRMT60 at CERN



C. T. Martin et al.

<u>TFGR W-TiC</u>

- Included in HRMT48 for AD-target design, Ir, Ta, TFGR,,,
- No noticeable damage
- Promising response

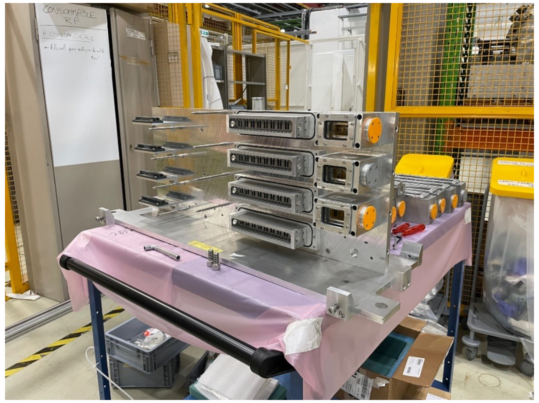
POT: 3.2×10^{13}～1.12×10^{14}
Beam size: 1mm × 1 mm
50 pulses, pulse duration 25 ns
dT=700℃,
Tensile stress: 1 GPa

Sample preparation

HRMT 60 under RaDIATE collaboration

More than 100 specimens were irradiated at HRMT60.

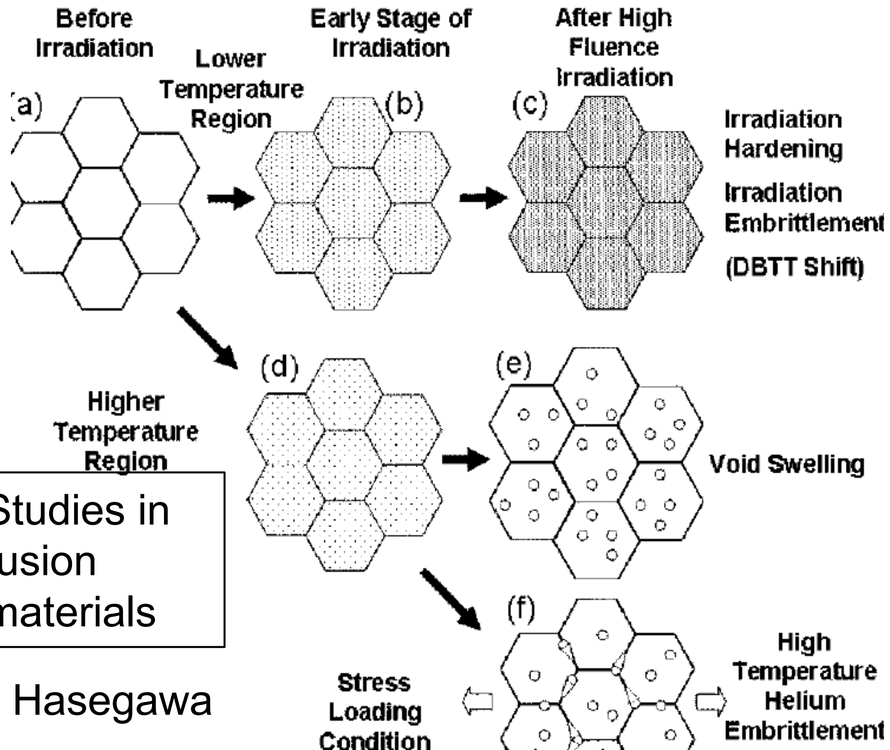
- Ti alloys from BLIP capsule
- Novel materials: TFGR-W, HEAs, Ti alloys, NITE SiC/SiC,,,



He embrittlement in high energy proton irradiation

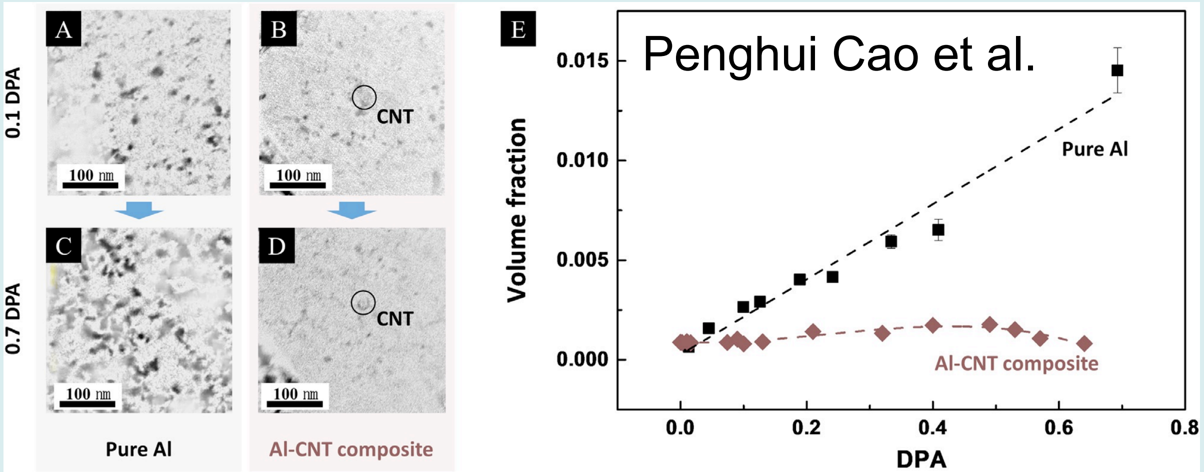
Irradiation Source	DPA rate (DPA/s)	He production (appm/DPA)	Irradiation Temp (˚C)
Mixed spectrum fission reactor	3×10^{-7}	1×10^{-1}	200-600
Fusion reactor	1×10^{-6}	1×10^{1}	400-1000
High energy proton beam	6×10^{-3}	1×10^{3}	100-800

- ✓High energy proton irradiation produces much larger helium than fission & fusion materials.
- ✓ He bubble formation leads fatal embrittlement in high temperature.
- ✓So far, no one could solve He embrittlement.



Studies in fusion materials

A. Hasegawa



Recently, it was reported "Carbon nanotube (CNT) Al composites exhibit greatly reduced He bubble formation",

These concepts were applied to tungsten under collaboration with J-PARC, FNAL, and BNL.

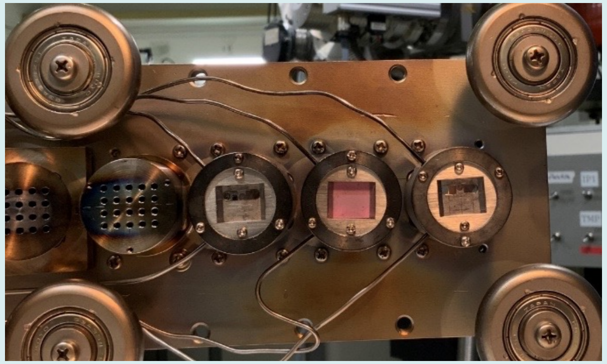
Preliminary results in W-CNT

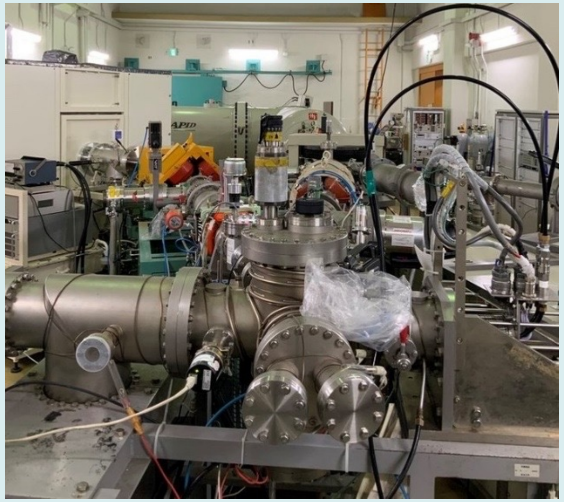
- W-CNT & W-TiC-CNT was manufactured in J-PARC.
- Multi wall carbon nanotubes
- Manufactured with powder metallurgy

Density (g/cc): W-CNT 16.96, W-TiC CNT 16.018

W-CNT

W-TiC-CNT





To form He bubble, samples were irradiated by He ions in HIT Tokyo Univ.

