The 6th RaDIATE Meeting, TRIUMF
Radiation Damage Studies II
(11:30-11:55, Dec. 10th, 2019)

Status of Ion Beam Irradiation at HIT facility

E. Wakai[1]
S. Kano[2]
T. Ishida[1*]
S. Makimura[1]
H. Abe[2]

1. J-PARC
2. Univ. of Tokyo

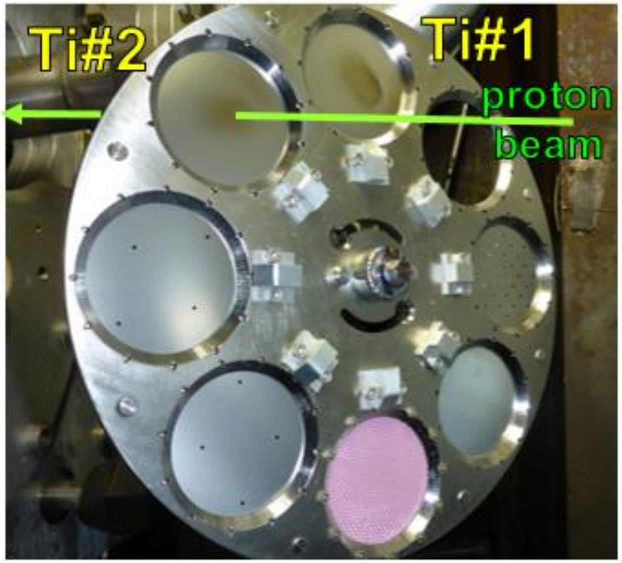
Background -1

T. Ishida, E.Wakai, D.Senor, A.Casella, D.Edwards et al., Nuclear Materials and Energy 15 (2018) 169-174.

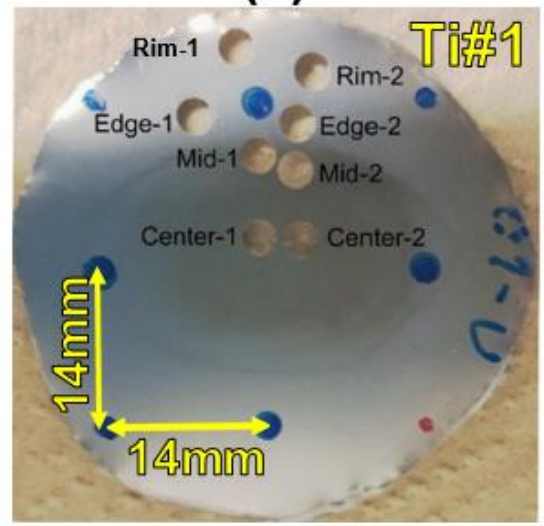
J-PARC OTR: 50μm-thick metastable-β (BCC) Ti-15V-3Al-3Cr-3Sn (ST) Irradiation Damage Analysis at PNNL

Ti-1: 1.4×10^{20}pot
0.12 peak DPA-NRT

(Ti-2: 5.2×10^{20}pot)
(0.29 peak DPA)



(a)



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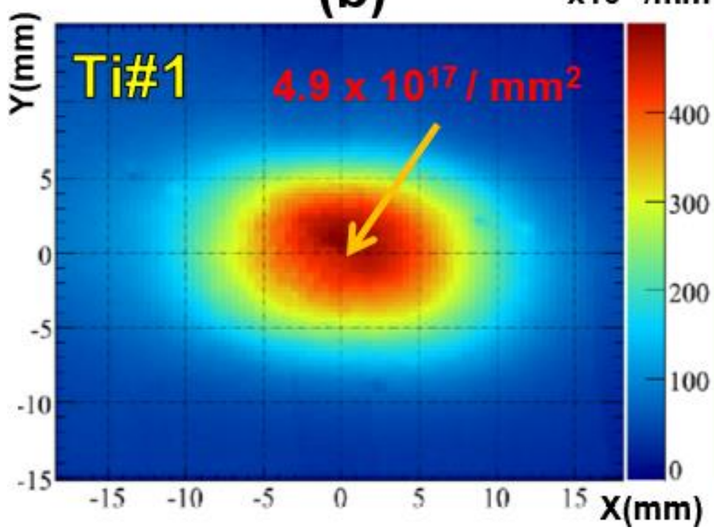
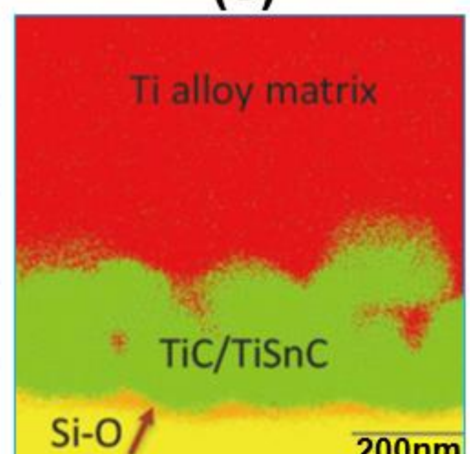
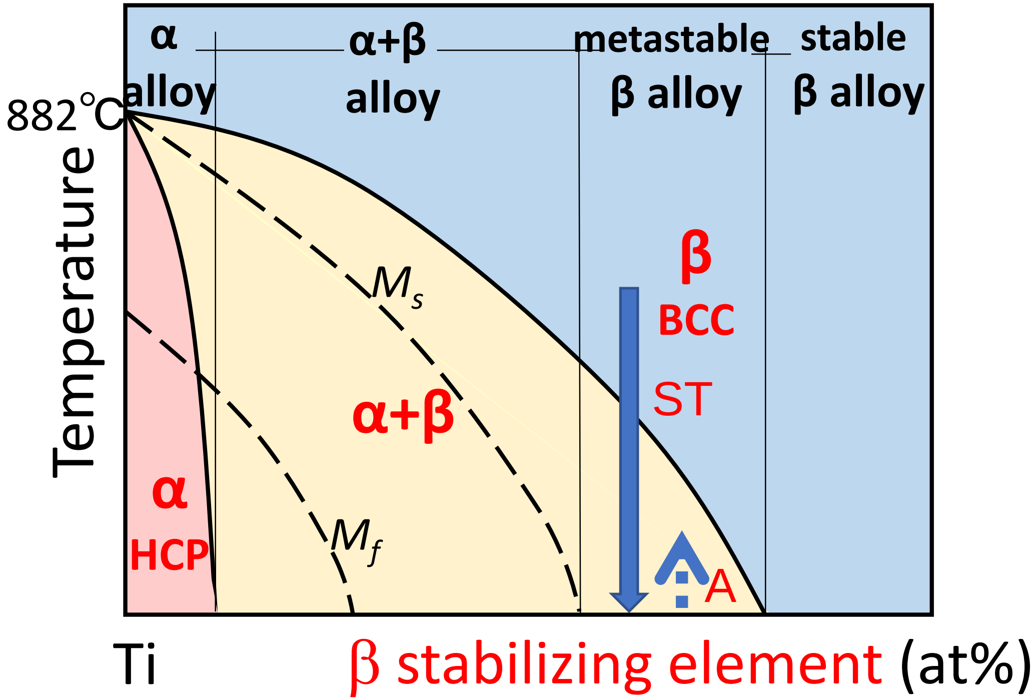


(c)



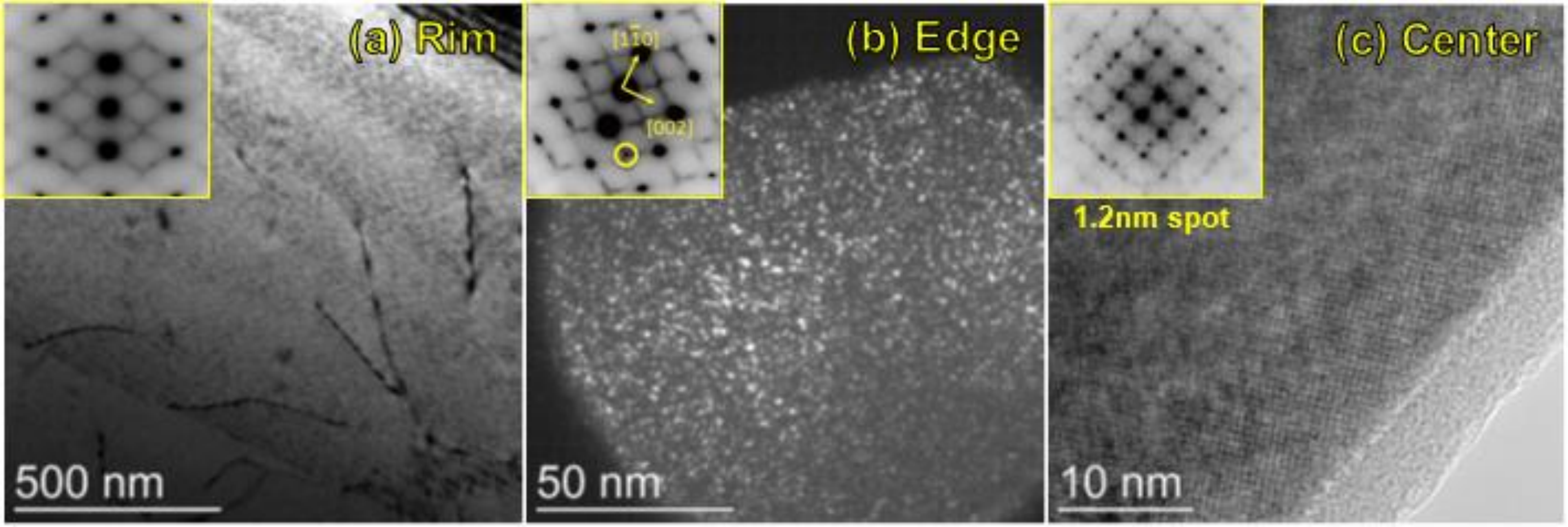


Mo equivalent (mass%) = Mo + 0.67 × V + 0.44 × W + 0.28 × Nb + 0.22 × Ta + 2.9 × Fe + 1.6 × Cr − 1.0 × Al

