

25th IAEA Fusion Energy Conference  
13–18 October 2014  
Saint Petersburg, Russian Federation  
MPT/1-4

# Neutron Irradiation Effects on Grain-refined W and W-alloys

A. Hasegawa<sup>a</sup>, M. Fukuda<sup>a</sup>, T. Tanno<sup>a,b</sup>, S. Nogami<sup>a</sup>,  
K. Yabuuchi<sup>a</sup>, T. Tanaka<sup>c</sup> and T. Muroga<sup>c</sup>

<sup>a</sup>*Graduate School of Engineering, Tohoku University, Sendai,  
980-8579 Japan*

<sup>b</sup>*Japan Atomic Energy Agency, Oarai, Ibaraki, 311-1393  
Japan*

<sup>c</sup>*National Institute for Fusion Science, Toki, 509-5292 Japan*

# Outline

*Dept. Quantum Science & Energy Engineering, Tohoku University*

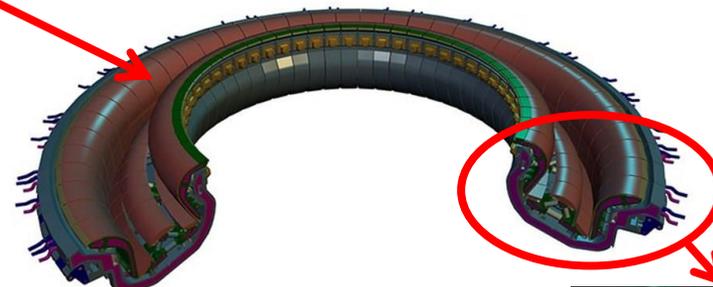
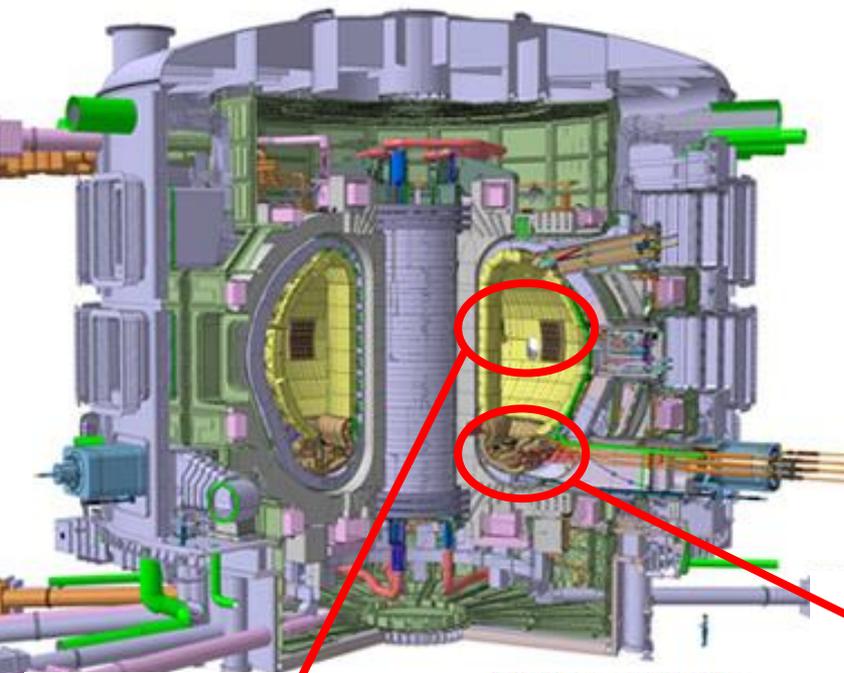
- 1. Introduction**
- 2. Summary of neutron irradiation effects of Tungsten based on previous data.**
- 3. Prediction of microstructural development of tungsten under fusion reactor irradiation conditions.**
- 4. Tungsten alloys development by grain refining and alloying for fusion application.**
- 5. Current status of material evaluation of unirradiated state.**

# Tungsten in Fusion Reactor

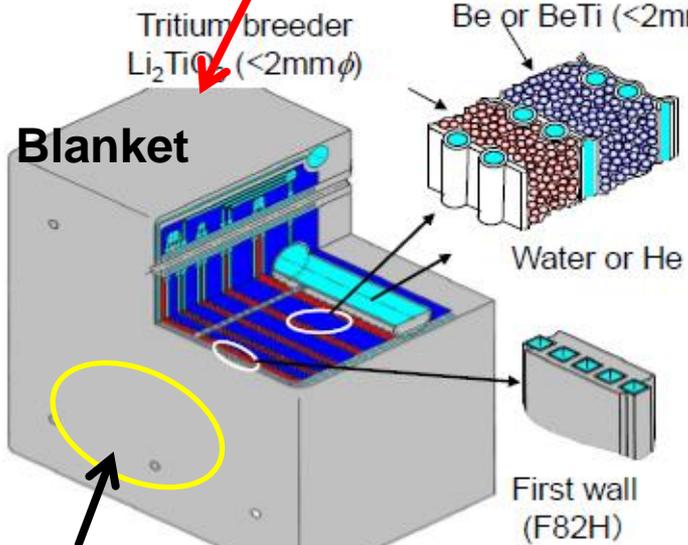
Dept. Quantum Science & Energy Engineering, Tohoku University

• Tungsten is candidate material for first wall and divertor components in fusion reactor.  
Resistance to High-Hear-Load and High-Density-Particle Irradiation

- High melting temperature
- High resistance to sputtering
- Low hydrogen retention

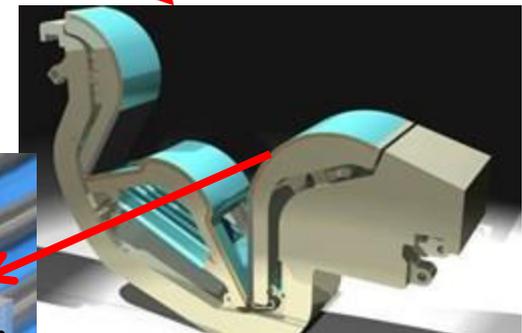


Divertor :  
Impurity exhaust  
apparatus



Coating on plasma facing surface  
(Thickness:1mm for TBR)

Cooling Block

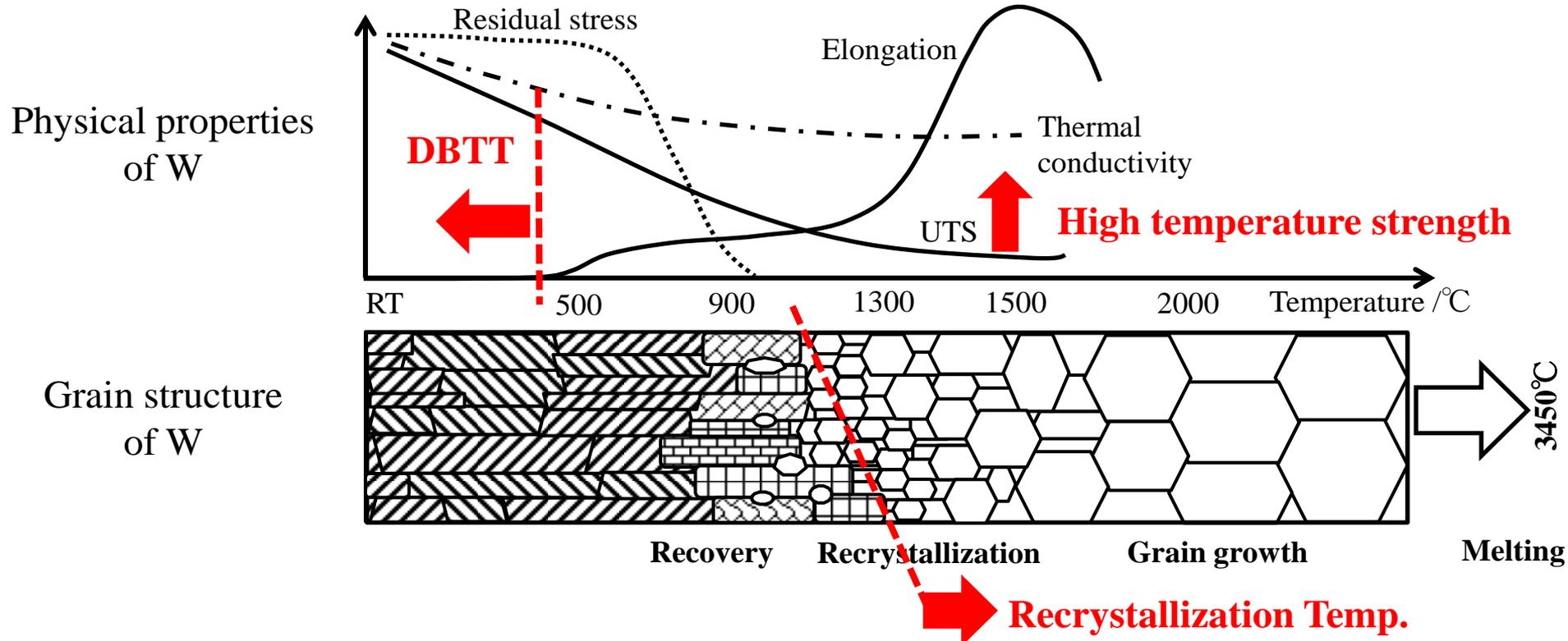


Cooling component

# Temperature Dependence on Microstructure and Physical Properties of Tungsten

4

Dept. Quantum Science & Energy Engineering, Tohoku University



## Strategy of material development of Tungsten

- **Decrease DBTT (Ductile-Brittle-Transition- Temperature)**  
Improve low temperature embrittlement.
- **Increase recrystallization temperature.**
- **Increase high temperature strength.**

# Improvement of Mechanical Properties by Microstructure Control

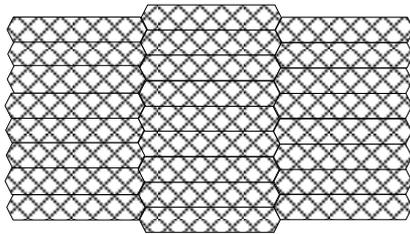
## Alloying

- decrease DBTT
- increase recryst. temp.

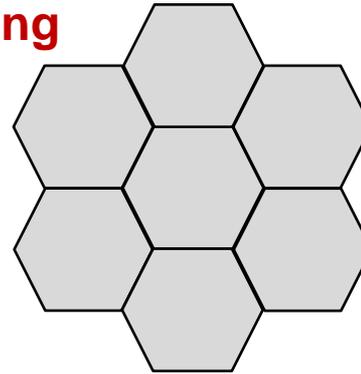
## Cold work

- decrease DBTT
- decrease recryst. temp.

