RADIETE COLLABORATION MEETING @TRIUMF

11TH DECEMBER, 2019

RESENT STATUS OF DEVELOPMENT FOR TUNGSTEN ALLOY, TFGR W-1.1%TIC, AS ADVANCED TARGET MATERIAL

HIGH ENERGY ACCELERATOR RESEARCH ORGANIZATION,
INSTITUTE OF PARTICLE AND NUCLEAR STUDIES

J-PARC CENTER, PARTICLE & NUCLEAR PHYSICS DIVISION

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- Development for TFGR W-TiC
  - Tungsten as target material
  - TFGR W-TiC
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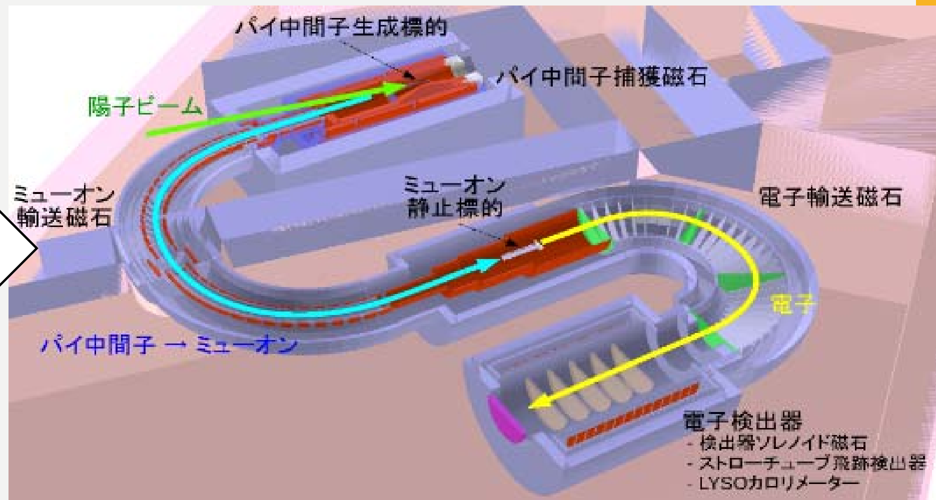
  - Present status of TFGR W-TiC
- Discussion to improve creep behavior in TFGR W-TiC
- Summary

PRIVATE AFFAIR

On 1st Oct. 2019

- I moved from "KEK, IMSS, Materials and Life Science Facility, Muon Science section" to "KEK-Institute of Particle and Nuclear Studies, J-PARC-Particle and Nuclear Physics division"
- Construction of Muon Target is completed, "COMET target" next

Muon Target @MLF, J-PARC
3 GeV, 1 MW
Graphite target



COMET target @hadron facility, J-PARC
8 GeV
P1: 3.2 kW, Graphite target
P2: 56 kW, SiC or tungsten target

THIS PRESENTATION IS SUPPLIED BY

- KEK-IPNS, J-PARC-Particle and Nuclear Physics division
- COMET collaboration
- Metal Technology Co., LTD
- Sunric Co., LTD
- Ehime University, CERN, HIT- Tokyo University,,,

Supported by

- MTC-KEK collaboration
- Grant-In-Aid, JSPS KAKENHI 19H01913
- Grant-In-Aid, The Small and Medium Enterprise Agency, METI
- Joint Usage/Research Center PRIUS, Ehime University, Japan

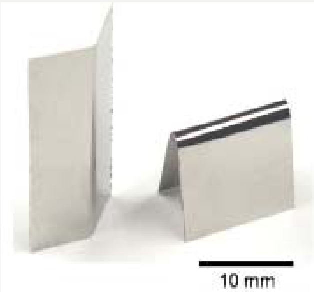
DEVELOPMENTS FOR TFGR W-TIC

TUNGSTEN AS TARGET MATERIAL

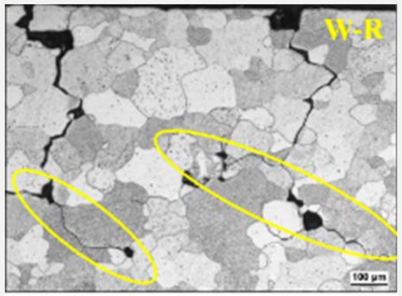
- Expected as the target material, due to <u>high density and high melting point</u>
- For use of <u>tungsten as muon production target</u>, the boundary between cooling material and vacuum in beamline disturb transportation of muon. Thermal radiation cooling is desired. <u>It should be used at high temperature</u>. (COMET, mu2e, MLF TS2,,,,)

✓ Tungsten is brittle, because grain boundary is weak.

