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# Damage Structure of Tungsten under He Particle Loading

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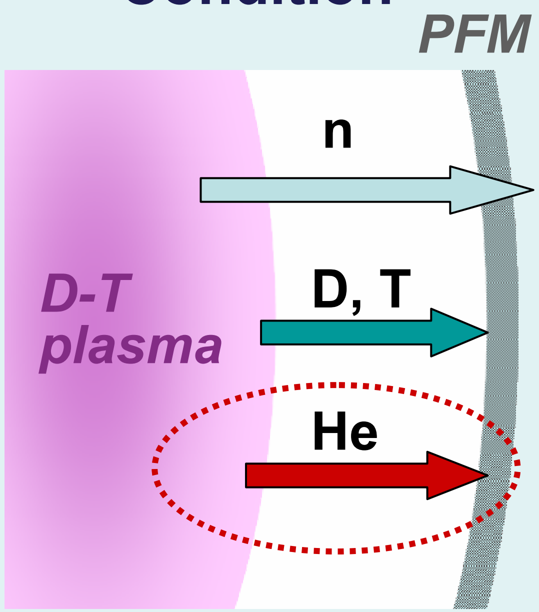
Advanced Diagnostics
for Burning Plasmas

# Background and Objectives

**Irradiation Environment for PFM in D-T Burning Condition**




***Suffer very strong irradiation not only of hydrogen isotopes but also of helium at D-T burning condition***

**In Metals：**

**Very strong He-Defects interaction**

⇓

**Heavy radiation damage**

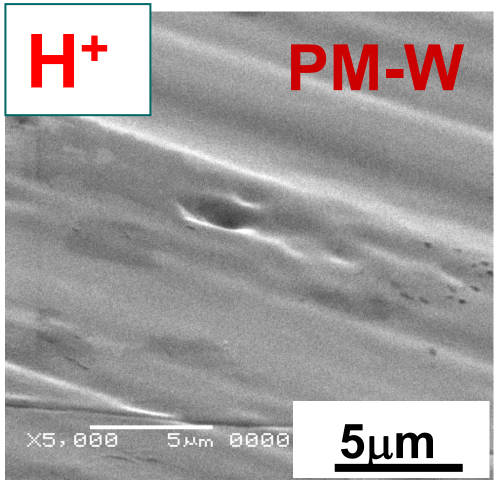
⇓

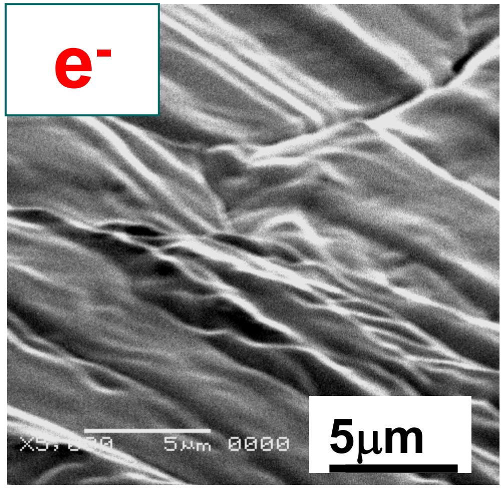
**He irradiation is an important event affecting functions, soundness, life time of the metallic PFMs**

