An Exploratory Production of Titanium-Based ODS Alloy Material

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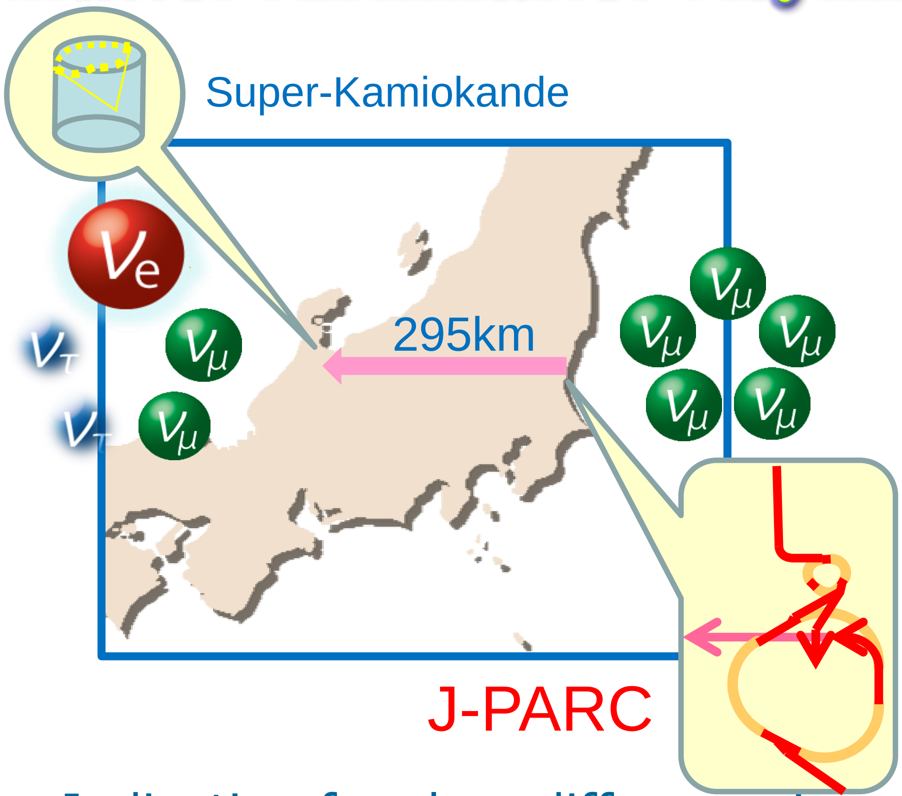
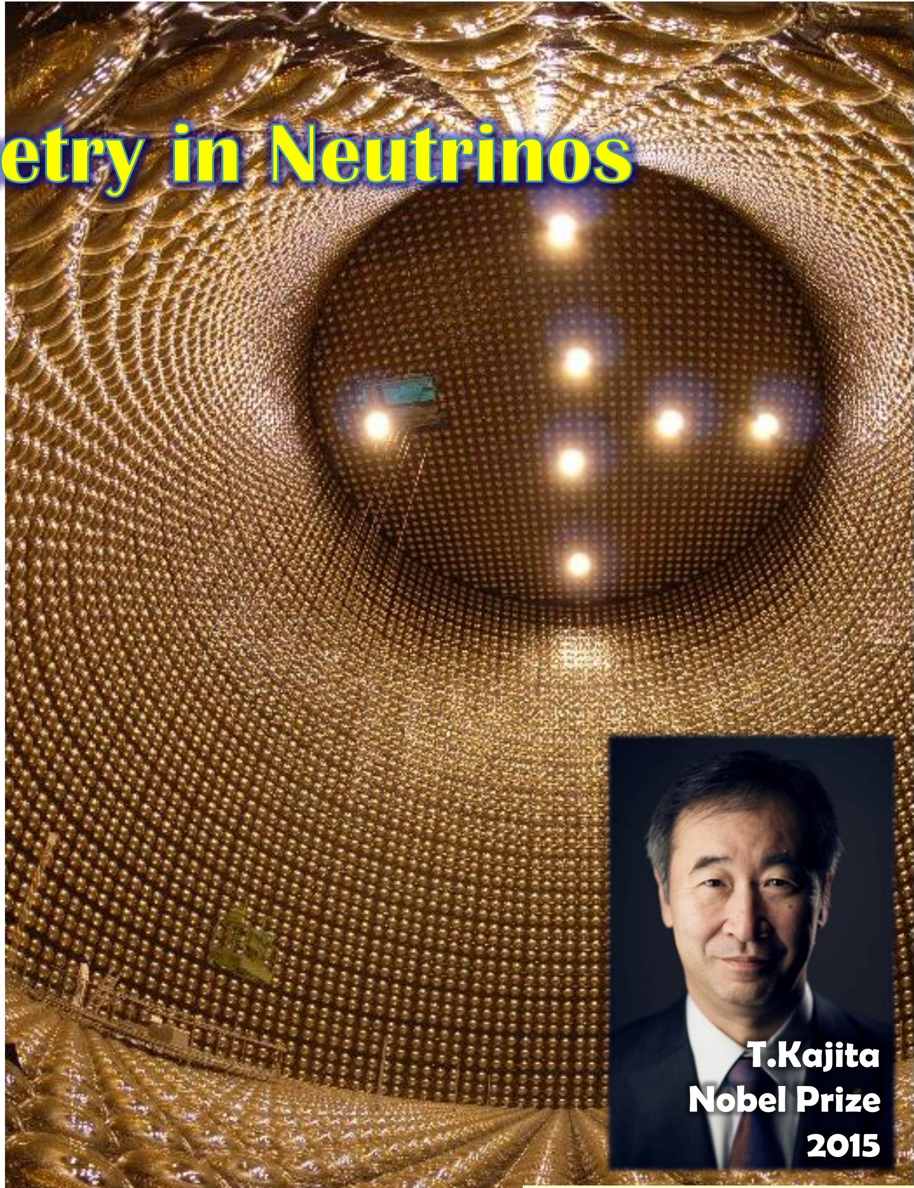
Another alchemists' dream !

1. Introduction

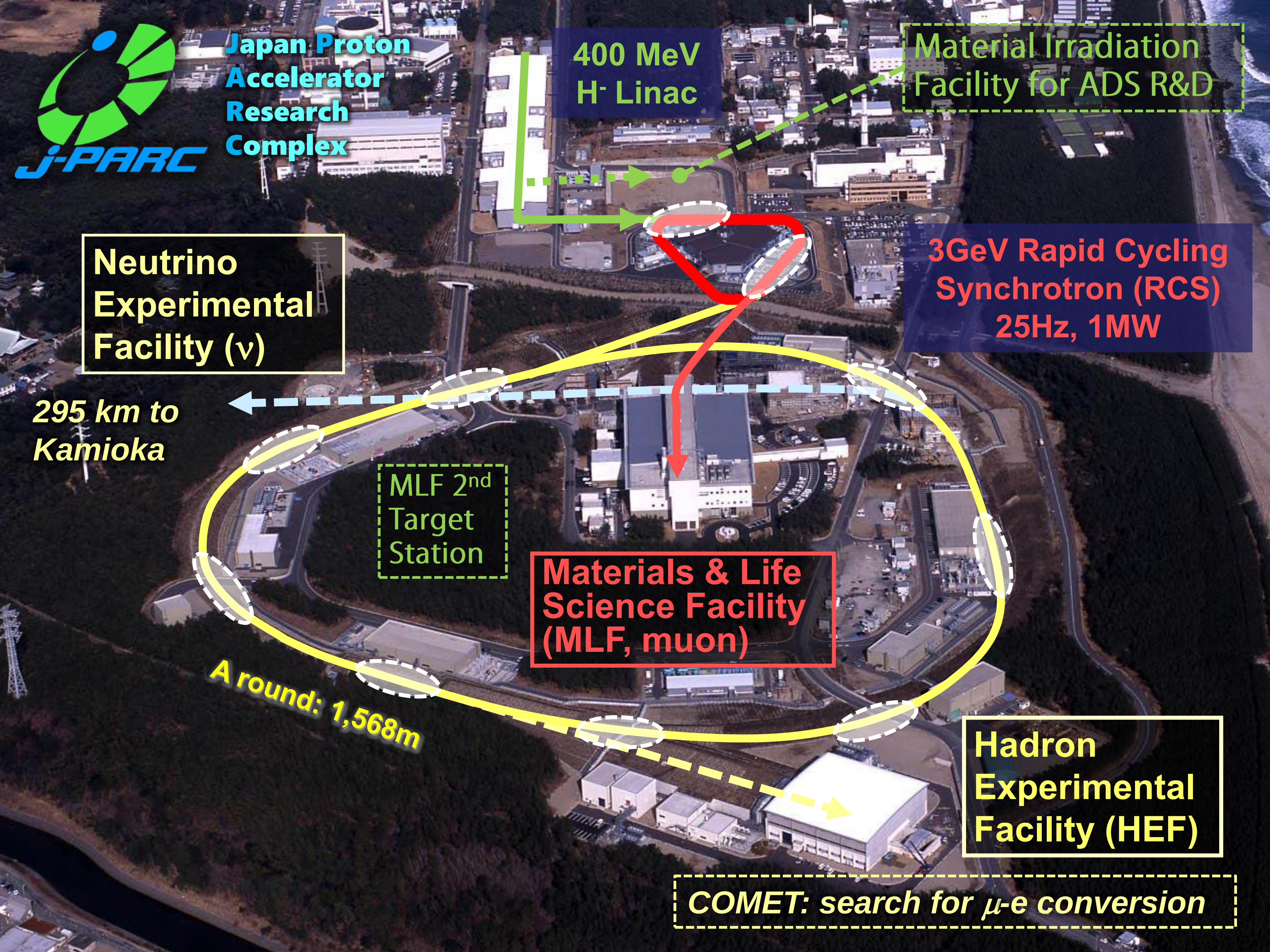
Matter-Antimatter Asymmetry in Neutrinos