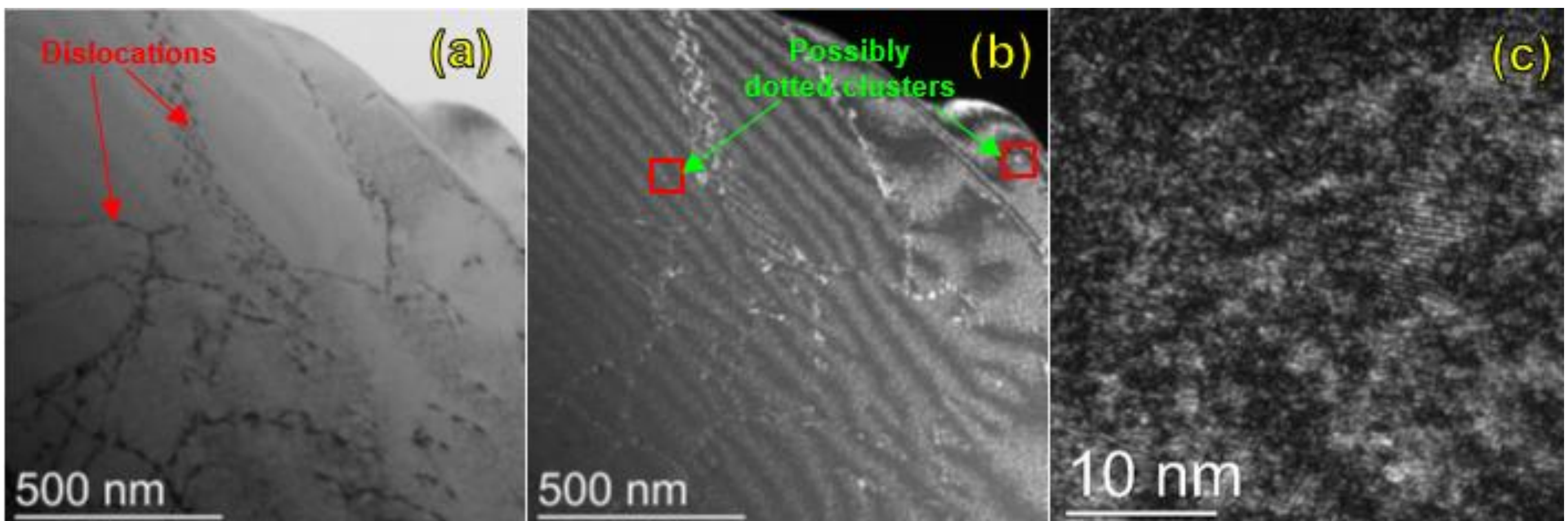
Grade	Heat Tr	0.2% YS	TS	TE
Ti-15V-3Cr-3Al-3Sn (metastable β)	ST	790 MPa	860 MPa	11%
	STA	1,280 MPa	1,400 MPa	7%
Ti-6Al-4V (α+β)	Anneal	900 MPa	1,000 MPa	13%
	STA	1,050 MPa	1,160MPa	5%

- The α+β phase and metastable β phase alloys are heat-treatable
- Solution Treatment and Aging (STA): maintain the alloy at a temp. higher than the β transus to dissolve the alloying elements
- Quench it to keep in a metastable supersaturated solid solution state.
- Aging at an elevated temp. for several hrs. generates a fine (less than 10 nm) scale precipitation in β-phase grains, *i.e.*, α phase for aging above ~500^{o}C, or ω-phase for aging between 400~500 ^{o}C.
- Whilst the fine scale precipitation makes the alloy stronger "precipitation hardening", there is evidence to suggest that the ω precipitation can lead to embrittlement "ω-embrittlement".

→ T.Ishida et al, https://arxiv.org/abs/1911.10198



Before irradiation, microstructures of 15-3 Ti exhibited a high density of nano-clusters of athermal ω and martensitic α'' phases. These were stable after irradiation.



Very low density of defect clusters were formed by irradiation about 0. 06 dpa.

<u>No significant changes on micro-Vickers hardness</u>

15-3 Ti alloy may have a high resistance for radiation damage, due to nano-scale precipitates working as *point-defect sink-cites*

Background -2

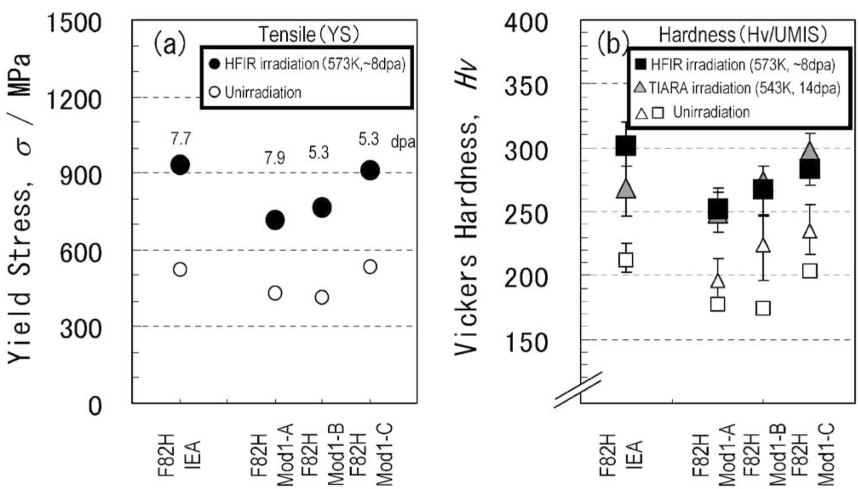
■ Correlation of Radiation Hardening Measurement Method

cm scale | **mm scale** | **Micro - Nano scale**

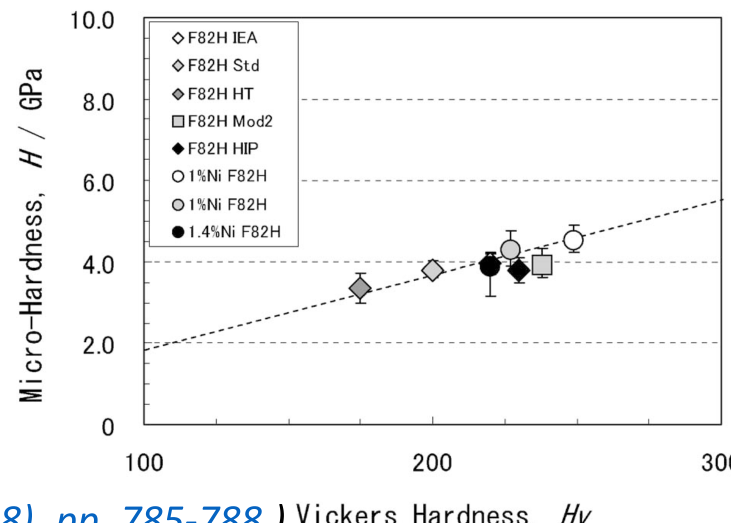
σy(MPa)≒3 Hv | Hv≒60×Hm(GPa)

Tensile Data ⟷ **Vickers Hardness Data** ⟷ **Micro-Harness Data**

Correlation-1



Correlation-2



(M. Ando, et al., J. Japan Inst. Metals, Vol. 72, No. 10 (2008), pp. 785-788.)

→ Low energy ion beam irradiation is useful in the evaluation of hardening behavior at high DPA region

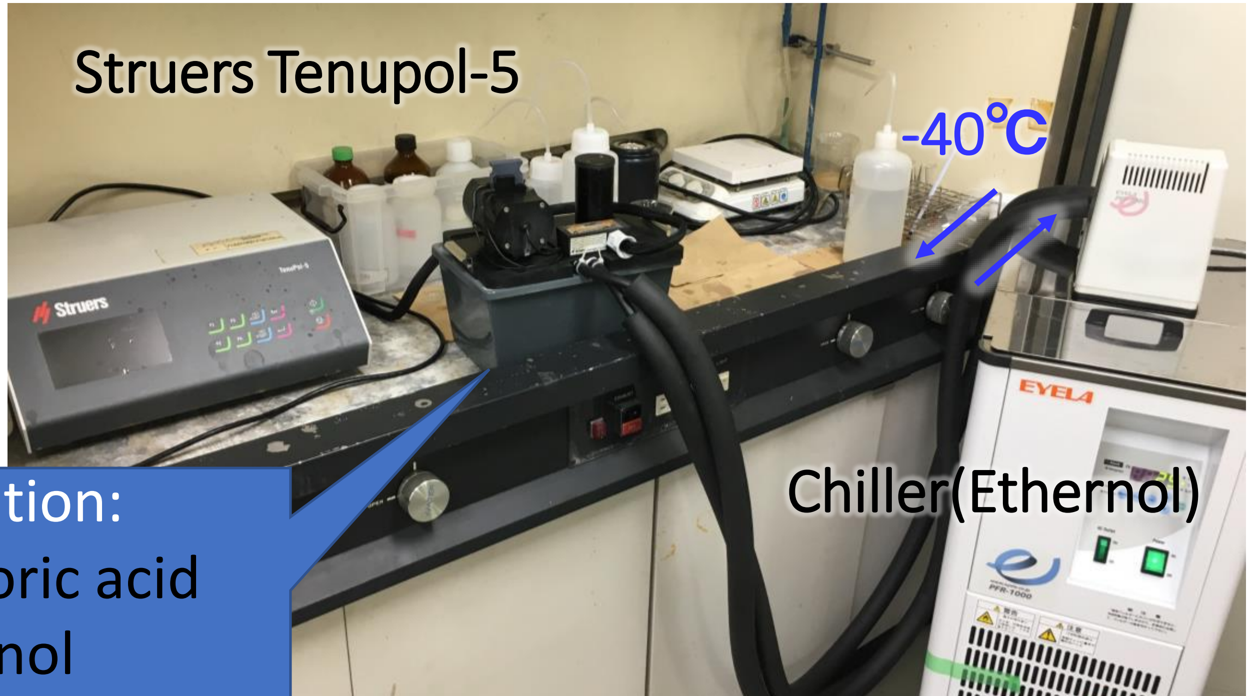
Objective

1. **Complement studies with high energy proton irradiation experiments**

2. **Estimate hardening behaviors in high DPA region and obtain DPA dependence for different alloys**

