DEMANDS FOR THERMAL SHOCK EXPERIMENTS OF PULSED-MUON-PRODUCTION TARGET MATERIALS

HIRADMAT WORKSHOP AT CERN

10TH JULY, 2019

J-PARC CENTER, MLF DIVISION, MUON SECTION

HIGH ENERGY ACCELERATOR RESEARCH ORGANIZATION, INSTITUTE OF MATERIAL AND STRUCTURAL SCIENCE (IMSS, KEK)

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2. Muon Target at J-PARC
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   - SiC-coated graphite
   - NITE SiC/SiC
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4. Summary and
   Expect to HiRadMat facility

- J-PARC, Muon

S. Makimura, N. Kawamura, S. Matoba, T. Yamazaki, K. Shimomura, and Muon Section

- J-PARC

T. Ishida, E. Wakai, and J-PARC

- NITE-SiC/SiC

N. Nakazato, H. Kishimoto, J.S. Park, and Muroran Institute of Technology
A. Sato, D. Tomono, K. Ninomiya, M. Aoki and Osaka University

- TFGR W-TiC, tungsten alloy

H. Kurishita, K. Niikura, H.C. Jung, M. Onoi, H. Ishizaki, and Metal Technology Co., LTD

- HiRadMat

M. Calviani, I. L. Garcia, C. Torregrosa, J. B. Descarrega, E. Fornasiere and CERN collaboration

- Irradiation, PIE, and SiC-coated graphite

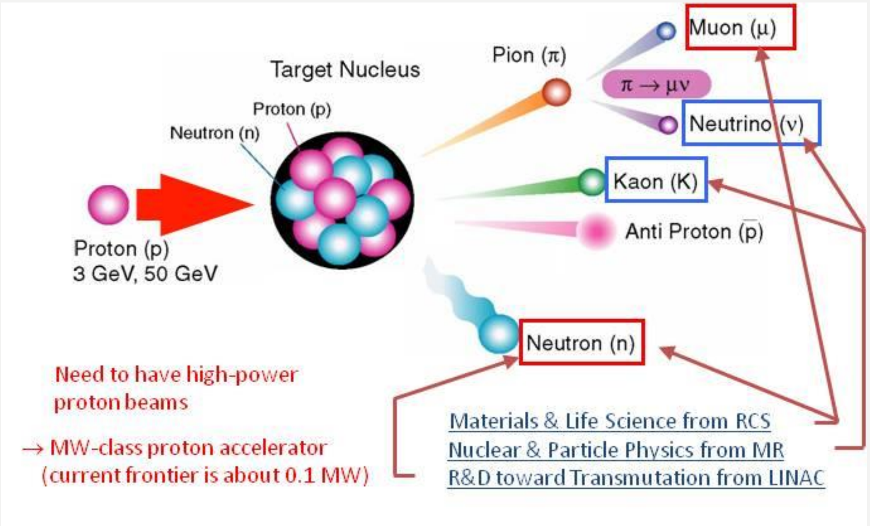
P. Hurh, K. Ammigan, N. Simos, D. Senor, A. Casella, and RaDAITE collaboration

MUON SCIENCE & PULSED MUON

MUON

- Muon is classified with a lepton.
- It is one of the most popular cosmic rays
- A cosmic muon reach on your palm in a second.
- μ+: like the isotopes of hydrogen
- μ -: 200 times larger mass than electron

- A pion decays into a muon and a neutrino.
- The decay of the positive muon into a positron and two neutrinos occurs after a mean lifetime of 2.2 μsec



TOPICS EXPLORED BY MUON VARIOUS & UNIQUE SCIENCE

Material and Life Science (μ+)

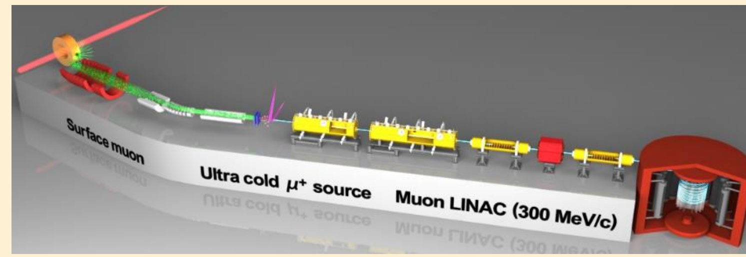
Superconductor Hydrogen in matter
Ion diffusion Biomaterial etc. etc.

by μSR

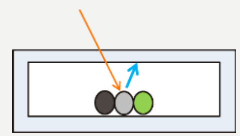
For ex.
Many useful semiconductors (GaN, TiO_2 etc.) exhibit n type conductivity. Why?
↓ Muonium ~ Hydrogen
Hydrogen impurity plays as a donor.

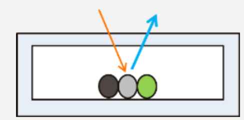
Fundamental Physics

Muon g-2, Mu HFS, μ-e conversion etc.



Electron



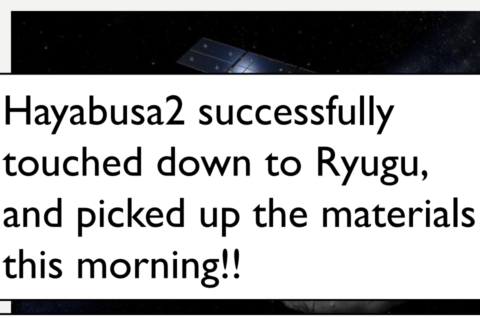


Muon

Sample from Ryugu can be kept in a vacuum

Nondestructive Element Analysis (μ-)

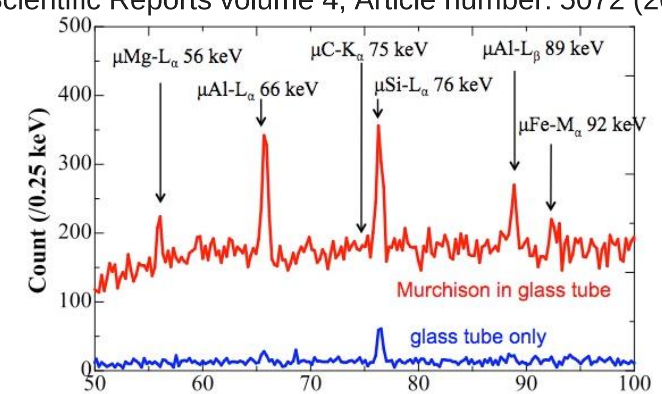
JAXA, University of Tokyo and collaborators



Hayabusa2 successfully touched down to Ryugu, and picked up the materials this morning!!

Asteroid explorer~Hayabusa2 (JAXA)

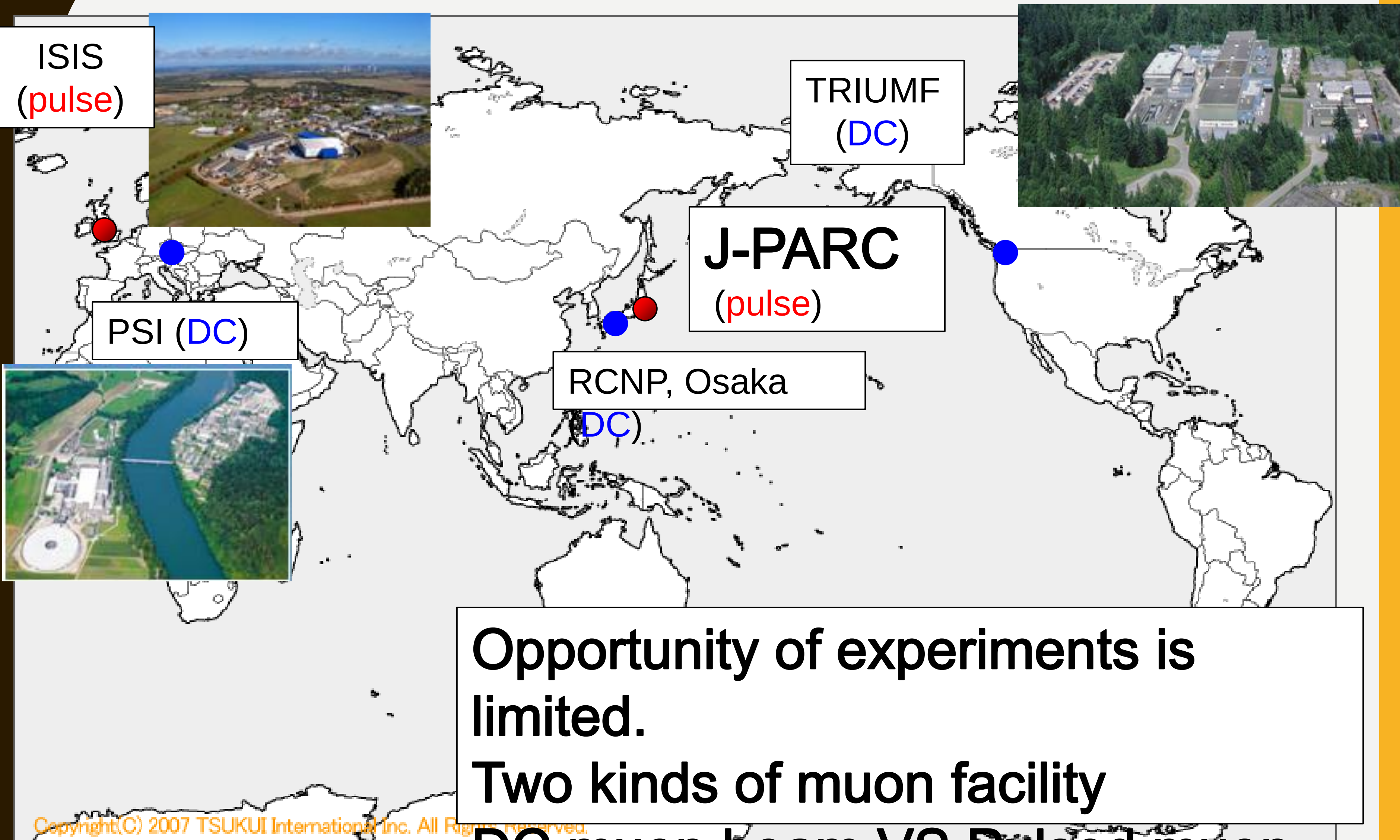
Scientific Reports volume 4, Article number: 5072 (2014)



Characteristic Muonic X-ray ~ Higher transmittance by heavy mass of negative muon

The scheme was successfully validated at J-PARC MLF

MUON FACILITIES IN THE WORLD

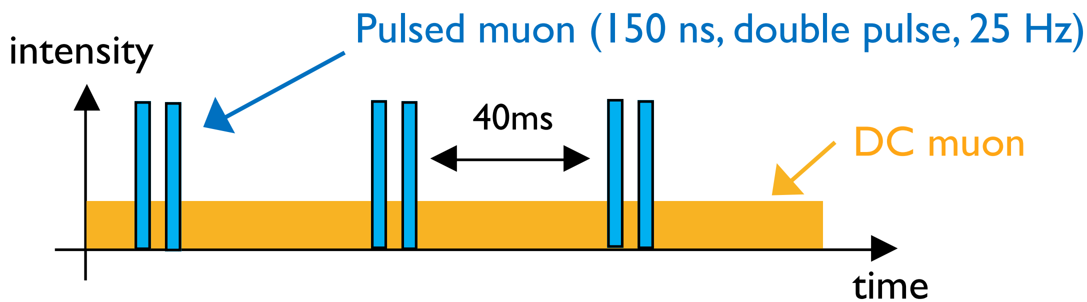


Opportunity of experiments is limited.
Two kinds of muon facility
DC muon beam VS Pulsed muon beam

CONTINUOUS DC MUON VS. PULSED MUON

Continuous DC muon

- Fine time resolution – Sub-ps order
  Fast muon spin depolarization
  High frequency (field) muon spin rotation is observable.
- Low peak intensity is beneficial to avoid saturation of the positron detectors.