Red: W, Green: C

TEM analysis in BNL

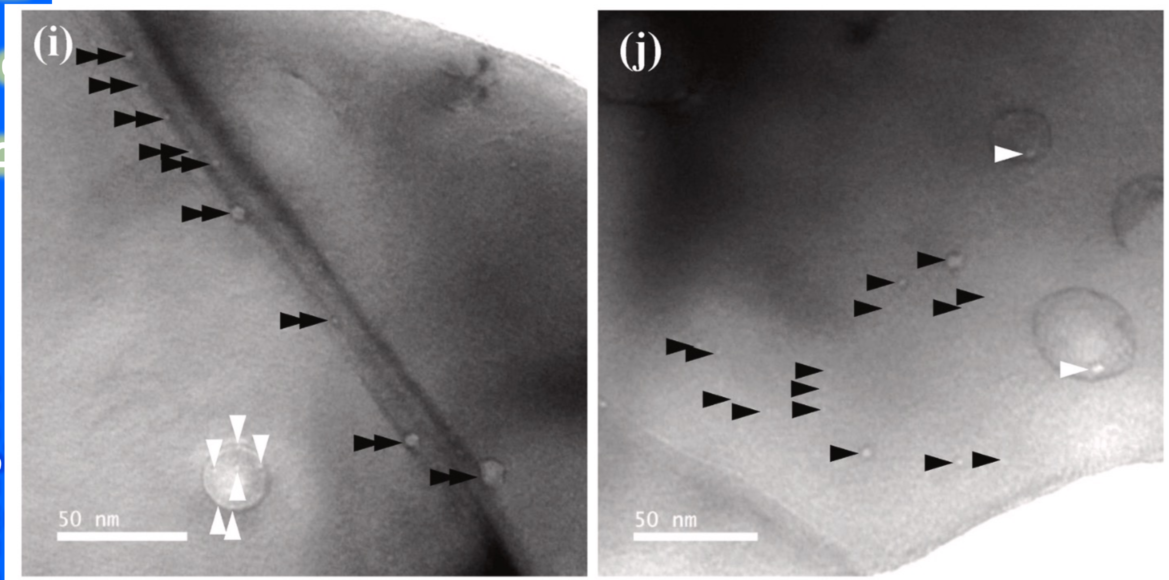
- EDS shows carbon clusters
- However, many pores exists. Improvement is necessary.

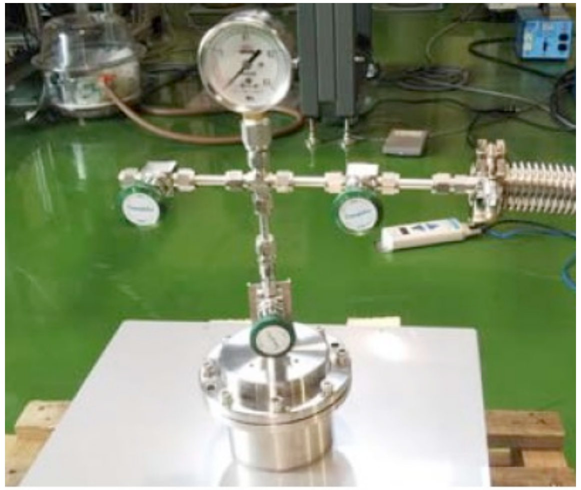
New technique: Helium embrittlement study w/o particles irradiation

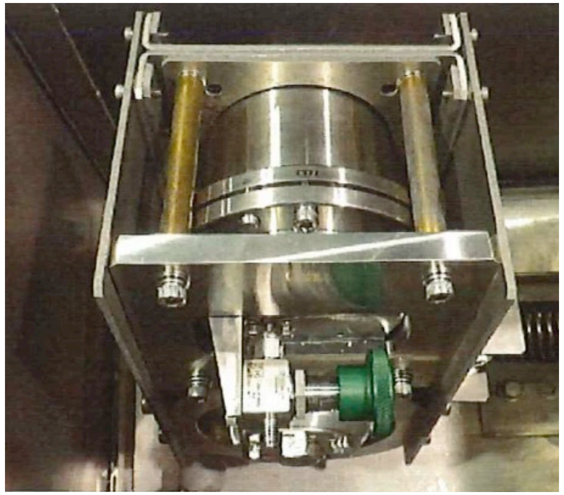
So far, to study Helium embrittlement,

- High energy proton or neutron irradiation: Samples are heavily activated.
- Helium and heavy ion irradiation: Damage is localized. Hardness testing or TEM.

Recently, we established a new technique to introduce helium bubbles in bulk tungsten material.







T. Sakamoto et al., Vacuum 228 (2024) 113482

- Mechanical alloying was conducted in helium atmosphere.
- Helium can be replaced with hydrogen.
- W w/o impurity O_2 & N_2 showed a lower bending strength.
- Further studies are necessary.

MA	O2	N2	Bending strength	Run
1 atm	wt%	wt%	MPa	
Ar	0.84	0.097	438	Trial run
He	0.18	0.13	1380	Low vacuum
He	0.039	0.015	1188	High vacuum

2. Titanium alloy for Neutrino beam window

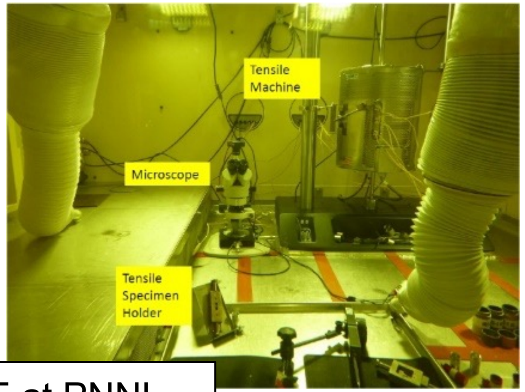
Materials are supplied by T. Ishida

Radiation damage studies in Ti-alloys

Press release Nov. 2020: “Why Does Titanium Alloy Beam Window Become Brittle After Proton Beam Exposure ?”

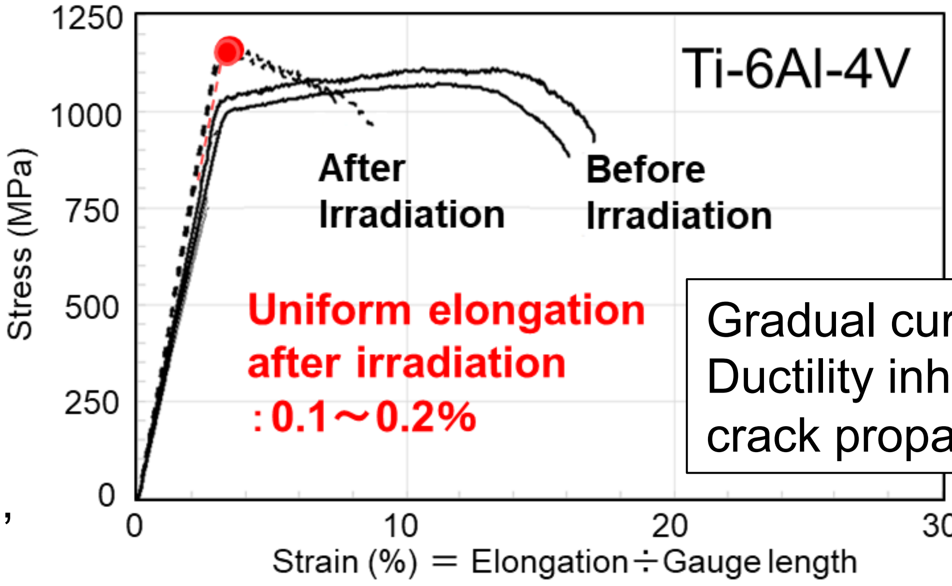
Ti-6Al-4V: widely used in industry
- Used in T2K beam window (B.W.)
- Will be used in LBNF B.W.