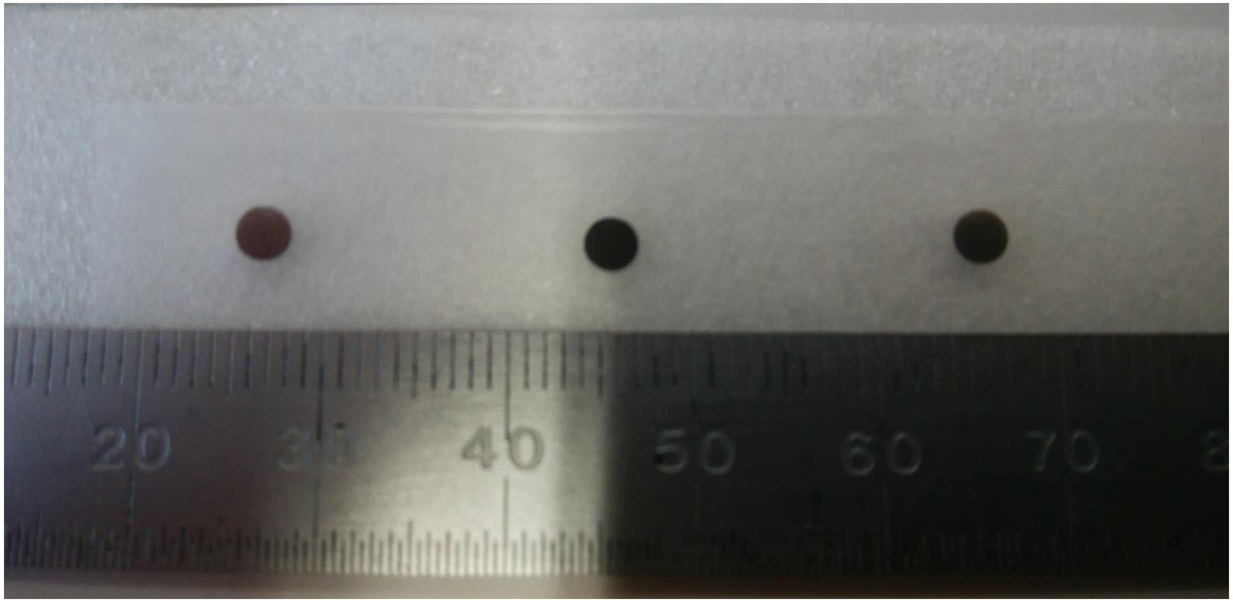
3. **Select material grades and heat treatments with higher radiation damage resistance**

Twin-jet Electro Polishing



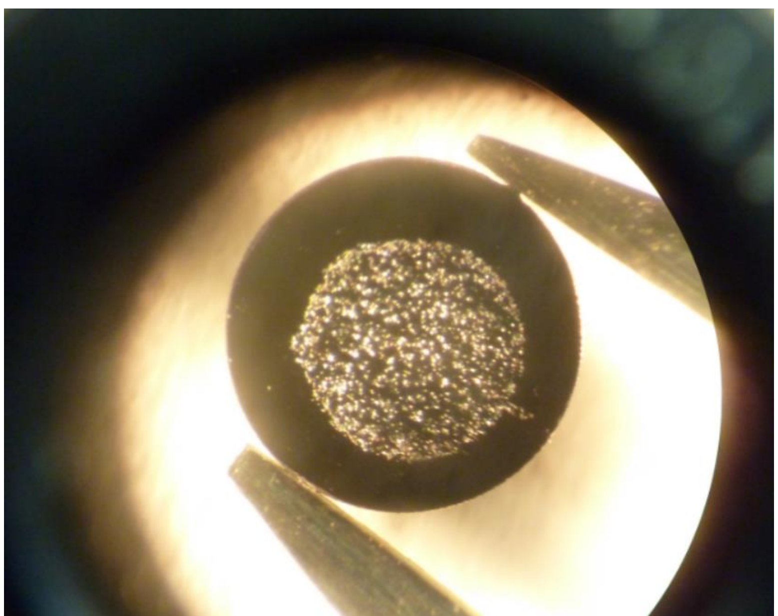
Solution:
6%Perchloric acid
35％Butanol
59％methanol

Specimen	Electro-polishing condition
Ti-15V-3Cr-3Al-3Sn (Metastableβ) Solution Treatment, not Aged (ST)	-40V, flow rate 22, 60-68 mA
Ti-15V-3Cr-3Al-3Sn (Metastableβ) Solution Treatment and Aged (STA)	-40V, flow20, 10sec, 68 mA
Ti-64 (α+β) Annealed (A)	-60V, flow22, 10sec, 106 mA
Ti-64 (α+β) STA (WQ -> Aging)	-50V, flow22, 10sec, 88 mA

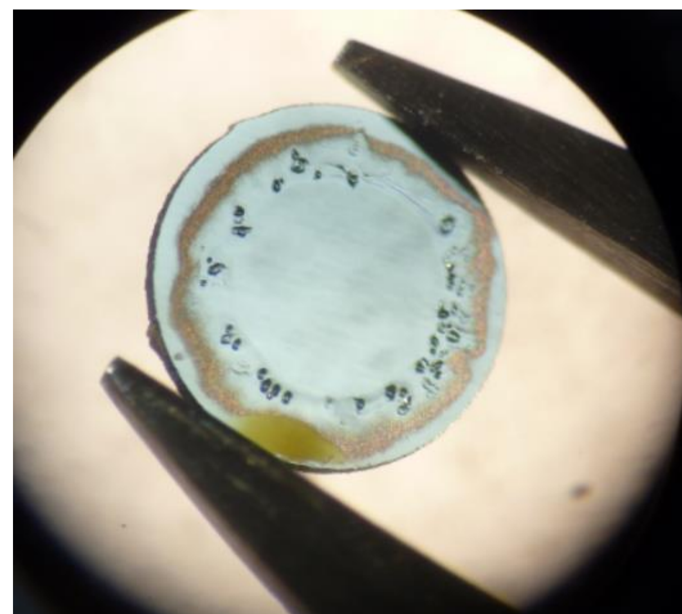


Φ3 × 0.2mm t

Pitting Damage (Not Used)