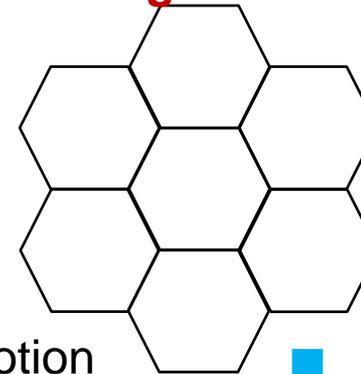
## Cold work



## Alloying



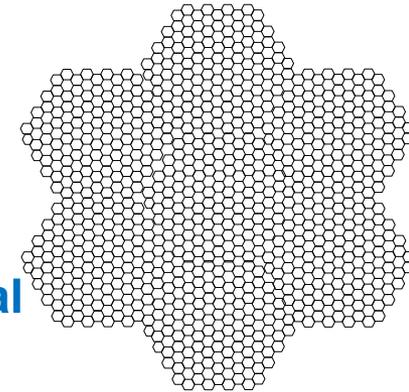
## Arc melting



- Decrease DBTT
- Increase recryst. temp.
- Increase high temp. strength

## Fine grain

- Powder Metallurgy
- Mechanical Alloying



## Fine grain by working and stabilized by dispersed obstacles

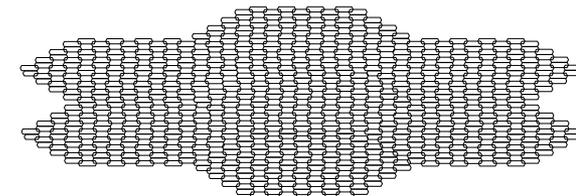
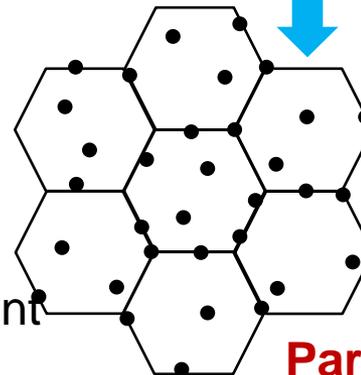
## Particle dispersion

## Particle (or hole) dispersion

- Obstacles of GB sliding and motion
- Improve high temp. strength & recrystallization behavior
- Stability of the obstacles

## Fine Grain

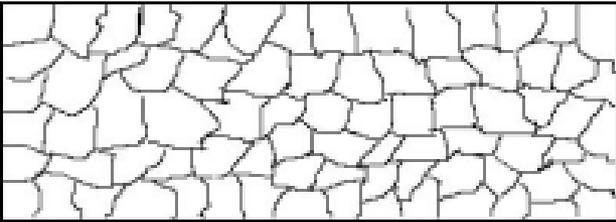
- Improve low temp. embrittlement
- Introduce defect sink



# Examples of Microstructure Control of W

PM W

Full Recrystallize (R)

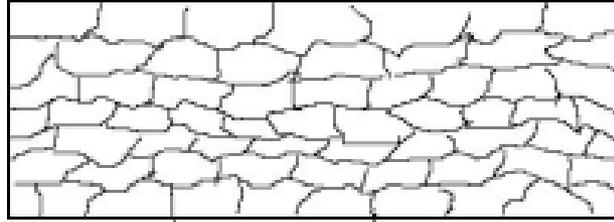


Coarse Grain

Grain size 100x100 $\mu$ m

PM W

Recrystallize (R)

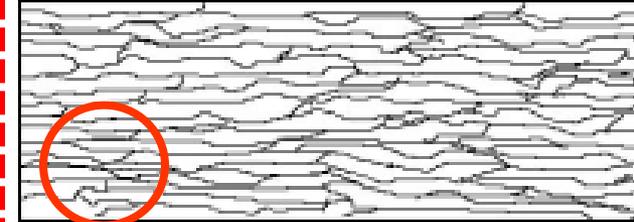


Coarse Grain

Grain size 100x50 $\mu$ m

PM W

Stress Relief (SR)



Layered Structure

Layer thickness < 10 $\mu$ m

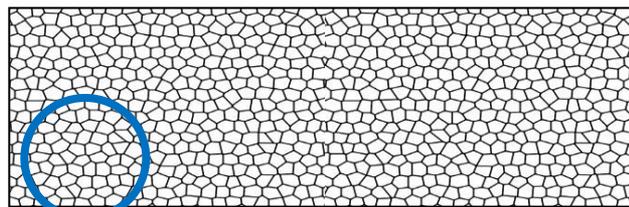
Arc Melt W(as-cast)



Coarse Grain

Grain size ~1mm

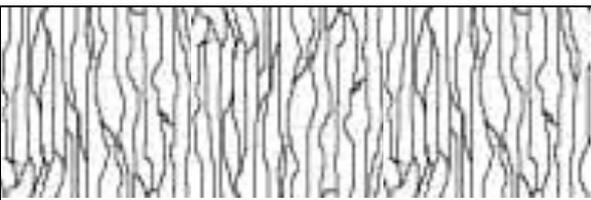
MA W-TiC UFG



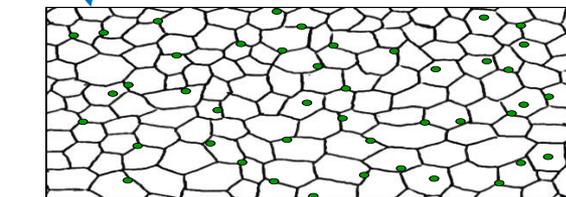
Ultra fine grain

Grain size 70-100nm

CVD W

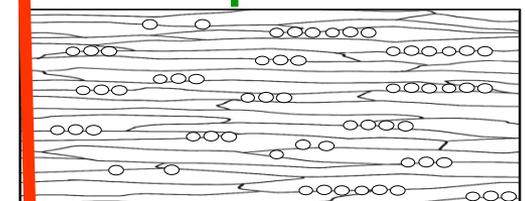


Columnar 100nm x 1 $\mu$ m



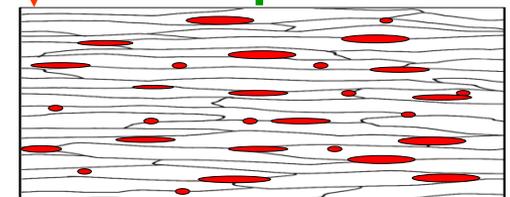
Ultra Fine Grain (<100nm) with Carbide precipitates on GB

K-doped W



Dope hole dispersion

La-doped W



La<sub>2</sub>O<sub>3</sub> particle dispersion

# Outline

Dept. Quantum Science & Energy Engineering, Tohoku University

1. Introduction

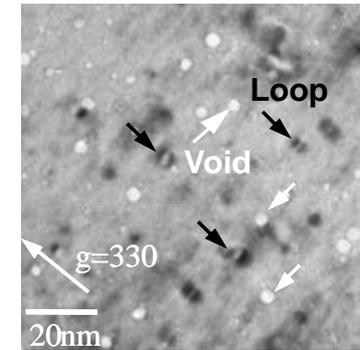
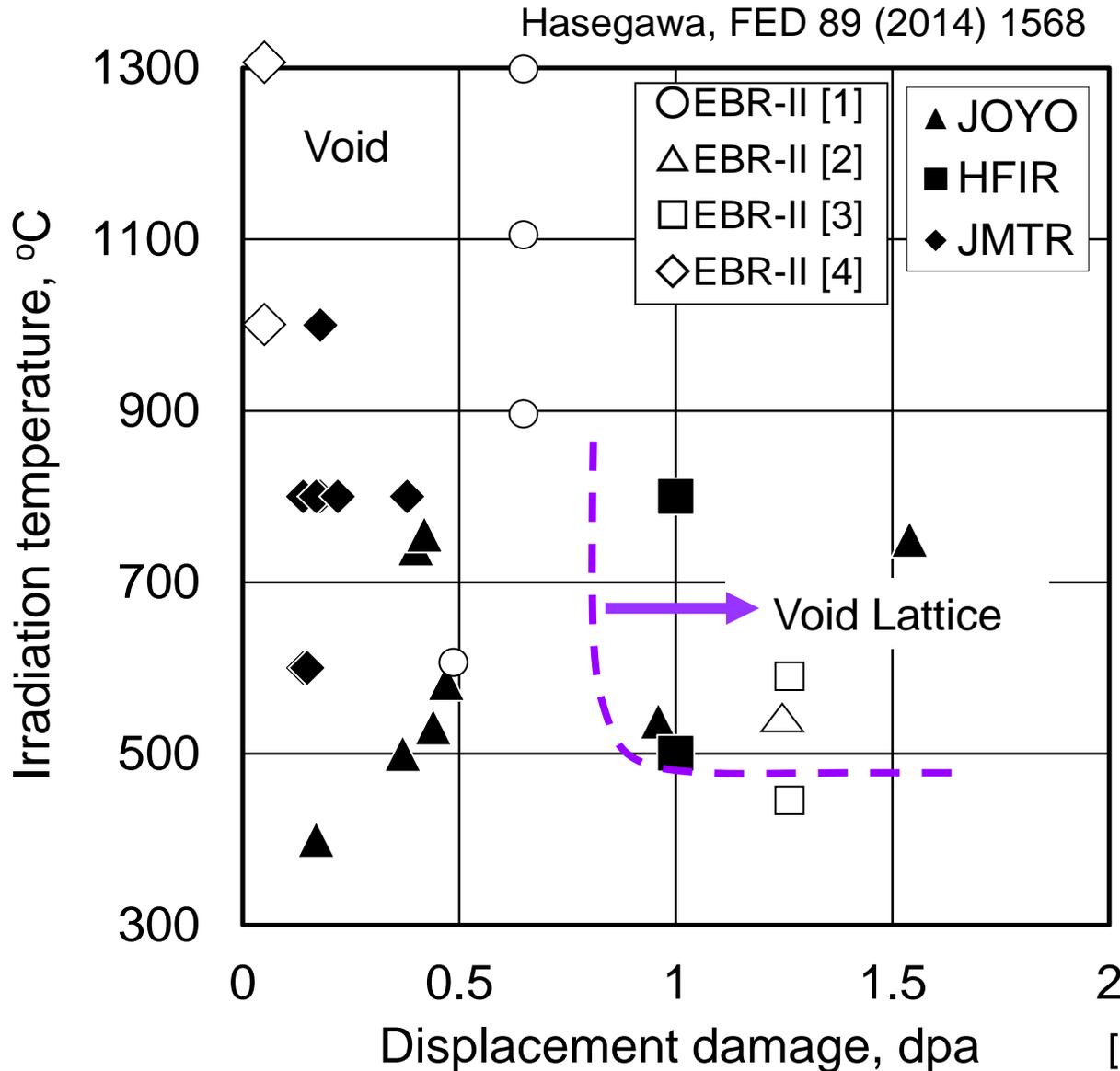
**2. Summary of neutron irradiation effects of Tungsten based on previous data.**

3. Prediction of microstructural development of tungsten under fusion reactor irradiation conditions.

4. Tungsten alloys development by grain refining and alloying for fusion application.

5. Current status of material evaluation of unirradiated state.

# Summary of Damage Structure Map of W



538 °C  
0.96dpa  
In JOYO

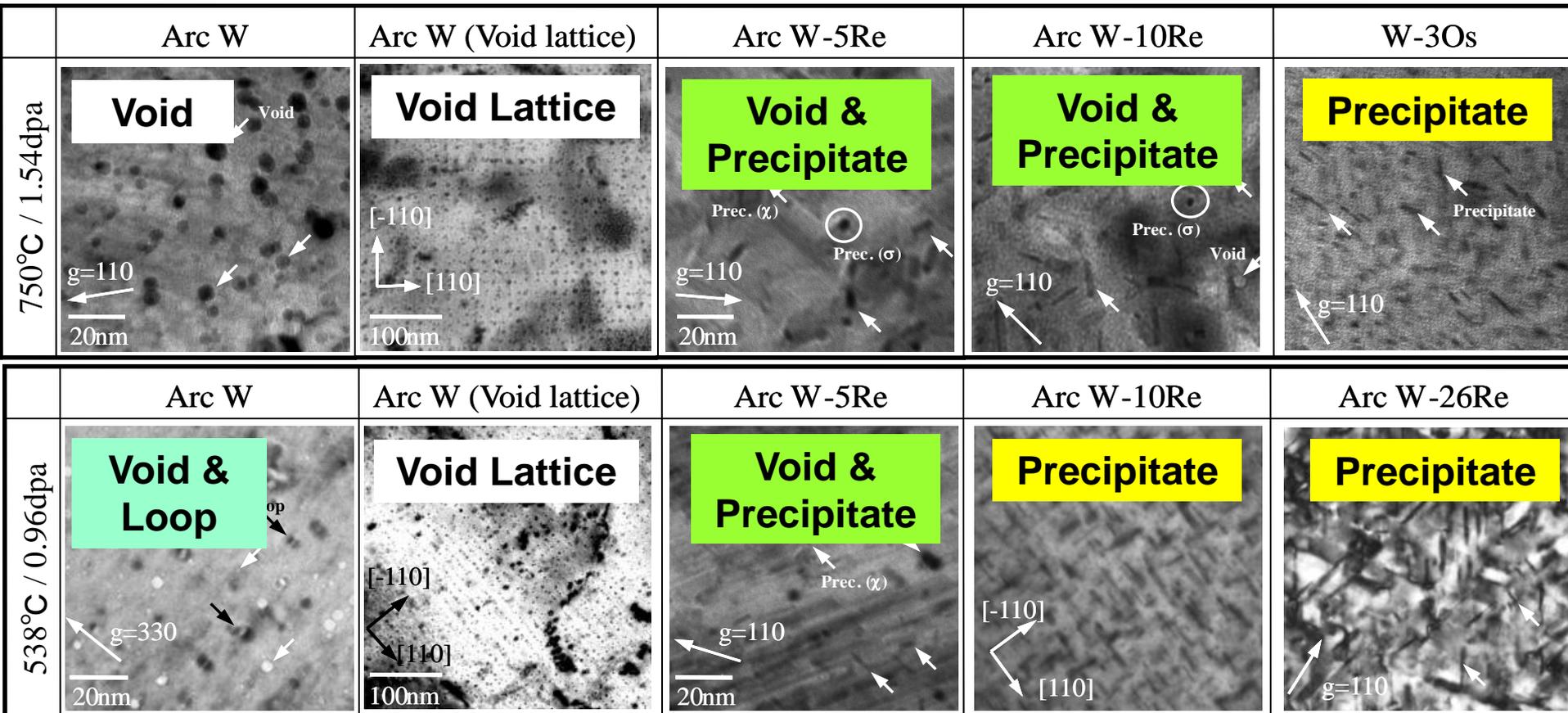
#Void was observed above 0.1dpa irradiation, above 400 °C up to 1300 °C .