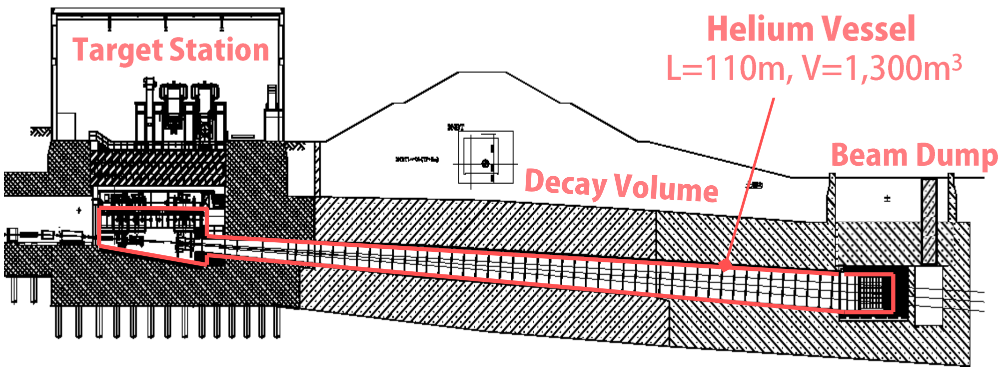


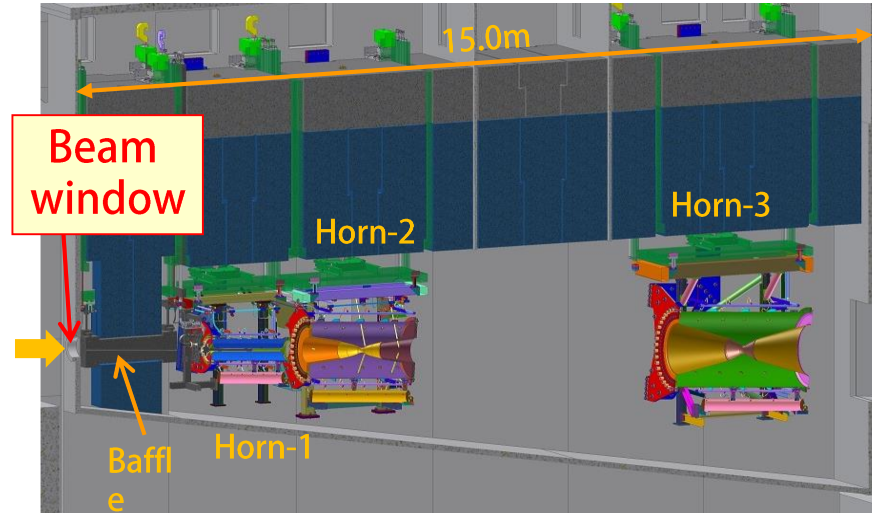
Super-Kamiokande
39mϕ x 41mH (50 kt)

- Indication for clear difference in "electron neutrino appearance" between neutrino and anti-neutrino beams
- Possible origin of our matter-dominant universe
- Need more statistics: *Higher Beam Power* (& Larger Far Detector)

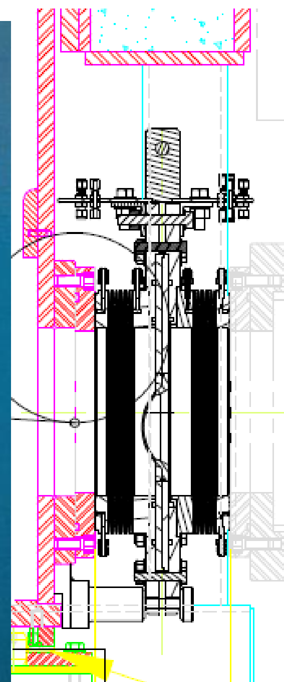

J-PARC
Japan Proton Accelerator Research Complex
400 MeV H- Linac
Material Irradiation Facility for ADS R&D
3GeV Rapid Cycling Synchrotron (RCS) 25Hz, 1MW
Neutrino Experimental Facility (ν)
295 km to Kamioka
MLF 2nd Target Station
Materials & Life Science Facility (MLF, muon)
A round: 1,568m
Hadron Experimental Facility (HEF)
COMET: search for μ-e conversion

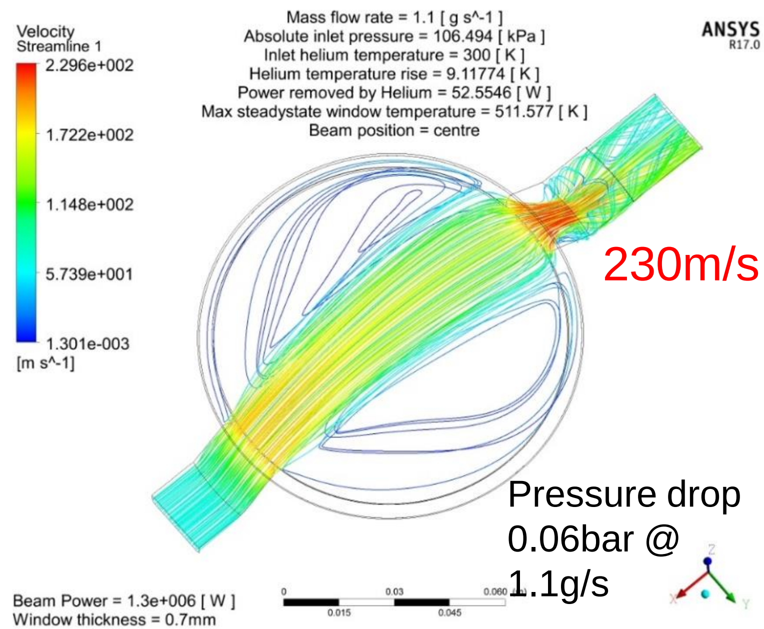
J-PARC Neutrino Beamline & Ti Beam Window