Pulsed muon

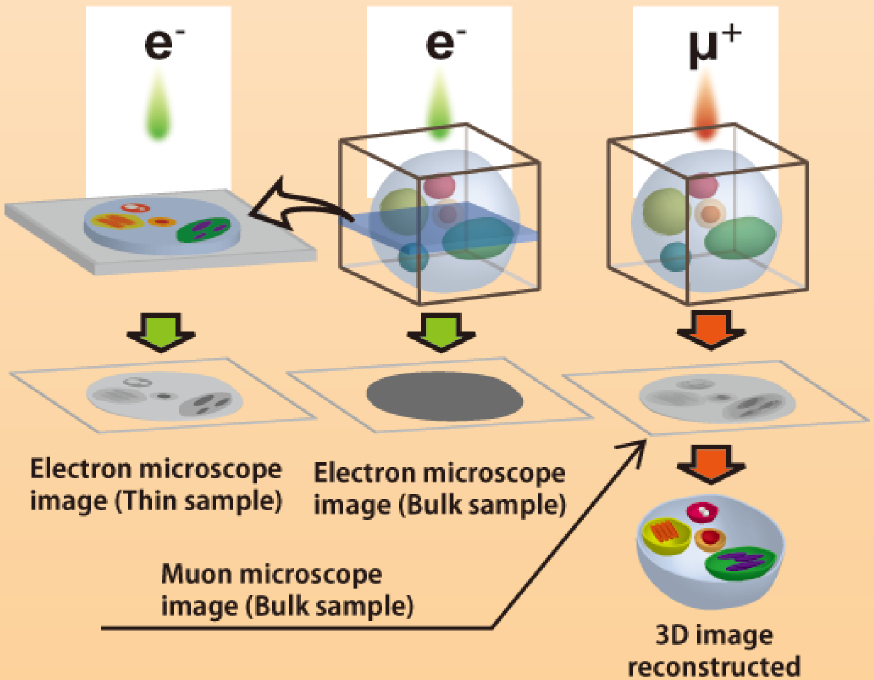
- Pulsed muon can be used with synchronized pulsed devices.
  e.g. laser resonant ionization of muonium
- Synchronizing measurement with muon incident can be conducted to reduce background

Pulsed muon and DC muon are complemental.
Demands for further peak intensity of pulsed muon!!

UNIQUE SCIENCE BY PULSED MUON 1

Transmission Muon Microscope

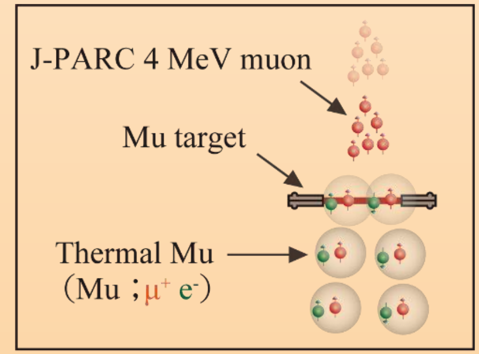
(Stage of feasibility study)



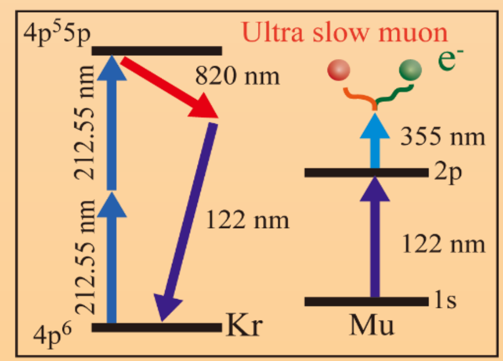
Electron Microscope can observe sample less than 1μm thickness.
Muon Microscope can observe 200 times thicker samples,
because of 200 times heavier mass,
enabling us to observe alive cell through a window!

Applying re-accelerated high-quality muons (Narrow time and energy width)

Ultra slow muon generation laser resonant ionization of Mu



Mu generator



Lyman-α laser generation and Mu dissociation by laser resonant ionizat method

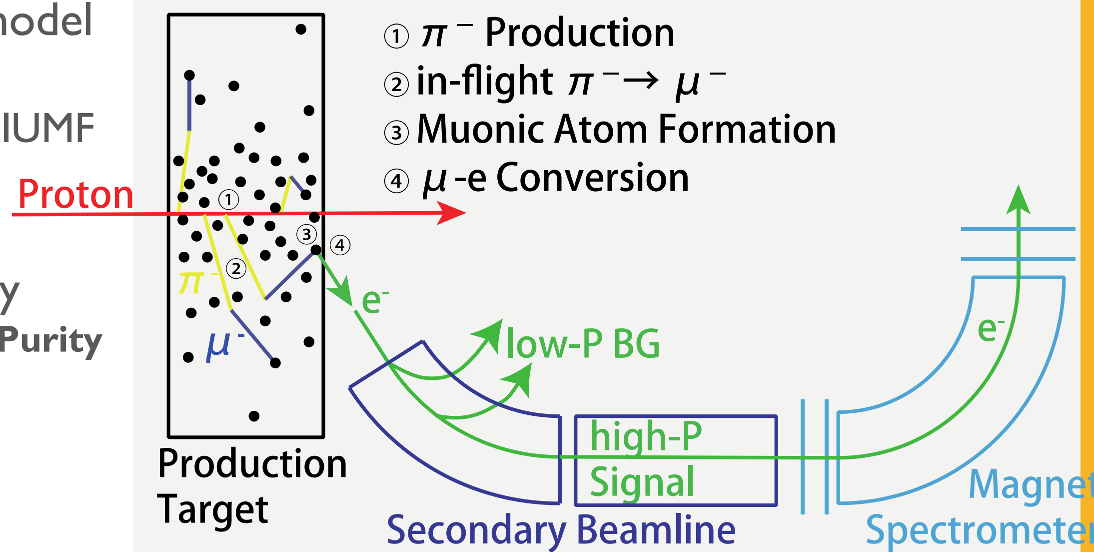
To obtain higher-quality muon beam, higher peak intensity of muon synchronized with laser is essential.

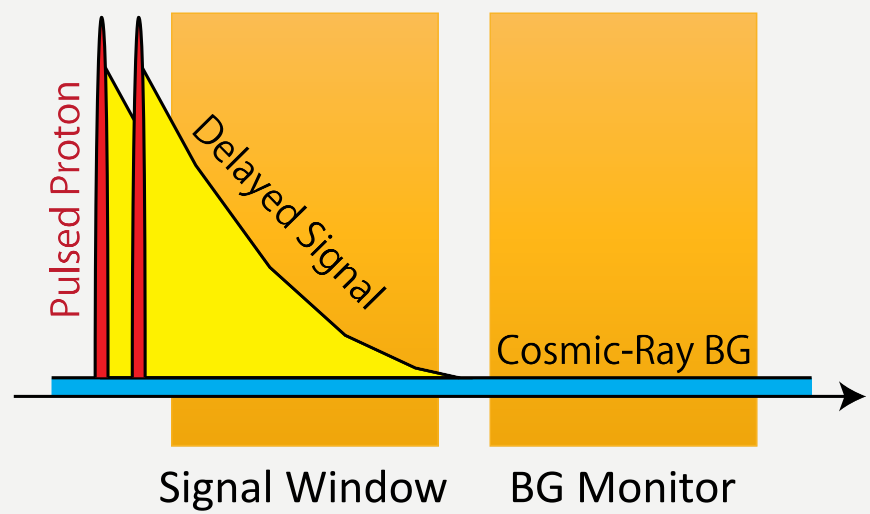
UNIQUE SCIENCE BY PULSED MUON 2 DEEME: M-E CONVERSION SEARCH

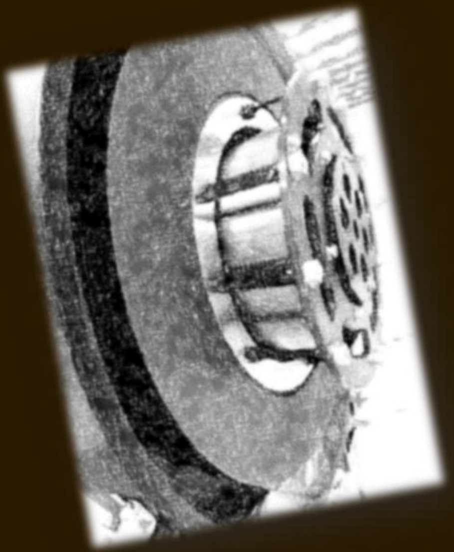
- Prohibited phenomena in standard model
- Current Upper Limits:
  - BR[μ^- Ti→ e^- Ti] < 4.6 × 10^{-12}@TRIUMF
  - BR[μ^- Au→ e^- Au] < 7 × 10^{-13}@PSI
- DeeMe @ J-PARC MLF Muon Facility

  RCS: World-leading High-Power High-Purity Pulsed Proton Beam
  - Novel Idea: μ-e Conversion in the primary production target itself
  - S.E.S: 1×10^{-13}(Graphite Target), 5×10^{-15}(SiC Target)
- Pulsed Proton Beam is a Key:
  - To reduce Prompt Backgrounds
  - To reduce/monitor Cosmic-Ray induced Backgrounds

Pulsed Proton Beam with SiC target realizes high sensitivity.



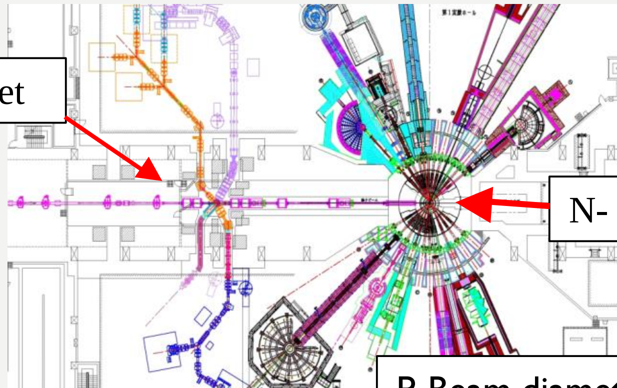


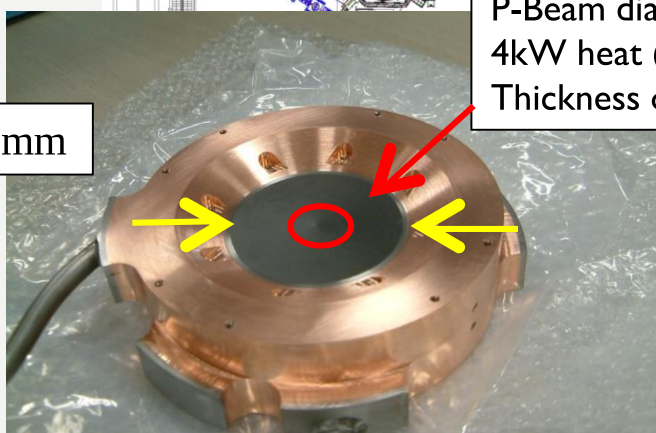


MUON TARGET AT J-PARC

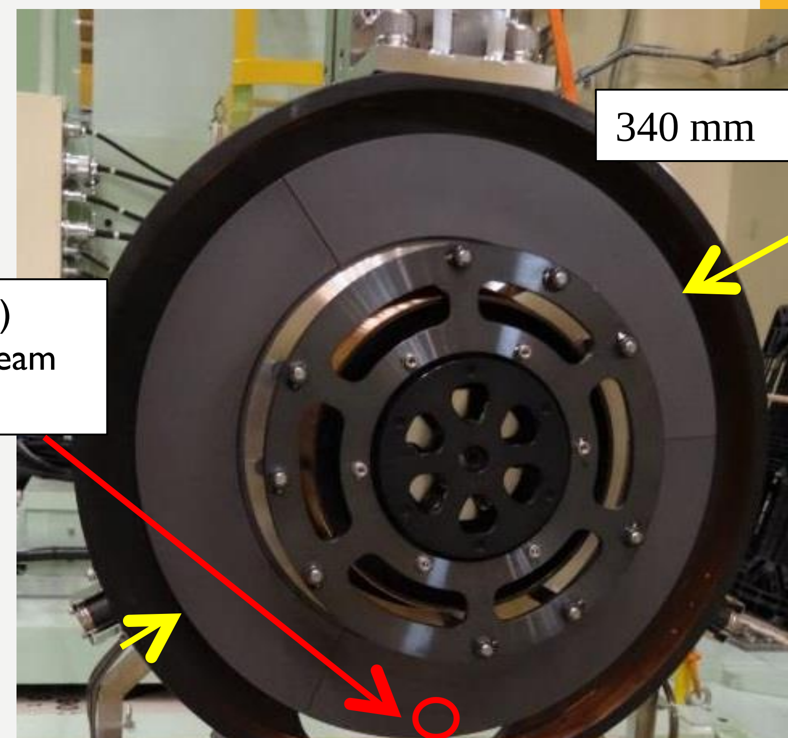
CURRENT MUON TARGET

- Target material is polycrystalline graphite, IG-430U.
- Fixed target was replaced with rotating target to disperse the radiation damage of graphite.