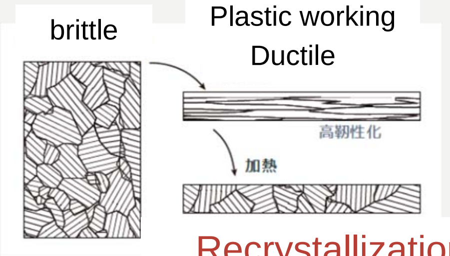
✓ Brittleness is improved by heavy plastic working.



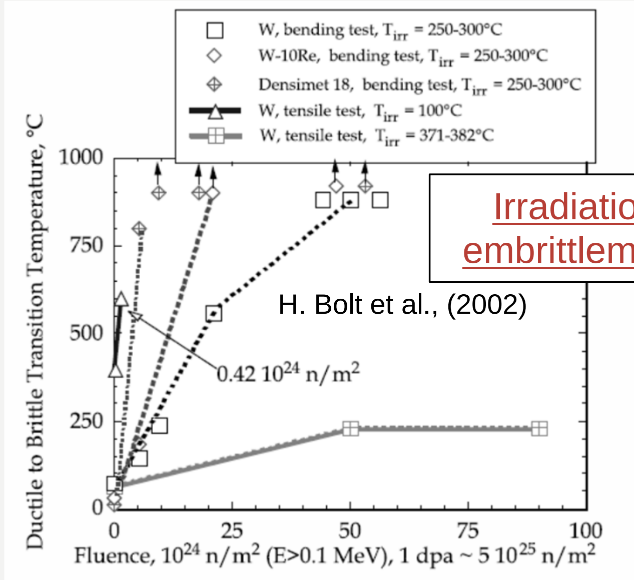
J.Reiser et al. JNM, 423 (2012) 1.



G. Pintsuk et al.



<u>Recrystallization embrittlement</u>



<u>Irradiation embrittlement</u>

H. Bolt et al., (2002)

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

- The use of W as target material is limited by

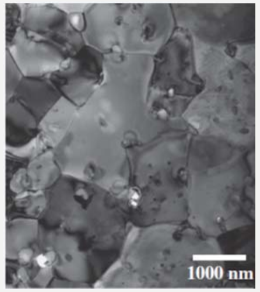
<u>"Recrystallization embrittlement" and "Irradiation embrittlement"</u>.

Since 2016

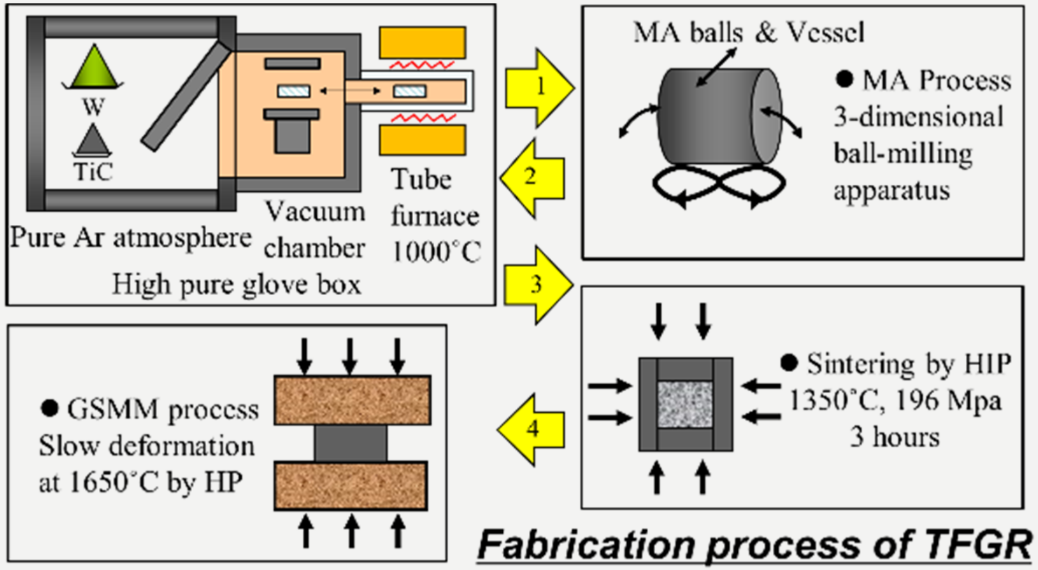
TFGR W-TiC

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.