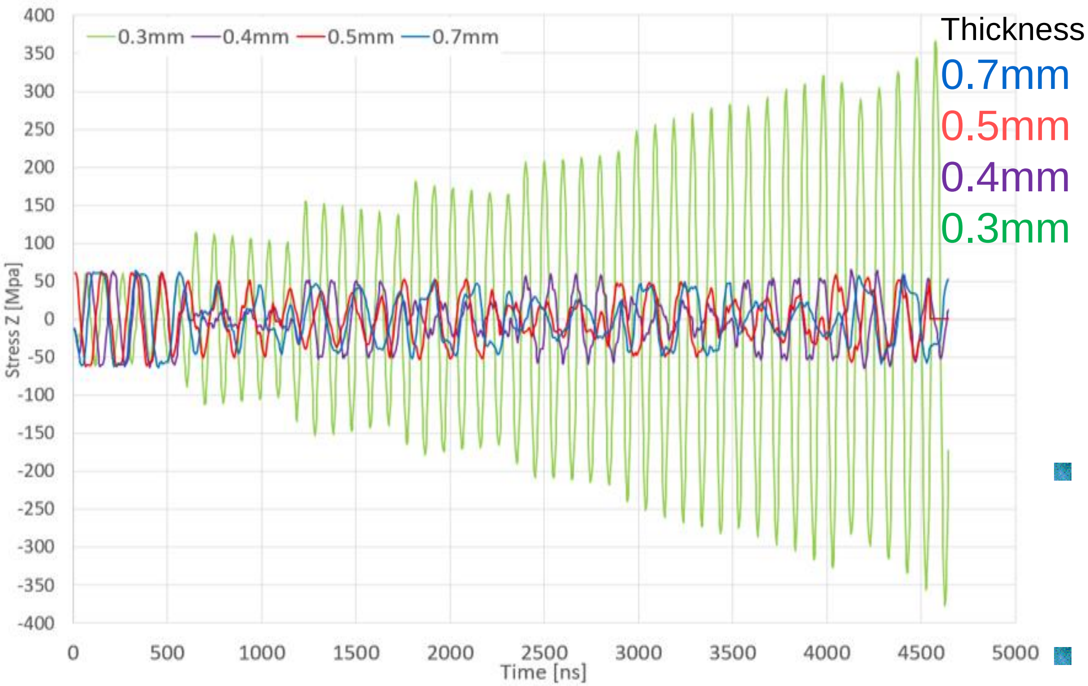


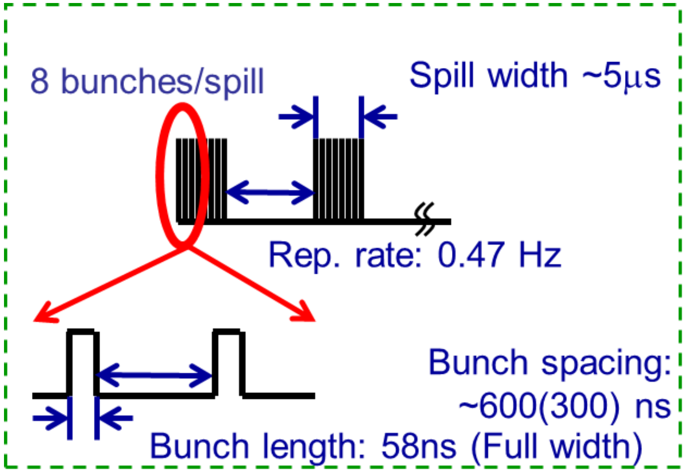
An $\alpha+\beta$ dual-phase titanium alloy
Ti-6Al-4V ASTM Grade 5

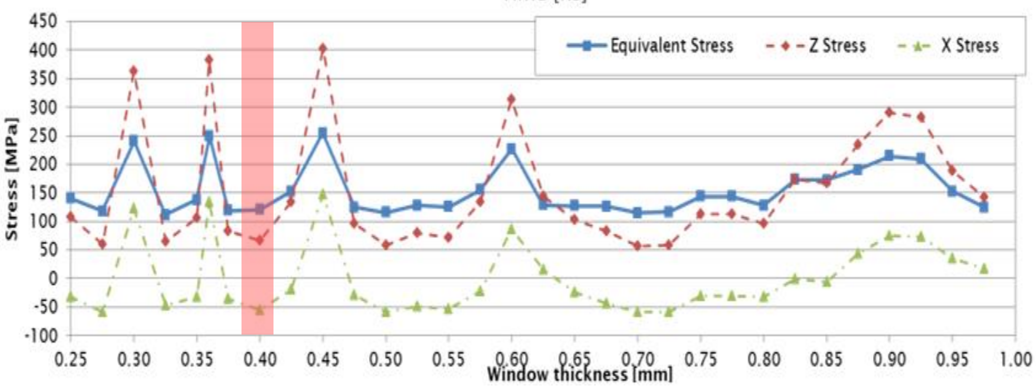




Stress in Beam Direction at Window Centre (1.3 MW beam operation)



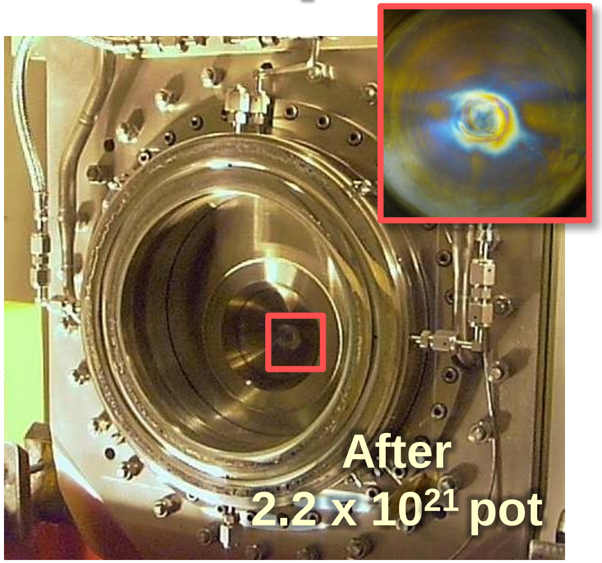




- Beam bunch structure generates stress resonance within material thickness
- Constructive interference at 0.30 mm (S.F.=1.5)
- Destructive interference at 0.40 mm (S.F.=2.6)
- 0.4 mm selected for next Ver.2 beam window
- Taking changes of E under high temperature into account: 0.39±0.01mm

J-PARC Neutrino Beamline Upgrade Technical Design Report
arXiv:1908.05141

Challenges for Target/Window Materials
Example: Neutrino Facility Ti-6Al-4V Beam Window



- **Thermal Shock**: >3x10^{14} 30GeV protons (1.3 MW flux) penetrare in a few mm^2 beam spot
  - Creation of periodic thermal stress wave in a few microsecond cycle
- **Radiation Damage**: irradiation hardening and loss of ductility with >0.1dpa
  - No higher data than 0.3 dpa exists
  - No known HCF data exists (Need >10$^{7\sim8}$ cycles)

Beam Power	PPP	Rep. cycle	POT / 100 days
485kW (achieved)	2.5 x 10^{14}	2.48 sec	0.9 x 10^{21}
1.3 MW (proposed)	3.2 x 10^{14}	1.16 sec	2.4 x 10^{21}

Energy Flux
1.3MW/(4mm)^2·π

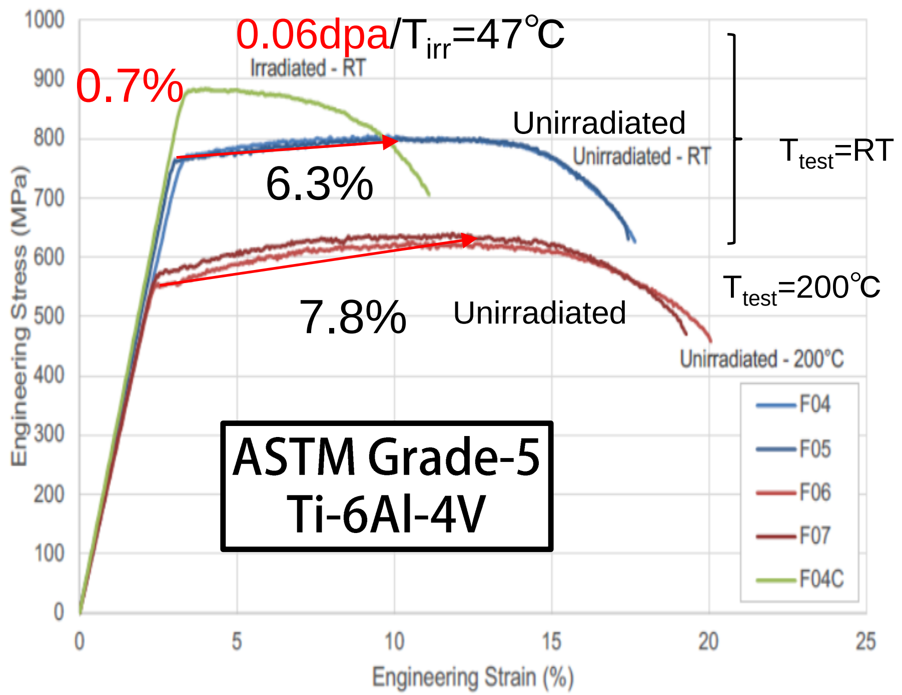
Thermal Shock
～ 200MPa

High Cycle Fatigue
～8M pulses/yr

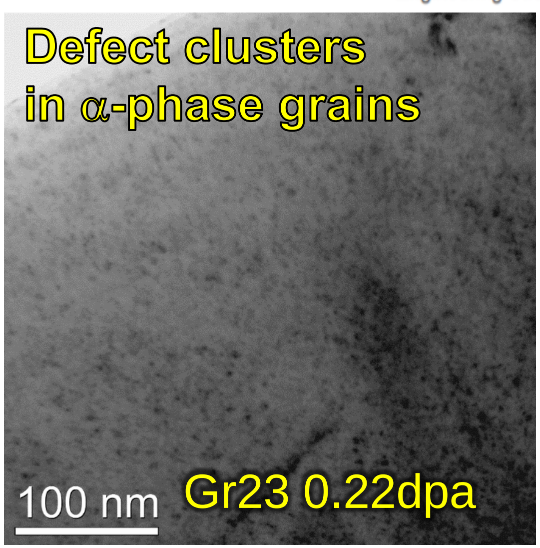
Radiation Damage
～2DPA/yr

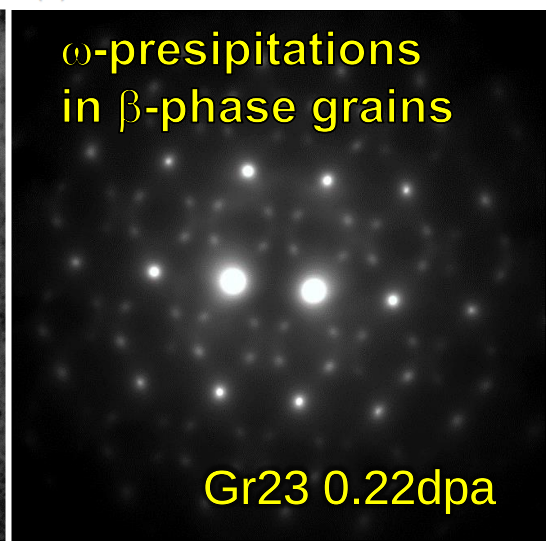
Innovation of target materials → *Breakthrough on Physics*

PIE on BLIP Titanium Specimens



- Ti-6Al-4V (most typical dual α+β phase alloy) showed increased hardness and a large decrease in ductility only with 0.06dpa
- Ti-3Al-2.5V (α-like α+β phase alloy) still exhibits uniform elongation (3%) after 0.22dpa irradiation





The radiation-induced ω-phase production in the β-phase could lead to greater loss of ductility in Ti-6Al-4V alloys in comparison with Ti-3Al-2.5V alloy with less β-phase.