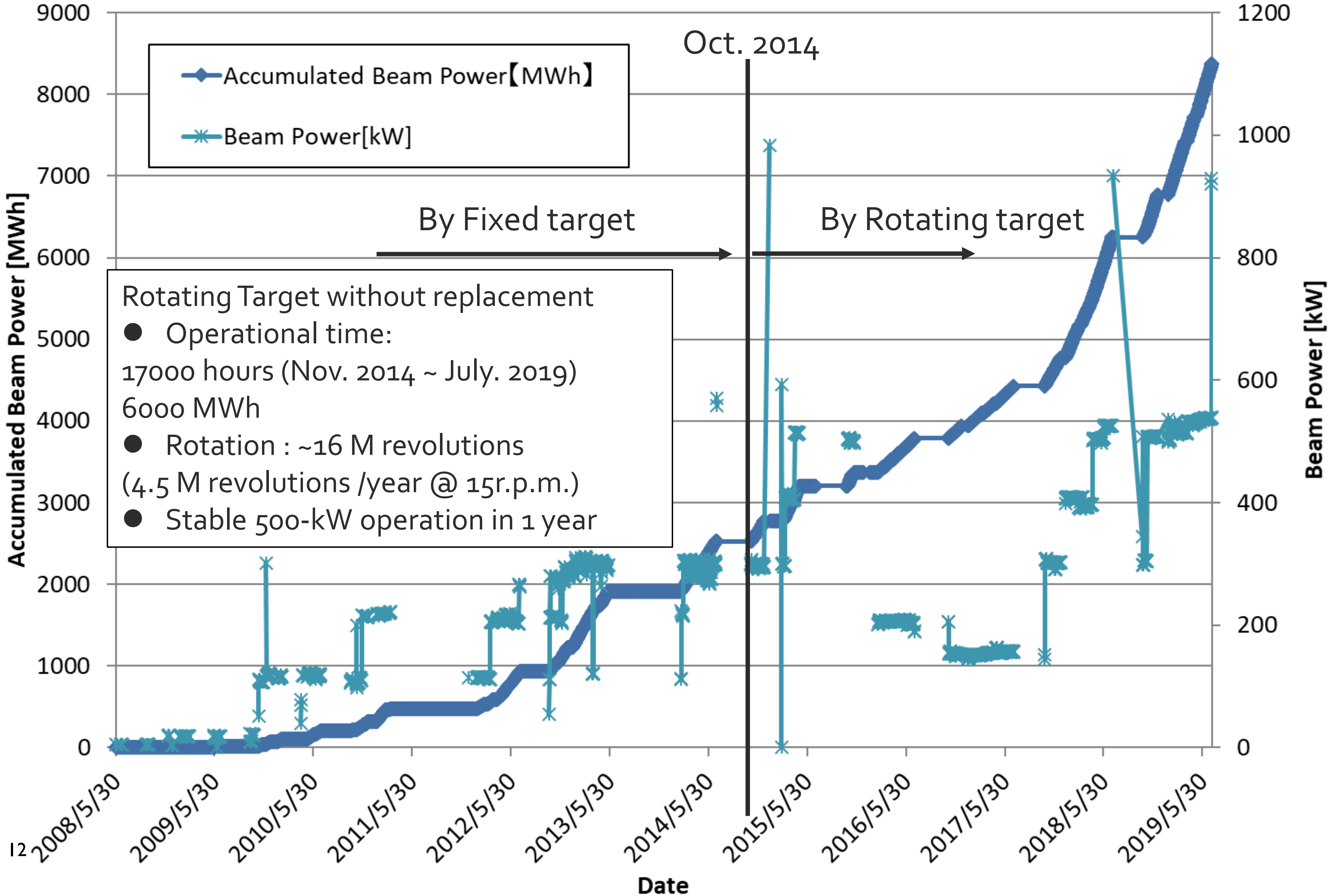
P-Beam diameter; 14 mm (2σ)
4kW heat @ 1MW proton beam
Thickness of graphite 20 mm



Fixed target, from 2008 to 2014
Water-cooling by thermal conduction
Lifetime: Irradiation damage of graphite
1 year at 1 MW operation

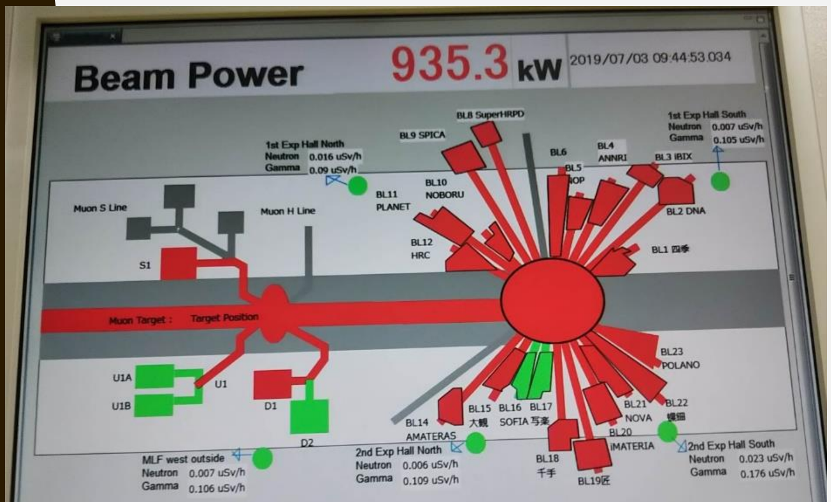
Rotating target, from 2014
Cooling by thermal radiation
Lifetime: Bearings
Aiming Lifetime: 10 years at 1 MW operation

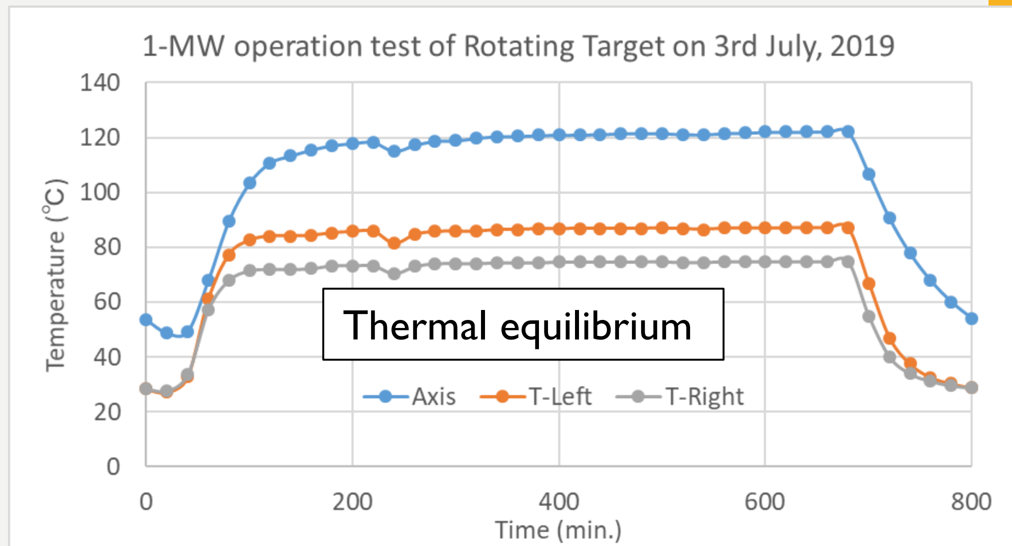
HISTORY OF BEAM OPERATION

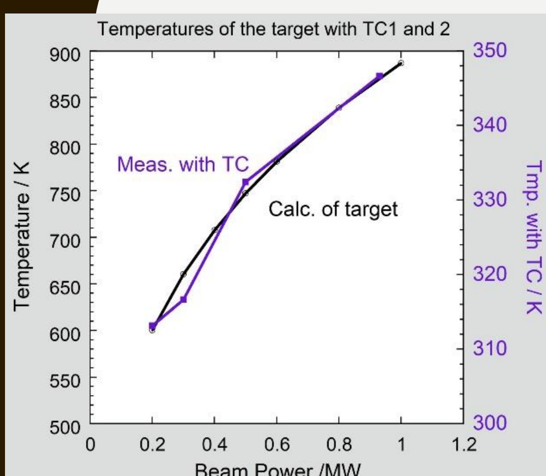


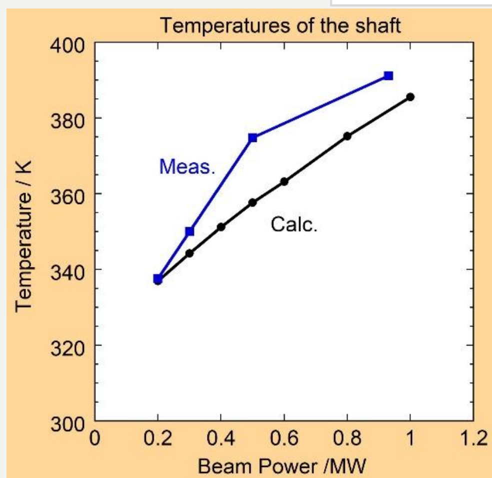
ALMOST 1-MW OPERATION TEST

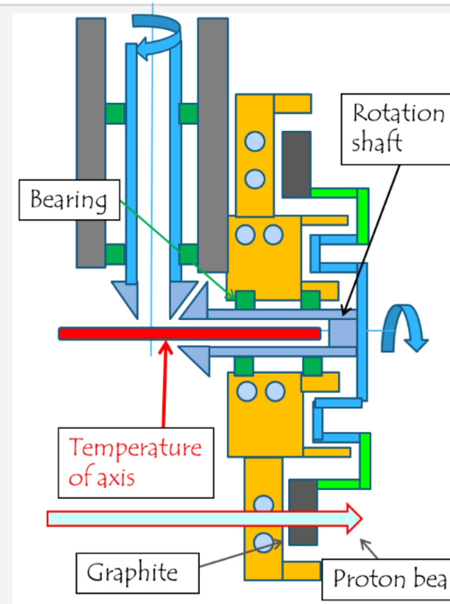
The 1-MW operation for 11 hours was successfully completed on 3rd July, 2019

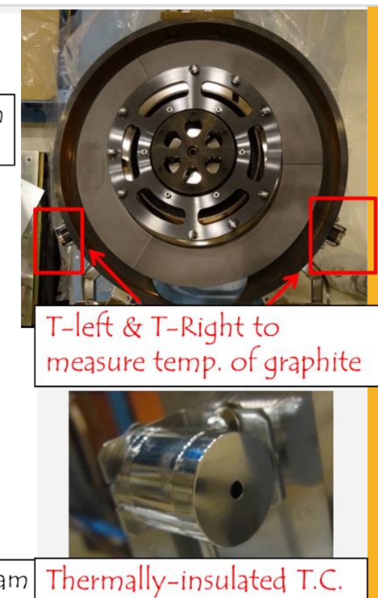










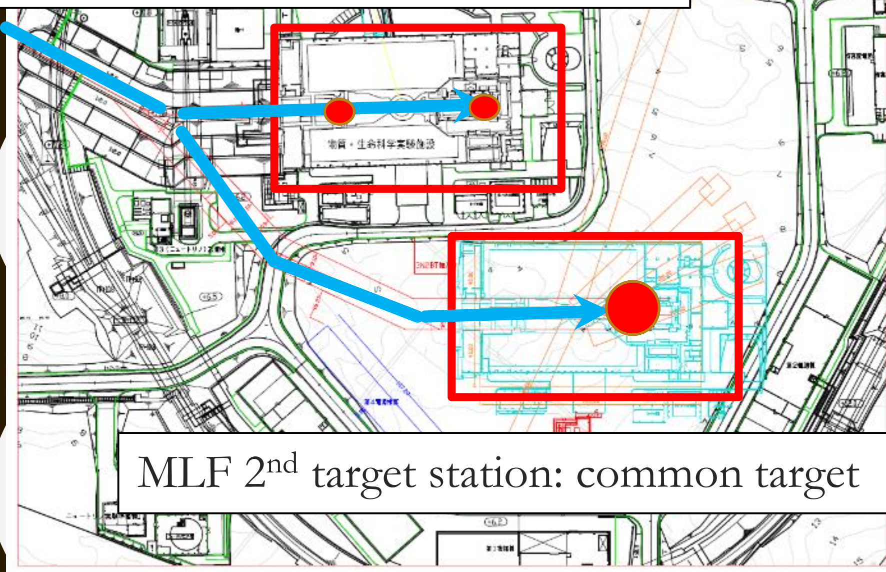


The results were in good agreement with predictions from the simulations.