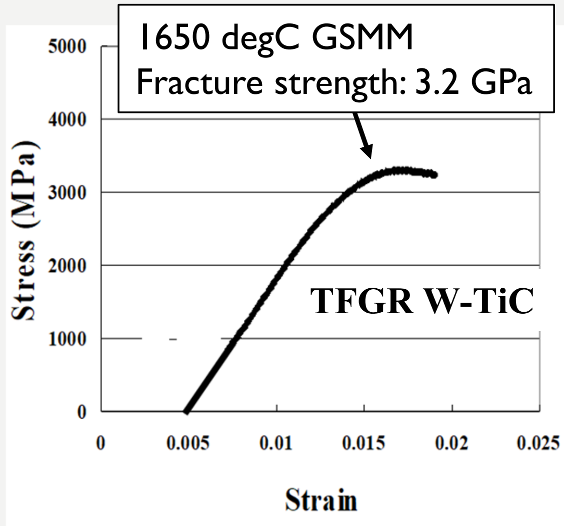
- ☐ Equiaxed, fine grains with TiC precipitates
- ☐ GB reinforced by TiC enrichment
- ☐ No recrystallization embrittlement
- ☐ High sink density: Resistance to irradiation is anticipated.
- ☐ DBTT (nil-ductility tem.) < RT



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



Fabrication process of TFGR



Ductility at RT

PURE W, ITS ALLOYS, AND TFGR W-TIC

Pure W

W with solution
(e.g. W-Re)
Solid-solution softening
Slight reinforce of GB

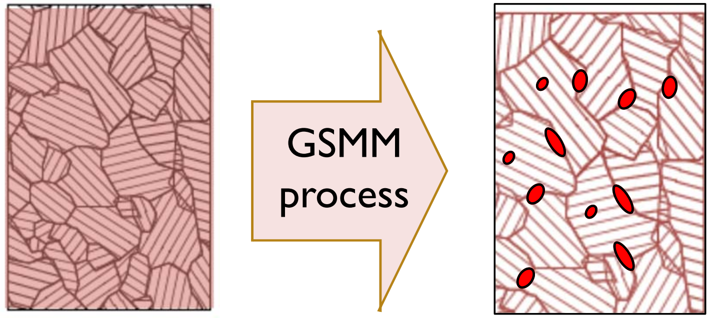
W with Dispersoids
(e.g. Y2O3 or K-doped W)
Increase of
Recrystallization temp.

Heavy plastic working and stress-relieved
to prevent crack propagation

Coarse and elongated (Radiation embrittlement)
Grain boundaries (GBs) are still weak.
Recrystallization embrittlement at high temperature

Pure W and Its alloy (Conventional)

Mechanical alloyed powder



Sintering as low
temp. as possible

Segregation &
Precipitation

Equiaxed, Fine grains (Radiation resistant)
The GBs are semi-coherent and reinforced.
No recrystallization embrittlement occur.

TFGR W-TiC

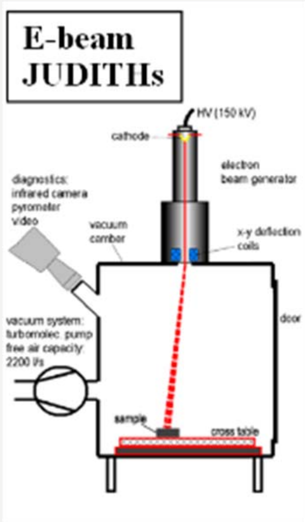
PURE W, ITS ALLOYS, AND TFGR W-TIC

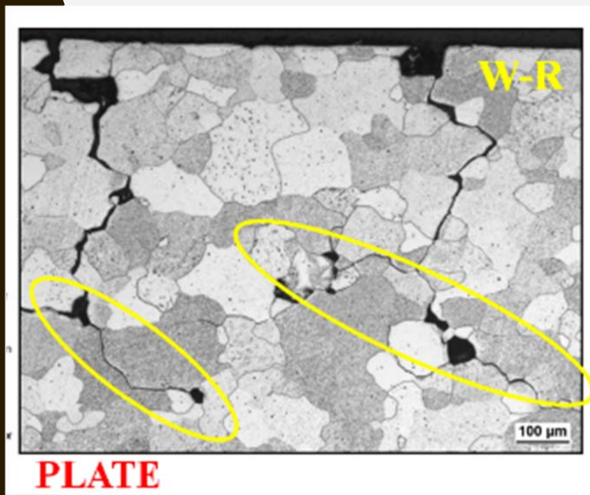
Characterization item	Pure W	W-Re	W-Y_2O_3 or K-doped	TFGR W-TiC
How Strengthened	Hot rolling etc. & Grain size	Hot rolling etc. & Solution	Hot rolling etc. & Dispersion	Segregation & Dispersion (Precipitation)
Recrystallization embrittlement				
Recrystallization embrittlement	Yes	Yes	Yes	No
Expected usable maximum temp.	1200-1300 ˚C	1500 ˚C	1500 ˚C	1800 ˚C (in UHV) or higher *
GB in the recrystallized state	Weak (not reinforced)	Slightly reinforced by Re	Very weak (not reinforced)	Reinforced by TiCx (Semi coherent)
DBTT in the recrystallized state	Higher than RT	Around RT	Higher than RT	Around RT
Irradiation embrittlement				
Grain appearance	Elongated, Coarse	Elongated, Coarse	Elongated, Coarse	Equiaxed, fine grains in recrystallized state
Radiation damage	Vulnerable	Vulnerable	Vulnerable	Resistant (anticipated)