→ T.Ishida et al (poster)

Objective

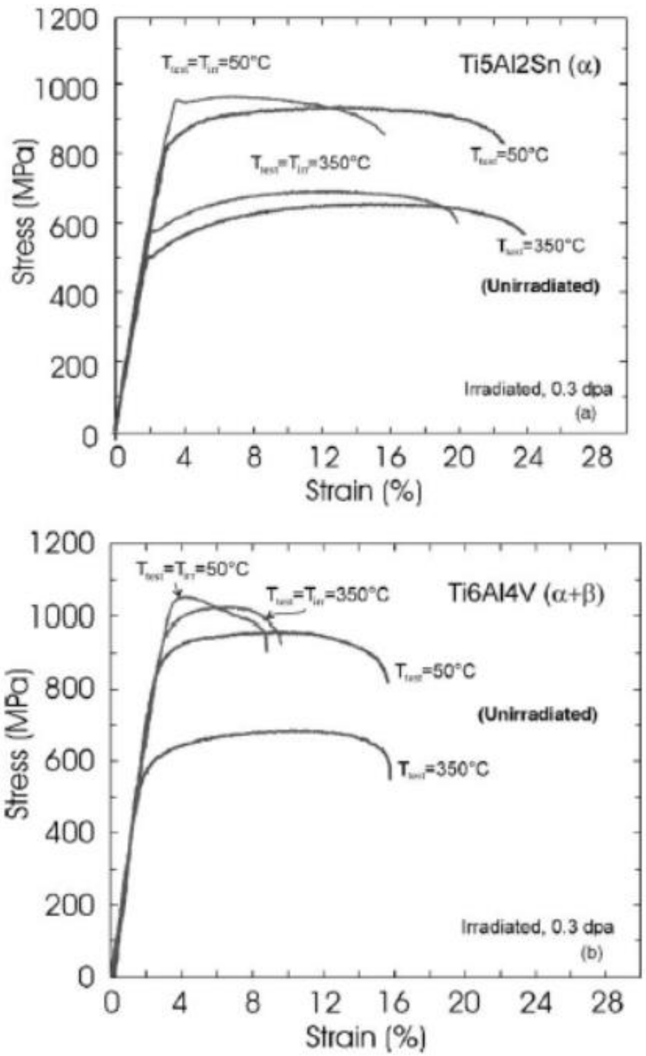


Fig. 2. Stress–strain curves for (a) Ti5Al2.5Sn (α) alloy and (b) Ti6Al4V (α + β) alloy tested at 50 and 350 °C in the unirradiated and irradiated conditions.

- High intensity proton beam penetration (~1MW beam power with 10^{14} protons-per-pulse) through the beam window in the area of a few tens of mm^2 area causes a few hundred MPa compressive stress, and initiates propagation of shock waves in a few microsecond cycle. Resultant high cycle fatigue can lead to failure of the radiation-embrittled beam window in a very short time scale.
- There are indications that single-HCP phase α-Ti alloys, such as Grade-6 Ti-5Al-2.5Sn, provide preferable ductility compared to the α+β dual phase Ti-6Al-4V.
- Recent work by some of the authors indicates that this is due to radiation induced ω-embrittlement that occurs only in the β phase.
- If improved radiation tolerance of the single α-phase alloy is realized, it can be adopted as the beam window material in future accelerators with upgraded power, which should bear damage levels of one to a few dpa-NRT within one year operation.

S. Tahtinen et al., JNM 307-311 (2002) 416-420

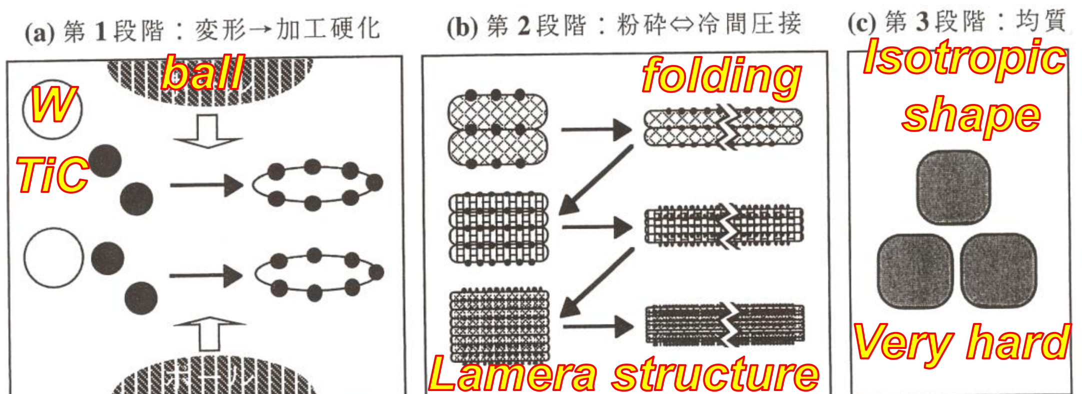
2. Reference Method

- By following the method developed on pure vanadium by one of the authors*, pure titanium powder with addition of ~2 wt-% yttrium is processed with mechanical alloying (MA).
- It aims to convert solute oxygen and nitrogen impurities, which are originally contained in the base powder (and will become harmful sources to decrease ductility), to form a large number of nano-scale precipitates, working as sink sites of radiation-induced defects.
- The MA process dissolves all added Y into the base Ti matrices. Subsequent thermo-mechanical treatments cause reactions between solute Y and O/N impurities, resulting in oxide (Y_2O_3) and nitride (YN) dispersed in the base Ti matrices as nano-scale precipitates.

Mechanically infuse kinetic energies into powders with equal to or more than 2 elements.

- ✓ *Forced solid solution state in RT, which never realized in equilibrium*
- ✓ *Fine-equiaxed crystal grains, typically 20~30nm in size*
- ✓ *Particle dispersion structure in a some~several nm in size*