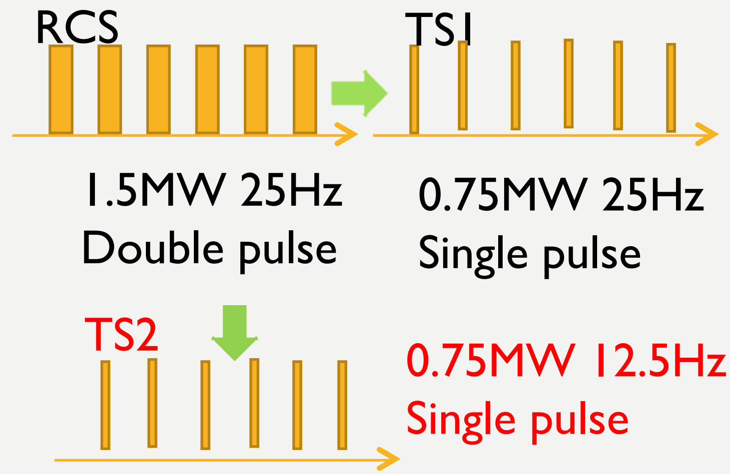
MLF 2ND TARGET STATION (TS2)

PROPOSAL OF FUTURE PROJECT WAS SUBMITTED

Current MLF facility: tandem target



MLF 2nd target station: common target



One of the plans for time structure

--Science by higher peak intensity

Extreme Condition
(High pressure, High external field)
Muon Micro Beam : small sample

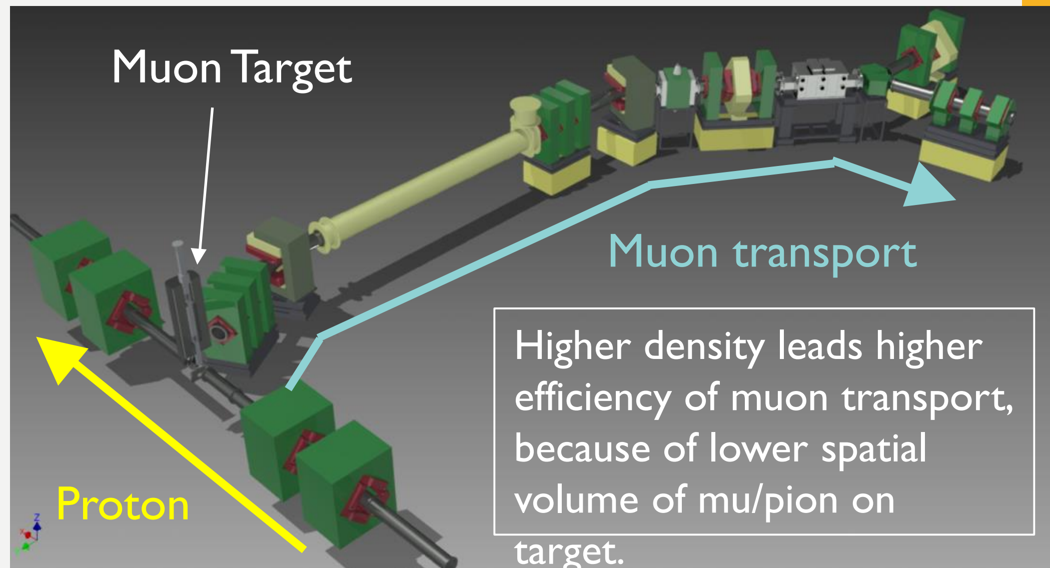
Bio Science （soft matter）
Muon Micro Beam : small protein crystal

Real Space, Real time （nonuniform system）
Muon Micro Beam, Stroboscopic measurement
Imaging, In situ, Operand, Element analysis
Industrial applications,

DEMANDS FOR NEW TARGET MATERIALS

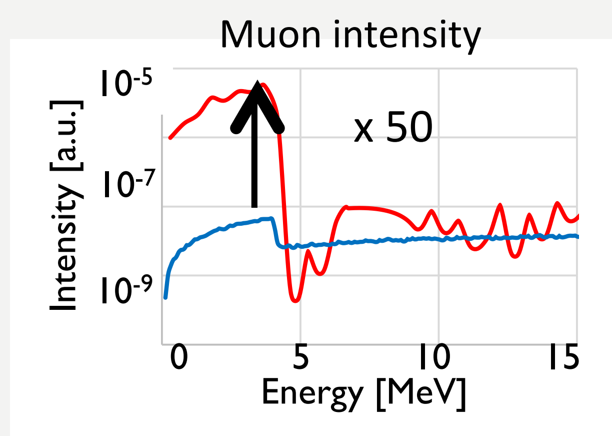
For MLF TS1:

- ☐ Graphite is the most popular and excellent target material.
- ☐ Demands for Graphite-substitute material
  - Higher oxidation resistance: SiC-coated graphite
  - Higher density: NITE SiC/SiC



For MLF TS2:

- ☐ Common target with Neutron target
- ☐ Tungsten is desired for higher efficiency of Muon and Neutron production.
- ☐ TFGR W-TiC tungsten: solution to recrystallize and irradiation embrittlement



TS1 vs TS2

	Density
	g/cc
W	19.3
Hg	13.5
Pb	11.4
PbBi	10.6
Fe	7.87

WHY HIRADMAT?

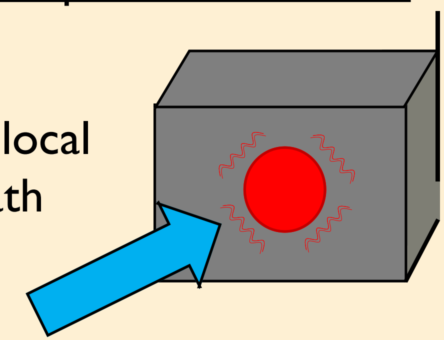
Rotating Target can disperse total heat density and radiation damage of target material. However,,,

Time width of proton beam for the pulsed muon is about 100 ns.
Too short to disperse the local heat density.
For application of new target materials,

Thermal shock resistance against H.E. proton beam is essential.

Unique aspect of H.E. proton irradiation

Thermal shock on local volume of beam path



Compressive stress *P* by thermal shock

$P \propto E\alpha\Delta T$

E; Young's modulus
α; Th. Expansion

* R.T.	Graphite	SiC	Tungsten
E (GPa)	11	440	411
α (ppm/K)	6	4.3	4.5

Graphite: Excellent thermal resistant material

Combination of proton-irradiation damage of materials must be considered as well.

HiRadMat is an unique facility to investigate thermal shock for higher peak intensity to new target materials.

SiC-coated graphite

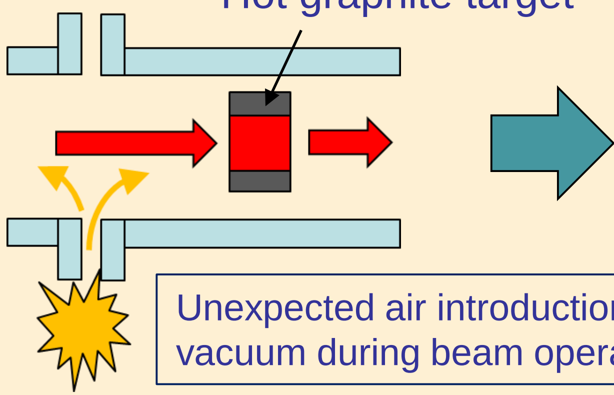
PREVIOUS RESEARCH AT HIRADMAT 1

Thanks to CERN collaboration
This program is supported by US-JP collaboration.

MOTIVATION OF SIC-COATED GRAPHITE

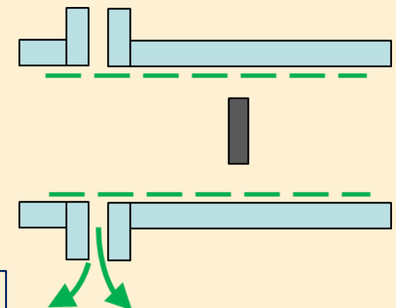
SiC-coating on graphite improves oxidation resistance.

In case of graphite target



Unexpected air introduction to vacuum during beam operation

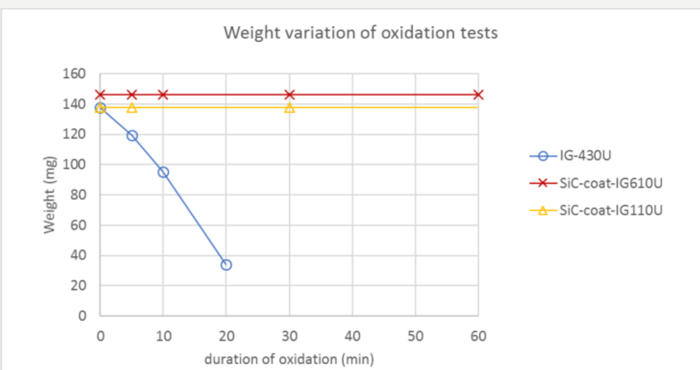
Graphite is easily oxidized at high temperature.



Graphite-oxidation contaminants complicate recovery.

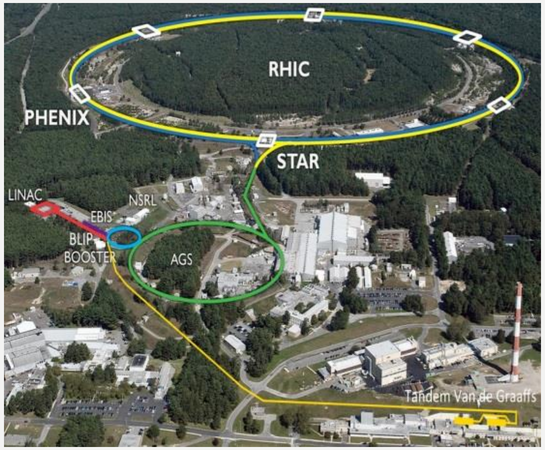
Oxidation tests of SiC-coated graphite

Much higher oxidation resistance

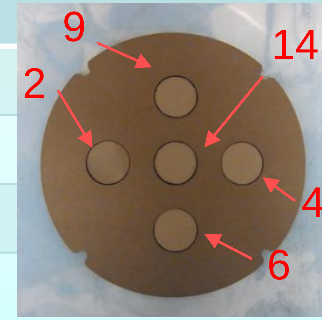


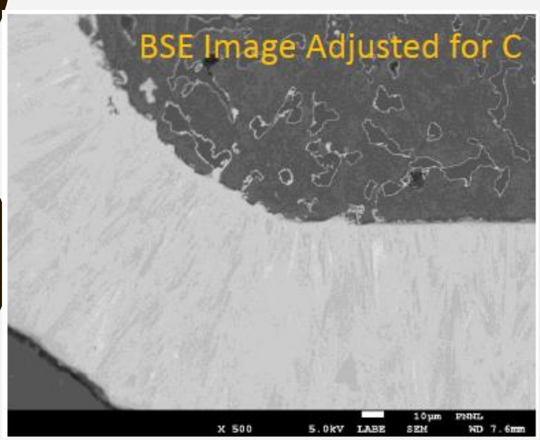
Volume loss was observed on graphite and not observed on SiC coated graphite.

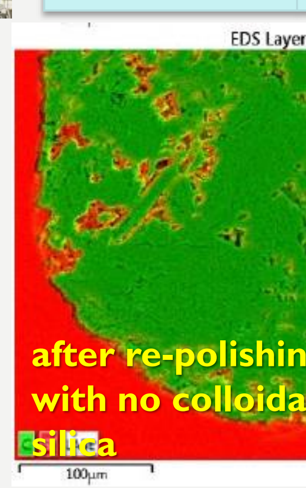
PROTON IRRADIATION AT BLIP, BNL AND PIE TESTS UNDER RADIATE COLLAB.



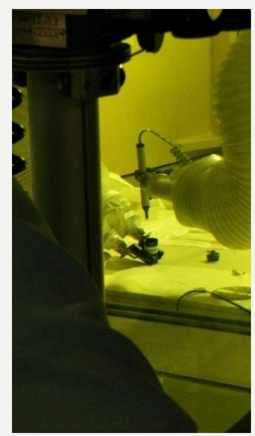
No.	Substrate Material	Crack	PIE (Newly revised, 14th March)
C-14 (Toyo-t)	IG-110U	Yes	SEM, TEM
C-9 (Toyo-t)	IG-110U	Yes	(TEM)
B-4 (Toyo-t)	IG-610U	No	HiRadMat
B-6 (Toyo-t)	IG-610U	No	SEM
No.2 (Ibiden)	ETU-10	No	SEM after TDS, 1300K TDS,







PIE testing at Pacific Northwest National Laboratory



SiC coated IG-110 were finally broken to pieces

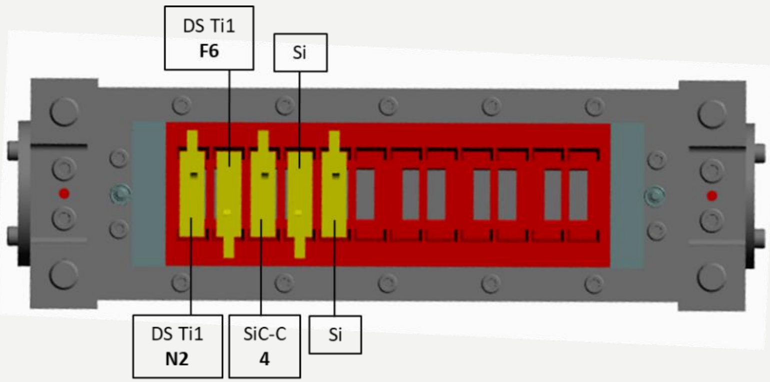
<u>SEM microscope analysis of cold specimens</u>
SiC coating deposited by CVD is intact.