#Loop was also observed above 400 °C . Upper limit of loop formation could not be confirmed.

#Void lattice formed after higher level irradiation (>1dpa).

# Damage Structure of W and W-Re Alloys

Irradiated in JOYO (first neutron spectrum reactor)



# Void lattice were observed in pure W. ( pure W  $\rightarrow$  W-1.5Re-0.05Os after 1.5dpa)

# Void formation was drastically suppressed in W-Re and acicular precipitates were observed above 5%Re.

# Outline

Dept. Quantum Science & Energy Engineering, Tohoku University

1. Introduction

2. Summary of neutron irradiation effects of Tungsten based on previous data.

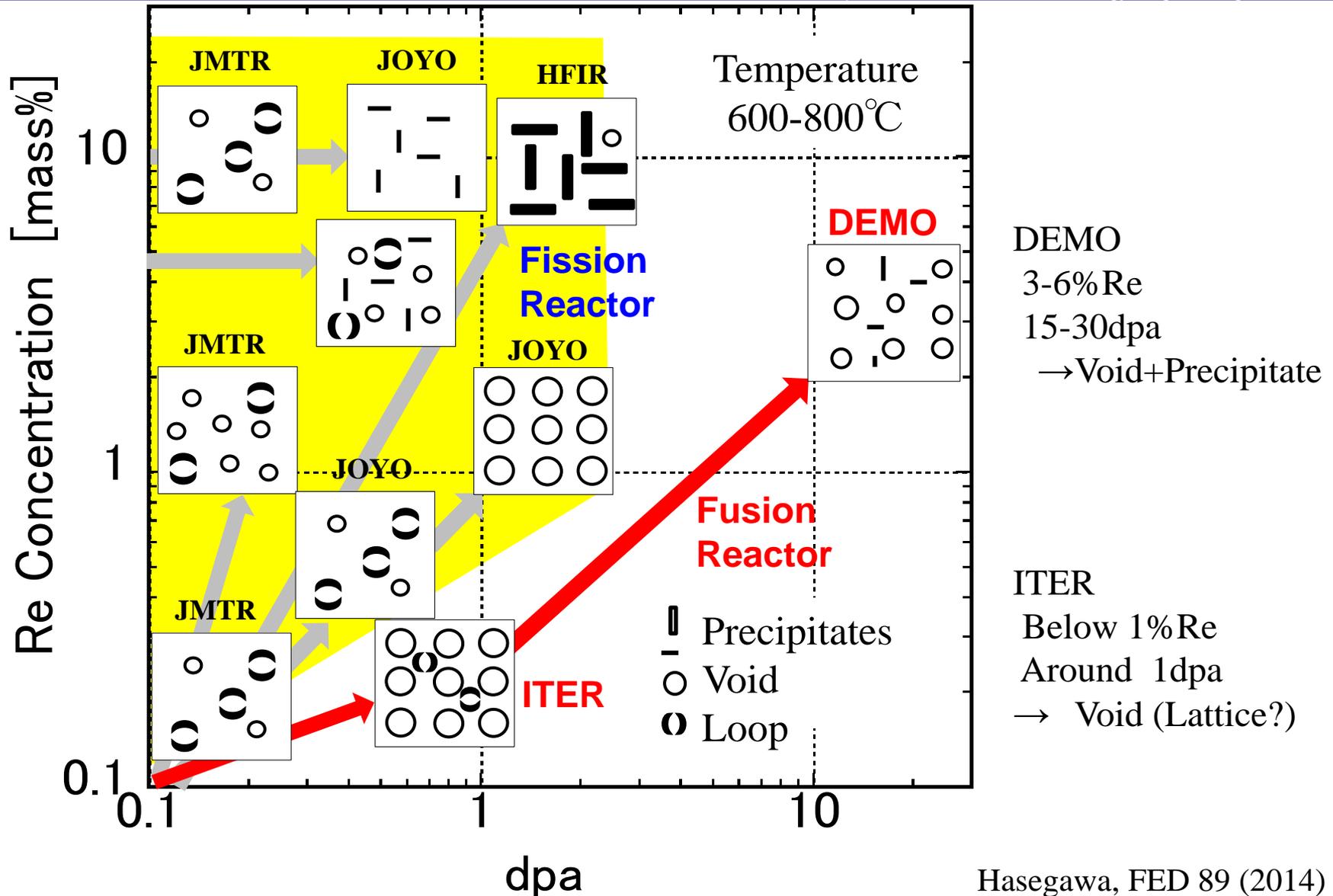
**3. Prediction of microstructural development of tungsten under fusion reactor irradiation conditions.**

4. Tungsten alloys development by grain refining and alloying for fusion application.

5. Current status of material evaluation of unirradiated state.

# Summary and Prediction of Microstructural Development of W<sup>11</sup>

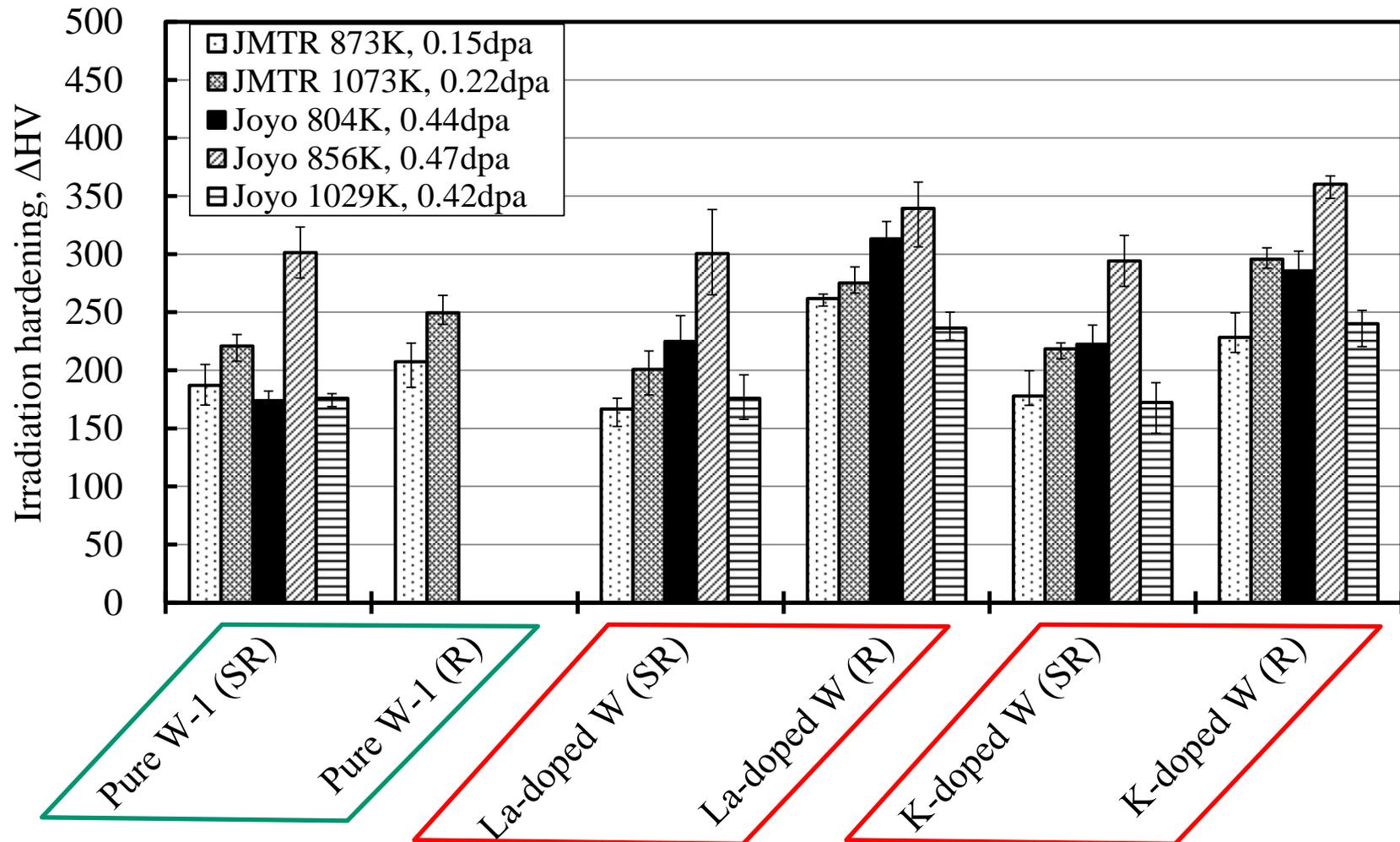
Dept. Quantum Science & Energy Engineering, Tohoku University



# Background of Grain Refined W Development

Dept. Quantum Science & Energy Engineering, Tohoku University

## Irradiation hardening of advanced W alloys.



# Defect Clusters in Matrix after JOYO Irradiation

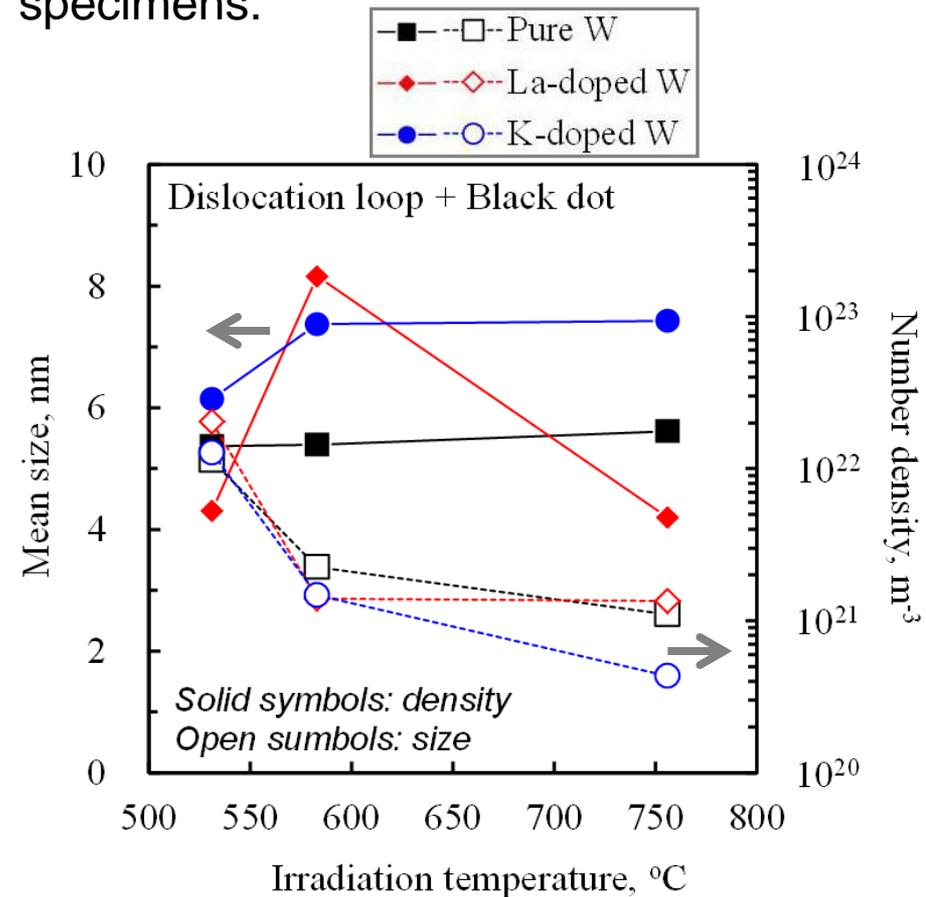
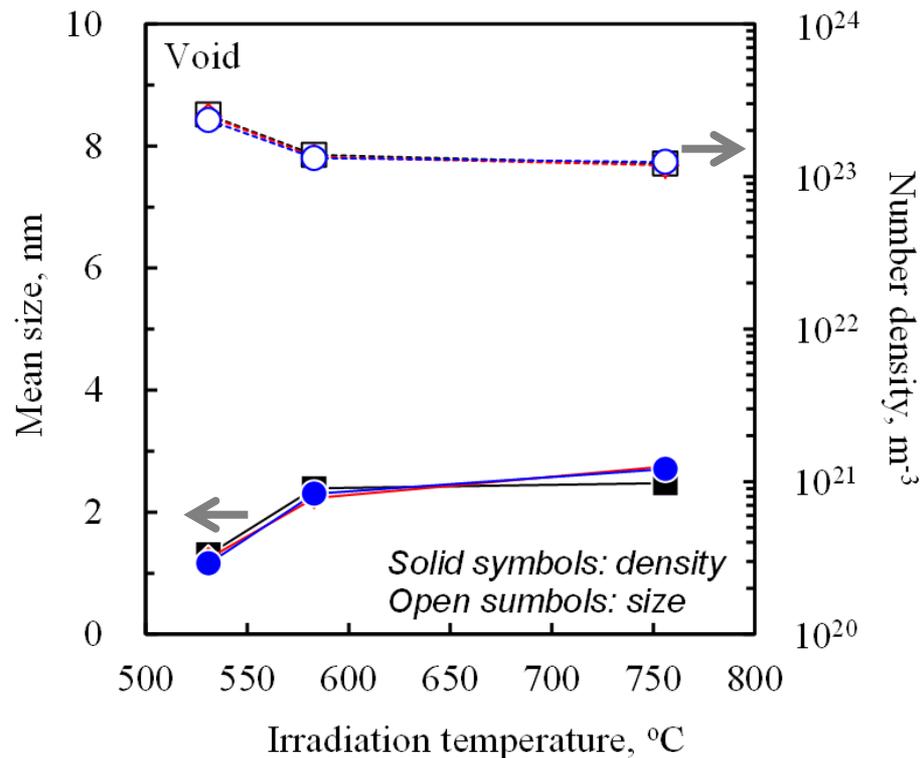
Dept. Quantum Science & Energy Engineering, Tohoku University