* Creep behavior must be considered, as discussed later

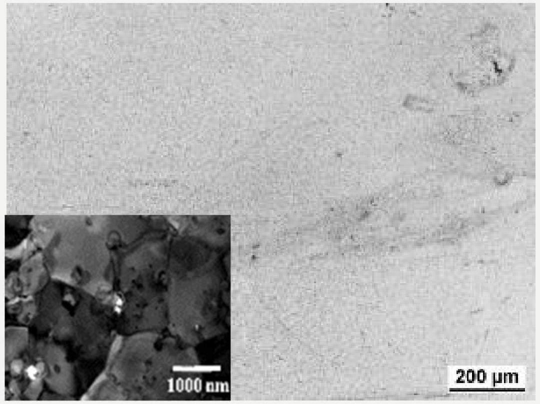
TFGR W-TiC

Electron beam heating,
$A = 16\ \mathrm{mm}^2$, $t = 1$ ms,
$P = 1.1\ \mathrm{GW/m}^2$
$\Delta T \approx 2000^\circ\mathrm{C}$
$T_{\mathrm{base}} = 100^\circ\mathrm{C}$, $n = 100$





TFGR W-1.1TiC/H-160ppmO

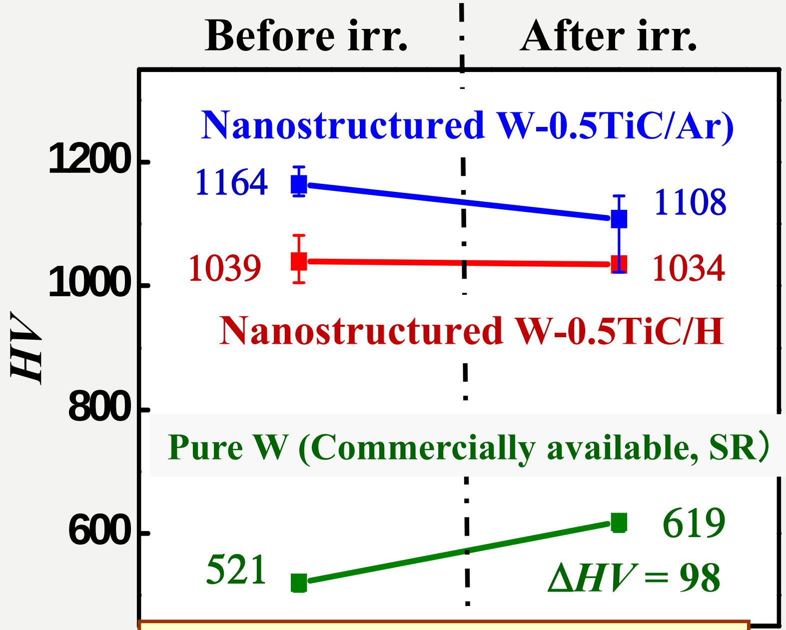


G. Pintsuk et al. Phys. Scr. T145 (2011) 014060.

Promising result of electron beam heating test.

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

Vickers microhardness



- No radiation hardening

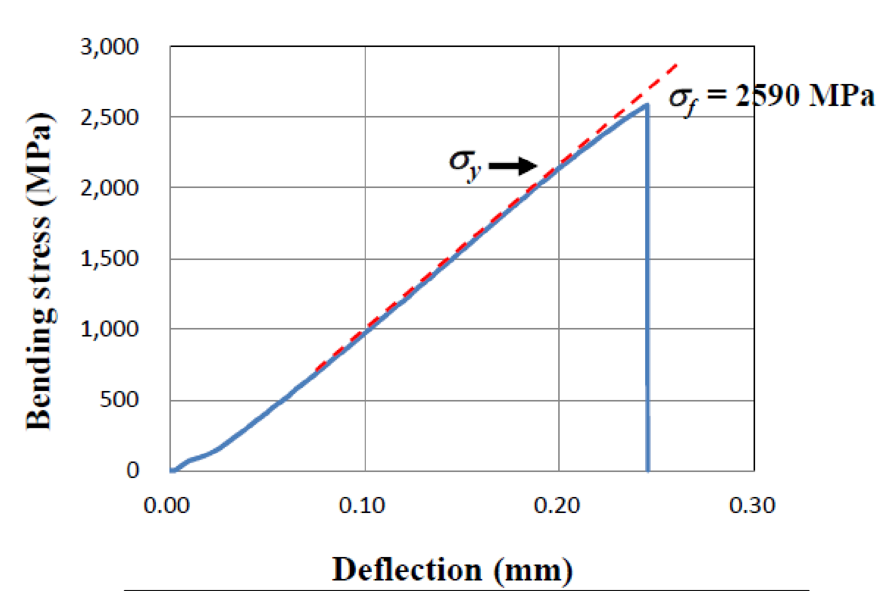
H.Kurishita et al. JNM 377 (2008) 34.