Good Condition

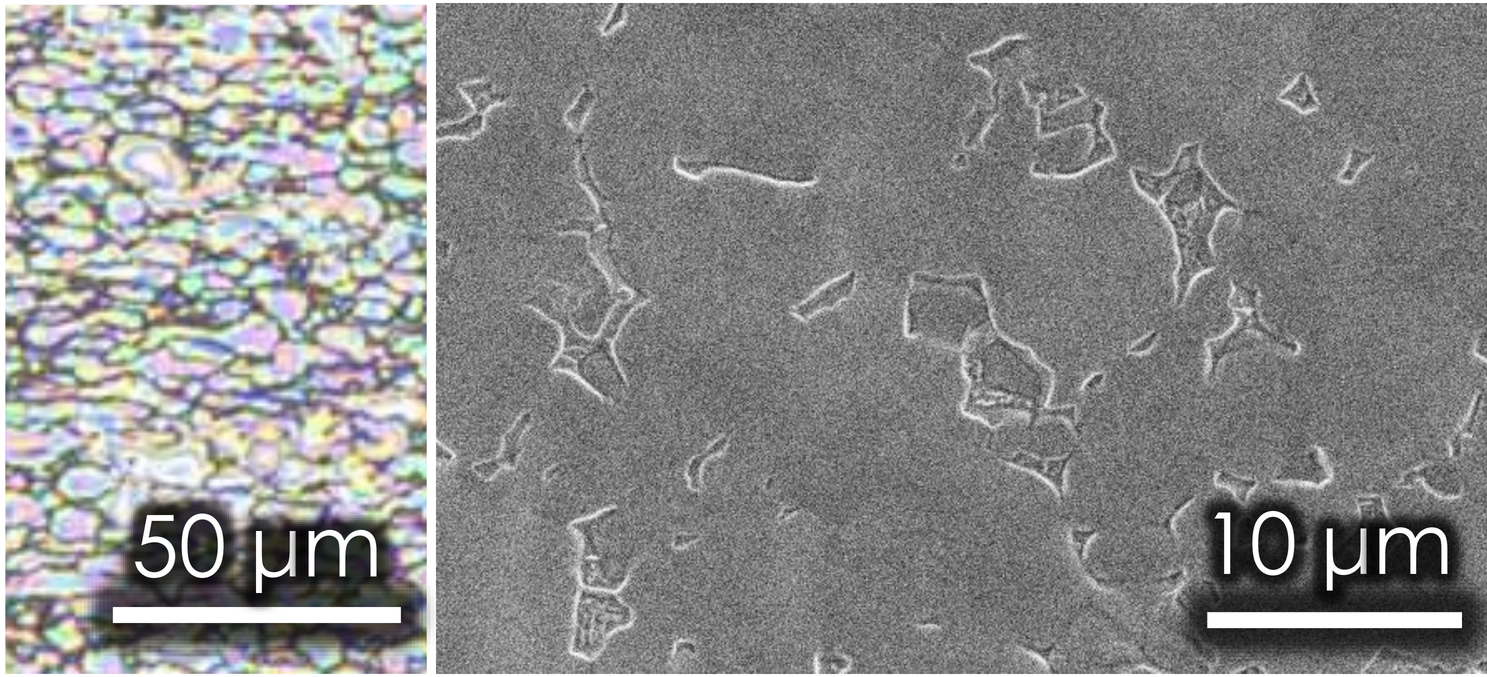


6-4

Heterogeneous corrosion spots

Precedential corrosion on beta phases

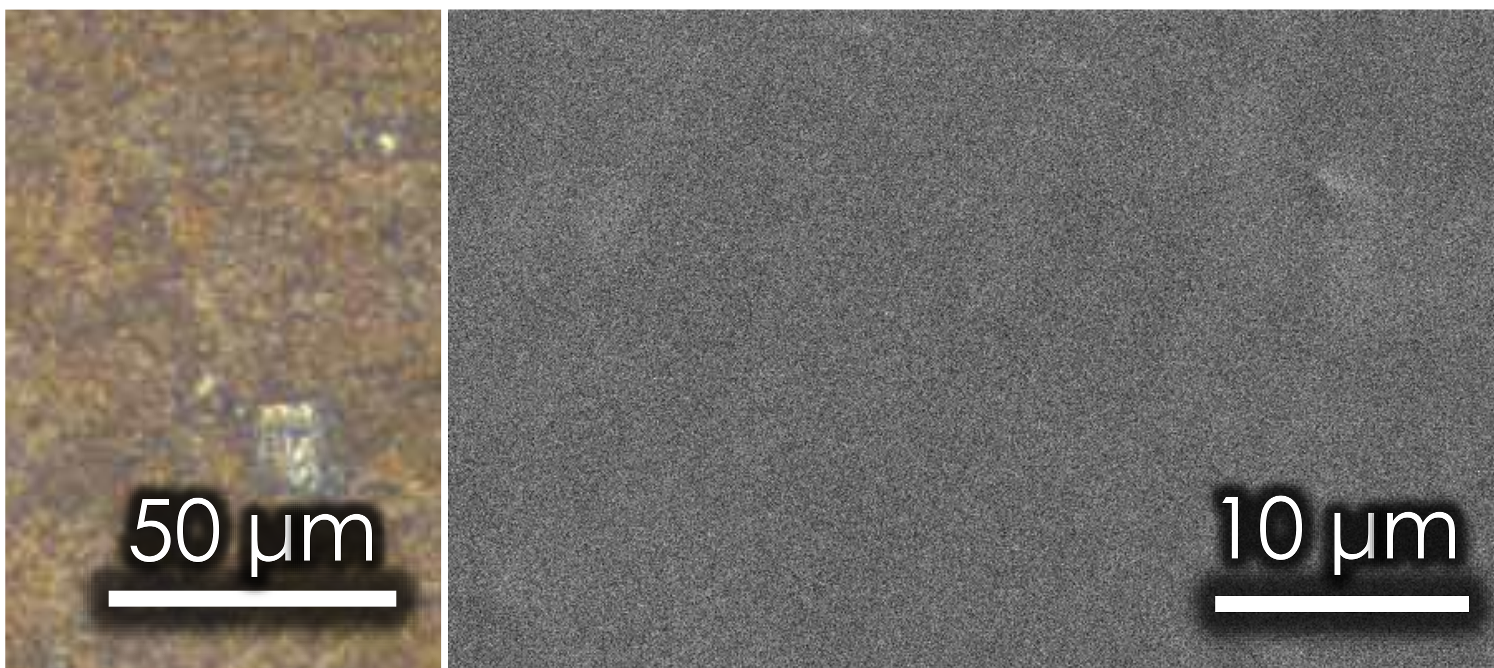
Depth: ~200nm



15-3

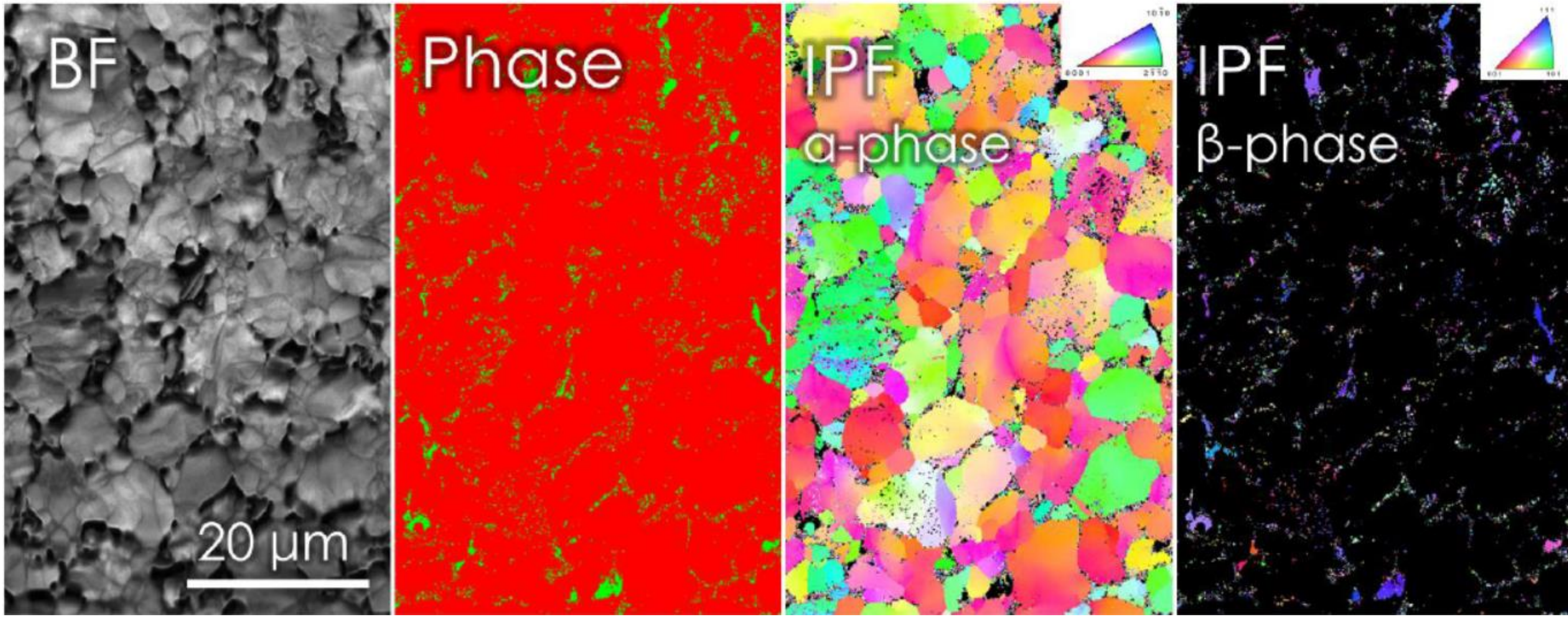
Smooth surface

No contrast



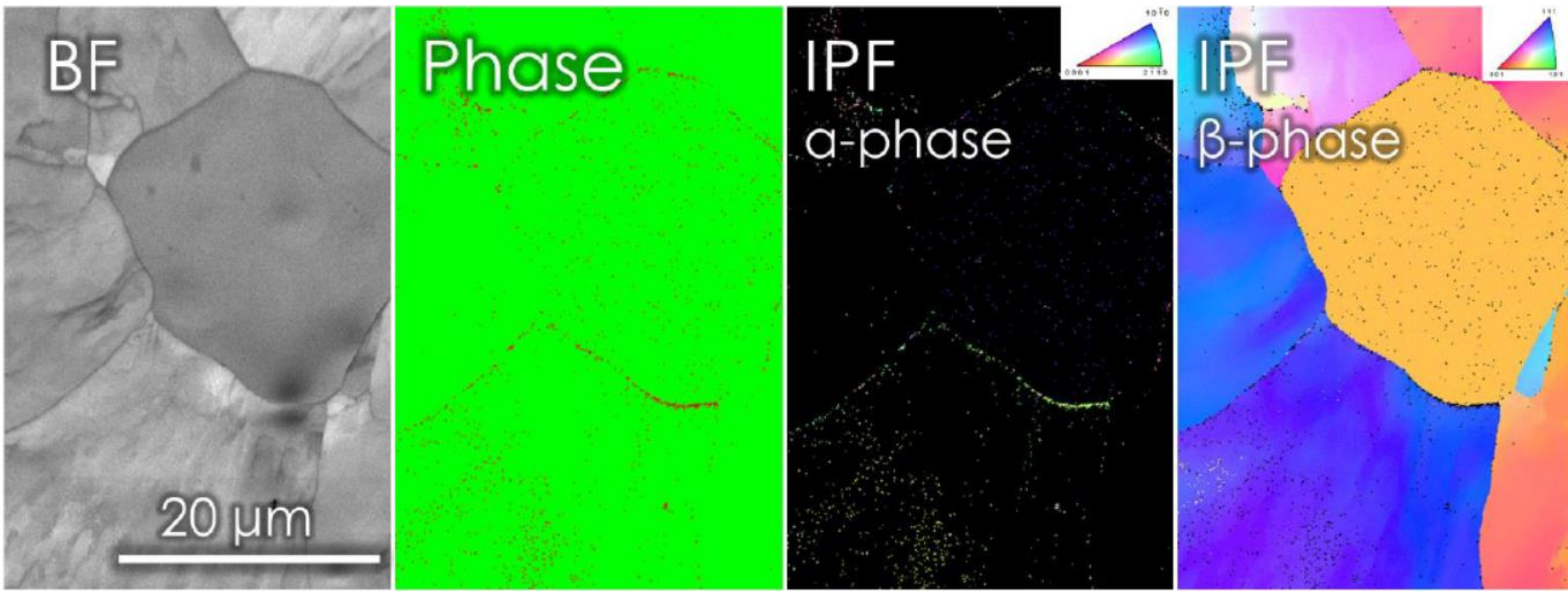
6-4

α: 95.2%
β: 4.8%

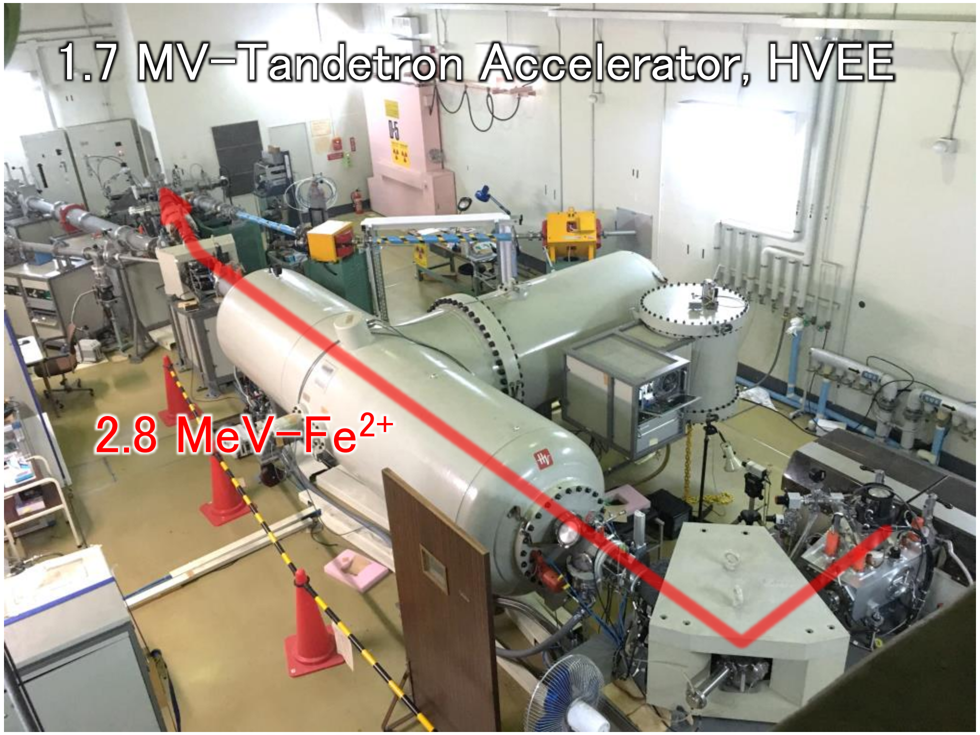


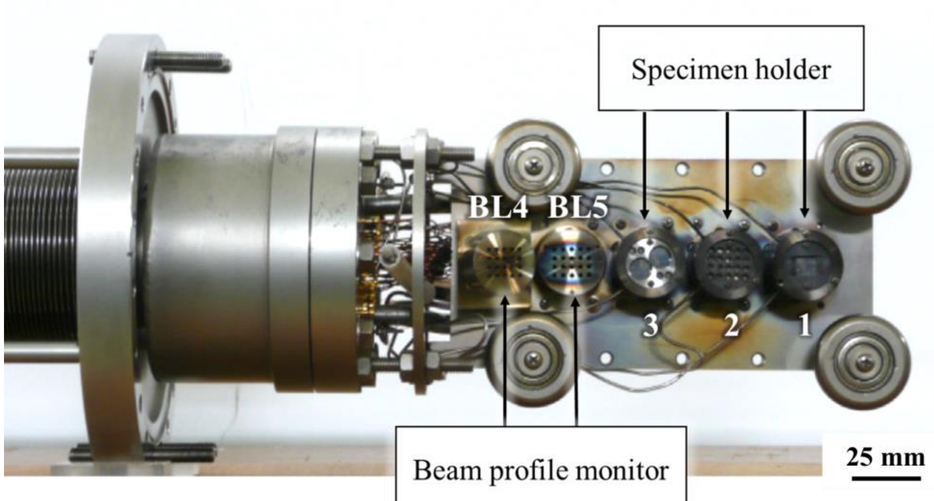
15-3