Fukuda, JNM 442 (2013) S273-S276

Calculate void swelling (%)

Irradiation conditions	Pure W	La-doped W	K-doped W
531°C, 0.44dpa	0.017	0.014	0.011
583°C, 0.47dpa	0.056	0.044	0.047
756°C, 0.42dpa	0.054	0.072	0.073

- Irradiation response of the advanced W alloys were almost the same as pure W.
- Matrix condition for defect clustering were considered to be similar between these specimens.



# Outline

Dept. Quantum Science & Energy Engineering, Tohoku University

1. Introduction
2. Summary of neutron irradiation effects of Tungsten based on previous data.
3. Prediction of microstructural development of tungsten under fusion reactor irradiation conditions.
- 4. Tungsten alloys development by grain refining and alloying for fusion application.**
5. Current status of material evaluation of unirradiated state.

# Fabricated W and W-alloys in LHD Project 15

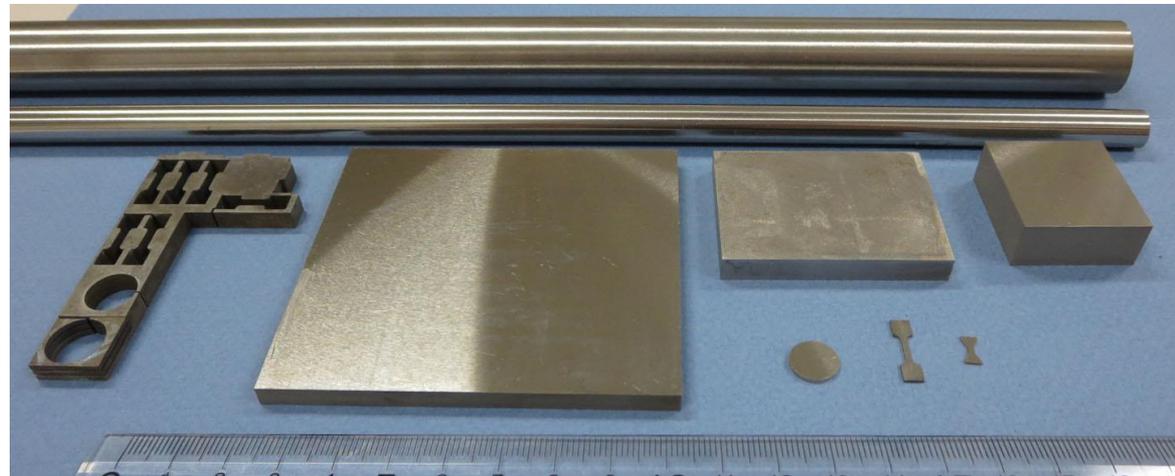
Dept. Quantum Science & Energy Engineering, Tohoku University

By A.L.M.T. Corp.

## Name of fabricated alloys

- Pure W
- W-1%Re
- K-doped W
- W-3%Re
- K-doped W-3%Re
- La-doped W-3%Re
- Pure-W
- K-doped W
- K-doped W-3%Re

- ① PM and hot rolled plates 5mmt or 7mmt      ② PM and swaged rod 20mmφ, 10mmφ



• Pure W 13mmt

## Example of impurities

	C [ppm]	O [ppm]	N [ppm]	Re [%]	K [ppm]	Al [ppm]	Si [ppm]
Pure W	10	< 10	< 10	—	< 5	< 2	< 5
W-1%Re	10	< 10	< 10	0.98	< 5	< 2	< 5
K-doped W	10	< 10	< 10	—	30	15	17

## Relative density

- Pure W : 99.0%
- W-1%Re: 99.1%
- K-doped W: 99.1%

$$\rho_0: 19.1 \text{ g/cm}^3$$

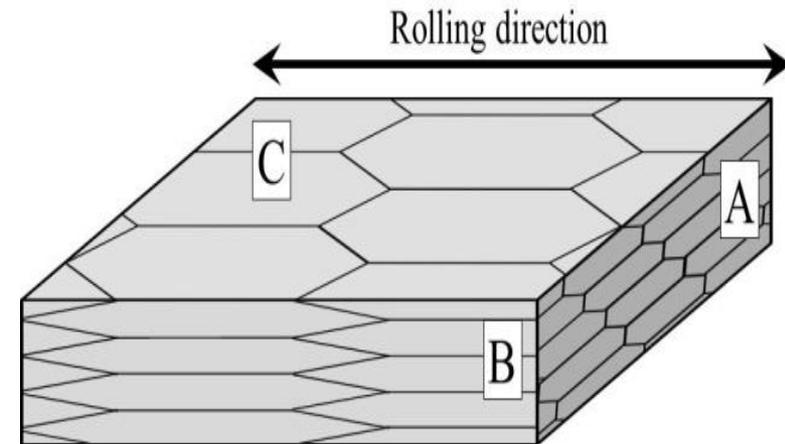
# Grain Structure (As received)

Dept. Quantum Science & Energy Engineering, Tohoku University

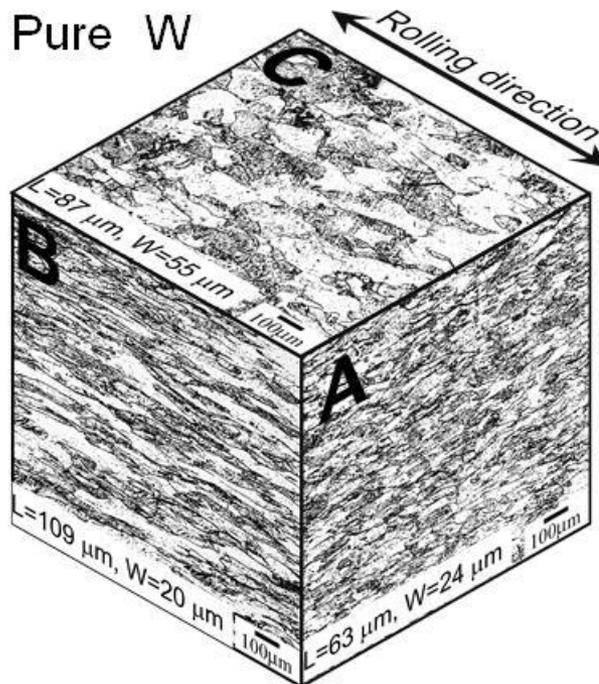
As received :

Heat treated at 900°C for 20min

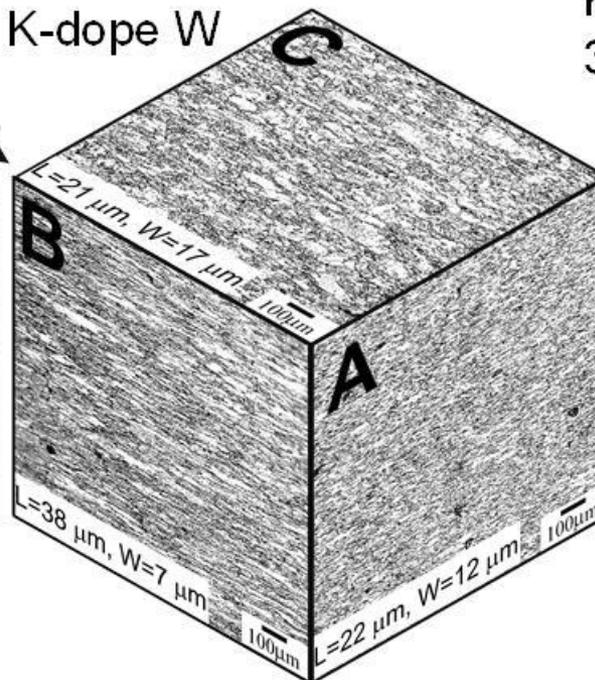
- Hot rolled structure  
Layered structure