<u>Visual check of irradiated specimens</u>
Crack was observed only on IG-110U substrate specimens, that was not designed for SiC coating

THERMAL SHOCK EXPERIMENTS AT HIRADMAT, HRT43

- SiC-coated graphite / Titanium Alloy (cold & irradiated at BLIP) exposed to thermal shock at HiRadMat on 1st Oct, 2018

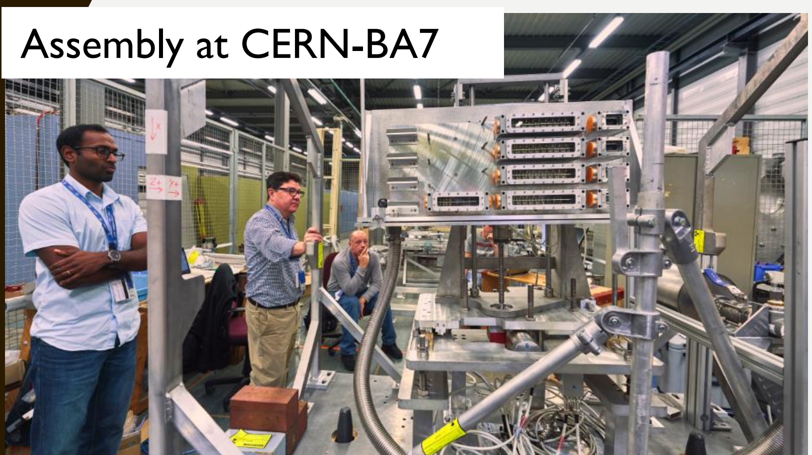
3 boxes with irradiated-specimens assembled at PNNL hot-cell (Exported to CERN)



BOX-3

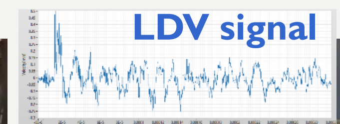
- Ti-6Al-4V Gr5
- Ti-3Al-2.5V Gr9
- SiC-coated Graphite IG-630U

Assembly at CERN-BA7



Sep.26

LDV signal



Oct.1(Mon)-2(Tue) 4:00am exposure completed !

- How did the SiC-coating on graphite behave ?
- ***How different between irradiated at BLIP / un-irradiated ?***

K.Ammigan, RaDIATE2018

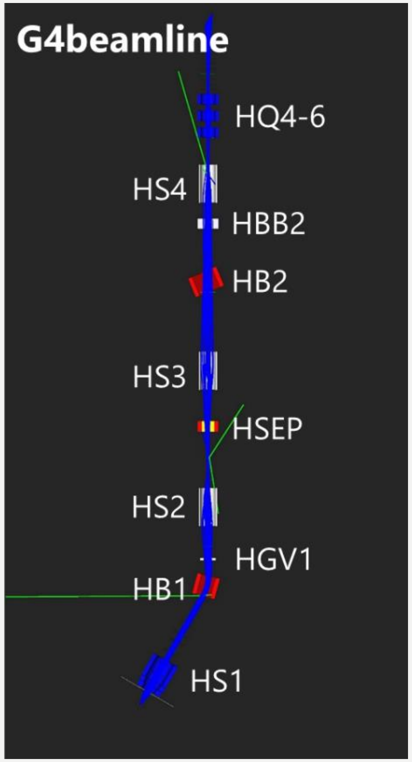
NITE SiC/SiC

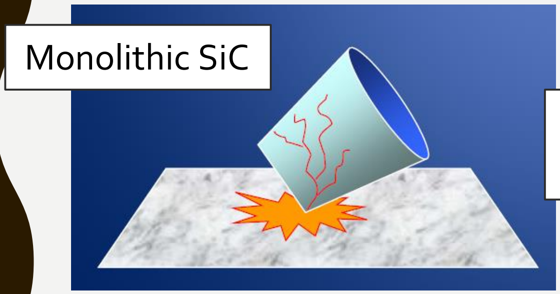
PREVIOUS RESEARCH AT HIRADMAT 2

Thanks to CERN collaboration
This program is supported by JSPS KAKENHI Grant Number JP16H03994.

MOTIVATION OF NITE SIC/SIC

- Higher transport efficiency by SiC target material, which is denser than graphite. (1.8 vs 3.2 g/cc)
- For DeeMe, 6 times larger efficiency can be expected.
- Beamline simulation for H line J-PARC, MLF through G4 beamline by T. Yamazaki
- 1.7 times larger surface-muon-yield at H-line experimental area.





Rate [1/s]

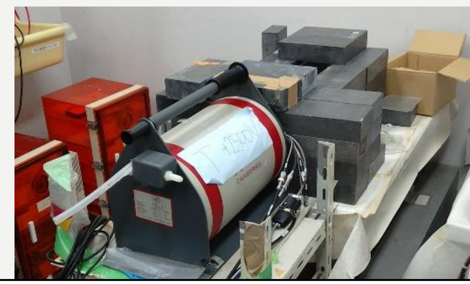
	Graphite ABLA	SiC ABLA
Proton 1MW	2e15	2e15
Surface μ (4π)	5.58e10	8.50e10
hs1ent	6.25e8	9.93e8
Focal point	1.62e8	2.75e8

- Monolithic Silicon Carbide cannot be used as target material, because it is brittle.
- NITE-SiC/SiC: developed at Muroran Institute of Technology for fusion material
- Pseudo-ductility to disturb the propagation of crack

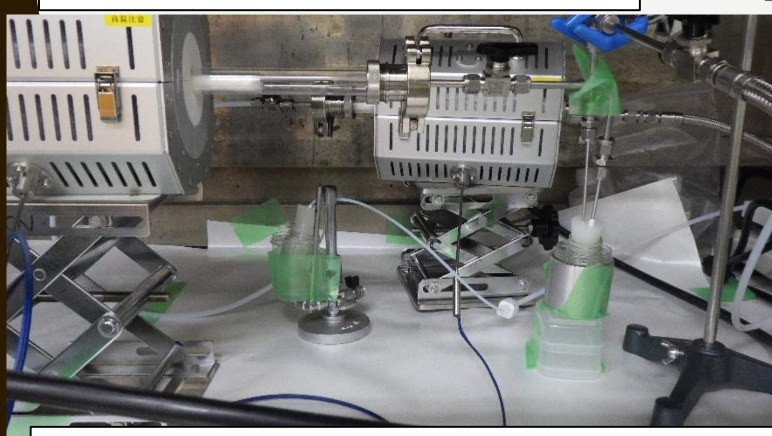
PROGRESS ON NITE SIC/SIC AS TARGET MATERIAL

- 392 MeV, 0.5 μA, 30 min. Proton-irradiation tests at RCNP, Osaka university
- Higher residual radiation dose (400 times higher dose than that of graphite after 1 year cooling)
- Tritium emission of NITE-SiC/SiC is completely different from CVD-SiC, probably, because of the effect of graphite matrix.

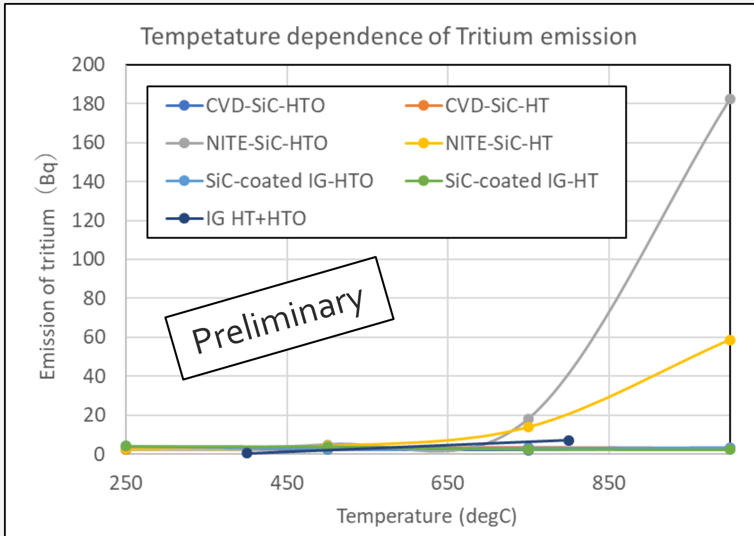
Sample holder at the irradiation test



Measurement of residual radionuclides for validation of simulation

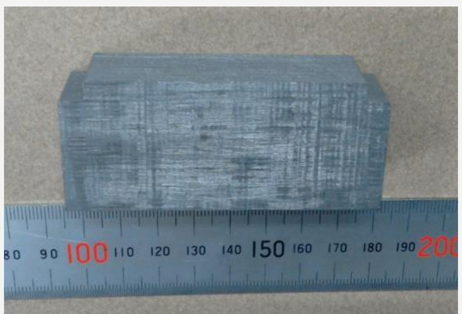


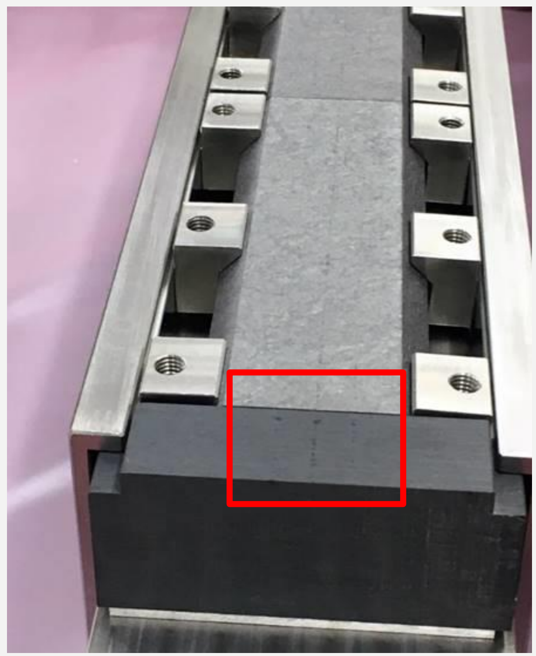
Two tube furnace and 3H traps, which can distinguish HT with HTO.



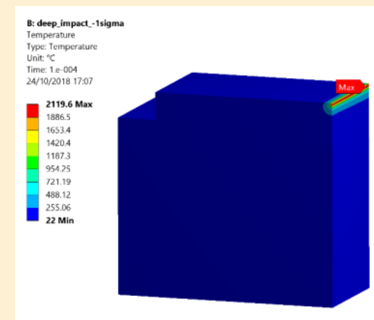
THERMAL SHOCK EXPERIMENTS AT HIRADMAT, HRT35

- To confirm the resistance to thermal shock resiatance
- NITE SiC/SiC was included in the experiments for coated low-Z absorbing materials for the Target Dump Internal in August 2017.
- PIE testing conducted by **J. Maestre C. Bahamonde and I. Lamas** (Results of PIE from Slides of IWSMT14 at Iwaki)

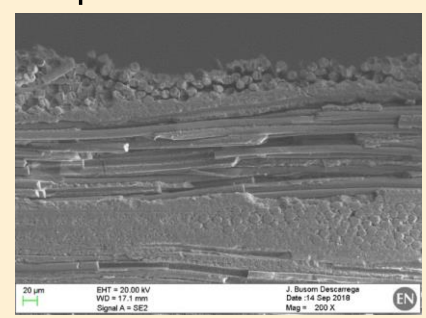




Stripe-like damage was observed.
440 GeV, 1.2E11 protons/pulse,
Gaussian, σ=0.3 mm.



Temperature rise: 2100 K



Results of SEM observation
(Top view, upstream, Deep impact)

Disturbance of crack propagation was observed.

TFGR W-TiC, tungsten

PREVIOUS RESEARCH AT HIRADMAT 3

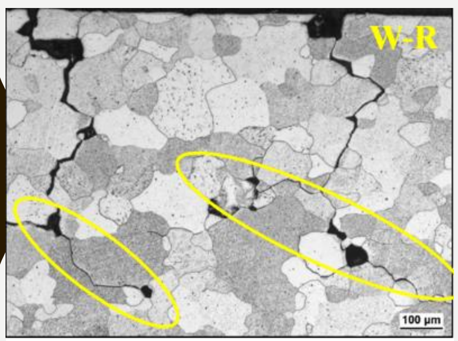
Thanks to CERN collaboration
This program is supported by MTC-KEK collaboration,
This program is supported by JSPS KAKENHI Grant Number 19H01913.