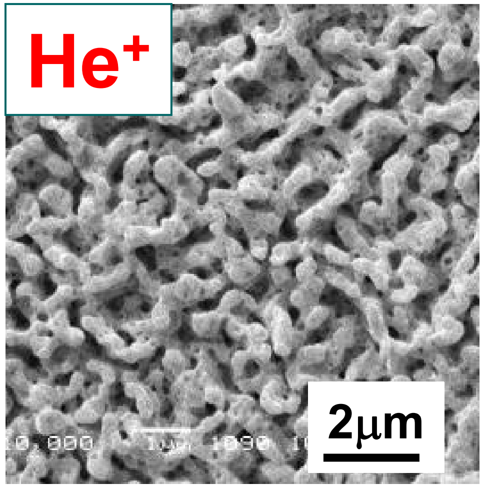
***Objectives*:**

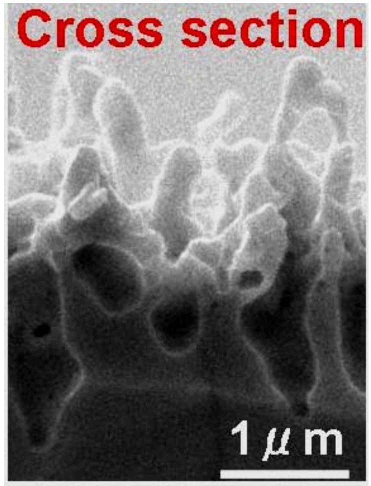
1. **To understand damage structure and its formation mechanism in W by low energy He ions irradiation at divertor relevant conditions (≧1073K).**
2. **To understand the effects on other physical properties such as optical reflectivity.**

# Comparison of Pulse Load of H, $e^+$, He




**1100℃→2700℃**
5.0 x $10^{23}$ H/$m^2$
2.0 s x 100 S
23.6 keV, H
PM-W(30x30x5mm)

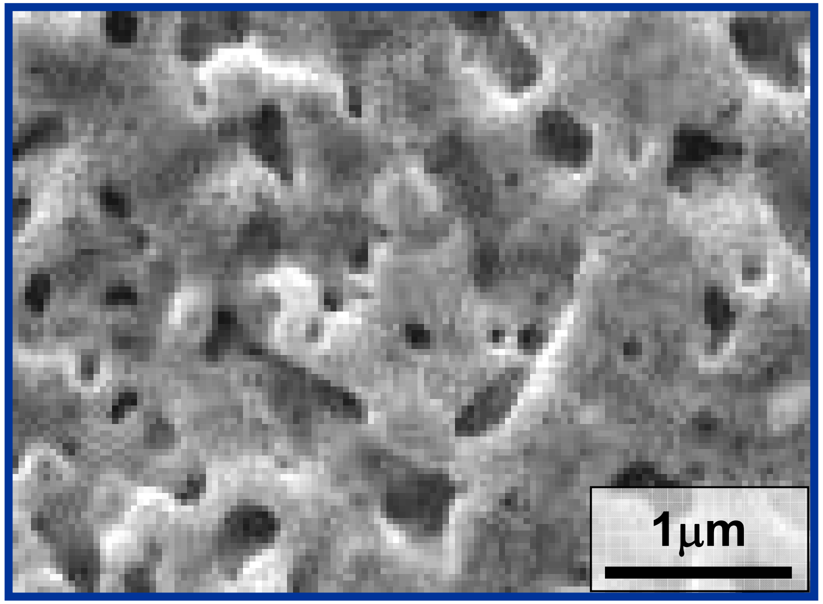
**1150℃→2700℃**
2.0 s x 100 S
34 keV, e
10 MW/$m^2$
PM-W(30x30x5mm)

**Max 2600℃**
3.3x $10^{23}$ He/$m^2$
3.5s/30s( 145S)
**18.7 keV, He**
WB-4(20x20x0.1mm)

- **$e^-$ beam & $H^+$:** very smooth surface, recrystallization, very low damage ⇐ **simple heating effects**
- **$He^+$:** very odd surface morphology (sub-micron projections), large cavities in/under the projections
  ⇐ **effects of He bubbles**