BNL-irradiated

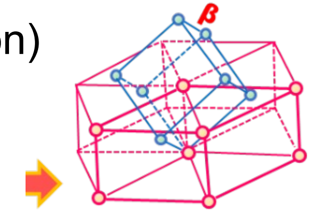


PIE at PNNL

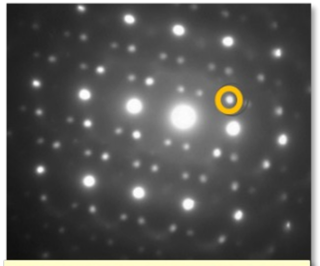
0.1 DPA (< 0.7 dpa@ 500 kW beam operation)



Gradual curve: Ductility inhibits crack propagation.



(Hexagonal)

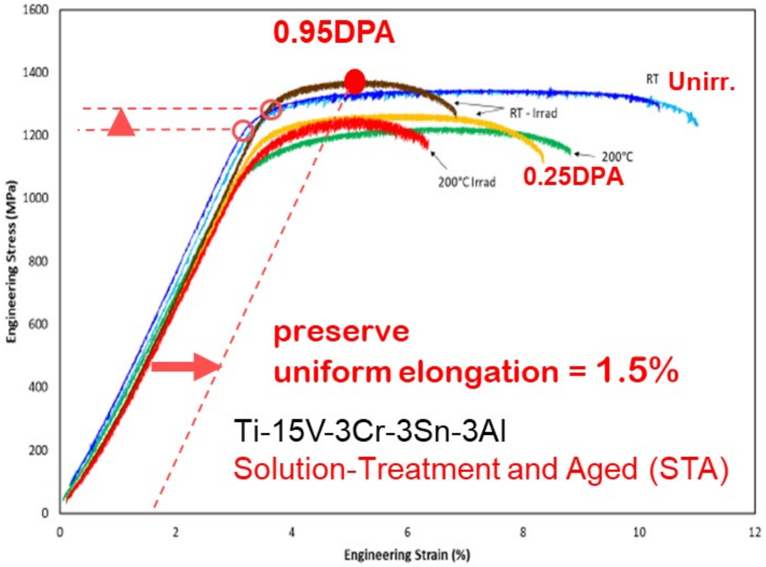


Radiation-Induced ω-phase

From the results of p+ irradiation at BNL and Post-Irradiation Exam (PIE) at PNNL, We found

The Ti-6Al-4V loses their ductility after slight irradiation by rad.-induced ω-phase.

And what do we do? – First choice, Ti-15-3-ST2A -



Promising sign at BLIP irradiation

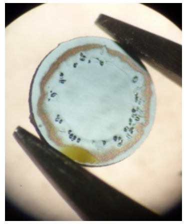
- ☐ The small ductility remained in Ti-15-3-STA after 0.95 dpa.
- ☐ In the upgrade of 1.5-MW beam, 2 dpa, Higher radiation resistance is needed.

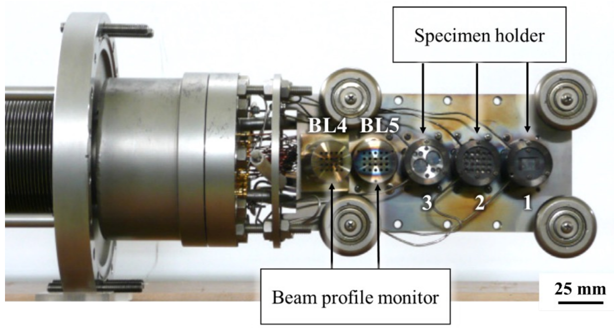
Nano-size precipitation by thermal treatment will absorb irradiation damage.

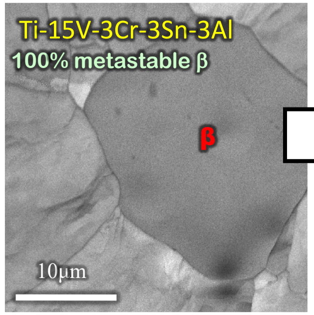
Ti-15-3-STA
(1 step aging)

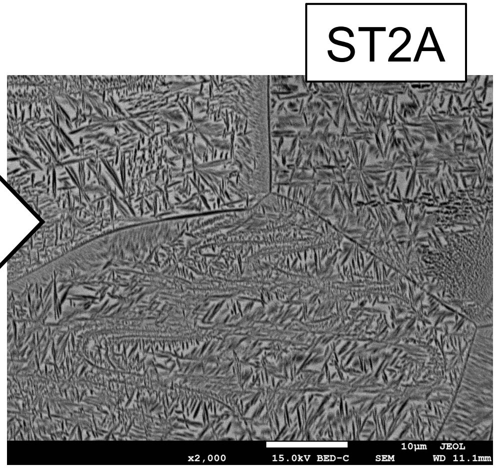
→

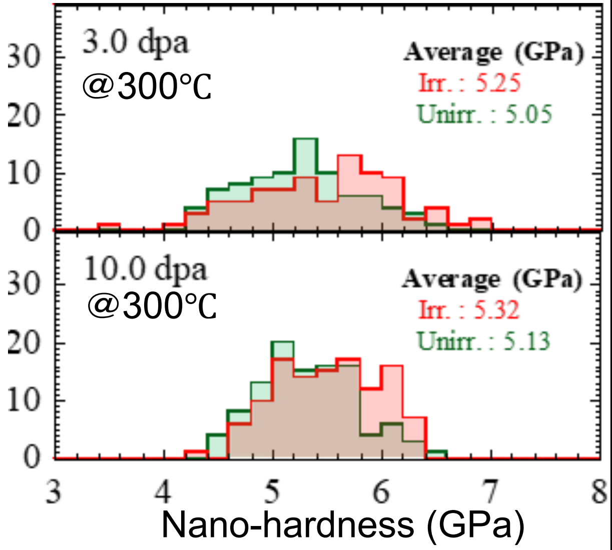
Ti-15-3-ST2A
(2 step aging)











- Local damage: Nano-hardness testing
- High DPA, no activation

Heavy ion irradiation at HIT, 10 dpa

ST2A showed higher resistance.