1. Deformation → Work hardening | *2.Fragmentation ←→ cold welding* | *3. MAed Powder*

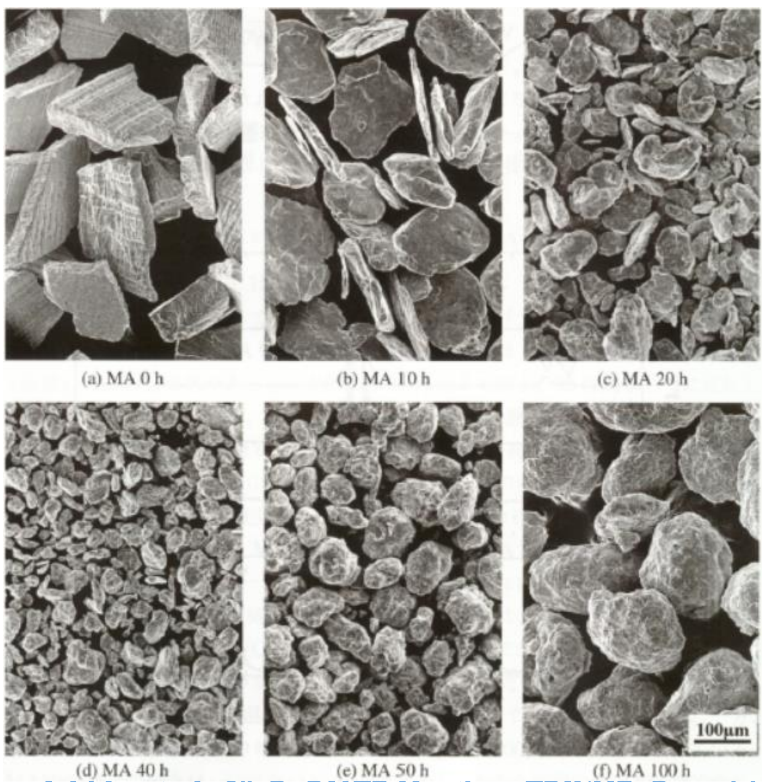


Vanadium-based ODS Alloy

4A	5A	6A
Ti	V	Cr
Zr	Nb	Mo
Hf	Ta	W

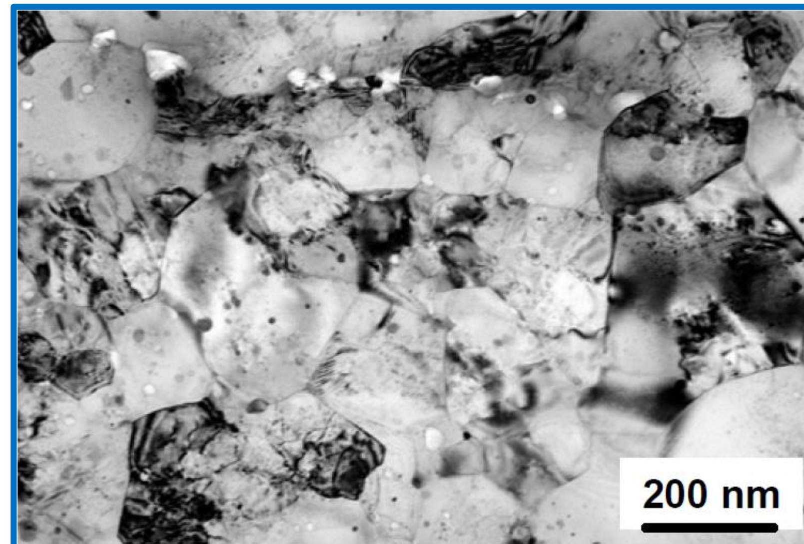
Group 4A/5A:

- High ductility
- Large chemical activity
- Large solubility of interstitial gas elements
- Resulting environmental embrittlement



Radiation-damage tolerance

✓ Micronized crystal grains
✓ Nano-scale precipitation



O/N impurities: 0.27±0.05wt%
(base powder & MA-HIP process)
→ Y=1.2±0.3wt%

Y=1.6~1.7wt%

* "Microstructure Control to Improve the Resistance to Radiation Embrittlement in Vanadium", H.Kurishita et al., J. Nucl. Mater. 343 (2005) 318-324.

3. Production Procedure

- Titanium: Toho-tech Co. TC-150, pass through a sieve with a mesh size of 150 μm
- Yttrium: Nippon Yttrium Co. 99.9% -20# powder

Impurities in Ti

Fe	0.02 %
Si	0.01 %
Mn	<0.01 %
Mg	<0.001 %
Cl	0.002 %
C	<0.01 %
N	0.01 %
O	0.09 %
H	0.02 %

→ Y for O/N in base powder : 0.40wt%
(0.33wt% in Y_2O_3 + 0.06wt% in YN)

Follow ODS-V: Y = ~2 wt%

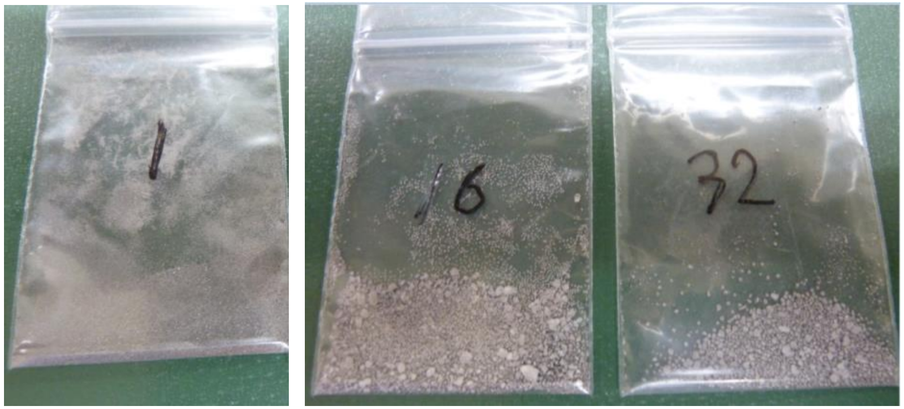
-RECIPE-

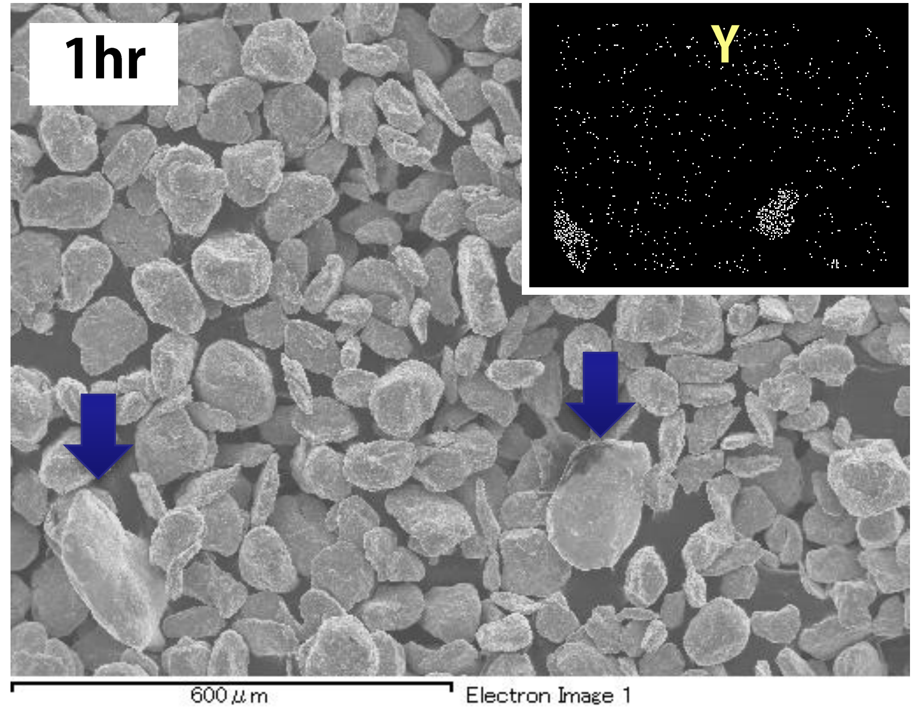
- Titanium powder: 75g
- Yttrium powder: 1.5g
- Stainless Steel 3/16" Balls: 300g

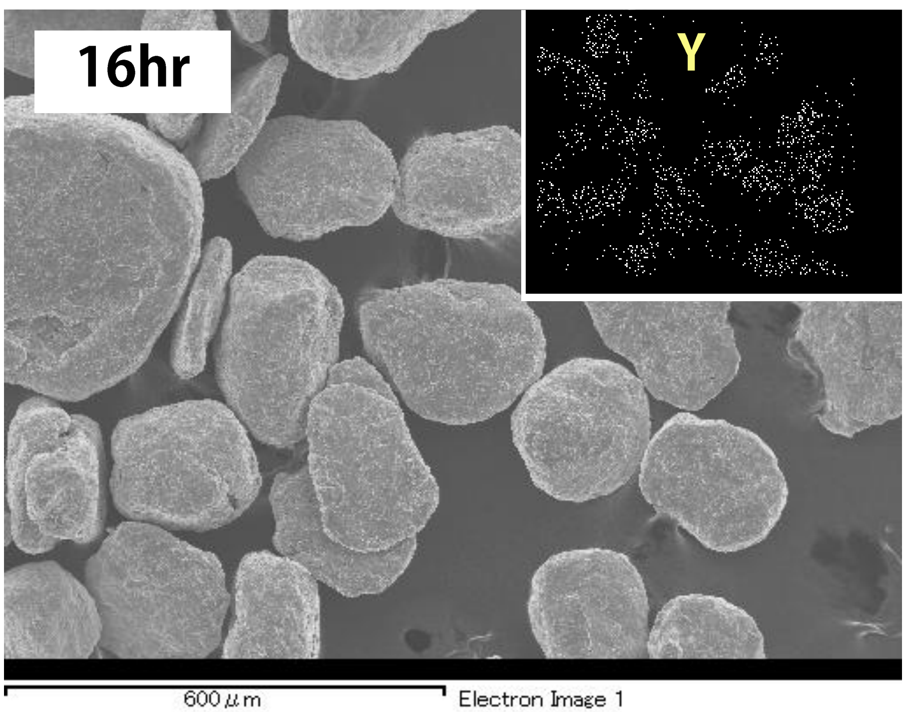
In a 250ml Hardened Stainless Steel Bowl
Planetary ball mill, 250 r.p.m.
Inspection at 1,2,4,8,16, 32 hrs.