# Repetitive Pulse Heat Loading by $He^+$

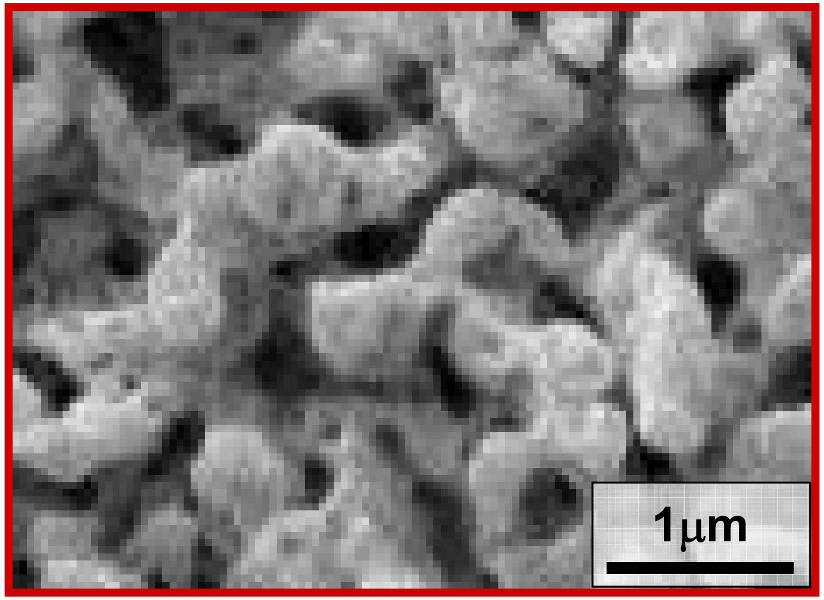
Tokunaga et al.

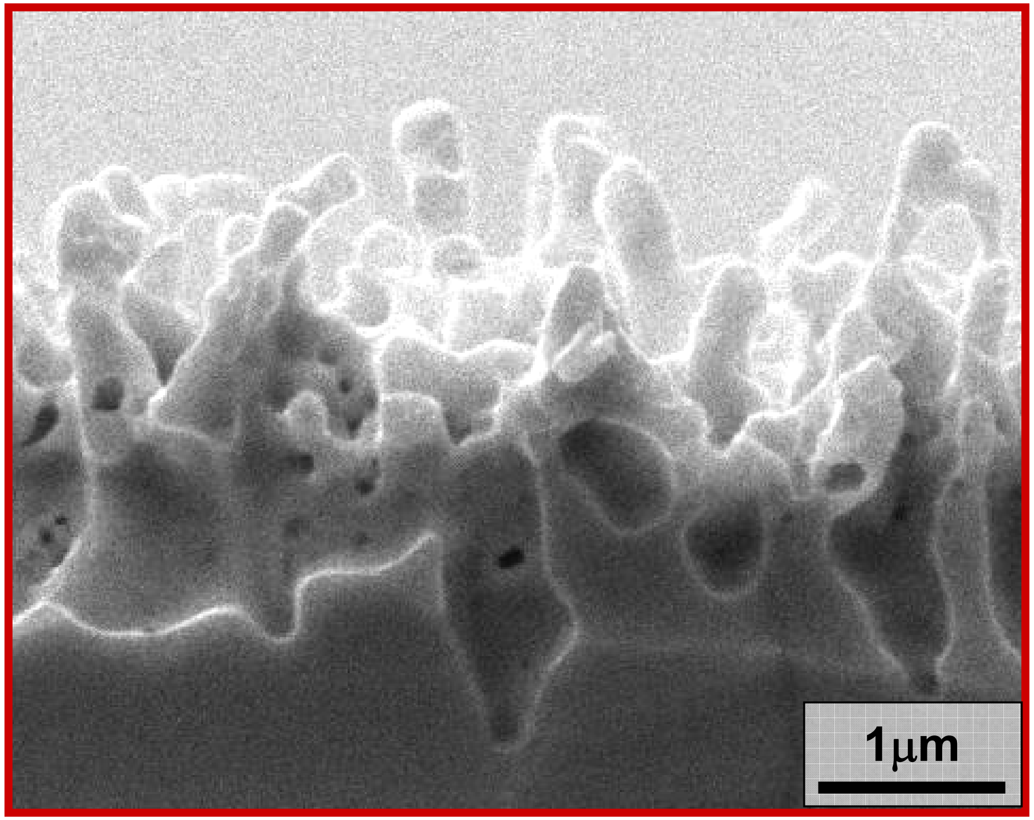



**PM-W** (20x20x0.1mm)

**18.7 keV-$He^+$** (PBEF at JAERI)

- $3.7 \times 10^{22}$ He/m², 3.5s/30s ( 8S)
- $3.3 \times 10^{23}$ He/m², 3.5s/30s ( 145S)
- ≤ 2600°C