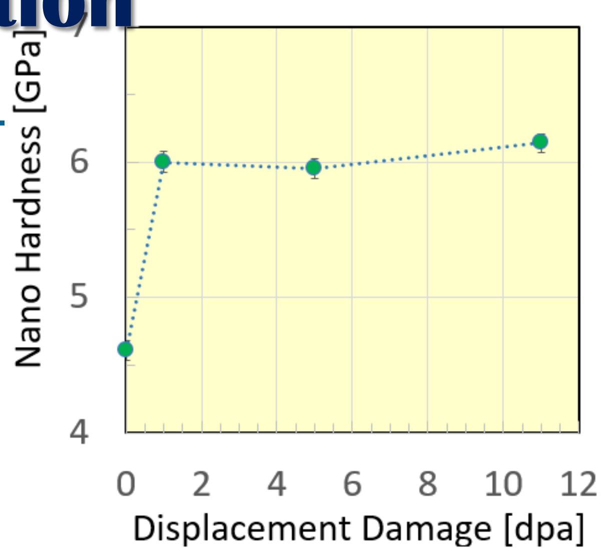
Contrasting Irradiation Behavior of Dual Phases in Ti-6Al-4V At Low-Temperature Ion Irradiation

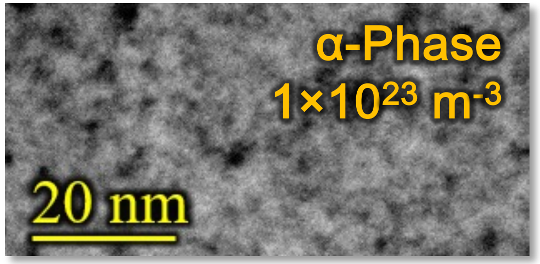
- Phase-dependent irradiation behavior of Ti-64 by Fe^{2+} ion beam at RT
- Nano-indentation hardness increases at 1 dpa and stays constant up to 11 dpa, due to the saturation of tiny defect clusters in the dominant α-phase
- Contrary *more than two orders fewer dislocations in the β-phase*
- Much less dislocations and absence of phase transformation in β-phase could be attributed to a strong sink effect or anomalous point defect recombination both originated from

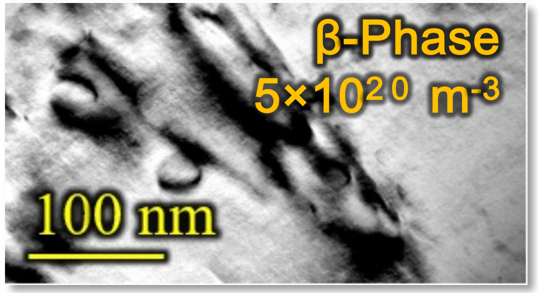
the ω-phase precursor

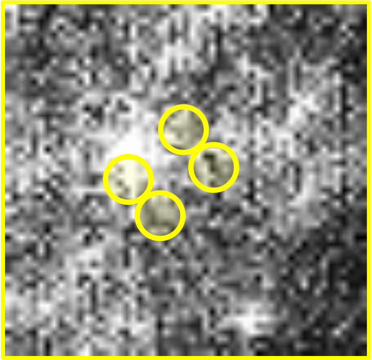
- **Published in Journal of Alloys and Compounds**

https://doi.org/10.1016/j.jallcom.2024.174701
https://arxiv.org/abs/2405.00517

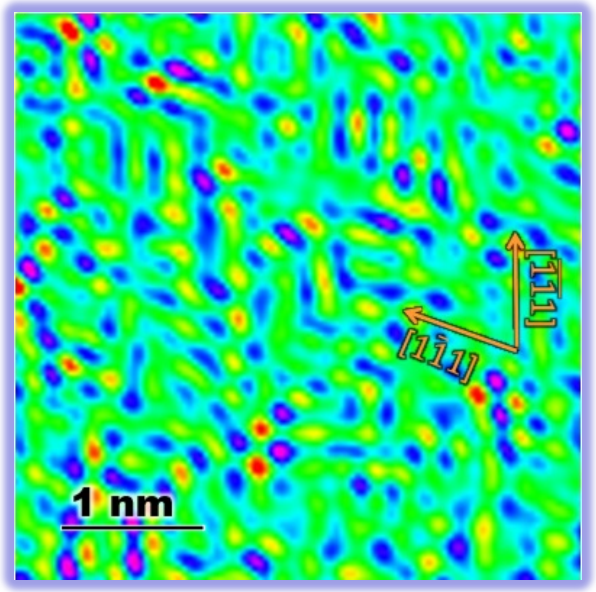








sub-nanometer-sized homogeneous lattice disorder within mother β-matrix



Phase Transformation of ω-phase Precursor in a Metastable β Titanium Alloy under Ion Irradiation at RT

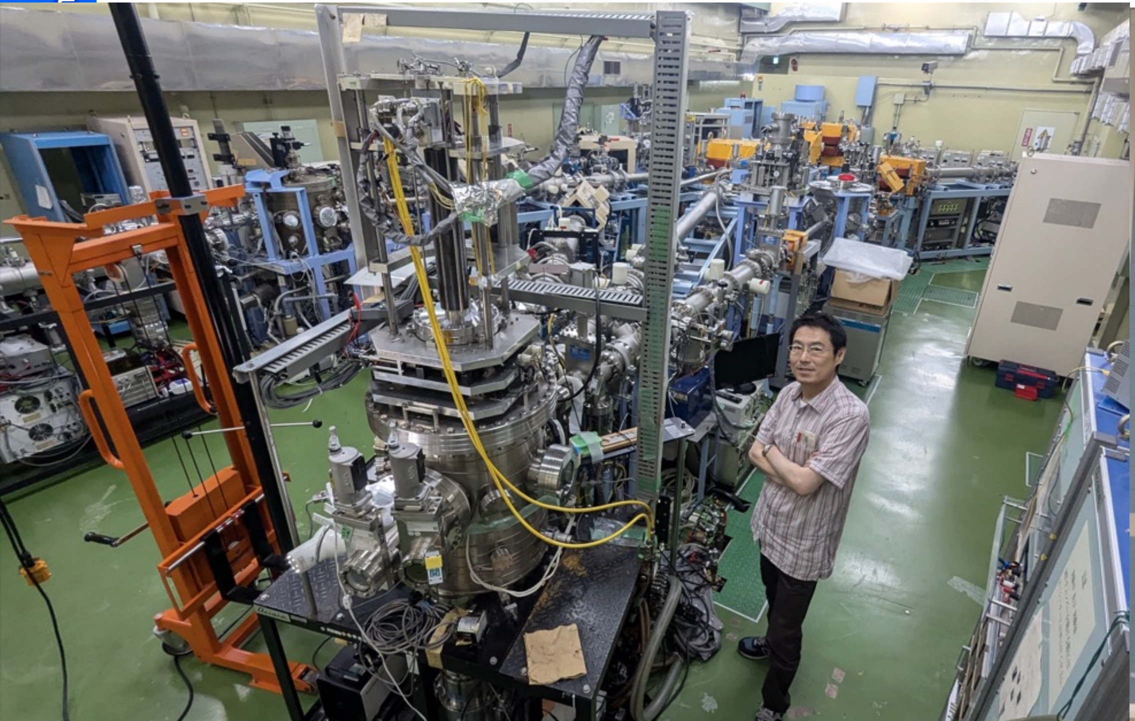
- Irradiation effects on phase transformation of ω phase and its precursors in a metastable β Ti-15V-3Cr-3Sn-3Al (Ti-15-3) to improve material properties
- Upon irradiation at RT, high number density nanoclusters corresponding to ω-like embryos formed from the precursor caused lattice disorder and developed with irradiation
- With continued irradiation, the ω-like embryos gradually disappeared, and dislocation loops were observed
- While irradiation hardening hardly occurred through irradiation

E. Wakai, T. Ishida, et al, HPTW2023, Riken, Nov.7.2023

Ion Beam irradiation Experiments

- Dual (Fe^{2+}/He^{2+}) beam irradiation at HIT (March 2024)
  - 40 appm-He/dpa
- Triple (Fe^{3+}/H^{+}/He^{+}) beam irradiation at TIARA (June 2024)
  - 400 appm-H/dpa, 100 appm-He/dpa