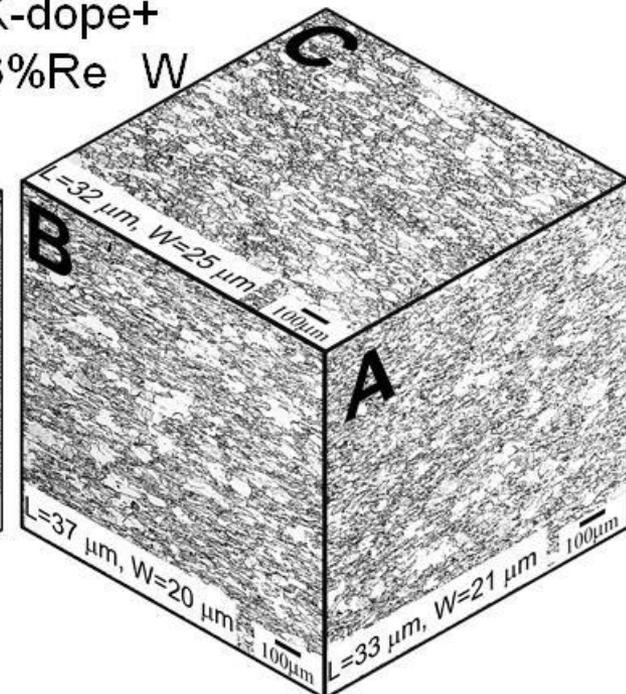
Pure W



K-dope W

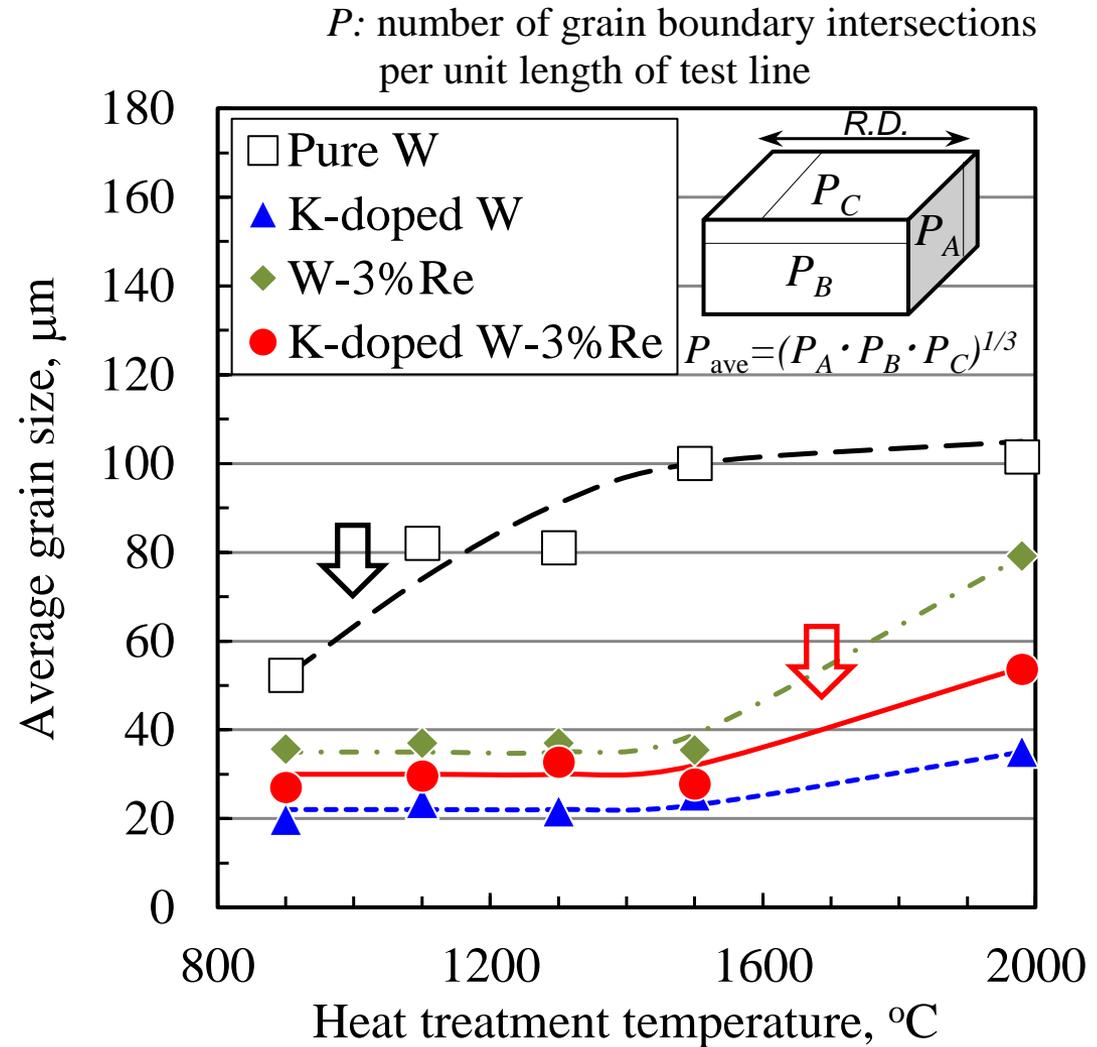
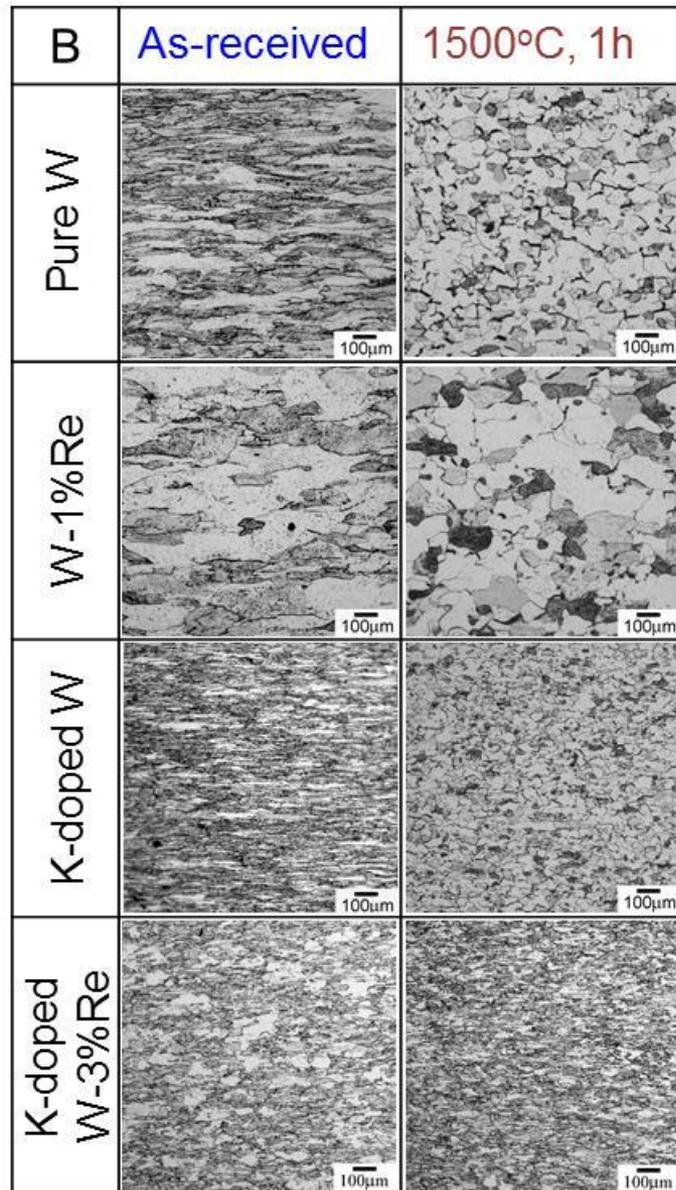


K-dope+  
3%Re W



# Recrystallized Behavior of Rolled Plate

Dept. Quantum Science & Energy Engineering, Tohoku University



# Temperature Dependence of Strength

- Strength increase by K-dope (at RT: 64% up, 1500°C: 36% up)
- Strength increase above 900°C by Re addition to K-dope W.

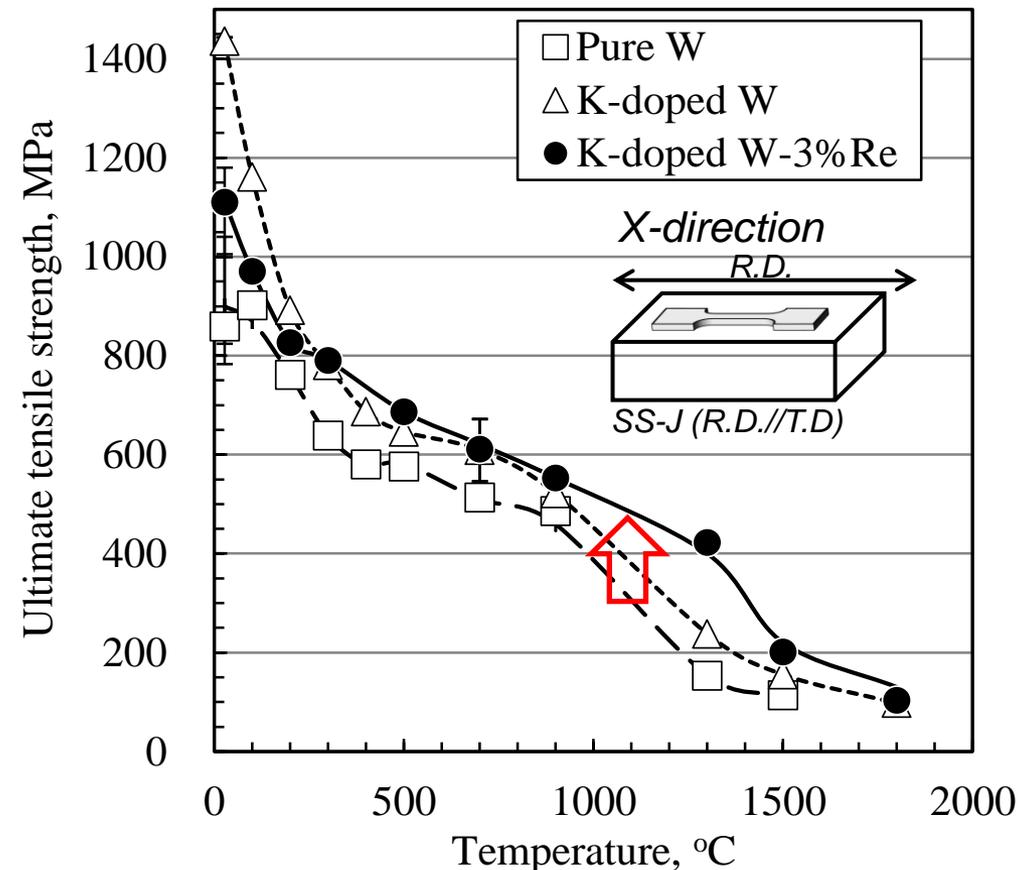
## K-doping

- Fine grain size
- Obstacles for dislocation and GB sliding, and recrystallization

## Re addition

Solute Re

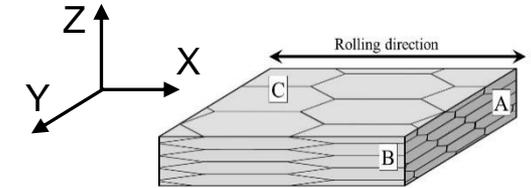
- Solution softening (<900°C)
- Solution hardening (>900°C)



# Anisotropic Tensile Strength

Dept. Quantum Science & Energy Engineering, Tohoku University

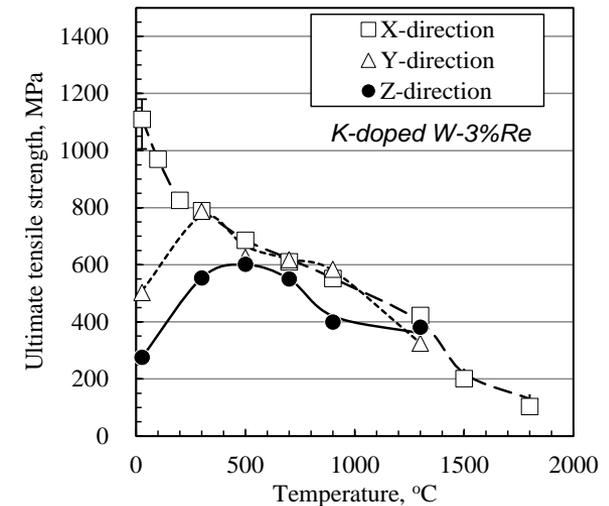
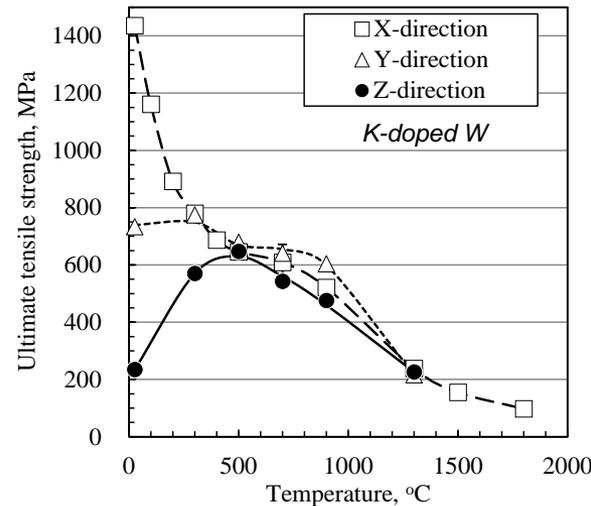
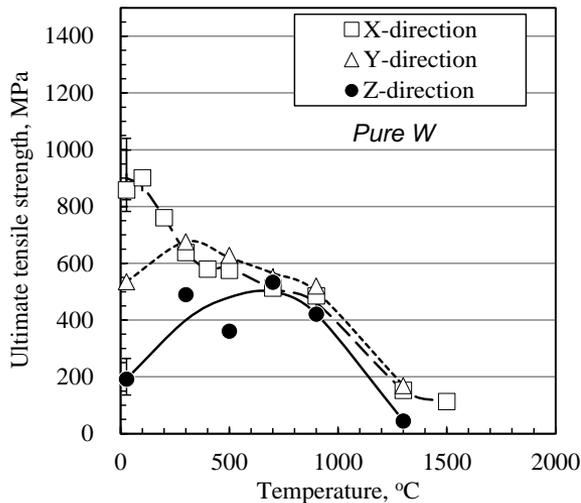
- Anisotropy was observed in the temperature range of R.T. - 500 °C.