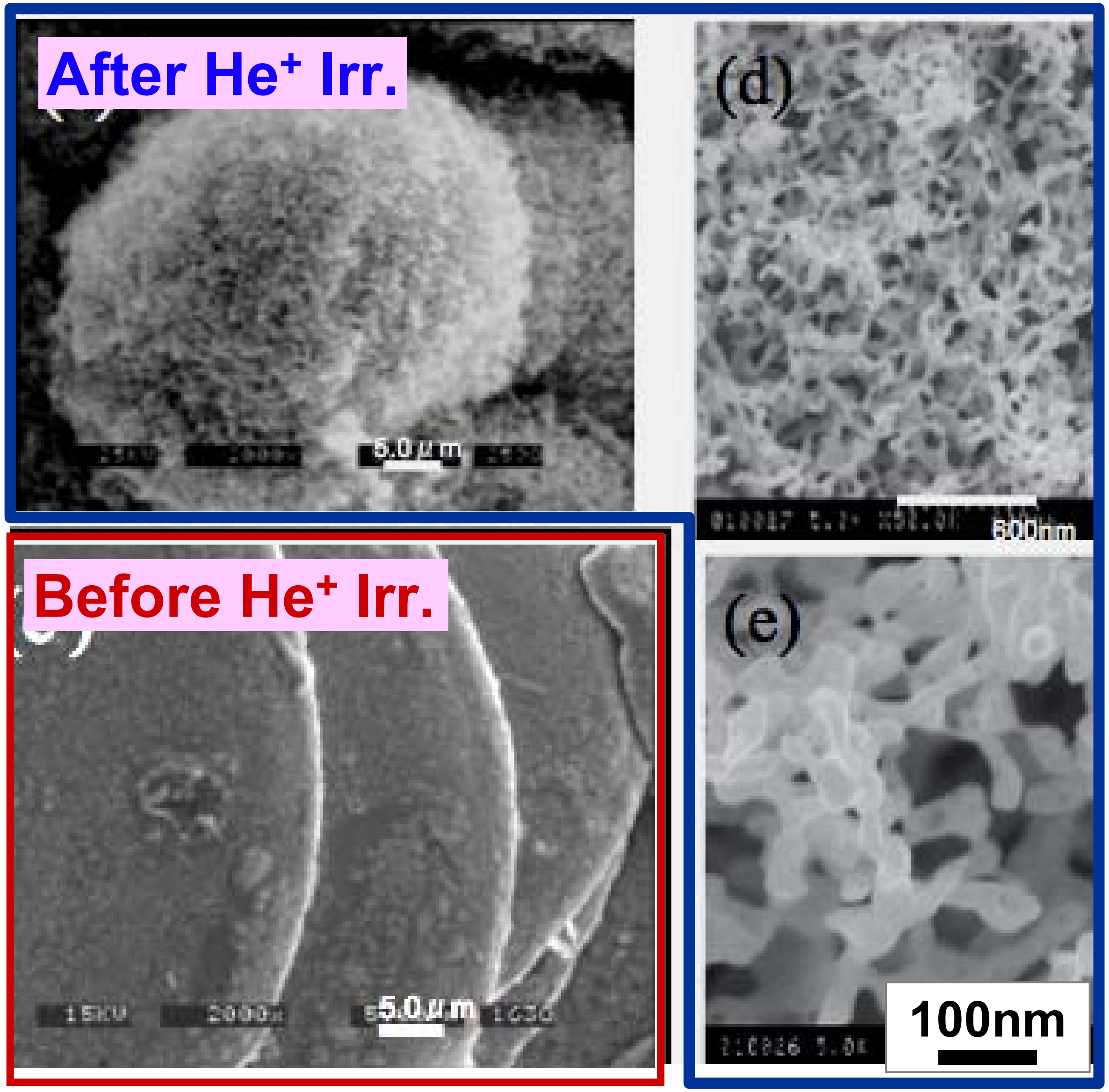


# Drastic Change of Surface by $He^+$ Irr.

**VPS-W@1250 K, 11.3 eV-$He^+$, $3.5\times10^{27}\ m^{-2}$**

**Takamura et al.**




S. Takamura et al., Plasma Fusion Res. 1(2006)051

- **Irradiated Surface is covered by nano-size fine projections with a few 10 nm in diameter**