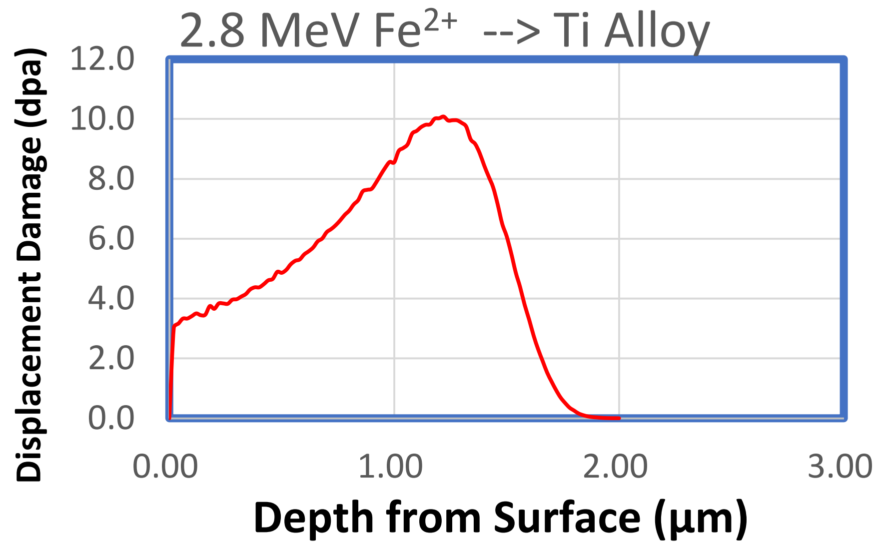
α: 1.1%
β: 98.9%



HIT: High Fluence Irradiation Facility of The Univ. of Tokyo

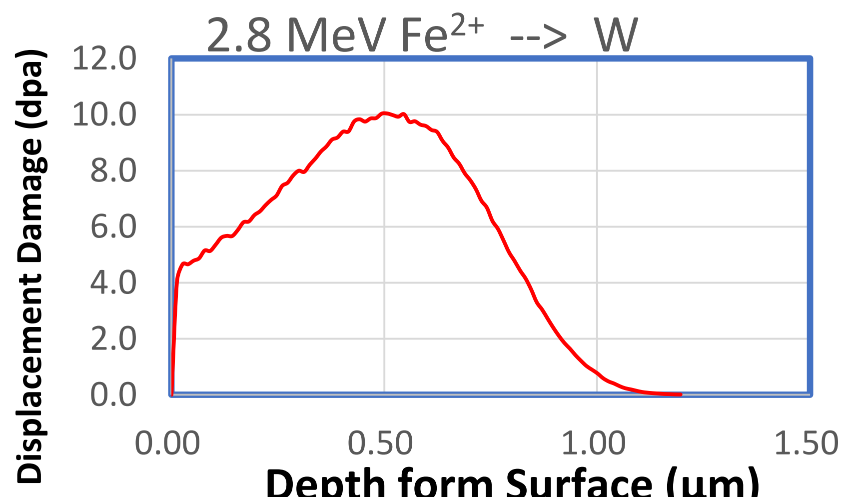






Current : ~8nA

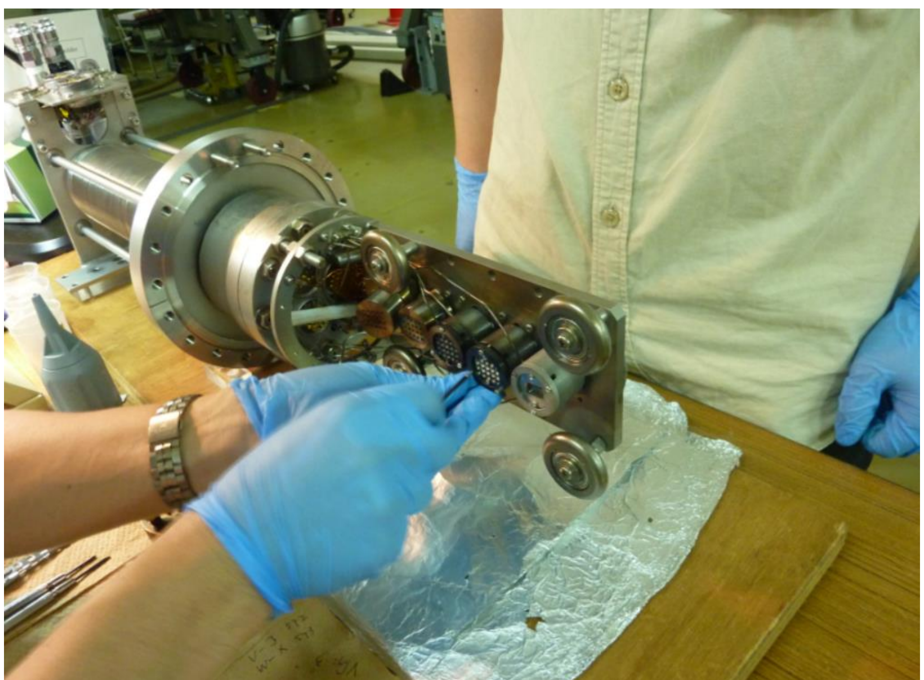
Dose rate : 9.5×10^{-4} dpa/s



Current : ~8nA

Dose rate : 3.3×10^{-3} dpa/s


FOIL



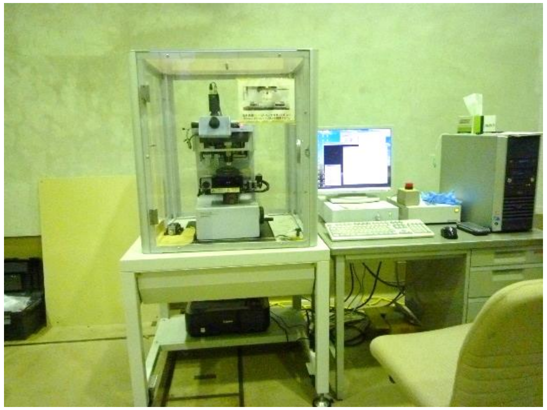
HIT Ion Irradiation Experiment's Matrix in August 2019 (3 days)