	500	1000	1500
X	200°C → Ductile →		
Y	300°C →		
Z	1300°C →		

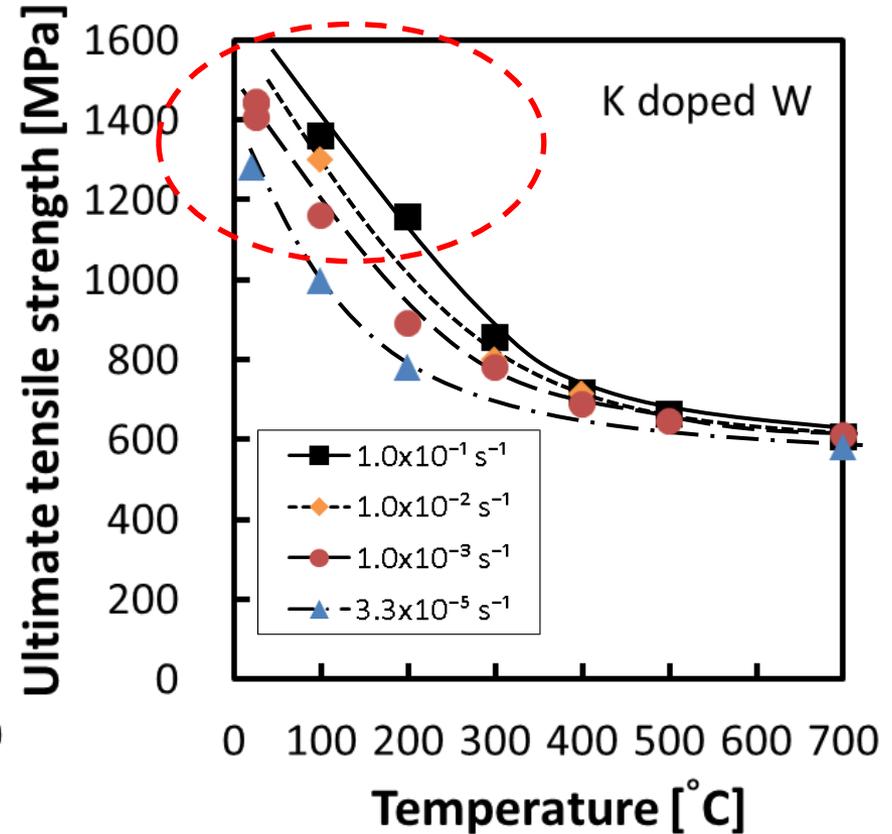
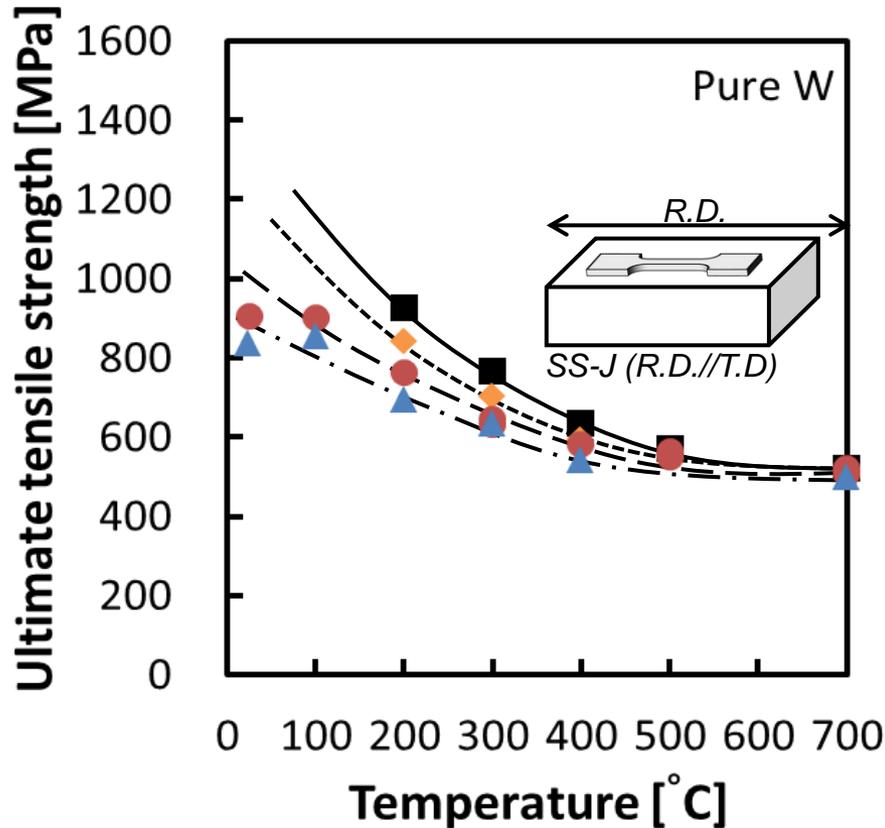
	500	1000	1500
X	R.T. →		
Y	300°C →		
Z	500°C → 1300°C →		

	500	1000	1500
X	100°C →		
Y	300°C →		
Z	500°C →		



# Strain Rate and Temperature Dependence of Strength

## Time dependent mechanical properties

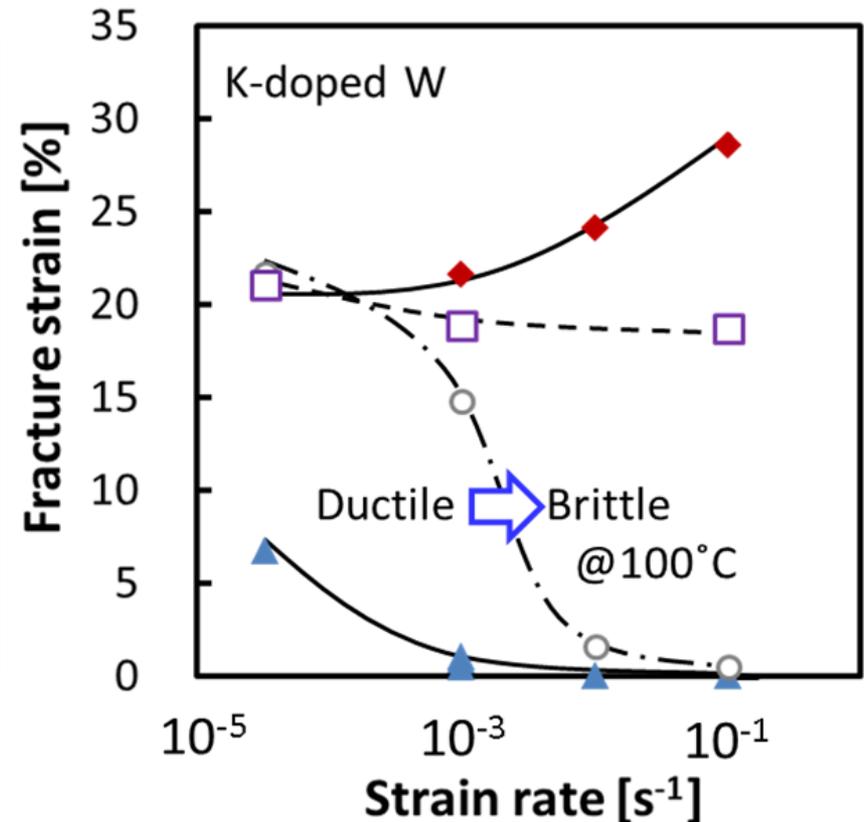
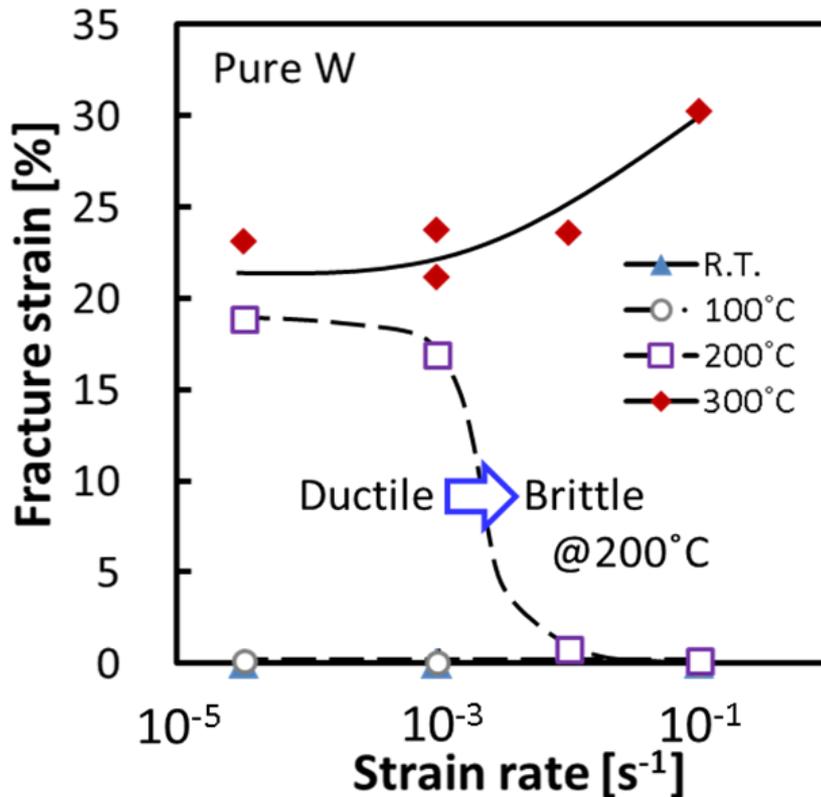
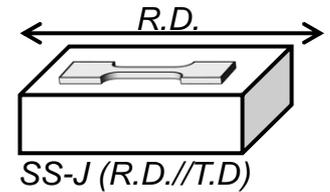


# Strain Rate Dependence of Tensile Behavior<sup>21</sup>

Dept. Quantum Science & Energy Engineering, Tohoku University

## Time dependent mechanical properties

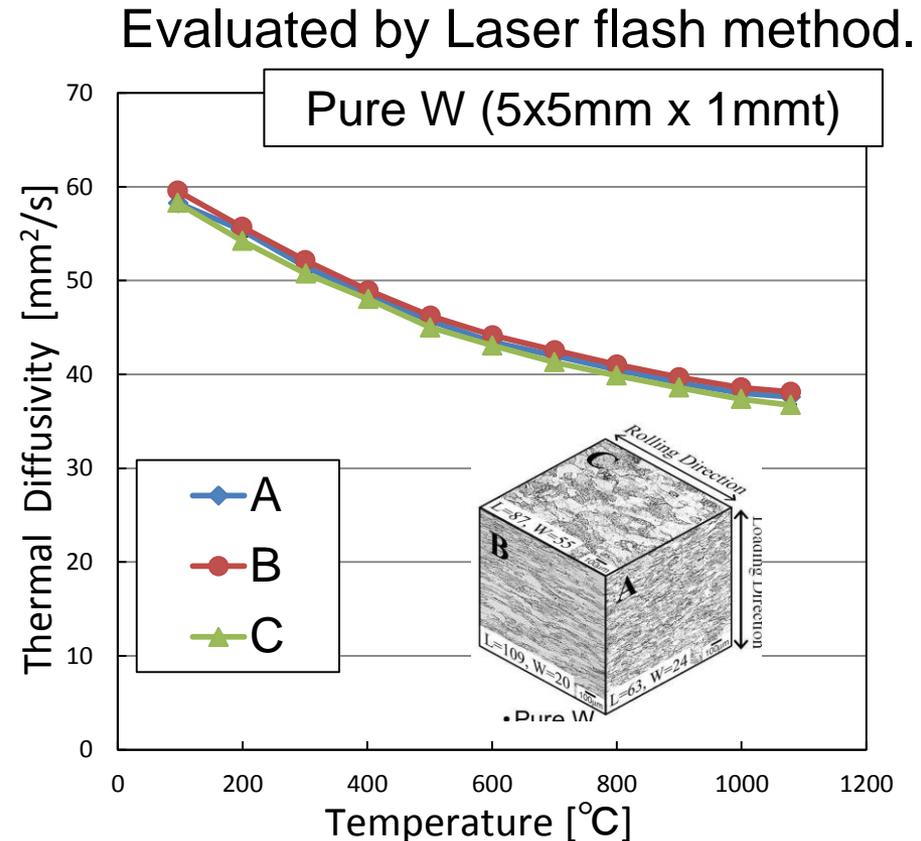
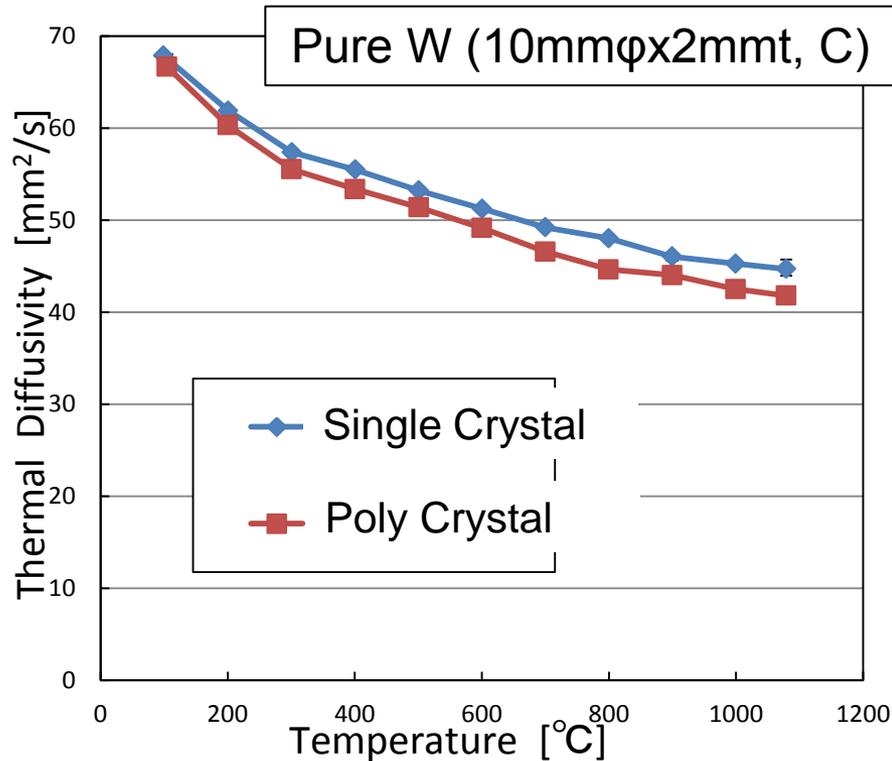
- DBTT of tensile deformation is decreased about 100 °C by K-dope process.