**How they were made?**

⇩

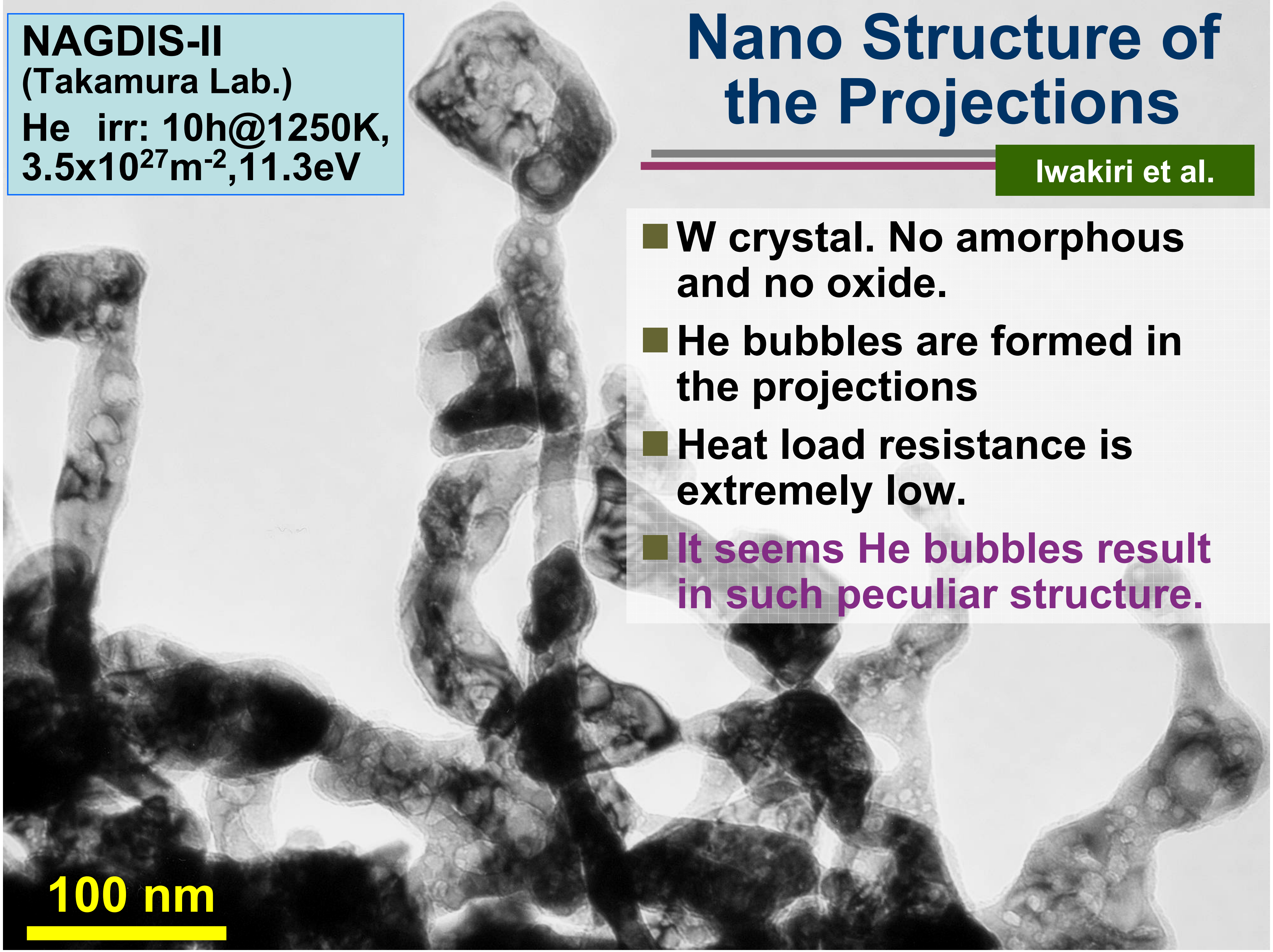
**TEM Observation**

# Nano Structure of the Projections

Iwakiri et al.

**NAGDIS-II**
(Takamura Lab.)
He irr: 10h@1250K,
$3.5x10^{27}m^{-2}$,11.3eV