TUNGSTEN AS TARGET MATERIAL

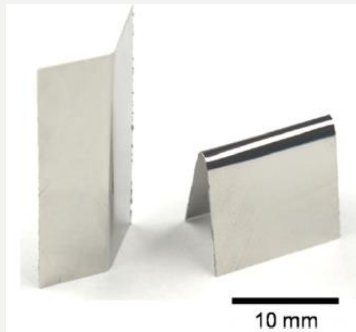
- Tungsten is expected as the target material.
- For use of tungsten as muon production target, the boundary between cooling material and vacuum in beamline disturb transportation of muon. Thermal radiation cooling is desired. It should be used at high temperature. (MLF TS2, COMET, mu2e,,,)

✓ Tungsten is brittle.

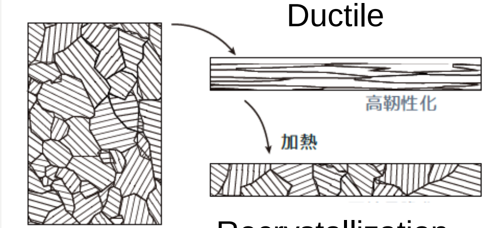
✓ Brittleness is improved by heavy plastic working.

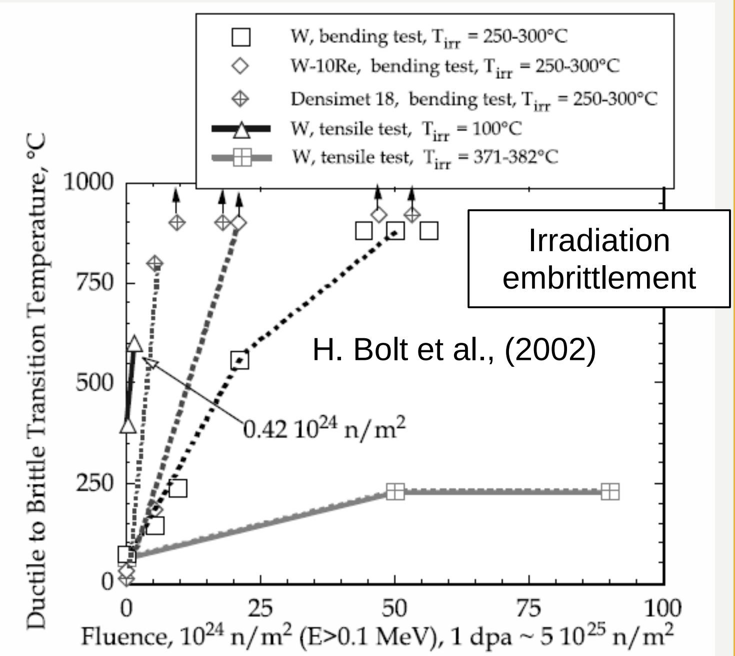


G. Pintsuk et al.



J.Reiser et al. JNM, 423 (2012) 1.



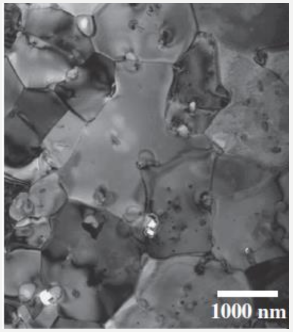


Ductile to Brittle Transition Temperature should be less than room temperature.

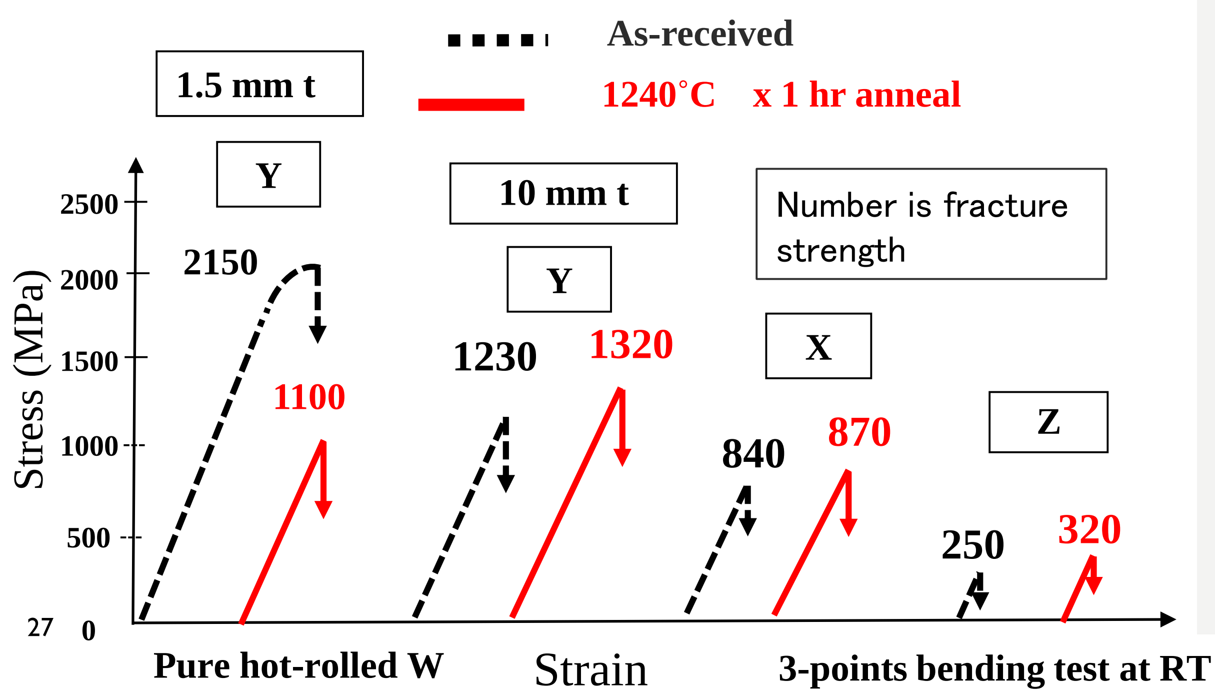
- The use of W as target material is limited by "recrystallization embrittlement" and "irradiation embrittlement".

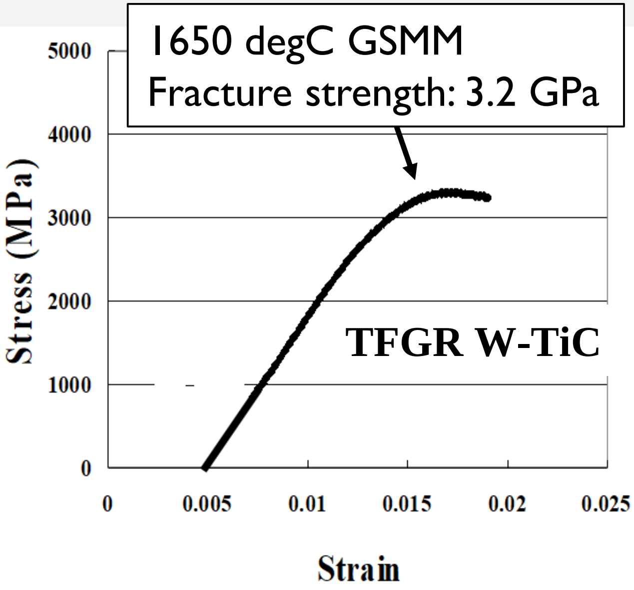
MOTIVATION OF TFGR W-TIC TUNGSTEN

- Toughened Fine-Grained Recrystallized W-TiC, (TFGR W-TiC) is developed by Prof. Kurishita at Tohoku University. Now the activities are transferred to KEK and Metal Technology Co. LTD collaboration.
  - ☐ Equiaxed, fine grains with TiC precipitates
  - ☐ GB reinforced by TiC enrichment
  - ☐ No recrystallization embrittlement
  - ☐ High sink density: Resistance to irradiation is anticipated.
  - ☐ DBTT (nil-ductility tem.) < RT



Mater. Trans. 54 (2013) 456-465.





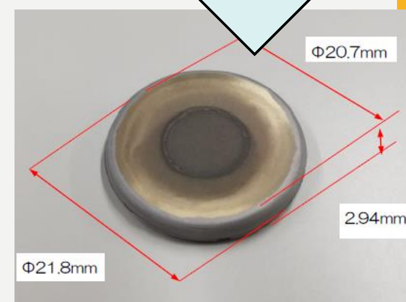
PROGRESS OF DEVELOPMENT FOR TFGR W-TIC

Purification of Powder

Mechanical alloying

Sintering

GSMM process



- The apparatuses for fabrication is ready.
- Revival of TFGR W-TiC is almost completed.

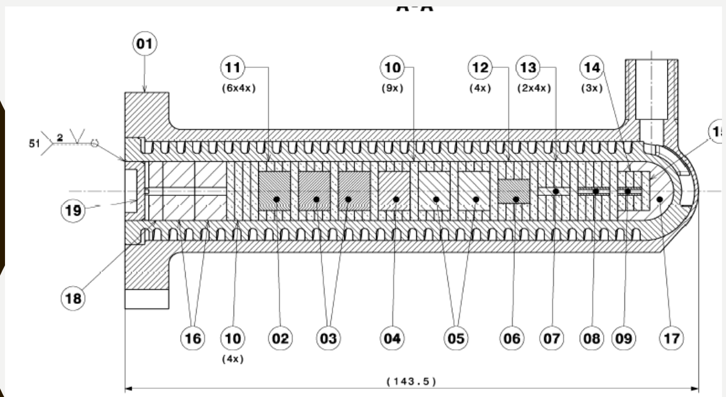
Future plans in up-coming three years:

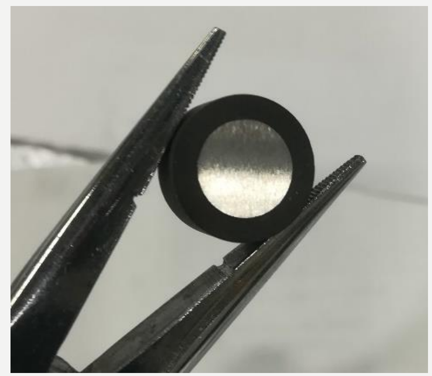
- Economical and Mass production under collaboration with industries
  Funding from Ministry of Economy, Trade and Industry was approved in June, 2019
- Higher-temperature resistance by W-TaC
  Funding, JSPS Kakenhi was approved in April, 2019
- Study for irradiation effect
  Ion-irradiation at HIT, Tokyo University in Aug. 2019, and H.E. proton irradiation under RaDIATE collaboration

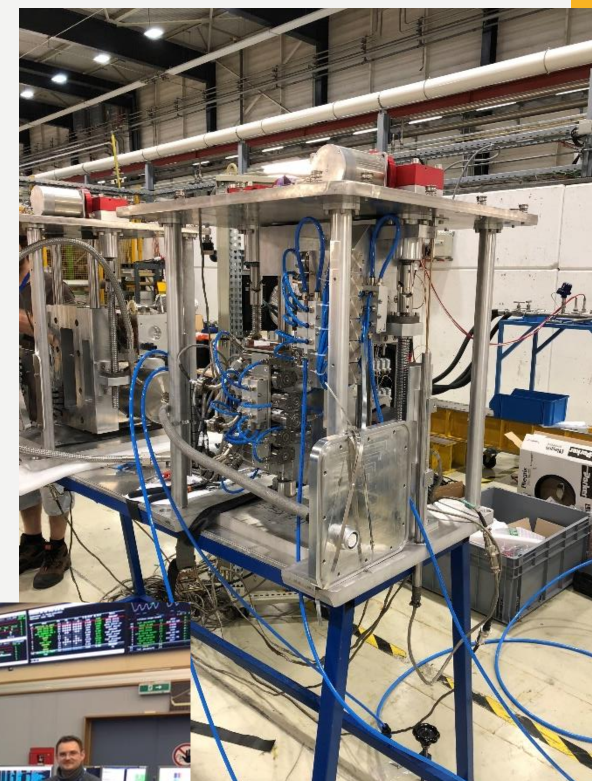
New Sintering & GSMM apparatus

THERMAL SHOCK EXPERIMENTS AT HIRADMAT, HRT48 PROTAD, SEP. 28, 2018

- TFGR W-TiC was included in HRT48.
- We are waiting for PIE testing.







Beam Parameters	
Beam energy	440 GeV
Max. bunch intensity	1.2×10^{11}
No. of bunches	1 – 288
Max. pulse intensity	3.5×10^{13} ppp
Max. pulse length	7.2 µs
Gaussian beam size	1σ: 0.1 – 2 mm

SUMMARY & EXPECT TO HIRADMAT FACILITY

SUMMARY &
EXPECT TO HIRADMAT FACILITY

Summary

- Pulsed muon contributes to various, brand-new and unique science in material science, fundamental physics, and industrial use.
- Higher peak intensity is desired in muon community, i.e. MLF TS2.
- Further thermal shock resistance is required to the new target materials.
- Developments of new target material, SiC-coated graphite, NITE SiC/SiC, and TFGR W-TiC are in progress, including thermal shock testing at HiRadMat.