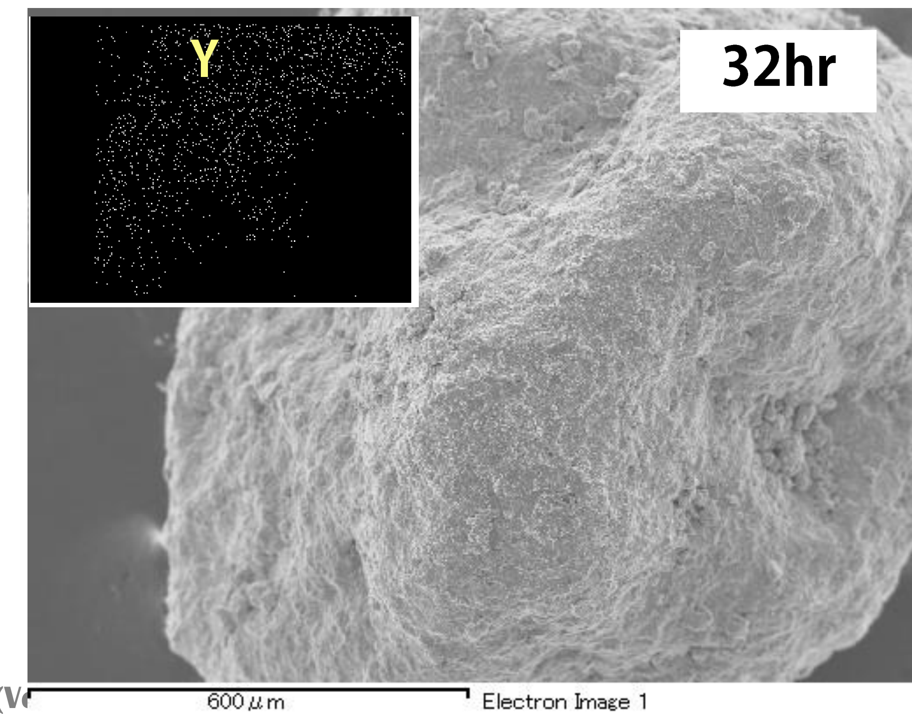
Mechanical Alloying

Powder SEM


1
16
32

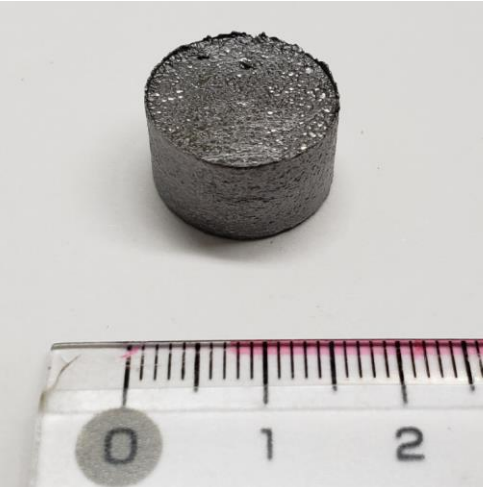

1hr
Y
600 µm
Electron Image 1

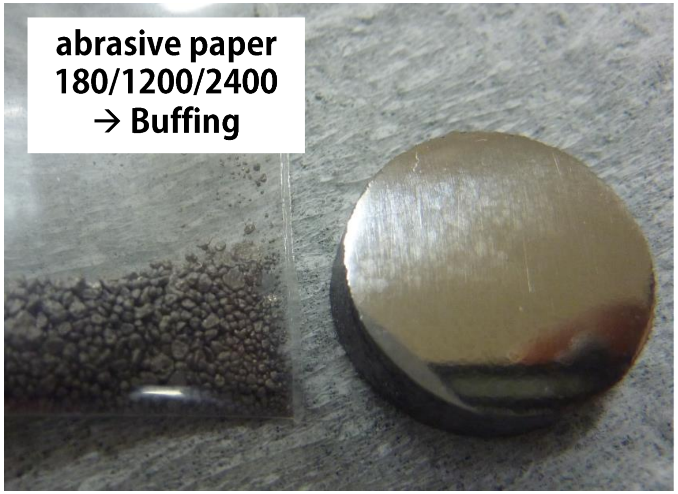

16hr
Y
600 µm
Electron Image 1

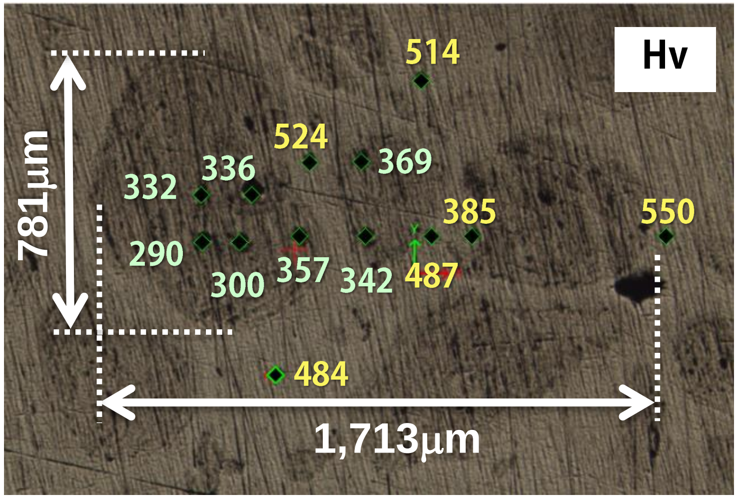

32hr
Y
600 µm
Electron Image 1

4. Alloy Characterization

Spark Plasma Sintering (Ed-Pas) at 800℃×15min

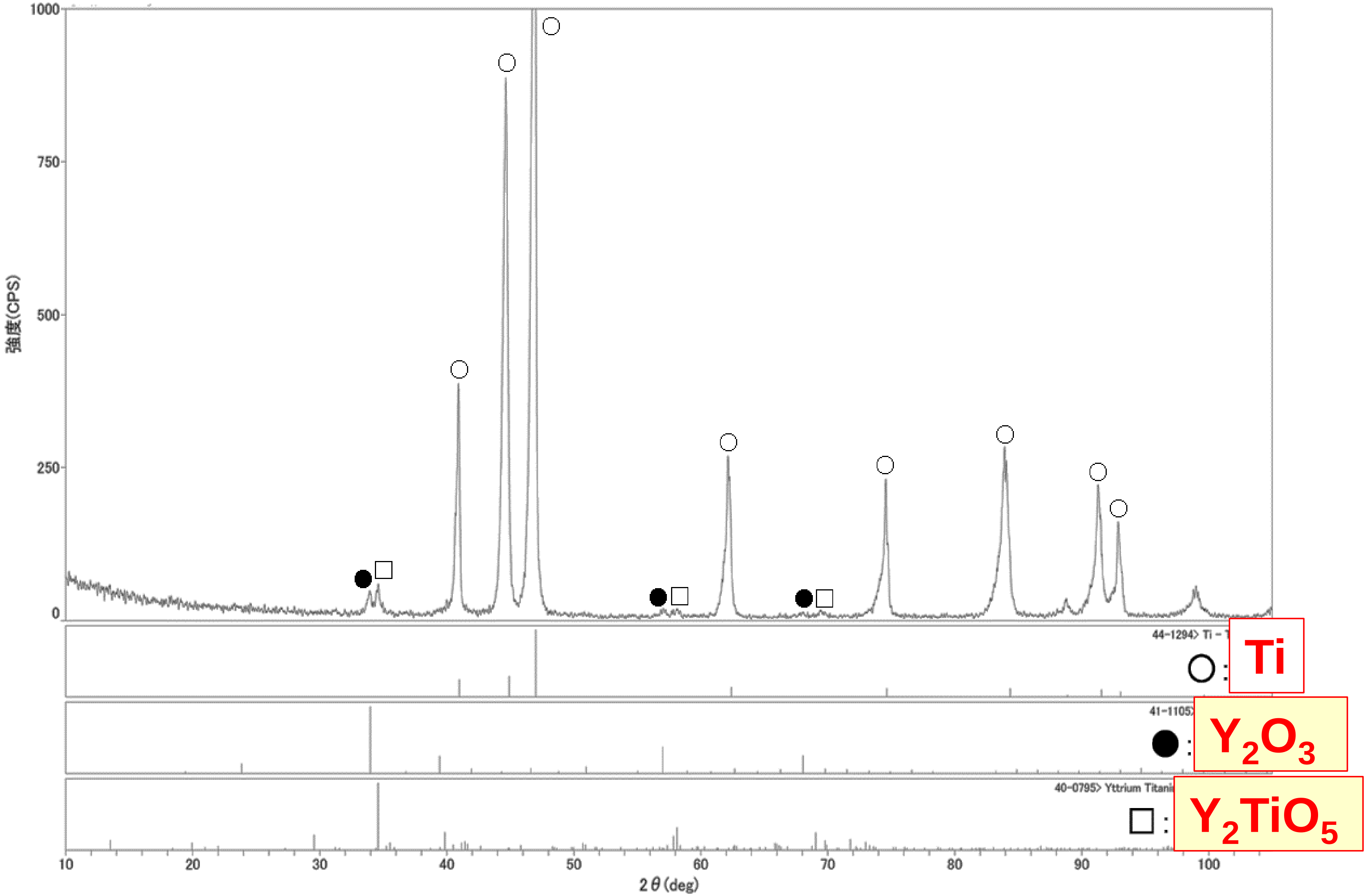