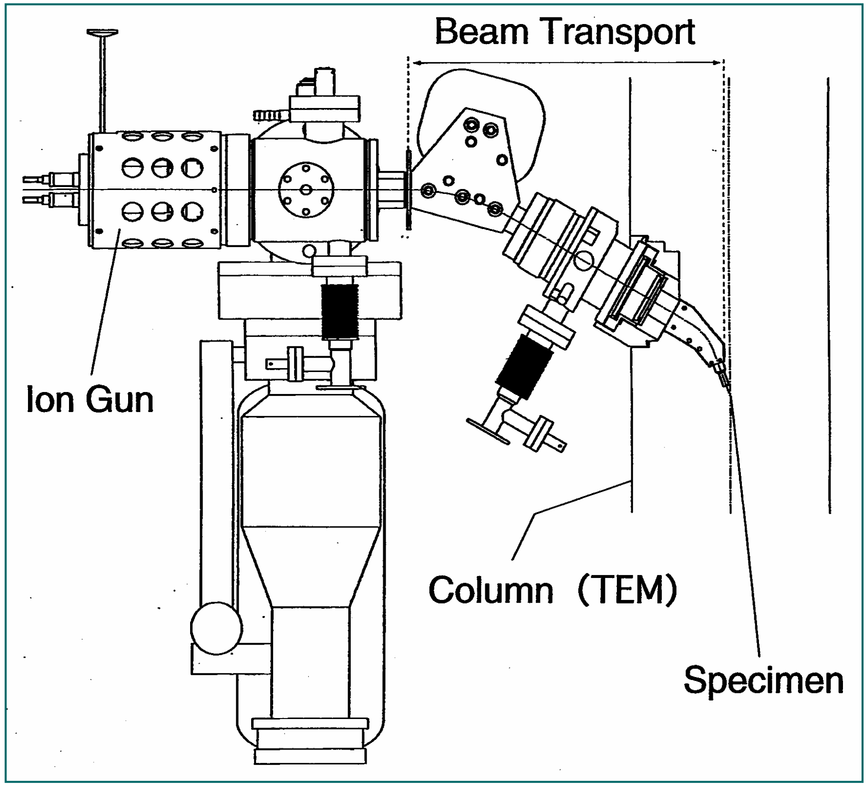
- W crystal. No amorphous and no oxide.
- He bubbles are formed in the projections
- Heat load resistance is extremely low.
- It seems He bubbles result in such peculiar structure.