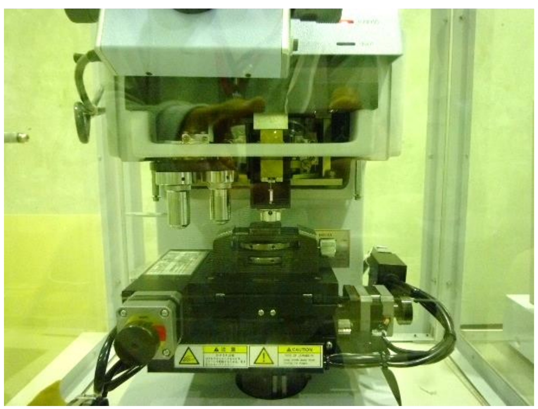
Specimen	1 dpa	5 dpa	10 dpa
Ti-15V-3Cr-3Al-3Sn Solution Treatment, not Aged (ST)	○	○	○
Ti-15V-3Cr-3Al-3Sn Solution Treatment and Aged (STA)	○	○	○
Ti-6Al-4V Gr23 ELI Annealed (A)	○	○	○
Ti-6Al-4V Gr23 ELI STA (WQ -> Aging)	○	○	○
W-TFGR	-	-	○
Pure-W	-	-	○

W-TFGR: "Toughened Fine-Grained Recrystallized" Highly-ductile Tungsten → S. Makimura

Nano Indentation Test

EIT Analysis (EIT: Elastic modulus of Indentation Testing)

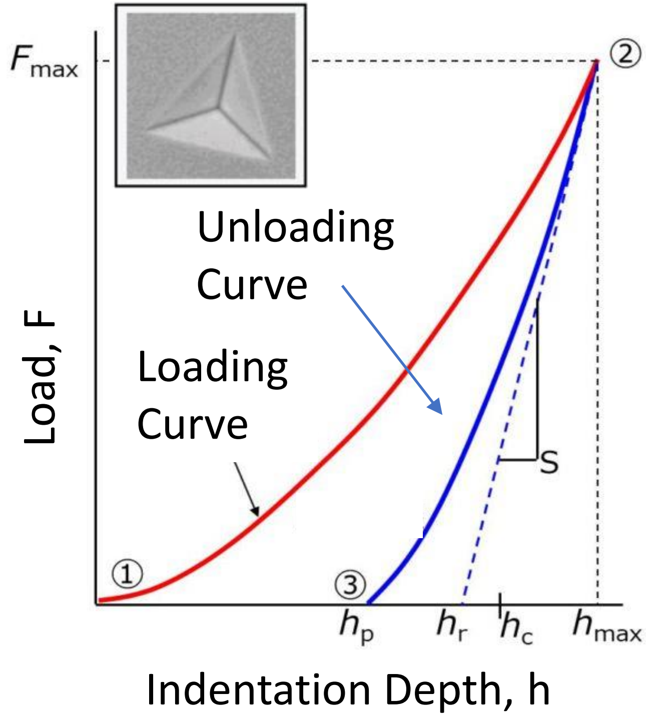


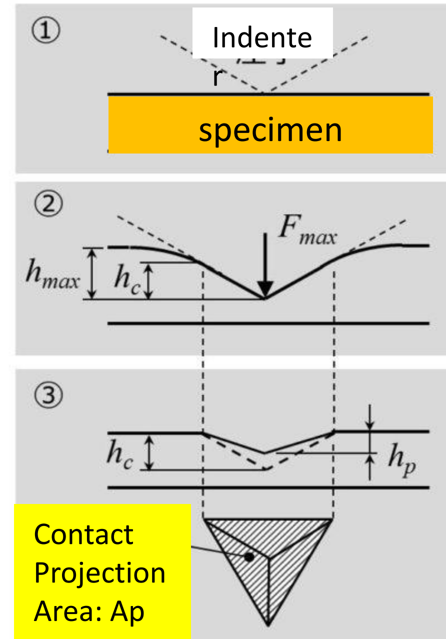


Shimazu- DUH-211S type, set in the University of Tokyo

Indenter : Berkovich(115º)

Indentation depth :150nm (fix)





$$h_c = h_{max} - \epsilon(h_{max} - h_r)$$

$$H_{IT} = \frac{F_{max}}{A_p(\boldsymbol{h_c})}$$

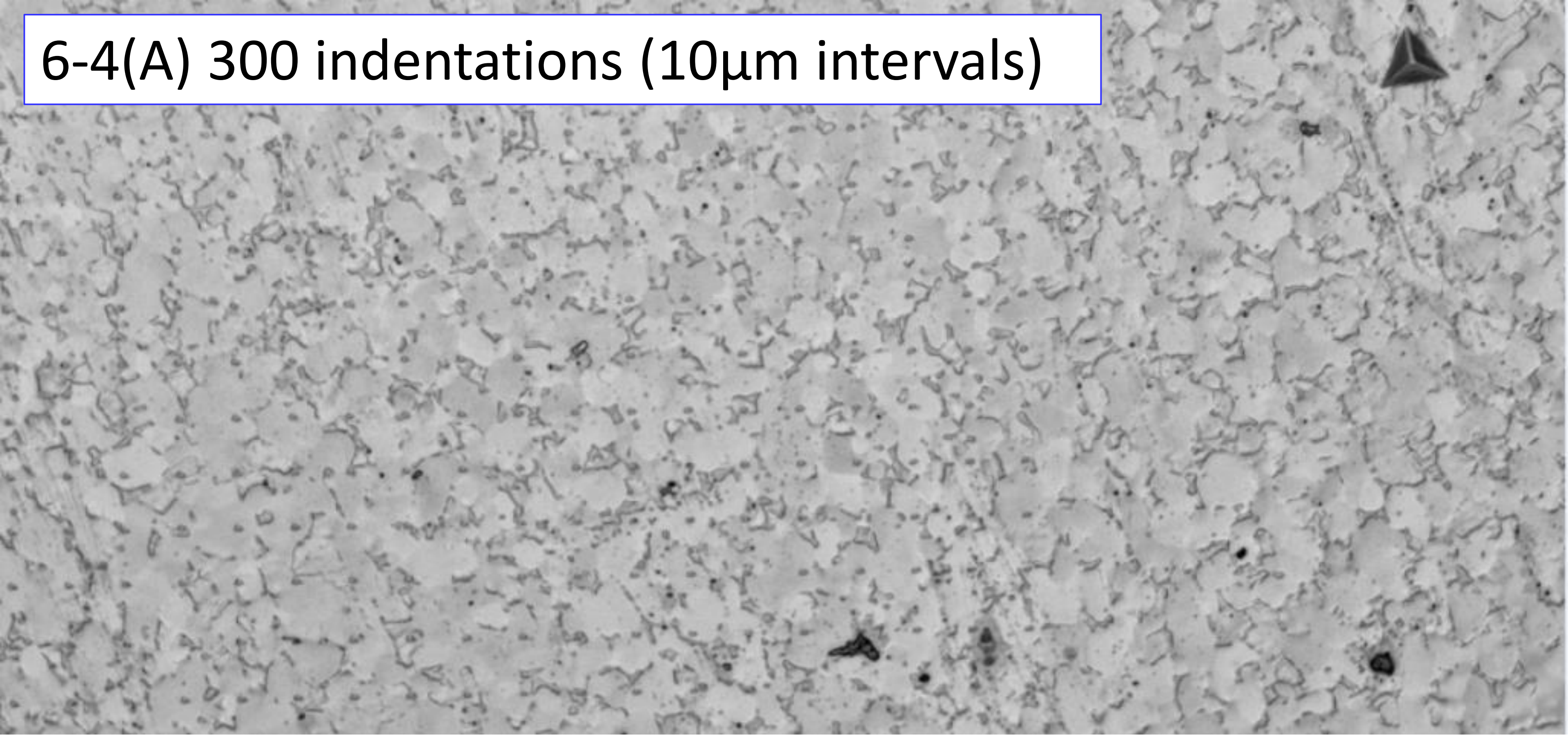
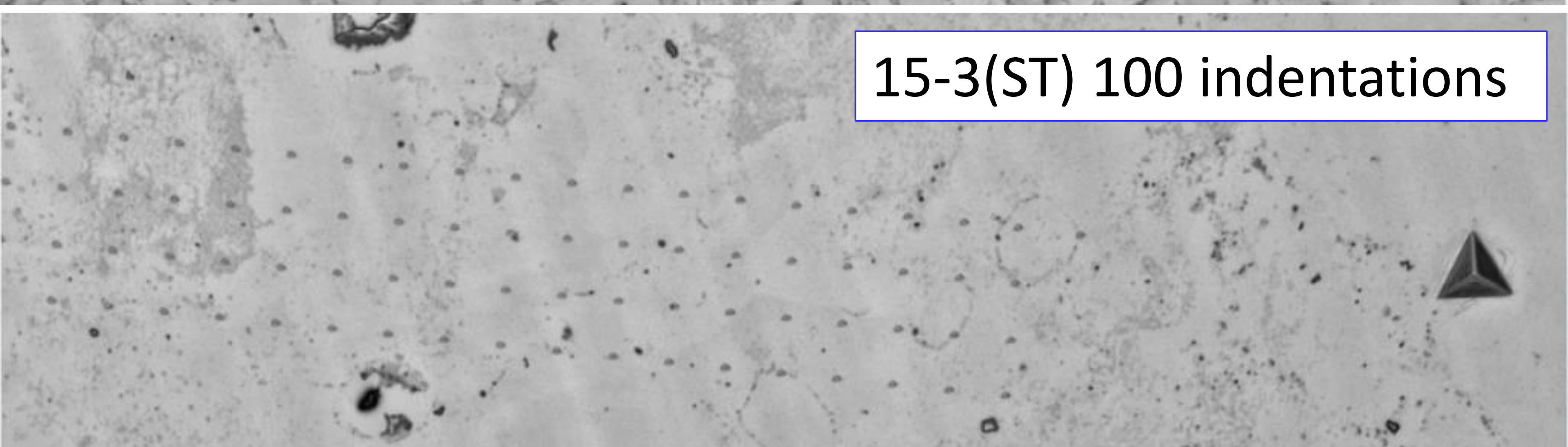
Er : Elastic modulus of composite of Indenter and specimen