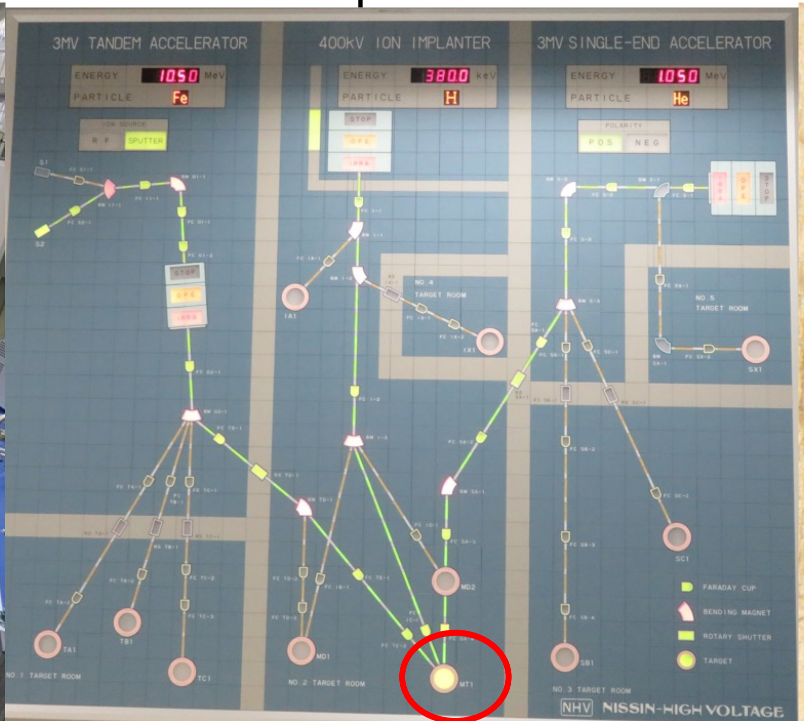
QST-TIARA Tripple Ion Beam Irradiation



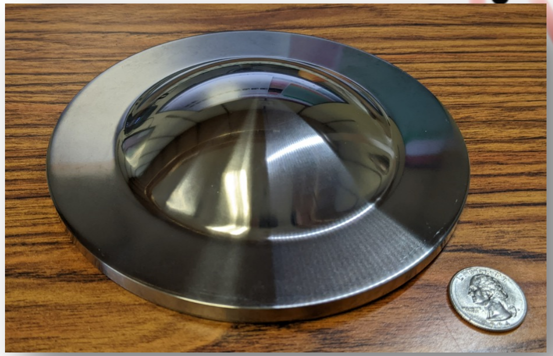
3MV TANDEM: Fe^{3+}

400keV ION Implanter: H^{+}

3MV Singleend Accelerator: He^{+}



Micro-structural control on Ti-15-3 ST2A for damage-tolerant beam window fabrication



- Despite successful prototype production, the coarse and uneven microstructure of the material was a challenge to improve
- Change of the thermo-mechanical process, which applies fast strains at high temperatures, has resulted in a finer, equiaxed microstructure

Launch of collaboration between KEK and NIMS on Thermo-Mechanical Processing on Ti-Alloys

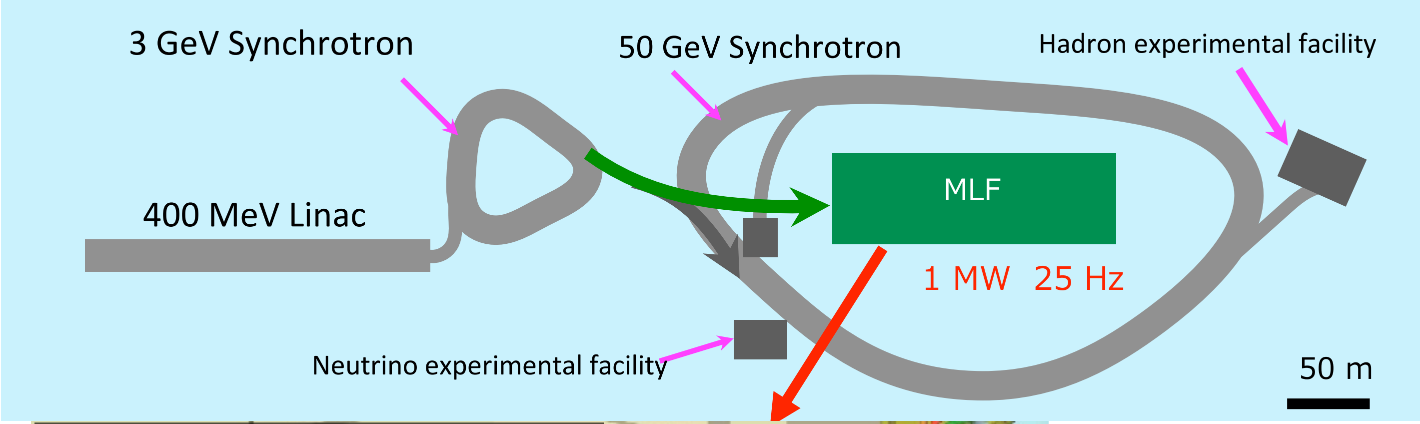
Specimen Foil Fabrication for Meso-scale Ultrasonic Fatigue Testing (Collaboration with UK)

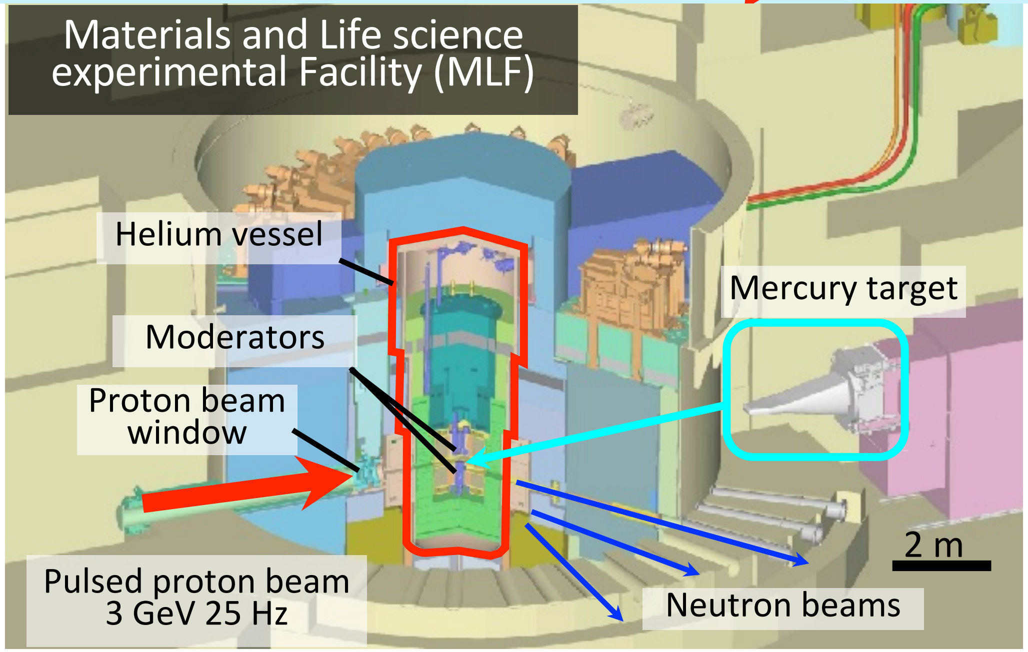
- Irradiate small “mesoscale” specimens of candidate radiation-resistant titanium alloys with proton beams (and helium) at the cyclotron accelerator of Univ. of Birmingham
- Carry out high-cycle fatigue strength measurements using ultrasonic vibration, at UKAEA-Material Research Facility(MRF)
- Expected to lead to an assessment of the service life of targets, windows at J-PARC HyperK/Fermilab LBNF
- Provide candidate grade materials, such as Ti-15-3 ST2A/ST, DAT54….
- R&D to fabricate both-side polished 150um-thick disk, cooperation with NIMS sample production experts / a NADCAP-certified company