My personal suggestion to HiRadMat as one of the users

- How to connect between the results of HiRadMat and the evaluation of actual long-term operation
- Saving time of PIE testing after irradiation, Upgrade of remote handling replacement?

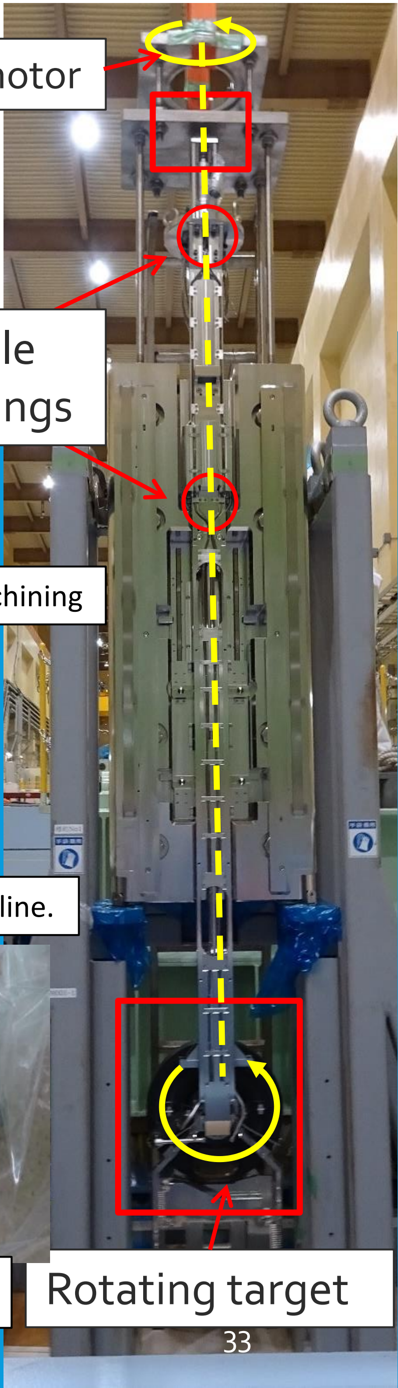
Expect to HiRadMat Facility

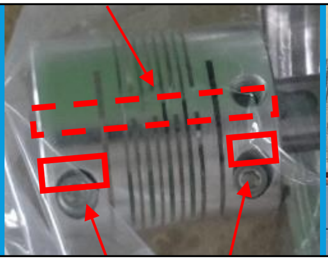
- HiRadMat is an unique facility to investigate thermal shock for higher intensity to new target materials.
- I always appreciate kind collaborations with CERN and RaDIATE.

BACK UP SLIDES

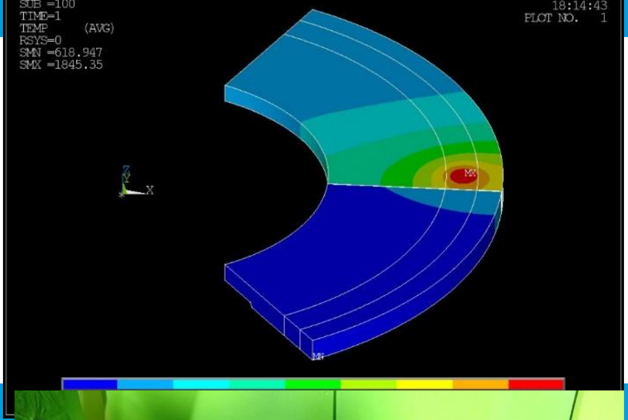
What happened to Muon Target?

- Rotating target has been successfully operated for 4 years.
- On this regular maintenance: replacement of rotation feedthrough from air to vacuum, the flexible coupling was broken.
- During operation, it was intact.
- We found the mistake of the machining process.
- We can replace the broken one.
- But the problem is that two couplings are used in the rotating target. The lower one cannot be replaced easily.





For beam operation, and replacement of target

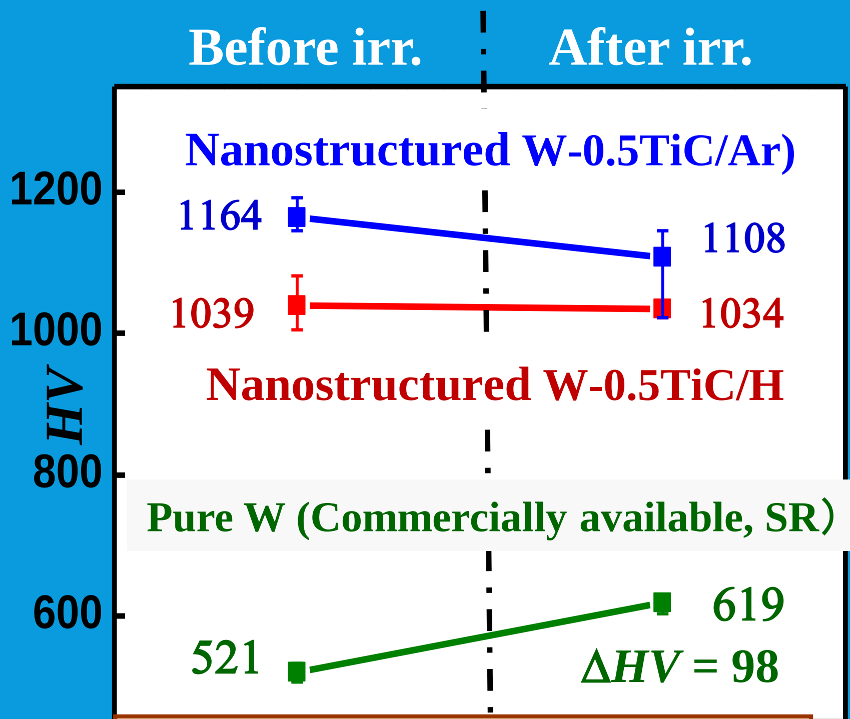


- It has been confirmed that the rotating target is not damaged with 500-kW proton beam even without rotation through FEM simulation . Furthermore Monitoring system is upgraded.
- The main issue is emission of tritium.
- The exhaust of vacuum pump is temporarily stored in a buffer tank system and is vented after measurement of concentration of RI.
- 500-kW operation is now going on.
- The target will be replaced with a new and appropriate one during next new-year maintenance interval.

Insufficient research for irradiation effect of W-TiC

T_{irr} = 873K, 2×10^24 n/m^2 (E_n >1 MeV), 0.08 dpa, JMTR

Vickers microhardness

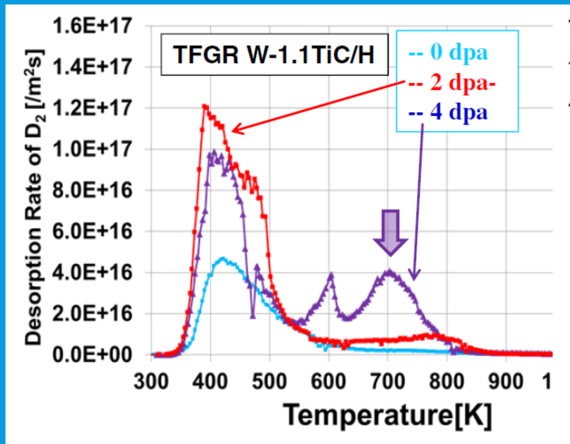


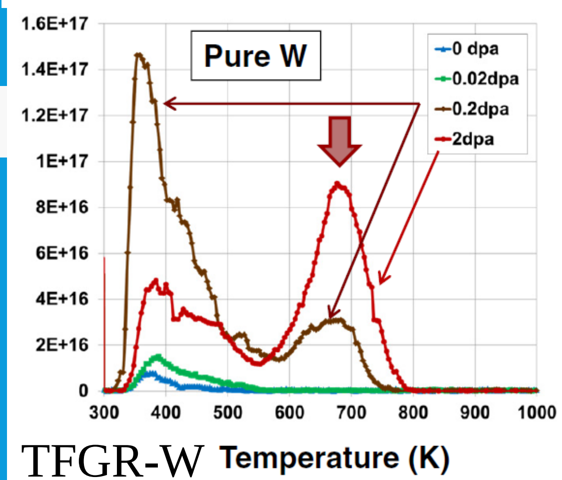
- **No radiation hardening**

H.Kurishita et al. JNM 377 (2008) 34.

W/O GSMM

T_{irr} = RT, 1-2×10^19 ions/m^2 (2.4 MeV Cu2+), 2-4 dpa, Tohoku Univ. IMR
T_{irr} = RT, 1×10^21 D2+/m^2 (2.0 keV D2+)





Thermal Desorption Spectroscopy of D2

The D retention in pure W is significantly increased by Cu2+ irradiation, while that in TFGR is relatively insensitive to the damage level.

Irradiation effect research of TFGR is mainly for D retention.

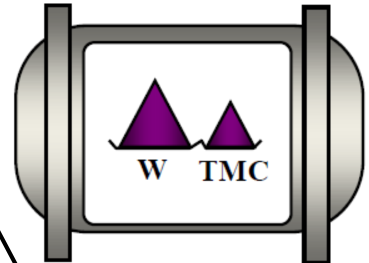
H.Kurishita et al. Phys. Scr. T159(2014)014032

Current status of W development (1)

Preparation and handling of purified powders

Setup of High-purity glove box (GB) with vacuum chamber/furnace for handing highly pure powders without gaseous contaminations

- **Purification and (remote) handling of starting powders (W, TiCx,) and MAed powders (W-Ti-C)**
- **Fully degassing of MA vessels and balls, HIP capsule etc. before use**



Powders in Ta boat for purification

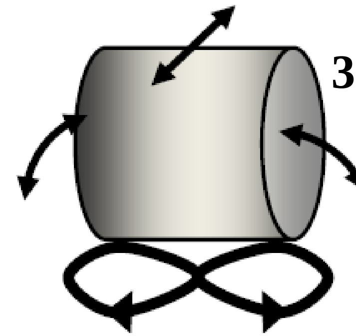
Comparison of impurity content in MAed powder

	High-Purity GB, KEK/MTC			Typical GB, KEK/NIFS		
Impurity	O	N	Mo	O	N	Mo
Content (wt%)	0.091	0.009	1.98	1.0	0.18	1.25

Current status of W development (2)

Mechanical Alloying (MA) in *vacuum*

MA: Introduction of ultrafine grains (~20 nm) and alloying by using high energy ball milling



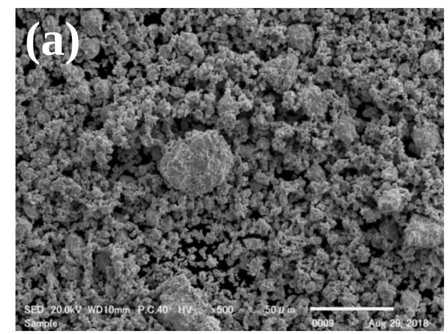
Powder with hard balls in a vessel (TZM)

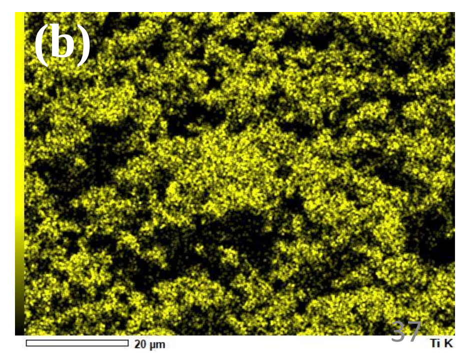
The MA Vessel is filled with
Previous work: Hydrogen, This work: Vacuum

- **Setup of 3MPDA high energy ball mill with cooling system (~ -20°C) to prevent strong adhesion of MAed powder to the vessel inner walls**

3MPDA: 3 Mutually Perpendicular Directions Agitation

Mo impurity (~ 2%) coming from TZM balls and vessels



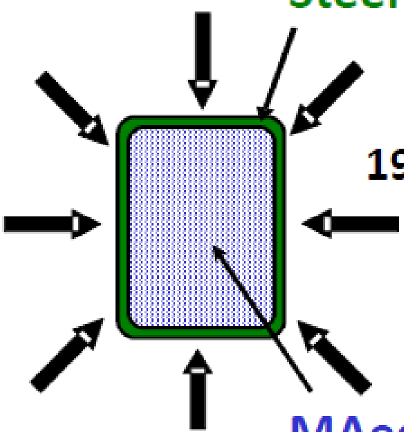


(a) SEM, (b) EDS(Ti) mapping for MAed W-1.2%TiC powder

Current status of W development (3)

HIP (Hot Isostatic Pressing)

HIP: Densification of MAed powder *without exposure to air*
Densified compacts with equiaxed, ultra-fine grains



Steel capsule filled with MAed W-1.2%TiC powder

HIPed W-1.2wt%TiC compact
Density : 17.92 g/cm^3

Chemical compositions (wt%) of W-1.2%TiC before and after HIP

	Ti	C	O	N	Mo
HIPed compact	0.88	0.23	0.038	0.010	---
MAed powder	---	---	0.091	0.009	1.98

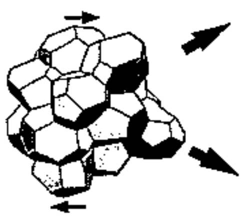
GSMM (Grain boundary sliding-based microstructural modification) process

GSMM : <u>Reinforcement of</u> intrinsically weak <u>GBs</u> in W *by enhancement and optimization of <u>TiC precipitation</u> and their segregation*

<u>Removal of the residual bubbles/pores through GB diffusion</u>

by using superplastic deformation, driven by GB sliding, where the recrystallized, equiaxed grain geometry is maintained and active grain rotation and extensive relative displacement of the adjacent grains are operative.