$$E_r = \frac{S\sqrt{\pi}}{2\sqrt{A_p(h_c)}}$$

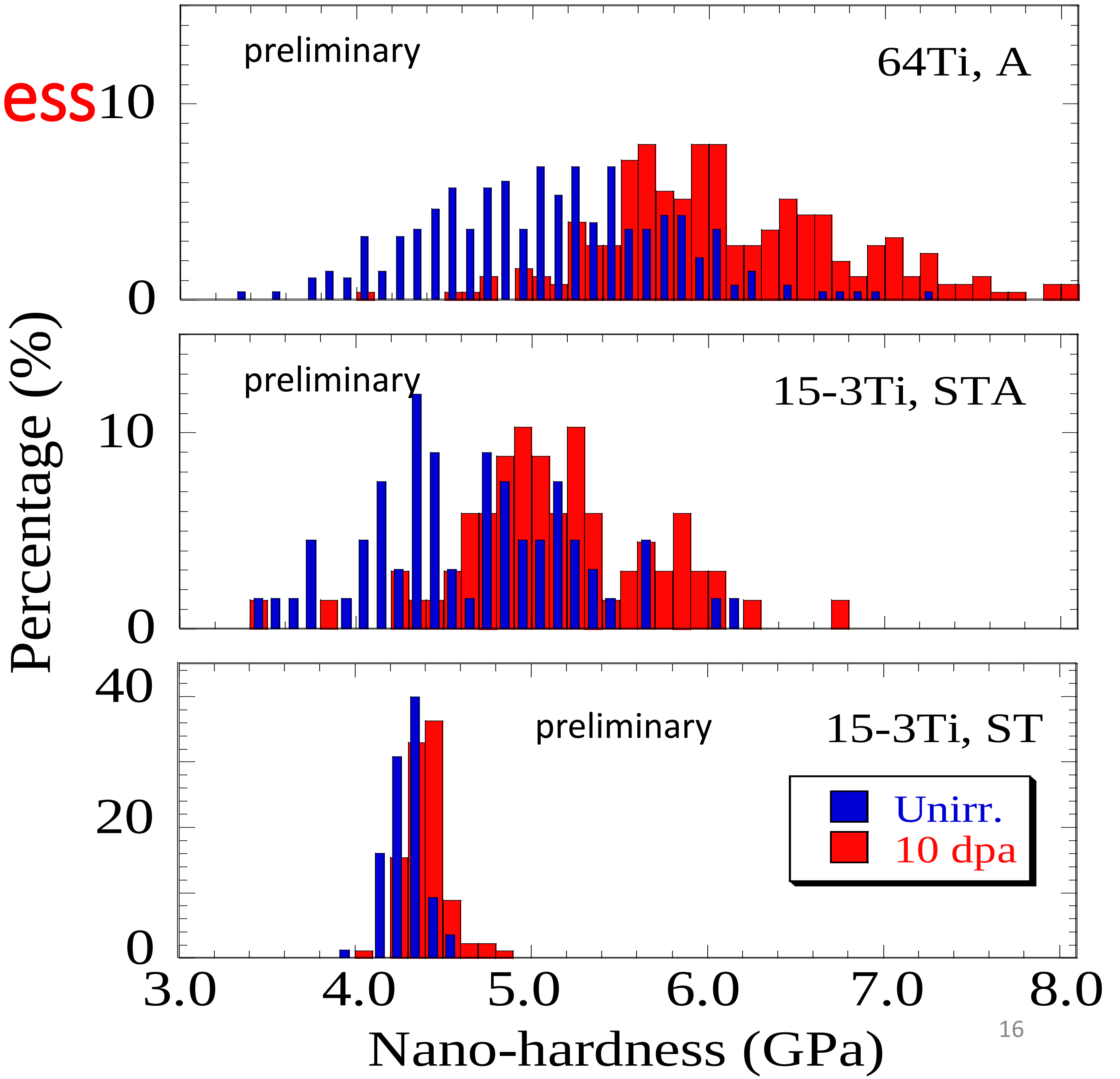
- The indentation modulus **EIT** of the specimen material is related to the reduced elastic modulus Er and the indenter modulus Ei:

$$E_{IT} = \frac{1-(v_s)^2}{\frac{1}{E_r} - \frac{1-(v_i)^2}{E_i}}$$

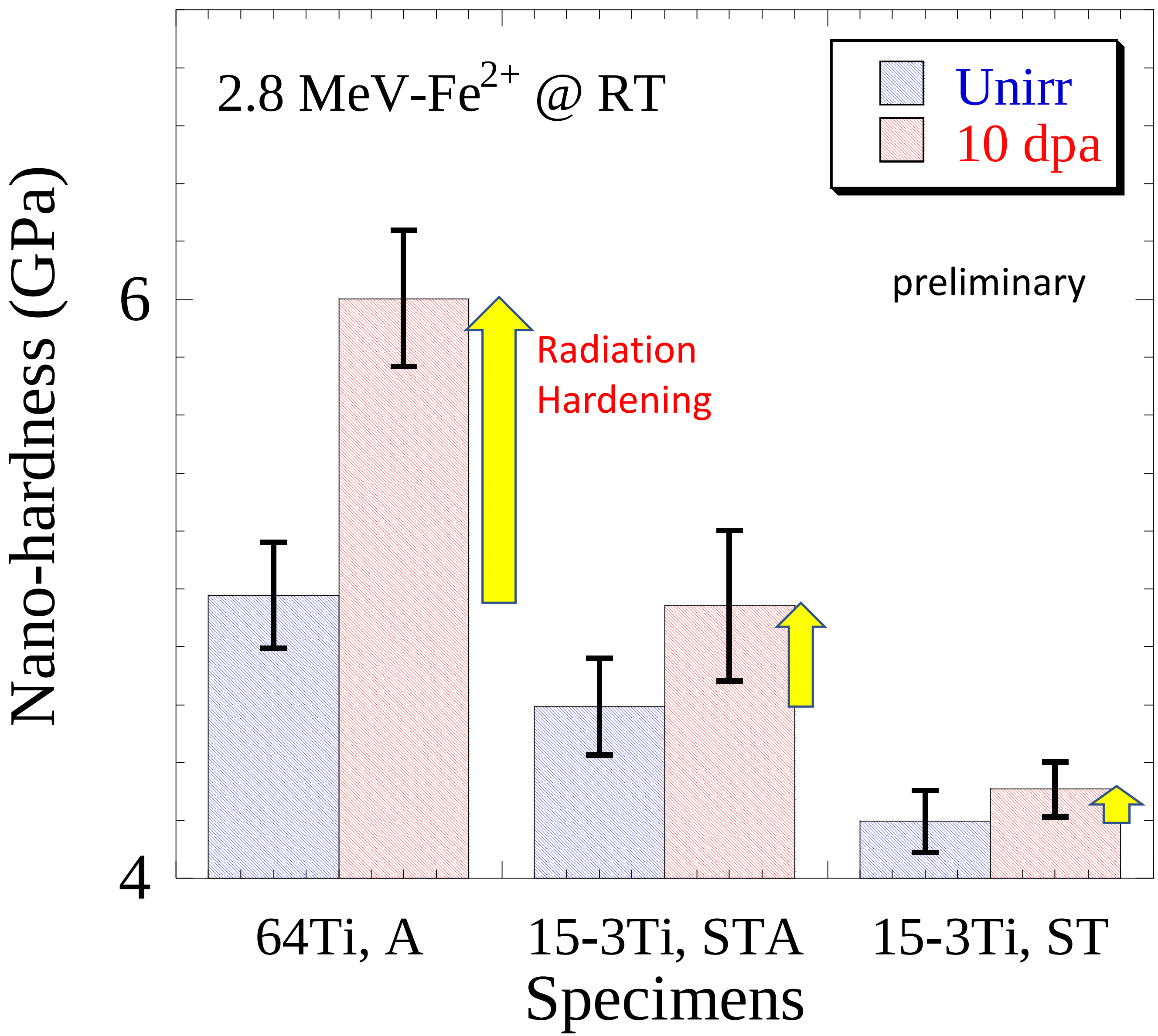
Ei , νi : Elastic modulus, Poisson's ratio of Indenter
Vs: Poisson's ratio of specimen,


6-4(A) 300 indentations (10μm intervals)

15-3(ST) 100 indentations

Result: Nano-hardness Distribution



Result: Ti Alloys – Averaged Nano-Hardness



Crystal orientation effect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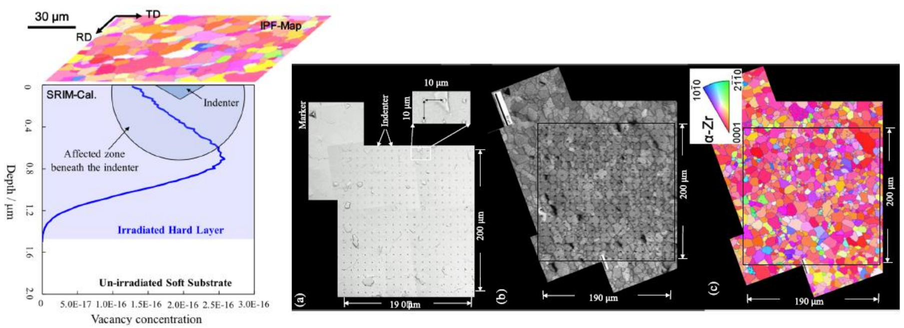
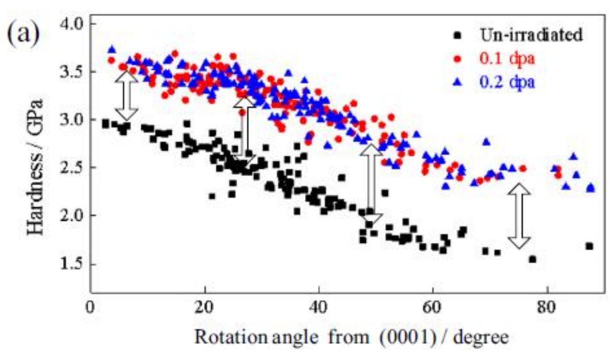
Surface orientation dependence of irradiation-induced hardening in a polycrystalline zirconium alloy