# Thermal Diffusivity

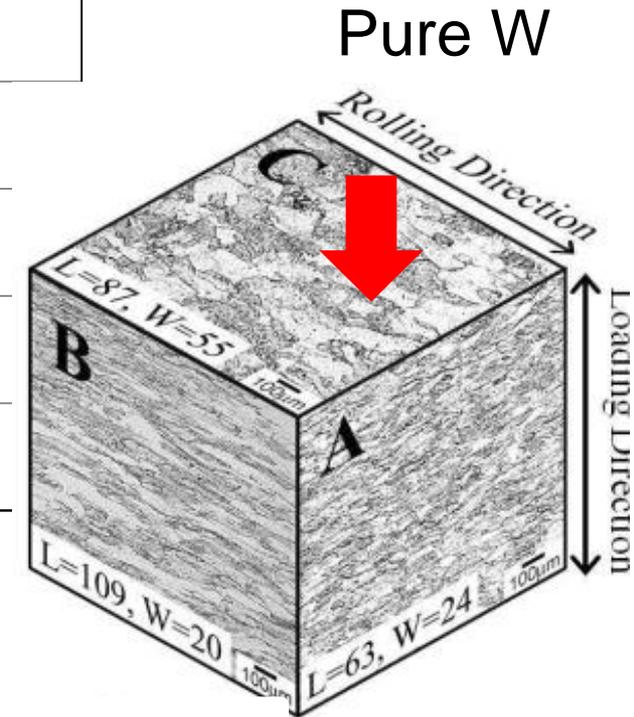
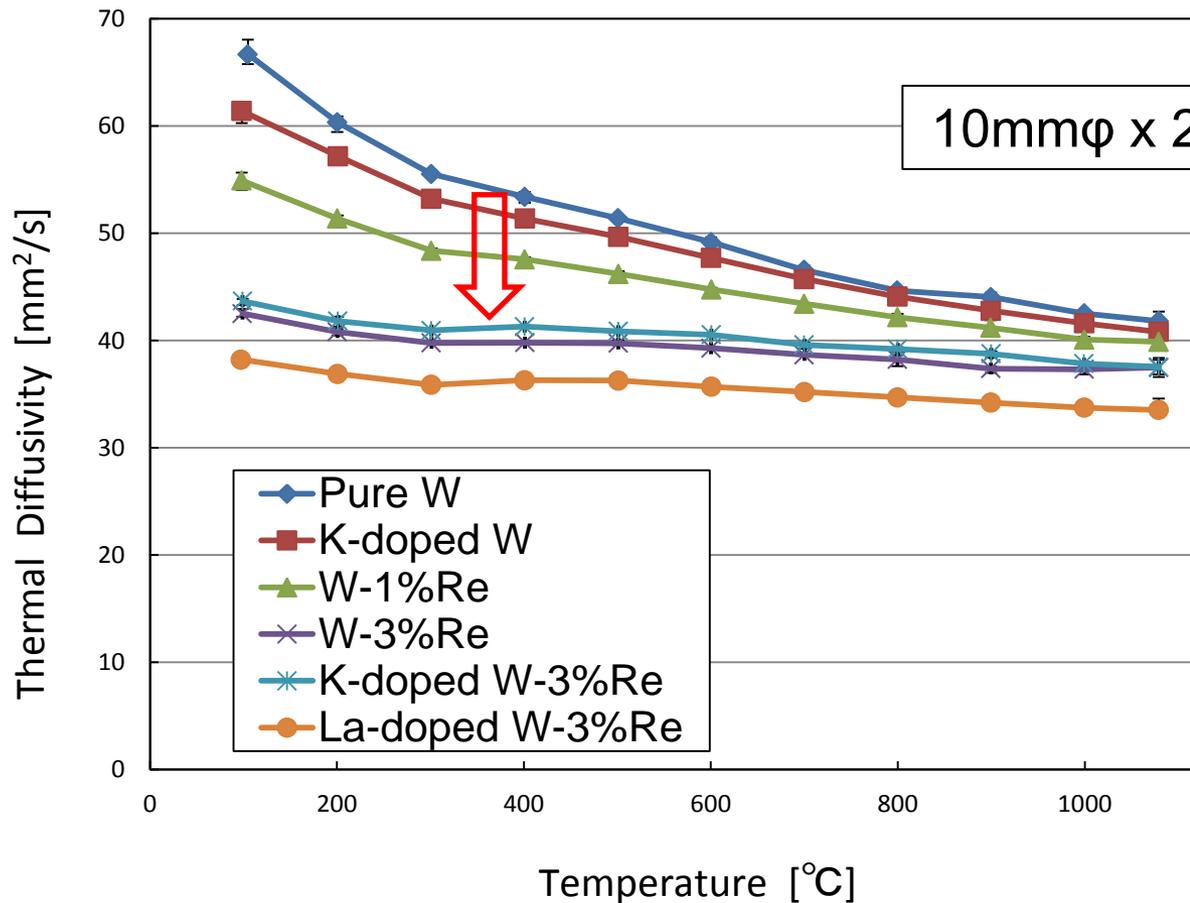
Dept. Quantum Science & Energy Engineering, Tohoku University

## Effects of grain boundary and microstructure anisotropy.



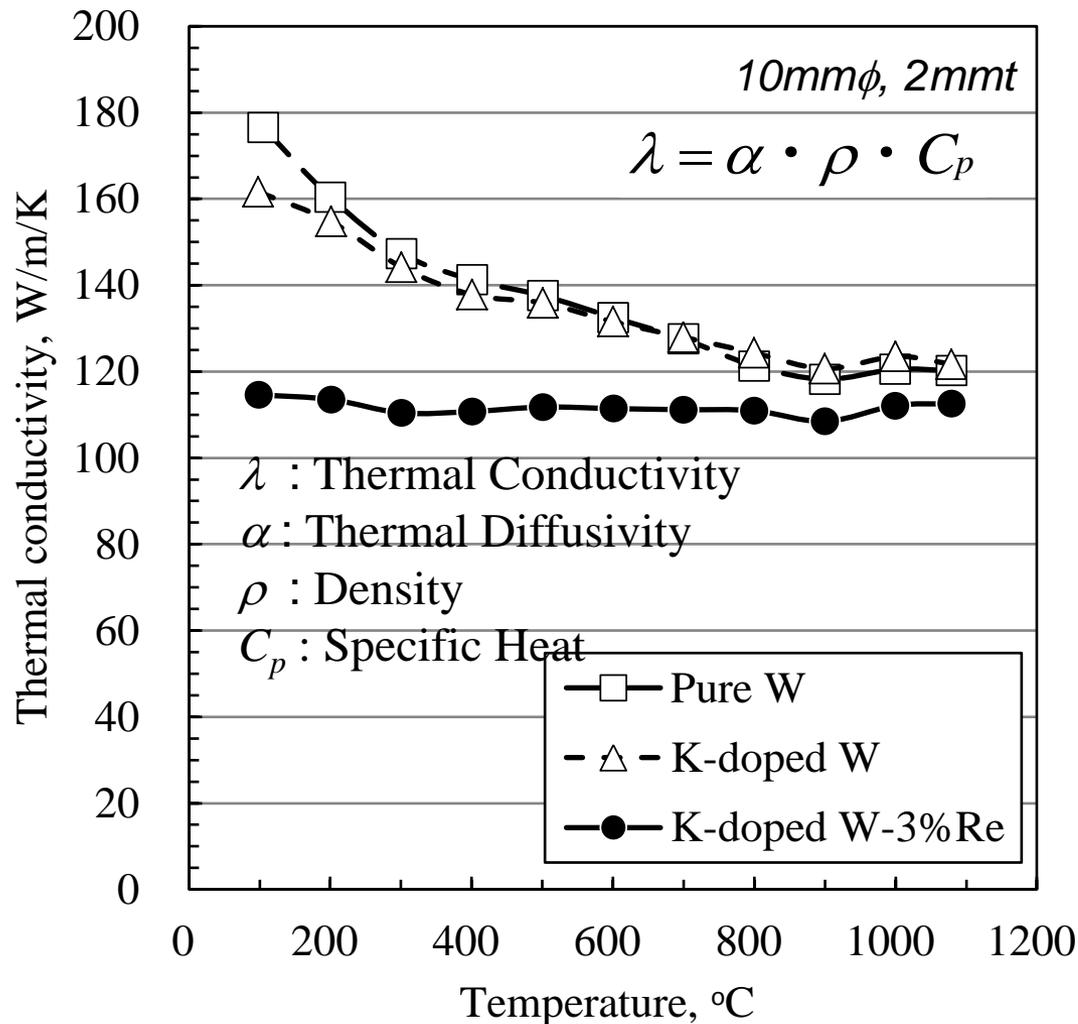
- TD of poly crystal is lower than TD of single crystal, but difference is small.
- Anisotropy of TD of pure W(poly-X) is not observed.

# Thermal Diffusivity of W and W alloys



- TD of W alloys are lower than pure W.
- Temperature dependence of TD was not significant by 3%Re addition.

# Thermal Conductivity of W and W alloys



$$\lambda = \alpha \cdot \rho \cdot C_p$$

$\lambda$  : Thermal Conductivity  
 $\alpha$  : Thermal Diffusivity  
 $\rho$  : Density  
 $C_p$  : Specific Heat