Limited, but promising result of HE-neutron irradiation.

Further studies are planned, HIT or next BLIP?

RECALL OF REPORT AT RADIATE CM, CERN, 2018

- The development of TFGR was transferred to KEK-MTC collaboration.
- Restoration of TFGR was realized.

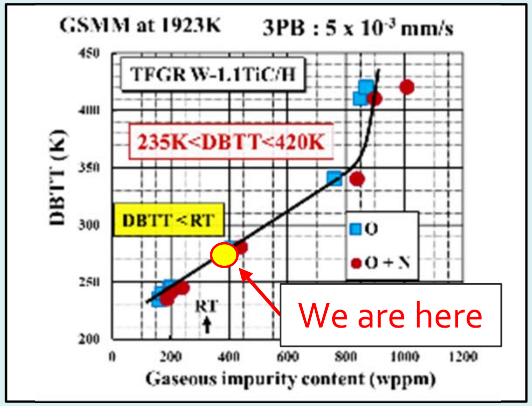


- <u>High fracture strength, 2.6 GPa</u>
- <u>Ductility at RT</u>

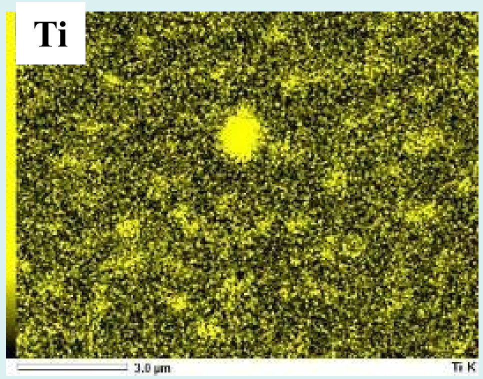
Construction of new dedicated apparatuses

High purity glove box

Mechanical Alloying in vac.



H. Kurishita, Phys. Scr. T159 (2014) 014032.



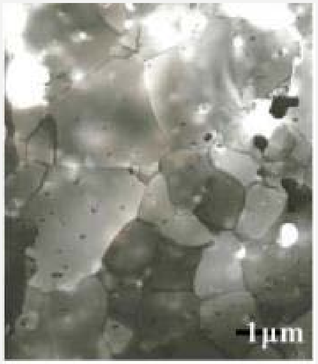
EDS~ Remaining Ti

- Oxygen impurity should be decreased.
- MA process for 72 hours was not sufficient.

Future Prospect of TFGR W-TiC

Pursuit of higher performance

W-TiC ⇒ W-TaC or ??



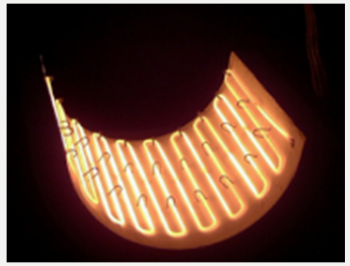
W-2.2TaC W-3.3TaC

Material	TaC	TaO_2	W_2C	G.S.	DBTT
W-2.2TaC	130	160	560	2.2 μm	330K
W-3.3TaC	80	130	–	1.2 μm	420K
	(nm)				

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

Developments for Mass production & Industrial use by Academic Industrial Collaboration

- Metal Technology Co., LTD
- Sunric Co., LTD.
- And several vendors as advisors



Objective

- Heater material in vapor deposition for manufacturing of semi-conductors
- Anode in lamp for lithography