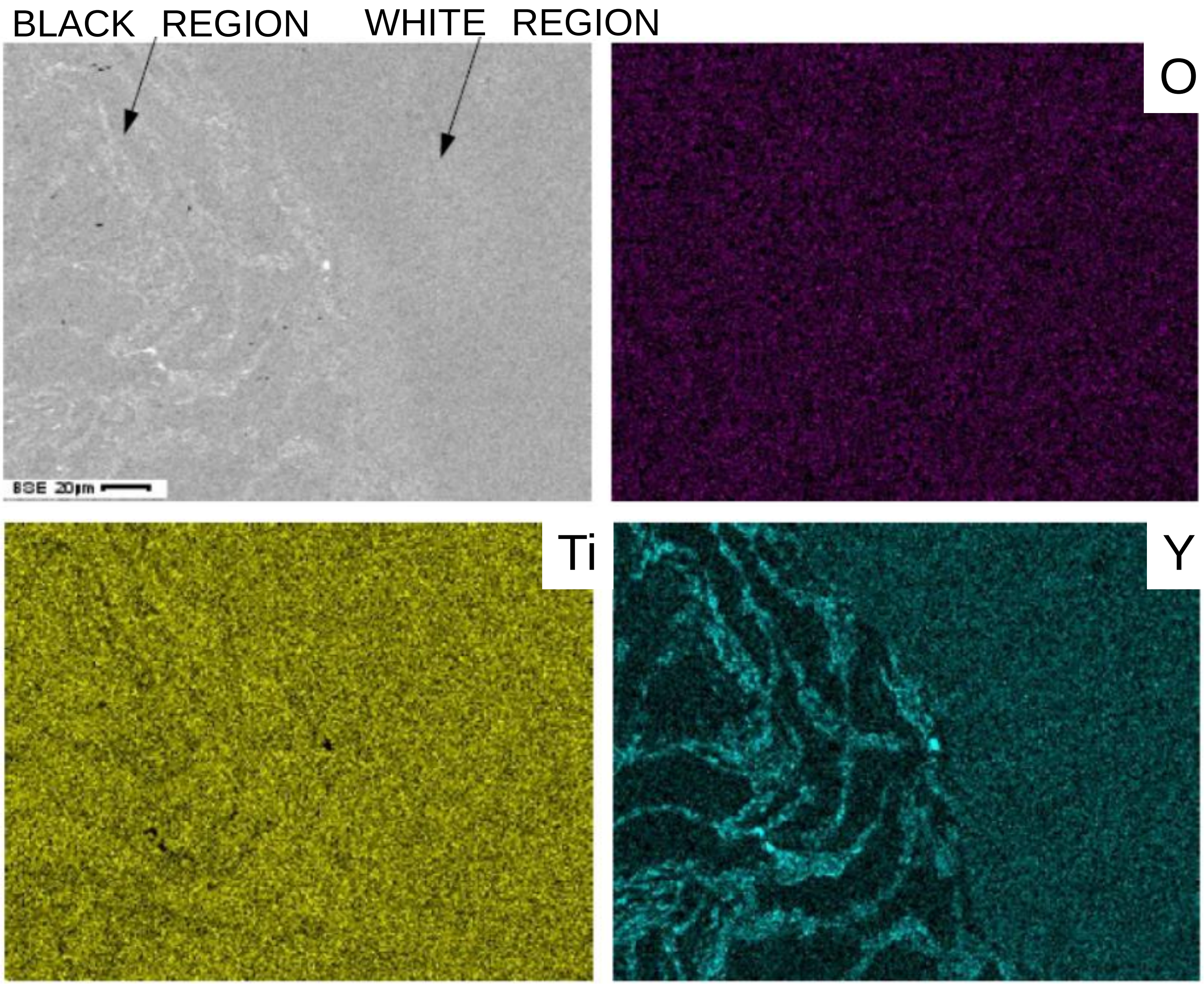
- Density = 4.50(bulk density)/4.52(apparent density)
  cf. Ti: 4.506
- Black(softer) regions with size O(0.5~1mm) surrounded by white(harder) regions.
- Correlation between hardness and oxygen concentration (max solubility: 15 wt%)

XRD



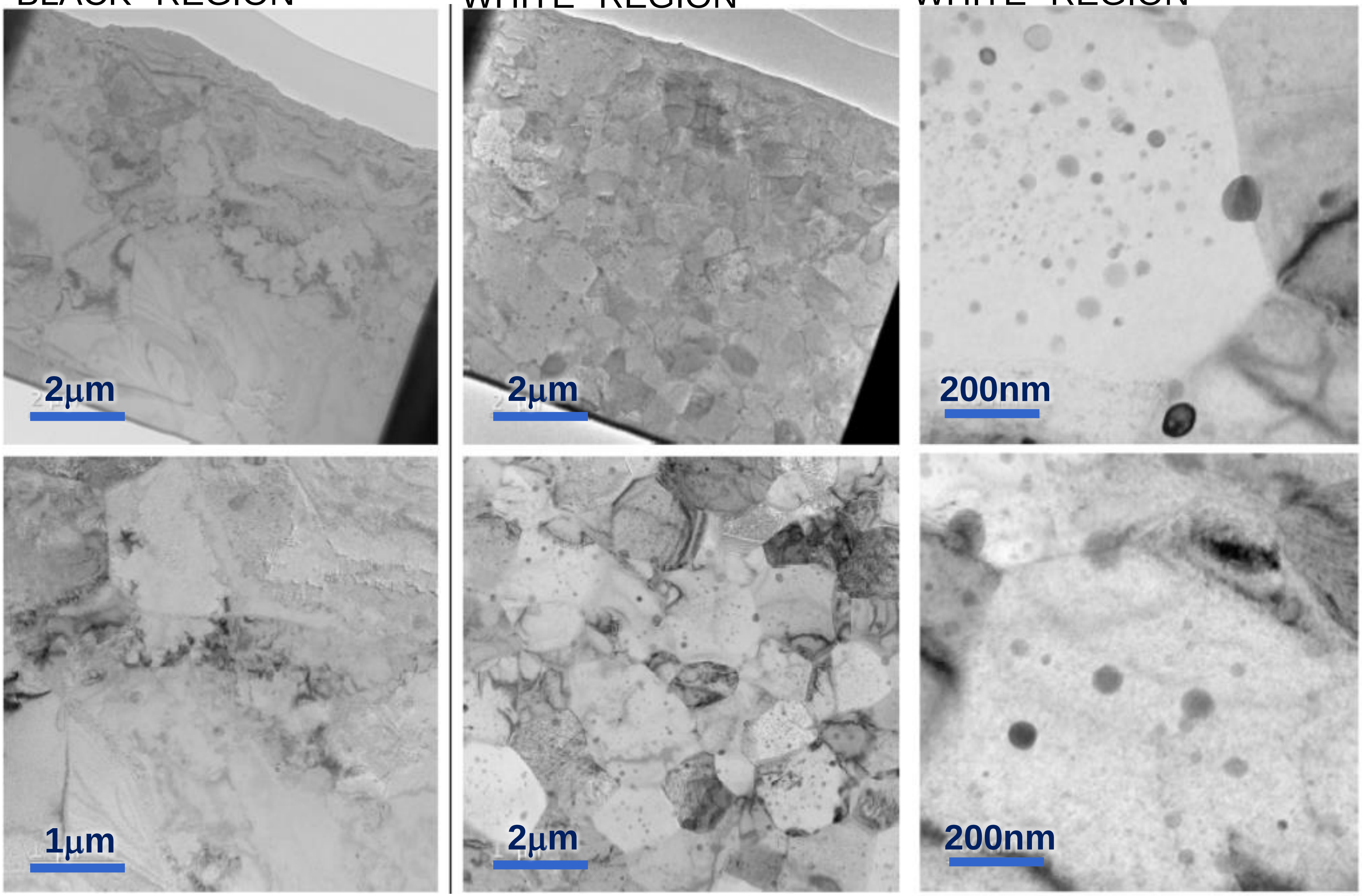
1000
750
500
250
0
強度(CPS)
44-1294> Ti - T
41-1105>
40-0795> Yttrium Titani
○ : Ti
● : Y2O3
□ : Y2TiO5
10
20
30
40
50
60
70
80
90
100
2θ (deg)