# In *situ* Obs. of Dynamical Damage Proc.

**Ion Injector**

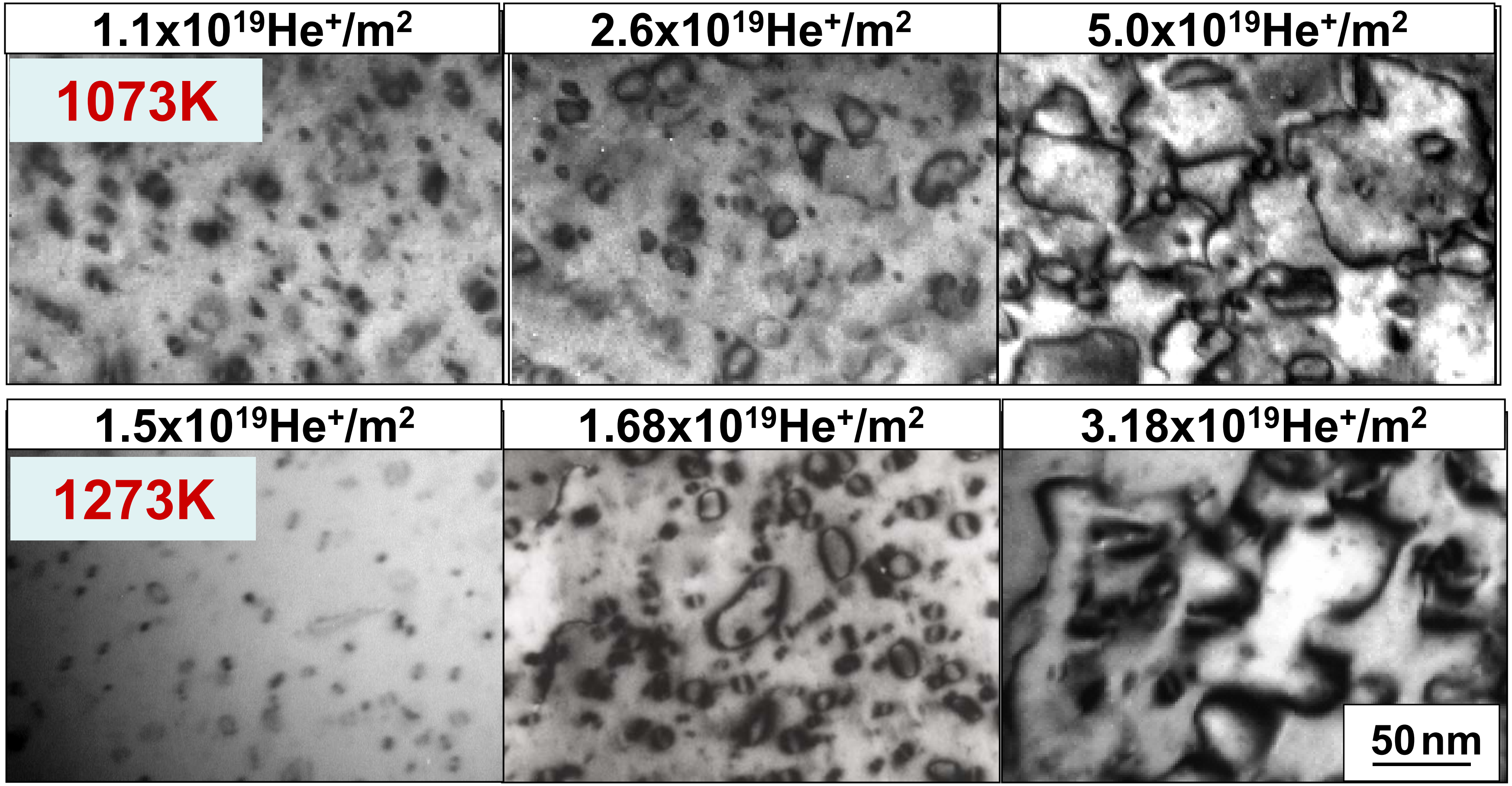
- **Acc. Energy : 0.1～10 keV**
- **Ion Species : H, D, He**
- **Ion Current (on the specimen):**
  **2 μA (@0.2keV)**
- **Record : films, video**




# Development of I-Loops under $He^+$-Irr.

Iwakiri et al.