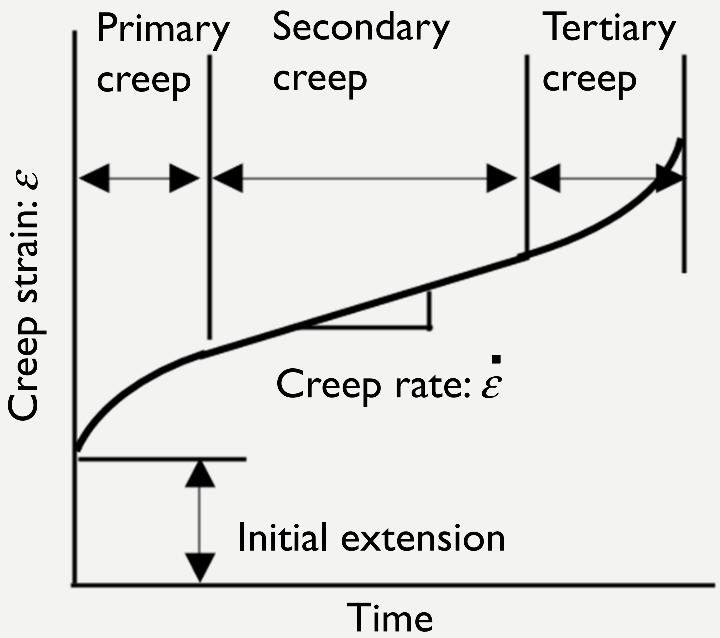
Supported by the Small and Medium Enterprise Agency, METI

Investigation of radiation damage

- Irradiation damage (dpa), by HIT-Tokyo-U
- Next BLIP or other neutron-irradiation?
- Investigation of He, Re, Os embrittlement by ??

DISCUSSION TO IMPROVE CREEP BEHAVIOR IN TFGR W-TIC

FUNDAMENTAL OF CREEP BEHAVIOR



Secondary creep

$\dot{\varepsilon}_s \propto (\sigma/E)^n \exp(-Q/kT)$

$\dot{\varepsilon}_s = \dot{\varepsilon}_0 (\mu\Omega/kT)(\sigma/\mu)^n (b/d)^p (D/b^2)$

n, p, Q are relevant to structure, stress region, and temperature region.

p, *d* are considered at 2nd formula.

Creep: Permanent deformation under Constant stress and Constant temperature

Primary creep (Skip in this presentation)

- Sliding length of Dislocation
  - Pure tungsten: large
  - With solution or dispersion: small

n : stress exponent

d : grain size, *p* : exponent of inverse grain size

Q: activation energy for creep

μ : rigidity

σ: stress

Ω : atomic volume

E: Young's modulus

b : Burgers vector

T: Absolute temperature

D: diffusion coefficient

k: Boltzmann constant

RELATIONSHIP BETWEEN STRUCTURES AND CREEP MECHANISM

Structure in W and its alloys	Steady state (Secondary) creep rate					
	Creep mechanism	Controlling mechanism	Stress exponent, n	Diffusion	Q	p
Pure W	Dislocation slip	Recovery of internal stress (disl.)	~ 5	Lattice, D_L	Q_L	0
			~ 7	Pipe, D_p	Q_p	0
Solution hardened (W-Re)	Dislocation slip	Solute atmosphere dragging	3 ~ 4	Mutual, D_m	Q_m	0
Dispersion hardened ($W\text{-}Y_2O_3$)	Dislocation slip	Recovery of inter. stress (particle)	> 6	Lattice, D_L	Q_L	0
TFGR W-1.1TiC (Toughened, fine grained, recrystallized)	Dislocation slip	Recovery of inter. stress (precipitate)	> 6	Lattice, D_L	Q_m	0
	Grain boundary sliding	Lattice diffusion	2	Lattice, D_L	Q_L	2
		GB diffusion	2 (1)	GB, D_{gb}	Q_{gb}	3

Secondary creep

$$\dot{\varepsilon}_s \propto (\sigma/E)^n \exp(-Q/kT)$$

$$\dot{\varepsilon}_s = \dot{\varepsilon}_0 (\mu\Omega/kT)(\sigma/\mu)^n (b/d)^p (D/b^2)$$

- n : stress exponent
- Q : activation energy for creep
- d : grain size
- p : exponent of inverse grain size

When the grain size is fine, the effect of grain boundary sliding must be taken into account.