Trend is the same as  
thermal diffusivity

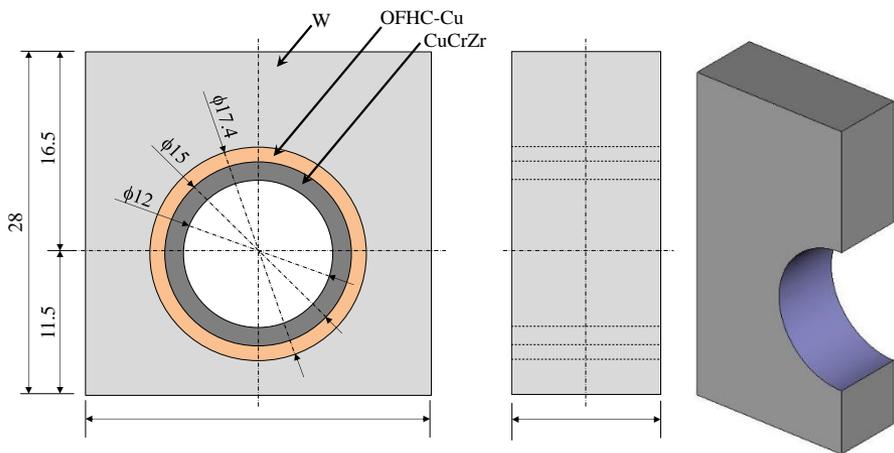
# Outline

Dept. Quantum Science & Energy Engineering, Tohoku University

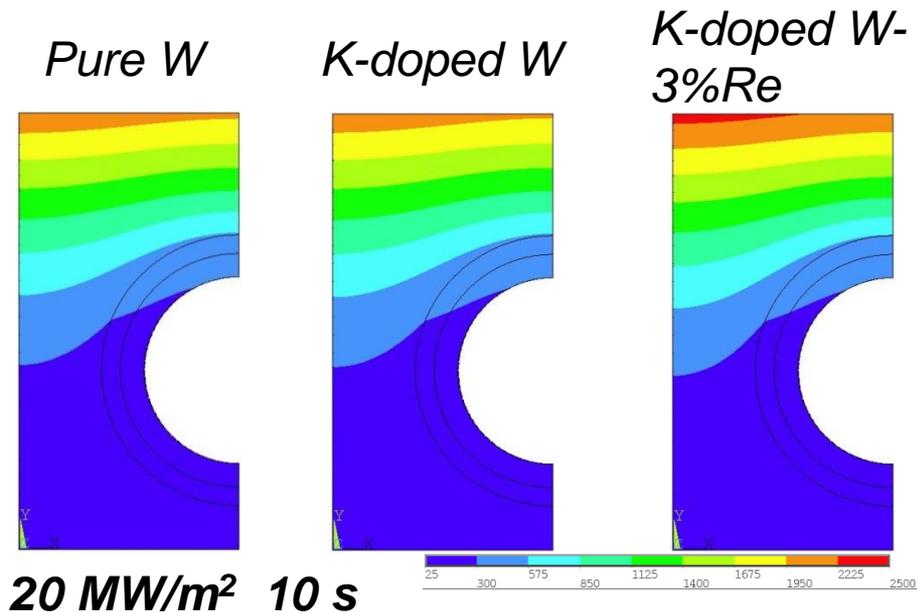
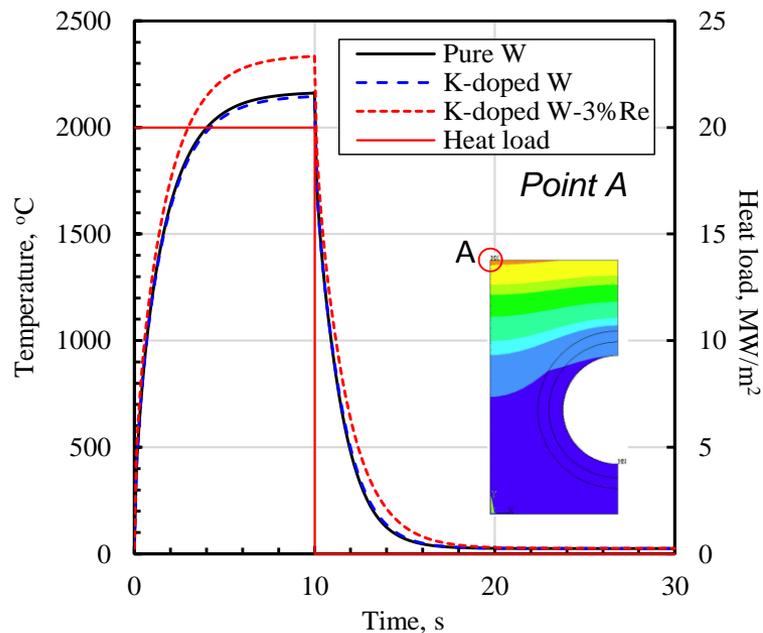
1. Introduction
2. Summary of neutron irradiation effects of Tungsten based on previous data.
3. Prediction of microstructural development of tungsten under fusion reactor irradiation conditions.
4. Tungsten alloys development by grain refining and alloying for fusion application.
- 5. Current status of material evaluation of unirradiated state.**

# Thermo-mechanical Analysis

Dept. Quantum Science & Energy Engineering, Tohoku University



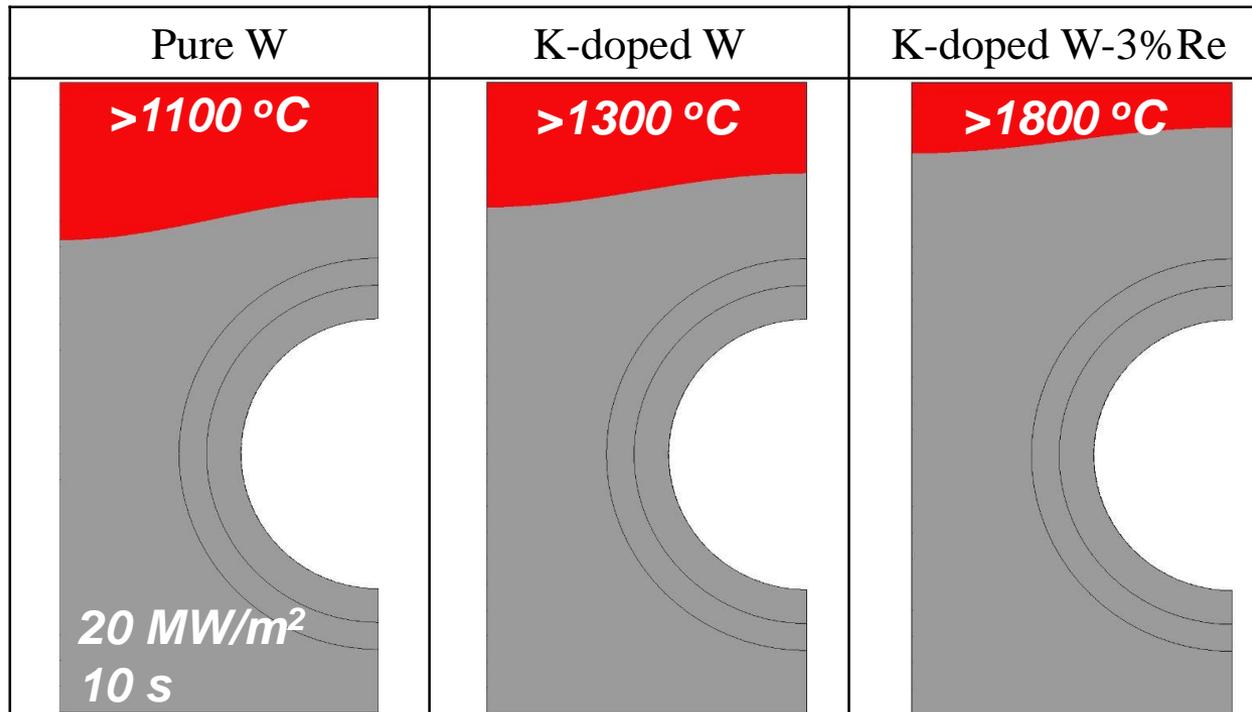
*Thermo-mechanical analysis of the W and its alloys mono-block tile under heat load using FEA is now in progress.*



# Recrystallization

Dept. Quantum Science & Energy Engineering, Tohoku University

- In the case of pure W, the depth of the recrystallized area was ~8 mm from the top surface.
- The depth of the recrystallized area were 6 and 3 mm from the top surface for K-doped W and K-doped W-3%Re, respectively.
  - Increase in recrystallization temperature by K and Re dope decreased recrystallized depth.



# Recrystallization

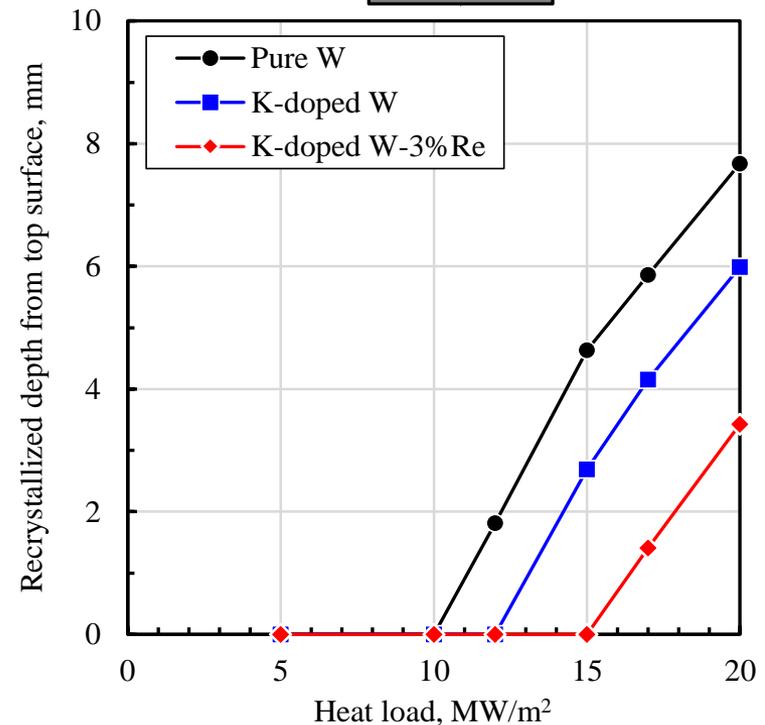
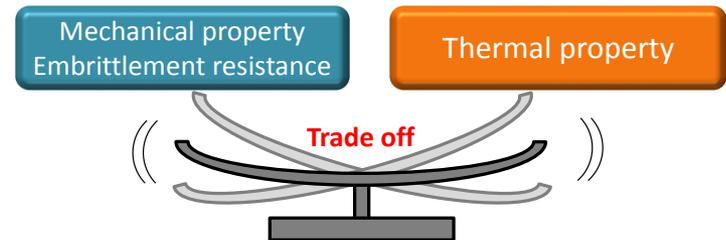
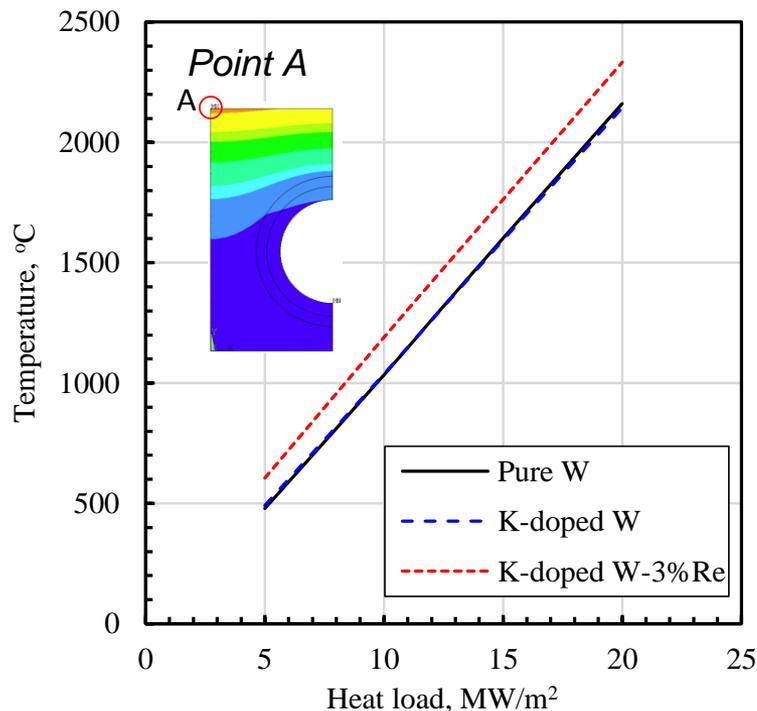
Dept. Quantum Science & Energy Engineering, Tohoku University

- The threshold temperature of the recrystallization was estimated as  $\sim 11$ ,  $\sim 12$ , and  $15 \text{ MW/m}^2$  for pure W, K-doped W, and K-doped W-3%Re, respectively.
- The recrystallization depth was linearly increased with increasing heat load.

Thermal conductivity decrement



Recrystallization temperature increment



# Summary

# Microstructural data of neutron irradiated Tungsten (W) obtained by neutron irradiated W up to 1.5dpa irradiation in the temperature range of 400-800°C were compiled quantitatively. Nucleation and growth process of these defects were clarified and a qualitative prediction of the damage structure development and hardening of W in fusion reactor environments were made taking into account the solid transmutation effects for the first time.

# Powder metallurgically processed pure W and W-alloys were fabricated to improve mechanical properties, recrystallization behavior and radiation resistance of W by grain refining and alloying processes.

# Mechanical property and thermal property of the alloys were obtained. Improvement of strength, low temperature embrittlement and recrystallization behavior of the W-alloys compared to pure W were demonstrated. [Neutron irradiation experiment of these materials using a fission reactor \(HFIR\) will start during 2014.](#)

# Trade-off between the thermal conductivity and mechanical property, embrittlement resistance by the structural control must be considered quantitatively to design diverter cooling component. The thermo-mechanical analysis of the diverter block made of the alloys considering thermal diffusivity and recrystallized temperature were performed by finite element analysis.

*Thank you for your attention.*