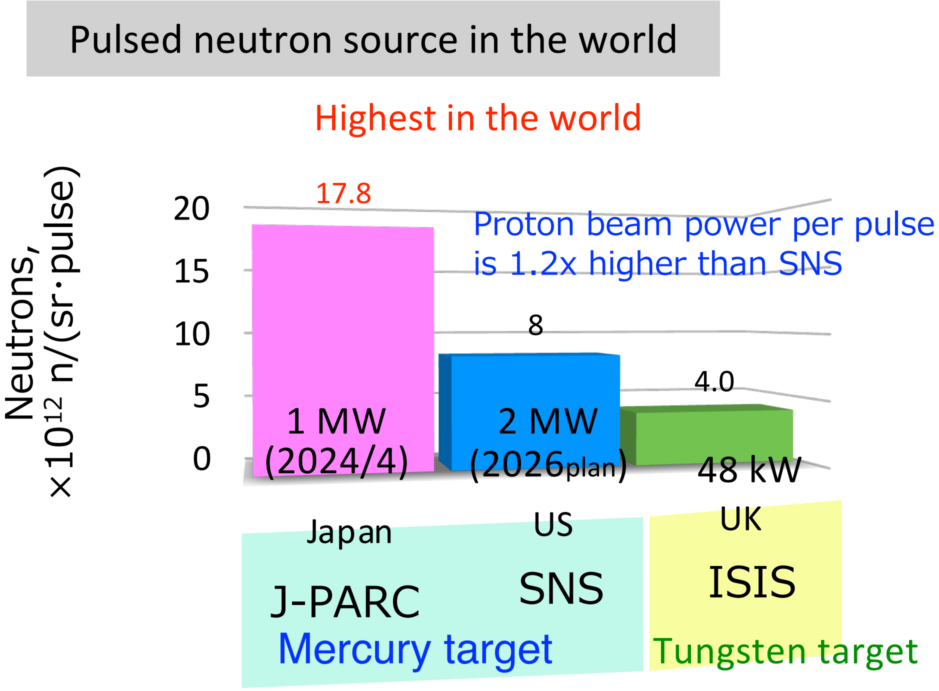
3. SS 316L for neutron target

Materials are supplied by T. Naoe

J-PARC pulsed spallation neutron source



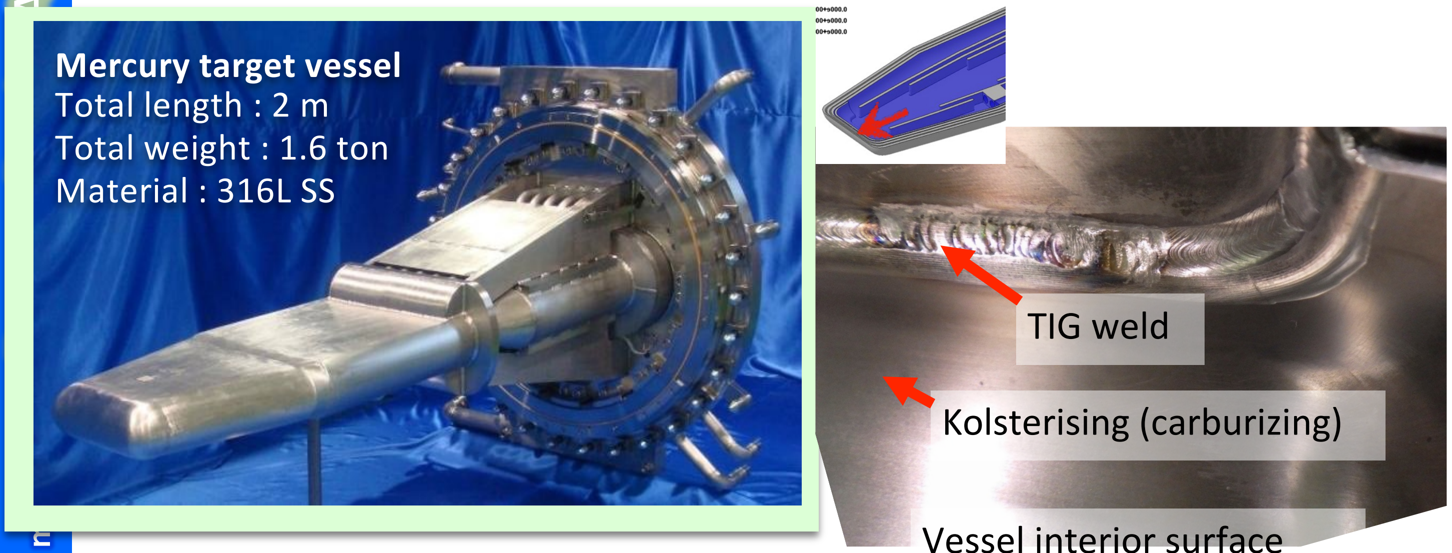


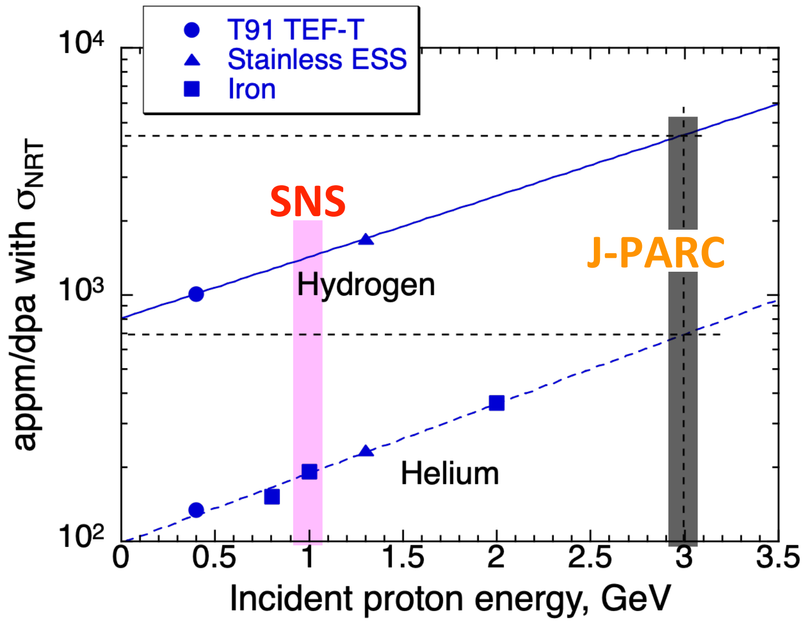


- Liquid mercury target
  Cooling performance, Neutron yield
- Pulsed proton beam
  1 MW, 25 Hz, 18x10^{12} n/(sr·pulse)
- Provides pulsed neutron beams to beam lines, operate 156 days/year and 1000 users/year

Stable operation is strongly required.

Mercury target vessel for spallation neutron source





Y. Iwamoto, et al., J. Nucl. Sci. Technol. 51 (2013)

- Target vessel which embrace liquid mercury target is made of 316L stainless steels with TIG welding
- Interior surface of the vessel was hardened by Kolsterising a kind of low temperature carburizing to reduce the cavitation erosion caused by beam induced pressure waves
- Total dose for 2 years operation is planned 7dpa

- Beam energy of J-PARC (3 GeV) is ca.3x higher than that of the SNS (1 GeV), which has the excellent PIE data
- Higher proton beam energy >> higher Hydrogen and Helium production
- Since the effects of gas production on mechanical properties are unclear, PIE for 3 GeV irradiated materials are required but much difficulties for PIE are remaining in J-PARC
- Effect of gas production on mechanical properties is evaluating by ion irradiation with indentation technique

Materials and conditions for ion irradiation tests

Base material: SS 316L (structural material for target vessel)

wt.%	C	Si	Mn	P	S	Ni	Cr	Mo		Fe
SS 316L	0.022	0.26	1.42	0.03	<0.001	10.1	16	2.02		Bal.