AIMING AT SIGNIFICANT ENHANCEMENT OF CREEP RESISTANCE IN TFGR W-TIC

Features of current TFGR W-TiC

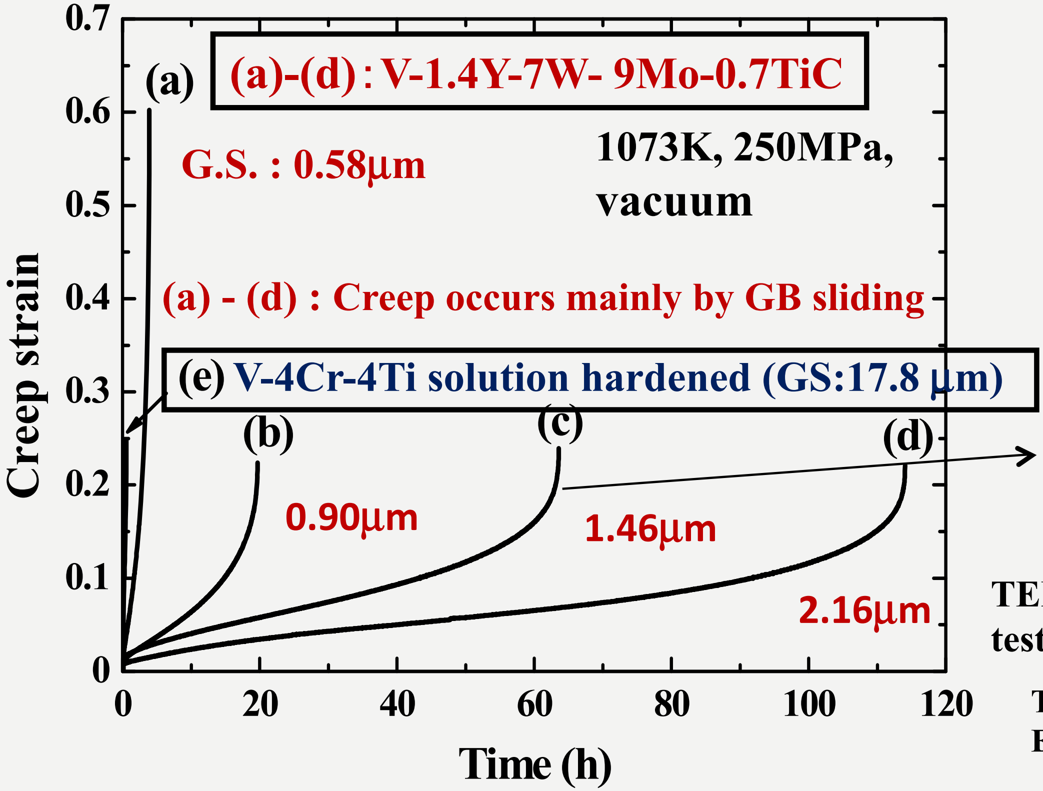
- Significantly reinforced GBs by segregation (enrichment) and precipitation of TiC_x at random GBs in the recrystallized state
- High density of sink sites for irradiation induced point defects
- Precipitation hardening
- Equiaxed fine grains (GS : 1.5 ~ 2 μm)

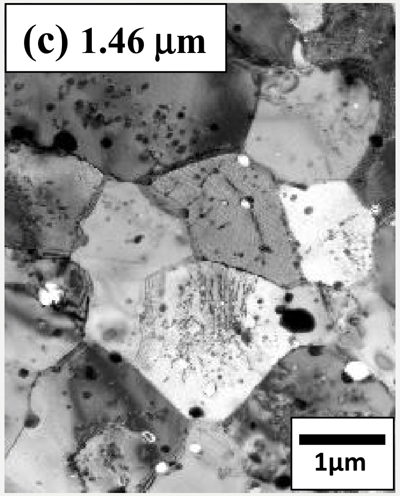
Need ＋ solution hardening

＋ adequate increase in grain size

Example of fine grained, solution and precipitation hardened V alloy

Creep resistance: Solution hardened: V-4Cr-4Ti << Solution & precipitation hardened: V-1.4Y-7W-9Mo-0.7TiC