Shear stress

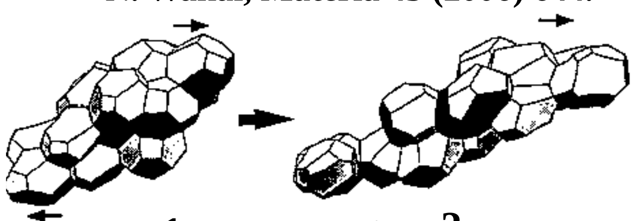


γ (shear strain)

$\gamma = 0$

<u>Superplastic deformation by GB sliding at high temperature</u>

N. Wakai, Materia 45 (2006) 644.

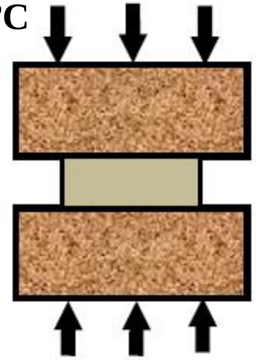


$\gamma = 1$ $\gamma = 2$

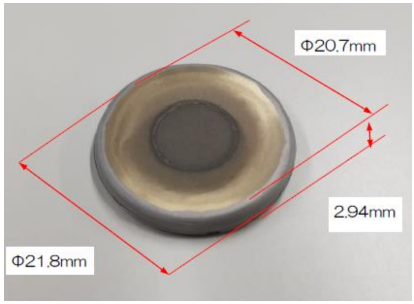
The number of GSMM operations at 1650°C attempted to date is ***10 times*** with various final loads

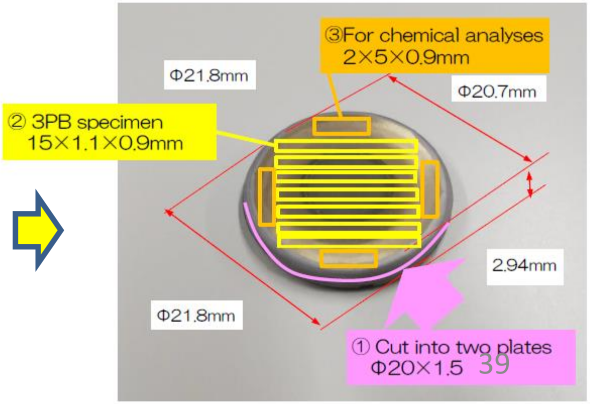
8ϕ x 20 mm

1650°C



Reduction ratio: 85.3%

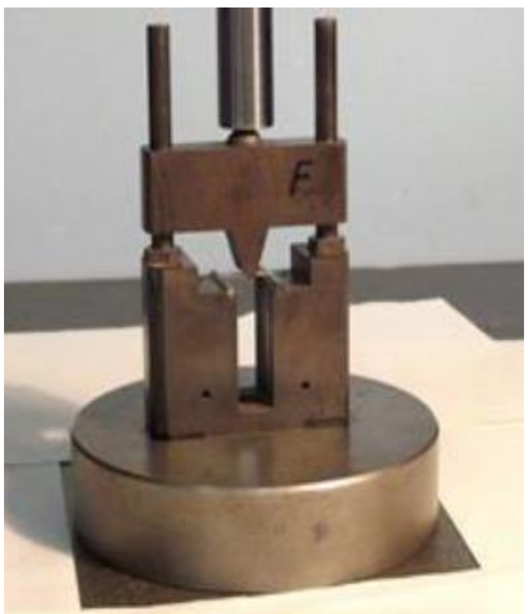




Three point bend (3PB) testing at RT

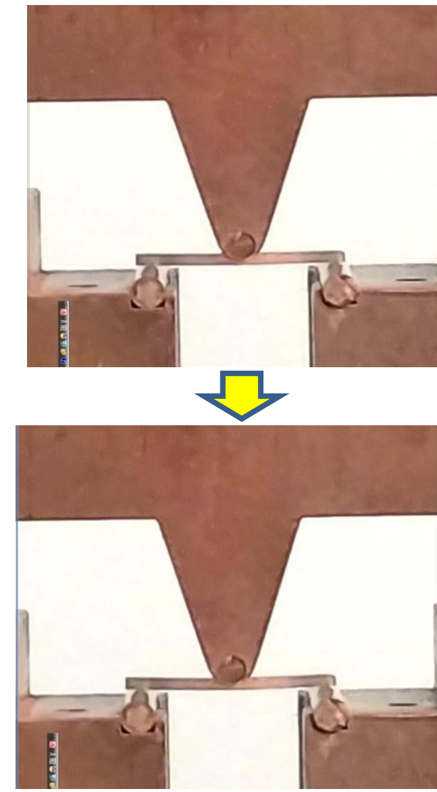
3PB testing : Demonstrate that the fabricated W alloys exhibit very high fracture strength (> 2~3 GPa) and appreciable ductility *at RT in the nanostructured, recrystallized state*

3PB specimen
1.1 mm x 0.9 mm x 15 mm



3PB test fixture used

Overview of 3PB testing at RT and 0.3mm/min

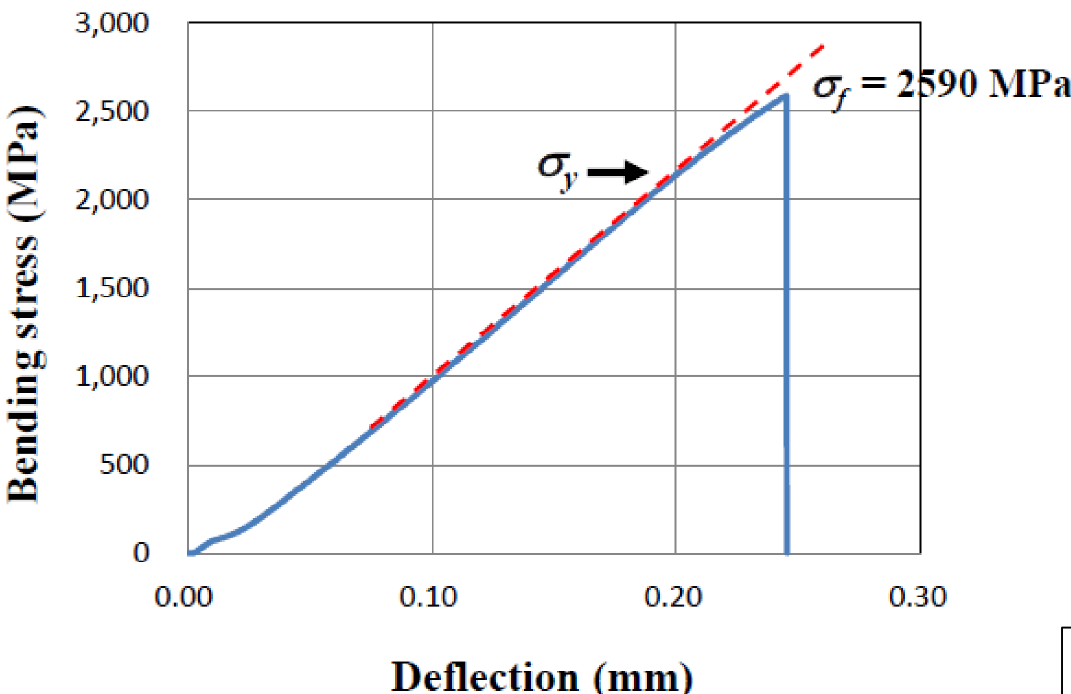


Specimen deflection during 3PB testing

3PB test results at RT

W-1.2wt%TiC in the nanostructured, recrystallized state

#8 specimen: MA-HIP-GSMM (RR : 82. 9%)



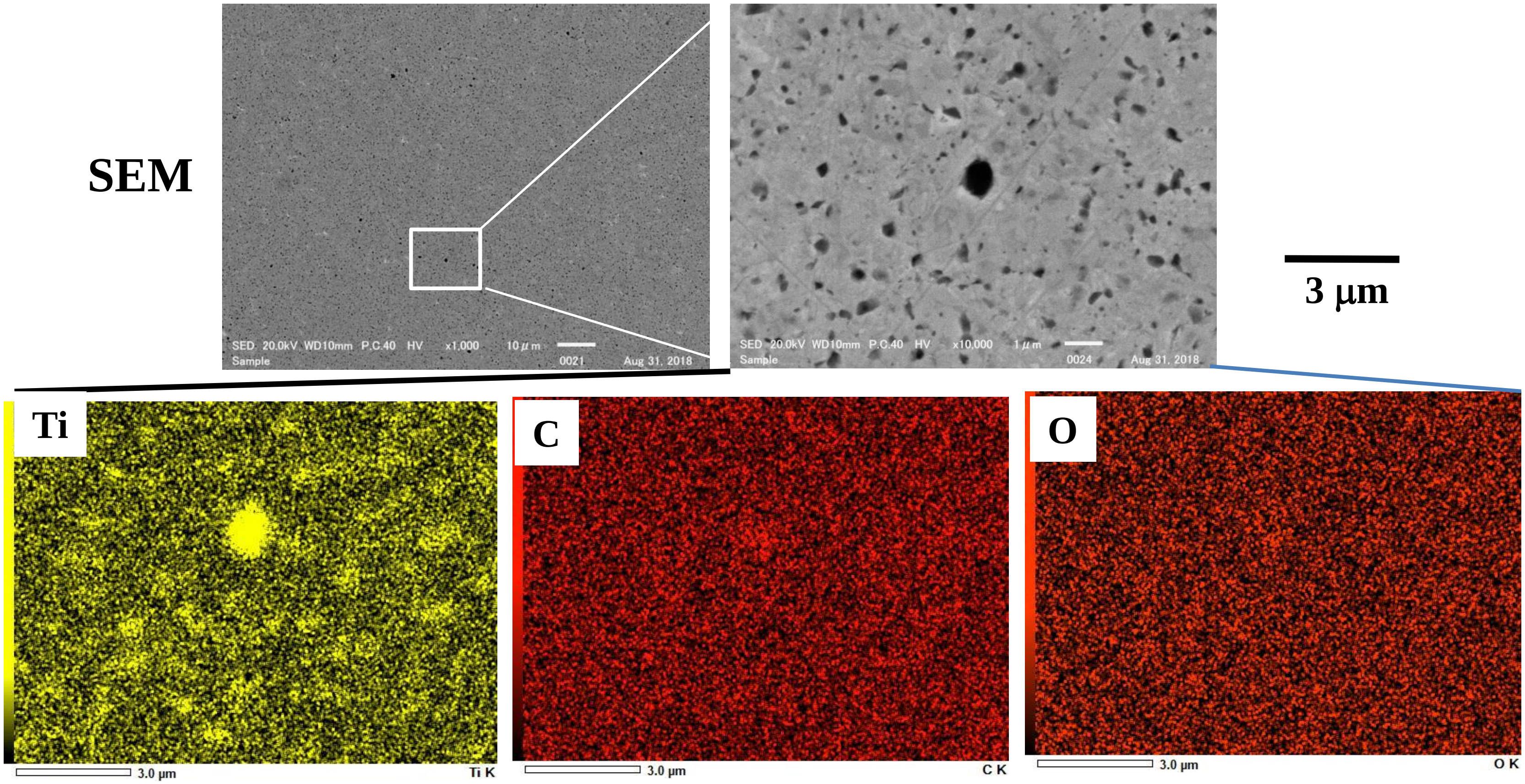
Very high fracture strength of 2590 MPa and slight ductility at RT

→ TFGR (Toughened, Fine Grained, Recrystallized) W-1.2%TiC alloy

In order to manufacture much more ductile W-1.2%TiC alloys, the microstructure of the #8 specimen has thoroughly been examined.

#8 specimen broken in two pieces after testing

Microstructures of #8 specimen SEM, EDS



A large precipitate of titanium oxy-carbide with approximately 1 μm diameter observed is attributable to locally insufficient progress of the MA process; the starting TiC phase has not completely been decomposed into Ti and C solutes in the W matrix by MA. The optimization of the MA process is required for ductility enhancement of W-1.2%TiC alloy.

TEM analysis by Prof. Sakamoto at Ehime University will come soon.