Specimen

SS 316L, cross sections of welded 316L and Kolsterising
W2 x L7 x t 0.7 mm

Temperature

200degC (max temp. on vessel at 1MW operation)

Conditions

HIT@Univ. Tokyo

Single: Fe^{2+}, 2.5 MeV, 2.5 nA,
1, 3, 5, 7, 10 dpa

Dual(planned): 5 dpa and various He appm

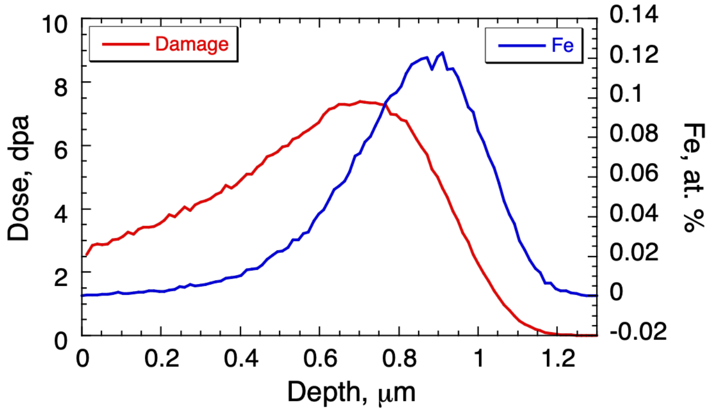
TIARA@QST Takasaki

Triple: Ni^{3+}, 12 MeV, 28 nA 5.4 dpa
H^+ 0.38 MeV, 3.5 nA 4800 appm
He^+ 1.05 MeV, 8.0 nA 1000 appm

Electron beam welded 316L

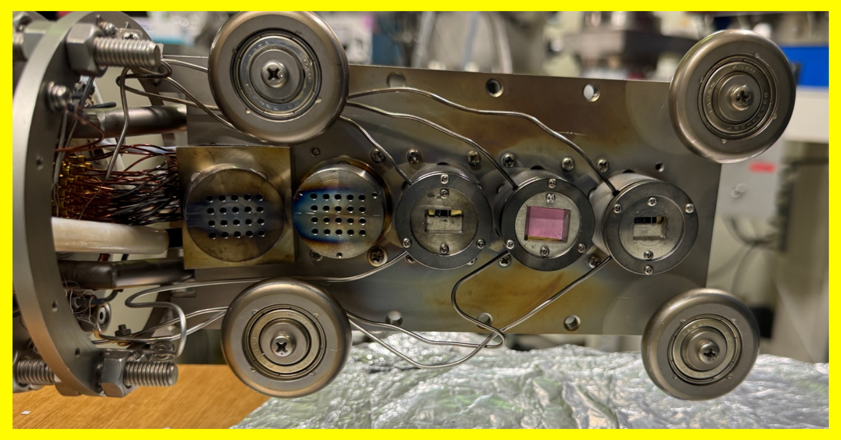
$$H_u = \frac{L_{max}}{26.43 d_{max}^2}$$

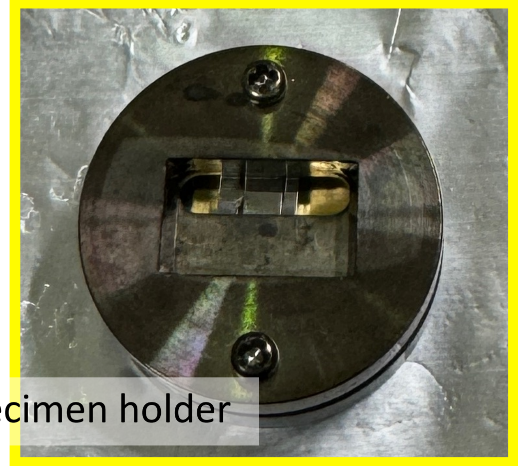
Hu: Universal hardness [GPa]
(HMT:Martens hardness [N/mm²])



Calculate damage distribution for HIT 7 dpa experiment

Hardness after ion irradiation at HIT





Specimen holder

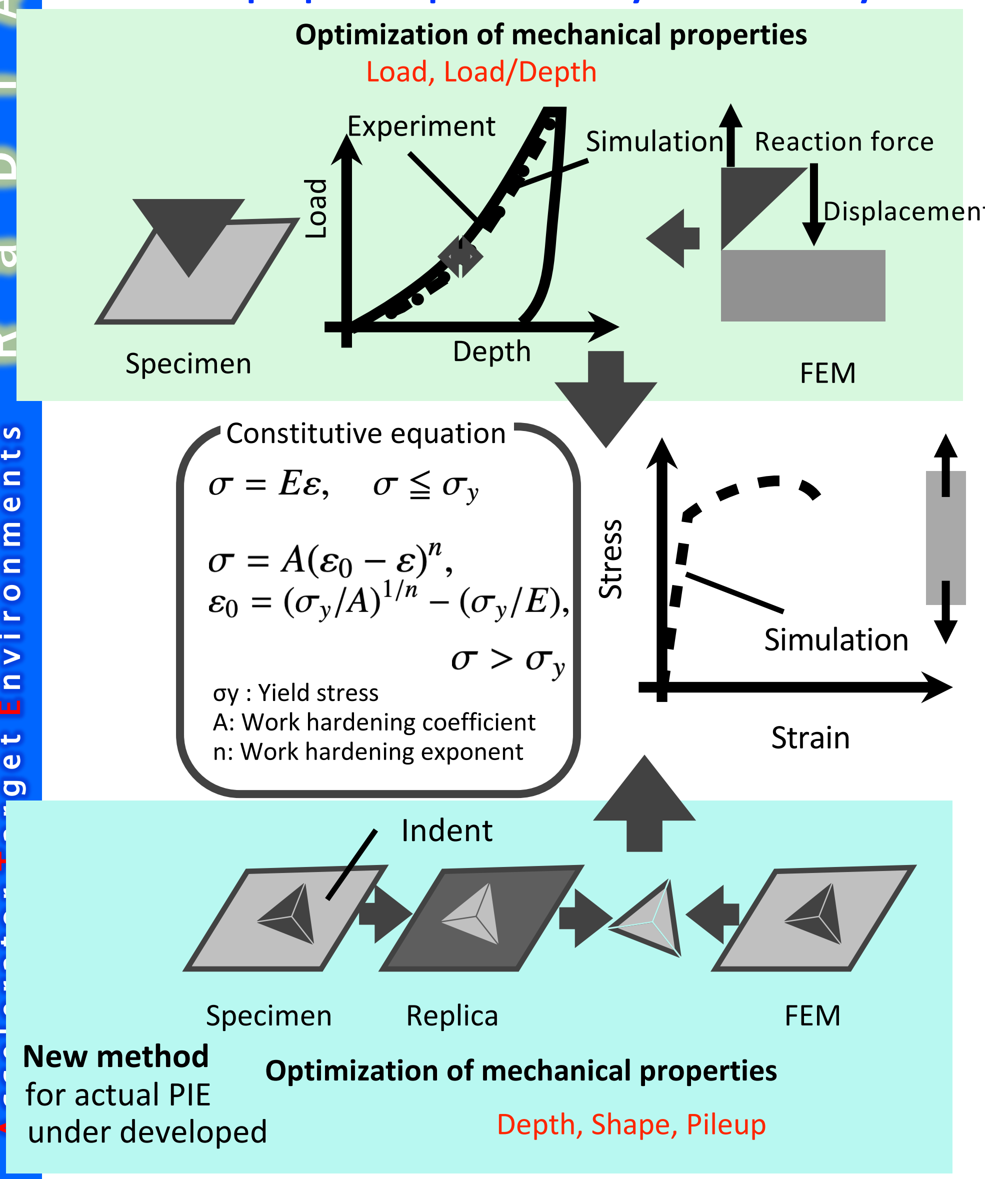
深さ0.15での試験は，HITで普段　やっている条件
30mNは，中性子源でこれまで他の照射試験でデータを蓄積している条件