Elemental Mapping



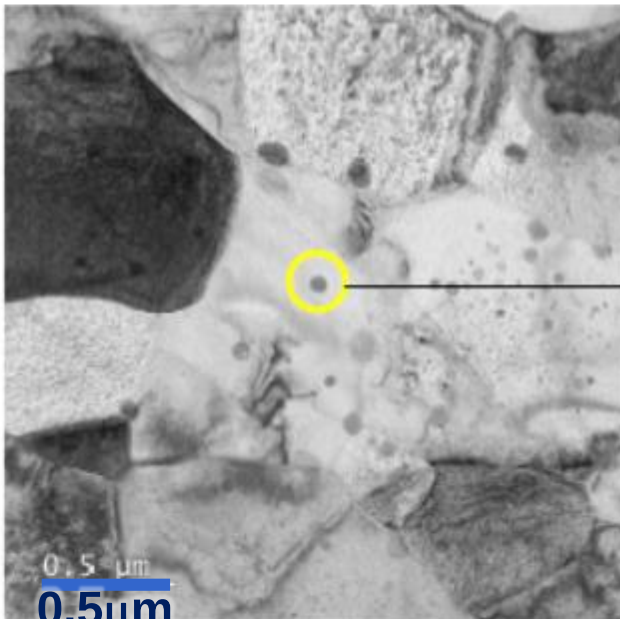
TEM

BLACK REGION　　WHITE REGION　　WHITE REGION

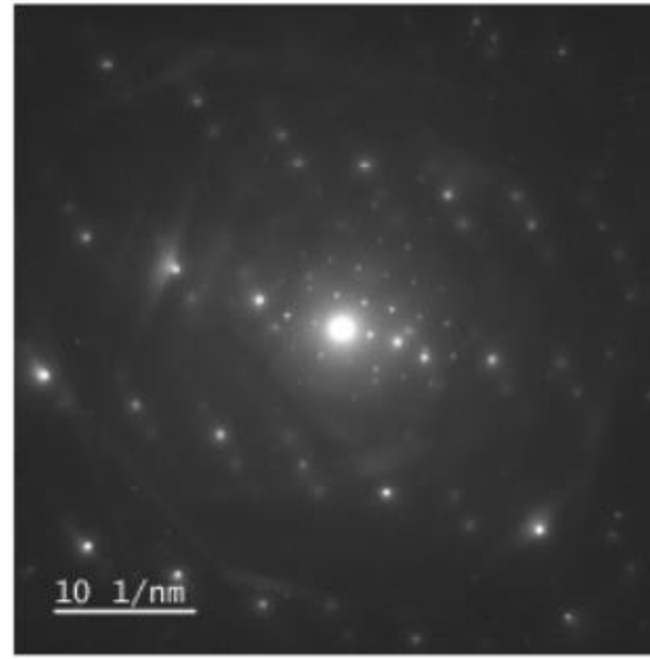


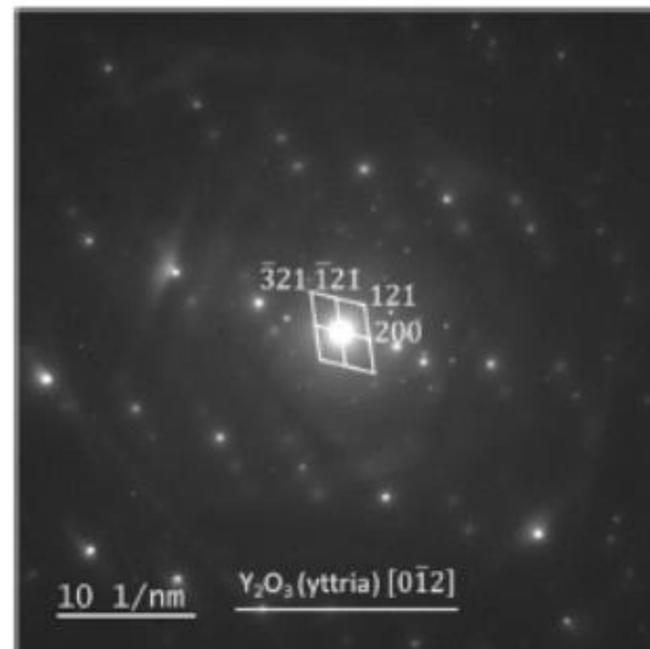
Identification of A Precipitate

WHITE REGION



同定した析出物





5. Discussion and Next Step

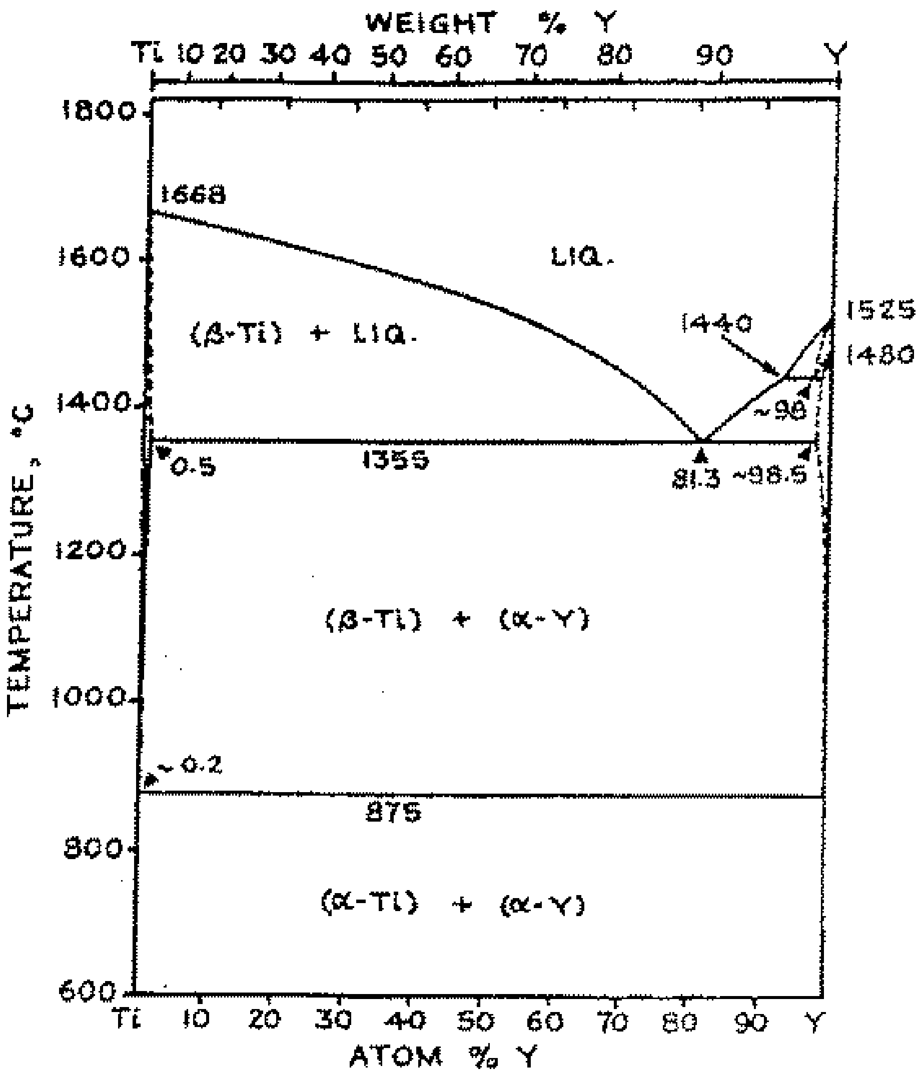
- The 1st exploratory production of ODS-Titanium alloy has been performed.
- Production of Y_2O_3 & Y_2TiO_5 has been identified successfully by XRD.
  - ✓ $Y_2Ti_2O_7$=1x(Y_2O_3) +2x(TiO_2), typically observed in ODS steels, are not observed. This may due to much higher solubility of oxygen into titanium than that into Fe. (Oxygen was not enough to form the yttria)
- Heterogeneous two regions appear in the produced alloy: white (hard) regions and black (soft) regions. Elemental Mapping exhibits the former is well MAed, while the latter not enough.
  - ✓ Difference of hardness in these regions may come from a variation of grain size and dispersion of precipitates
- In white (MAed) region, fine, equiaxed grains have been observed with less than 1μm in size. The precipitate particles are in sphere shape, seemingly larger than those typically observed in ODS alloys. This may limit the number density of the precipitates, and thus dispersion strengthening (and radiation resistance ?)

➢ Seek condition to realize homogeneous MA,

➢ Improve retrievable ratio of MAed powder
  - ✓ Avoid adherence to bowl/balls, exchange balls a few times during MA process

THANK YOU FOR YOUR ATTENTION !

Ti-Y equilibrium diagram



Mechanical Properties As func of Oxygen concentration