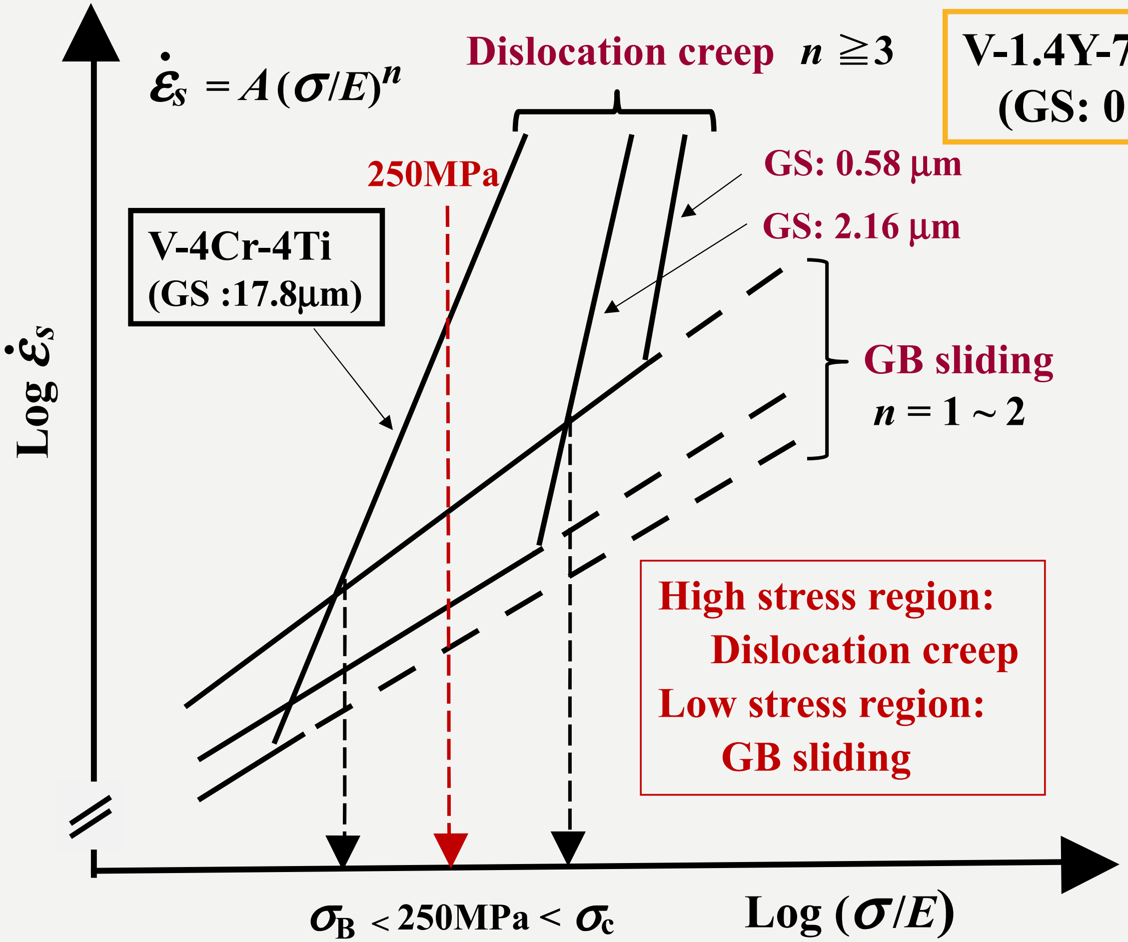
TEM image taken after creep testing at 1073K and 250MPa

T. Sakamoto et al. Mater. Sci. Eng. A 528 (2011) 7843

Effects of grain size on the entire creep curve at 1073K and 250MPa for V-1.4Y-7W-9Mo-0.7TiC together with (e) V-4Cr-4Ti with grain size of 17.8 μm (Nifs heat 2).

<u>Slight increase in grain size significantly enhances creep resistance of fine grained, solution and precipitation hardened V alloy</u>

Relationship between steady state creep rate, $\dot{\varepsilon}_s$, creep mechanism and grain size in two V alloys



T. Sakamoto et al. Mater. Sci. Eng. A 528 (2011) 7843

Schematic illustration explaining the observed creep resistance for fine-grained, solution and dispersion hardened V–1.4Y–7W–9Mo–0.7TiC with different grain sizes and coarse-grained, solution hardened V–4Cr–4Ti alloy

<u>TFGR W-TiC is:</u>

- Fine-grained
- Dispersion (Precipitation) hardened
- Slightly-solution hardened (Ti, C, Mo)

<u>For increase of creep resistance,</u>

- ◆ Addition of an appropriate element
- ◆ Control of grain-size

Subsequent effects, e.g. thermal conductivity, radiation resistance should be confirmed.

<u>Confidential experiment will be reported in a few years.</u>

SUMMARY

- The use of W as target material is limited by “Recrystallization embrittlement” and “Irradiation embrittlement”.
- Now the activities of TFGR W-TiC are transferred to KEK and Metal Technology Co. LTD collaboration.
- Developments are in progress under collaboration with academic and industries.
- The creep resistance of TFGR W-TiC can be improved by addition of another element and control of grain size.

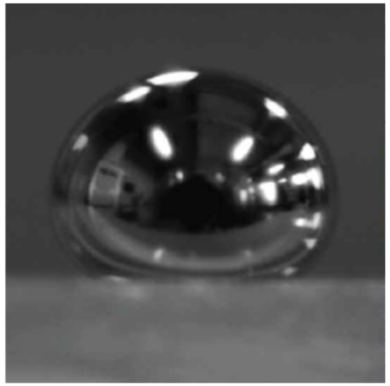
Thanks for your attention and,,,

8th High Power Targetry Workshop

May 25-29, 2020
Venue : RIKEN Wako campus

Themes for the workshop include :

1. R&D to support concepts
2. Radiation damage in target material and related simulations
3. Post-irradiation examination
4. Target design, analysis and validation of concepts
5. Target facility challenges
6. Construction, fabrication, inspection, quality assurance
7. Operation of targets and beam dumps
8. Multipurpose use of targets and beam dumps



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http://indico2.riken.jp/event/3102/   hptw2020-loc@ml.j-parc.jp