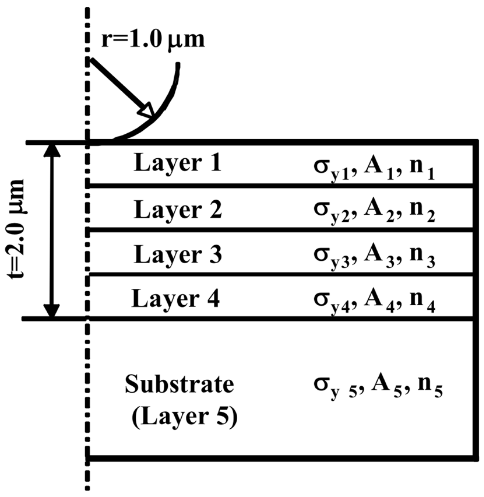
荷重/深さ-深さの傾きは硬度と相関があり，変曲点以前の傾きから表層のみの硬度を評価できる

- Surface hardness change was measured using Bekovich indenter by depth control (0.15μm) and load control (30 mN)
- Planning to obtain L-D curves by spherical indenter for inverse analysis to predict mechanical properties from indentation

Data analysis and future plan

Mechanical properties prediction by inverse analysis





Inverse analysis for material properties prediction

- T. Wakui, et al., J. Soc. Mat. Sci., Japan 51 (2002)
- T. Naoe, et al., J. JSEM, 5 (2005)
- M. Futakawa, et al., J. JSEM, 4 (2004)
- Naoe, et al., Int. J. JSME A, 48 (2005)

Multilayer model for ion irradiation

- Compare the experimentally obtained load and depth curve and FEM result, and optimize material properties in the constitutive equation for FEM model by Kalman's filter with iterative simulation to close the experimental result
- Multilayer model can be adopted to consider the thin surface layer hardness change by ion irradiation
- Spherical indenter which continuously changes the contact angle to the specimen surface is used for this method, but now procedure for Berkovich indenter is under developing for actual PIE test in Hot-cell

4. Superconductor for magnets

Materials are supplied by M. Yoshida

RaDIATE

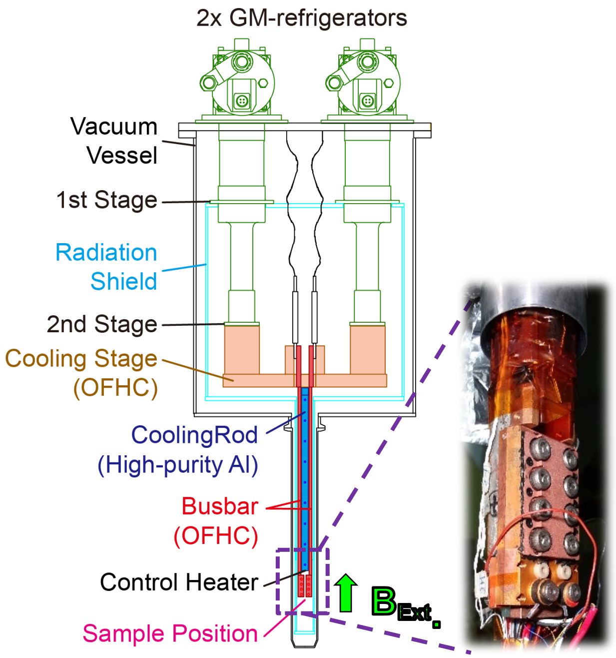
Radiation Damage In Accelerator Target Environments

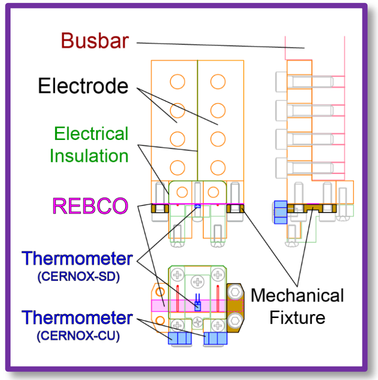
Neutron Irradiation Tests on ReBCO conductor

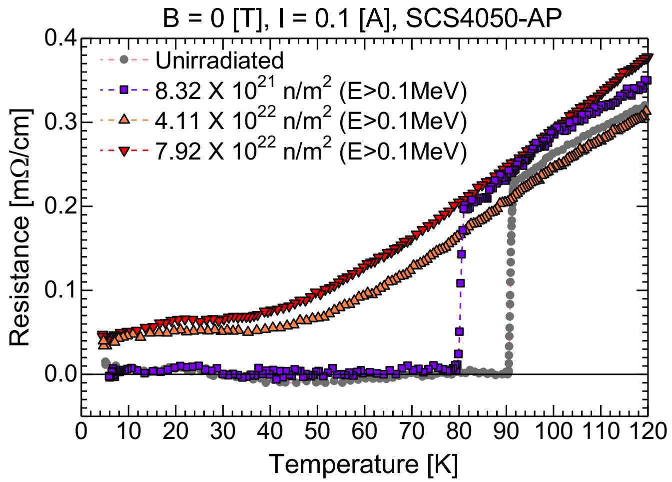
M. Yoshida, M. Iio and J-PARC Cryogenics Section

- Aim to characterize the neutron irradiation effects of the practical high temperature superconductor, ReBCO
- Neutron irradiation at JRR3 and BR2 reactor is performed under the GIMRT program of the IMR, Tohoku Univ.
- PIE with an external field up to 15.5 T at IMR-Oarai.

- Degradation of GdBCO was observed at around 10^{22} n/m^2
- YBCO and other samples irradiated with lower fluence of 10^{21} n/m^2 will be investigated.





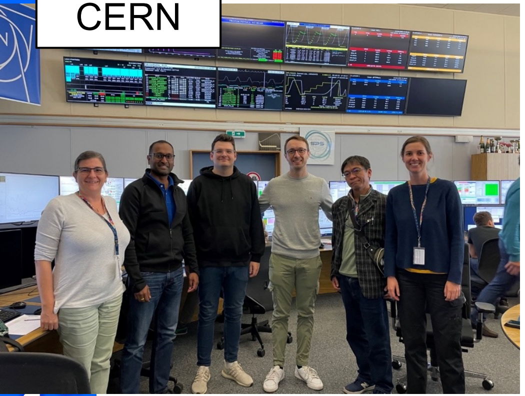


M. Iio, M. Yoshida, T. Nakamoto, T. Ogitsu, M. Sugano, K. Suzuki, and A. Idesaki, "Investigation of Irradiation Effect on REBCO Coated Conductors for Future Radiation-Resistant Magnet Applications,"IEEE Trans. Appl. Supercond., vol. 20, no. 6, Sep. 2022, Art. no. 6601905.

RaDIATE

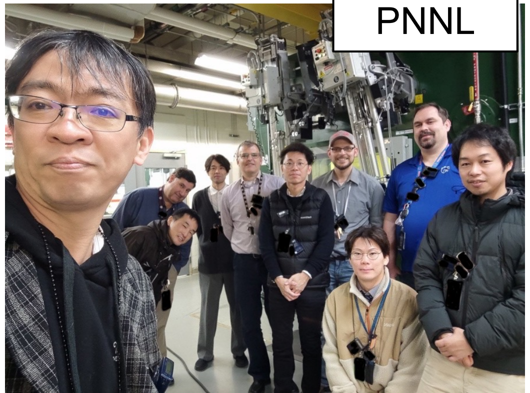
Summary

- J-PARC RaDIATE is organized to tackle radiation damage studies for each experimental facility.
- Investigation in Tungsten alloy, titanium alloy, SS316L, superconductor, and other materials are in progress.
- Your collaboration is welcome.


CERN


UK-RAL

Thanks for your attention.


PNNL


FNAL


BNL

Radiation Accelerator Tar