H.L. Yang [a,*], S. Kano [a], J. McGrady [a], J.J. Shen [a,1], Y. Matsukawa [b], D.Y. Chen [c], K. Murakami [d], H. Abe [a,*]

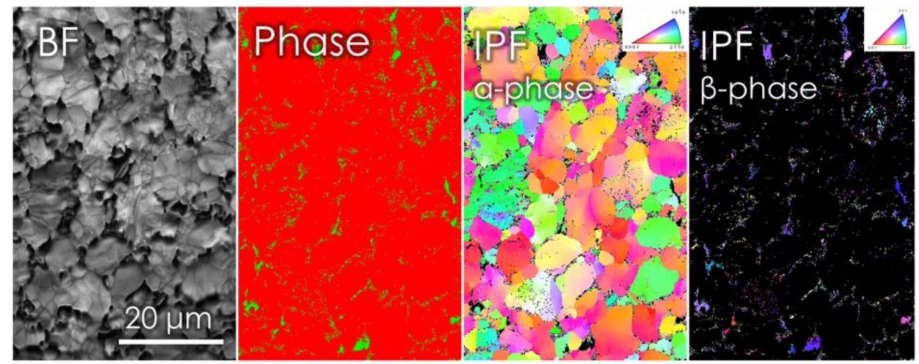
[a] Nuclear Professional School, School of Engineering, The University of Tokyo, 2-22 Shirakata Shirane, Tokai, Ibaraki 319-1188, Japan
[b] Graduate School of Engineering, Tohoku University, 2-1-1 Katahira, Sendai 980-8577, Japan
[c] Department of Nuclear Engineering and Management, School of Engineering, The University of Tokyo, 7-3-1, Tokyo, Hongo, Bunkyo, 113-8656, Japan
[d] Institute of Gigaku, Nagaoka University of Technology, 1603-1 Kamitomioka, Nagaoka, Niigata 940-2188, Japan

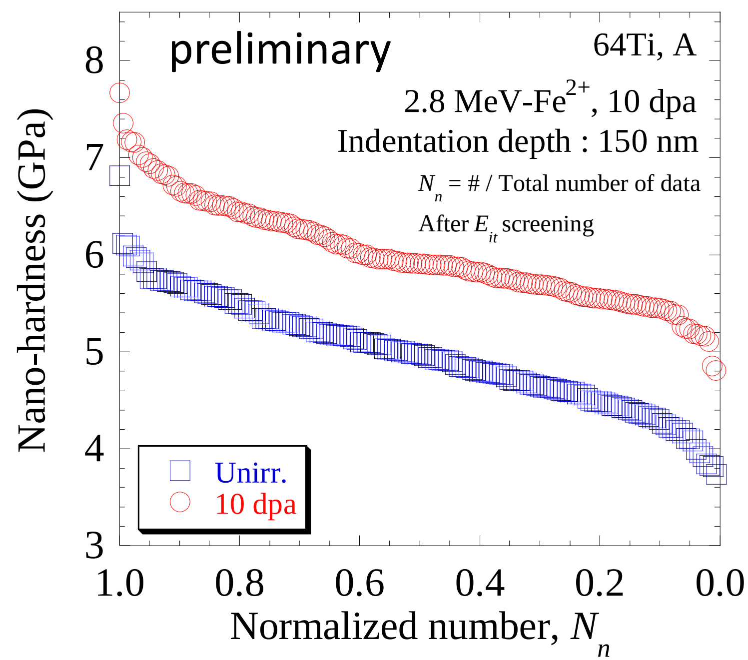




- Nano-hardness in hcp material is strong affected by the crystal texture.

64Ti, A

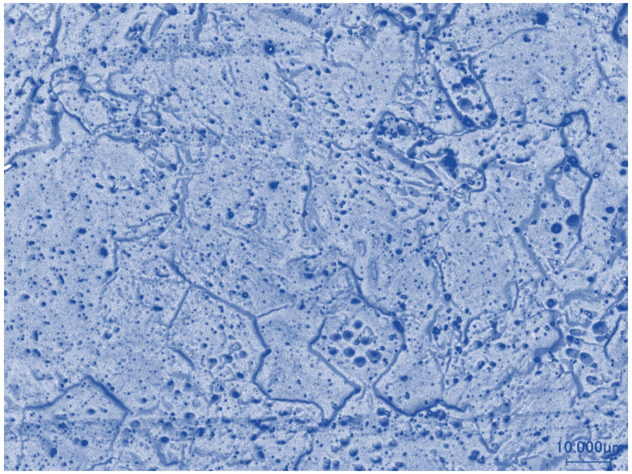




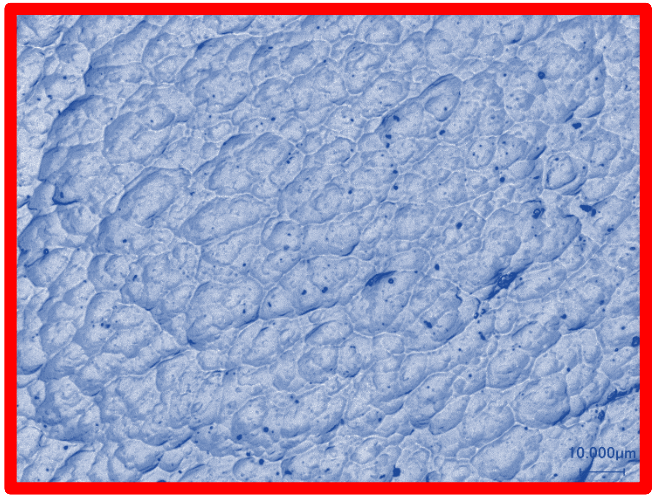
Near (0001) Near (2$\bar{1}\bar{1}$0)

TFGR-W irradiation test

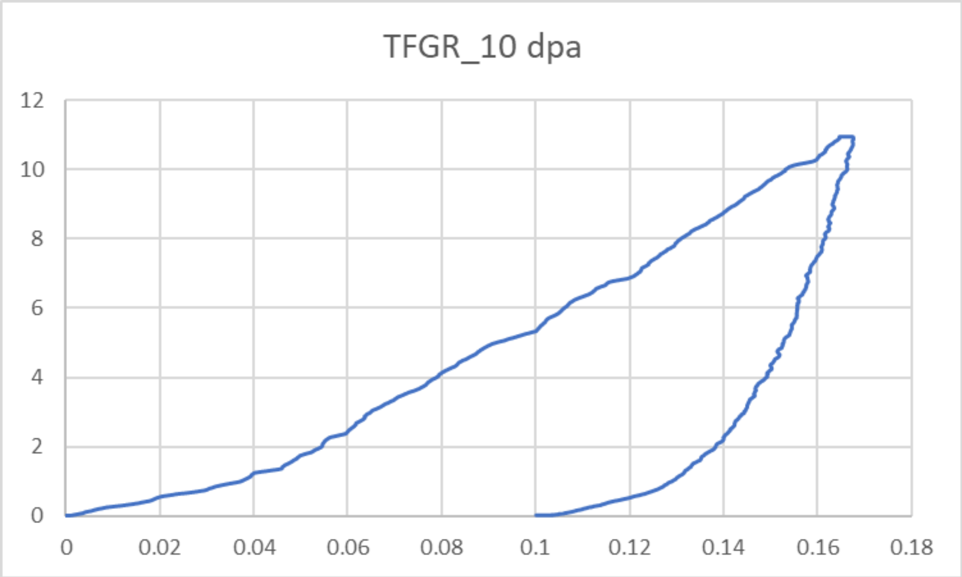
- Electro-polishing process could not be optimized on time.
- Many pivots were observed on the specimen surface
- Load-strain curve is not smooth, probably because indenter slides on surface
- Improvements on electro-polish process are on-going
- Results to be obtained at next irradiation scheduled on March 2020.



Pure tungsten: x1000



TFGR tungsten: x1000



Summary

1. High-DPA ion beam irradiation experiment has been conducted at the HIT facility on Titanium alloys and Tungsten alloys: $\alpha+\beta$ 64-Ti, metastable-β 15-3-Ti, pure W and TFGR-W were irradiated to 1, 5, and 10 dpa (W: 10 dpa only) by Fe^{2+} at ambient temperature.
2. Preliminary results of nano-hardness measurements for 64-Ti(A), 15-3-Ti (ST), and 15-3-Ti(STA) irradiated to 10 dpa were reported.
3. The radiation hardening of 15-3-Ti alloys were significantly smaller than that for 64-Ti alloy. Especially, hardness of 15-3-Ti(ST) stays the same within the statistical error after the irradiation to 10 dpa.
4. This may suggest that the metastable-β Ti alloys exhibit high defect sink strength, because of their nature to employ “precipitation strengthening”, nano-scale precipitates act as defect sink cites.
5. Next irradiation at the HIT facility will be conducted in March 2020 on aerospace grade metastable β Ti alloy(s), which has better creep property to higher temperature than 15-3 Ti (200^{o}C).
6. The TFGR-W will also be examined with optimized polishing.

Thank you for your attention.