**PM-W, 8 keV-$He^+$, In *situ* obs. under He ion irradiation**

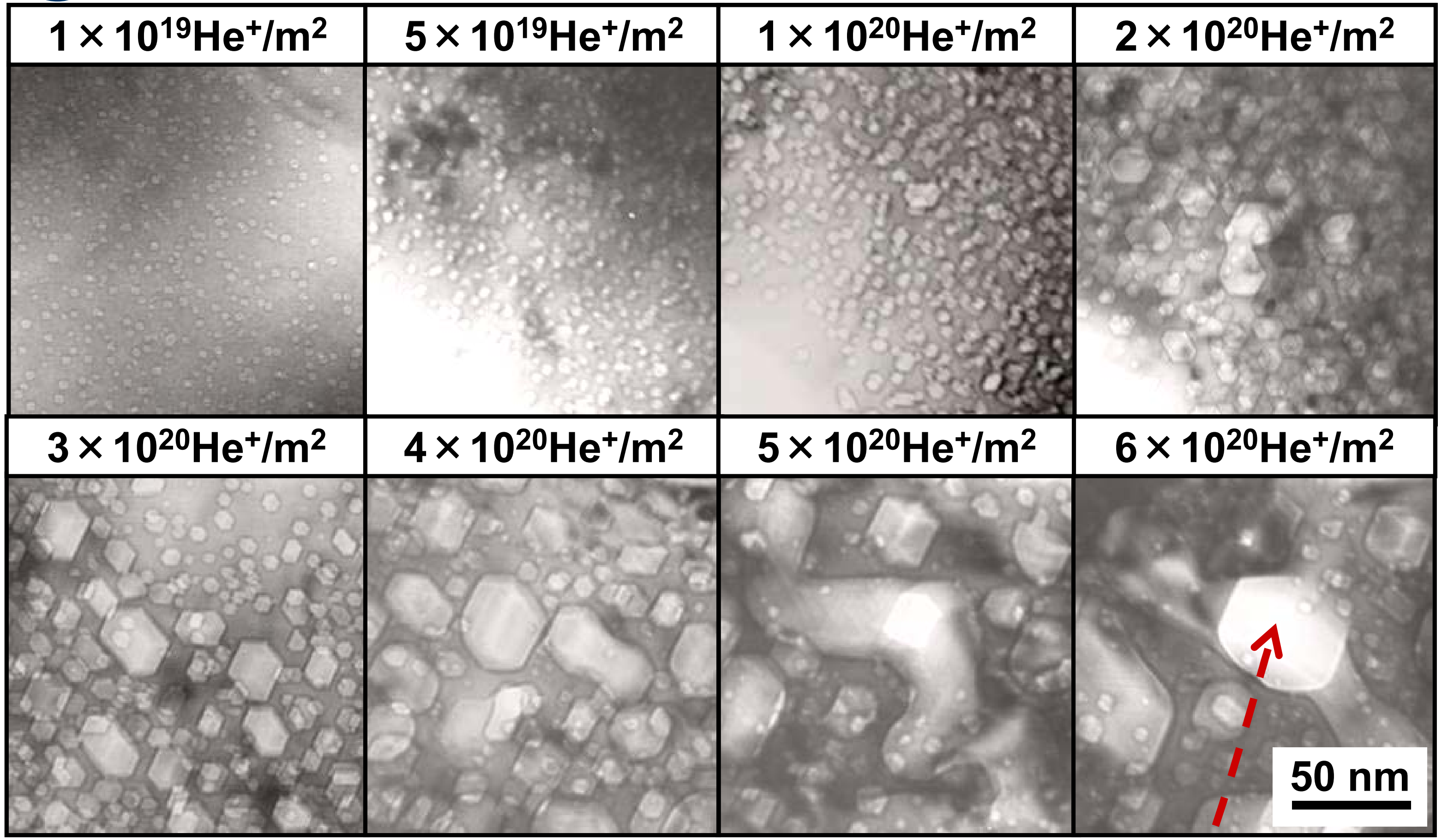



- Nucl. and growth of dislocation loops. ⇒ dis. network
- Such damage does not occur under $H^+$ irradiation at such high temp ← pinning of dislocation by He (He bubbles)

# Simultaneous Formation of He Bubbles

Iwakiri et al.

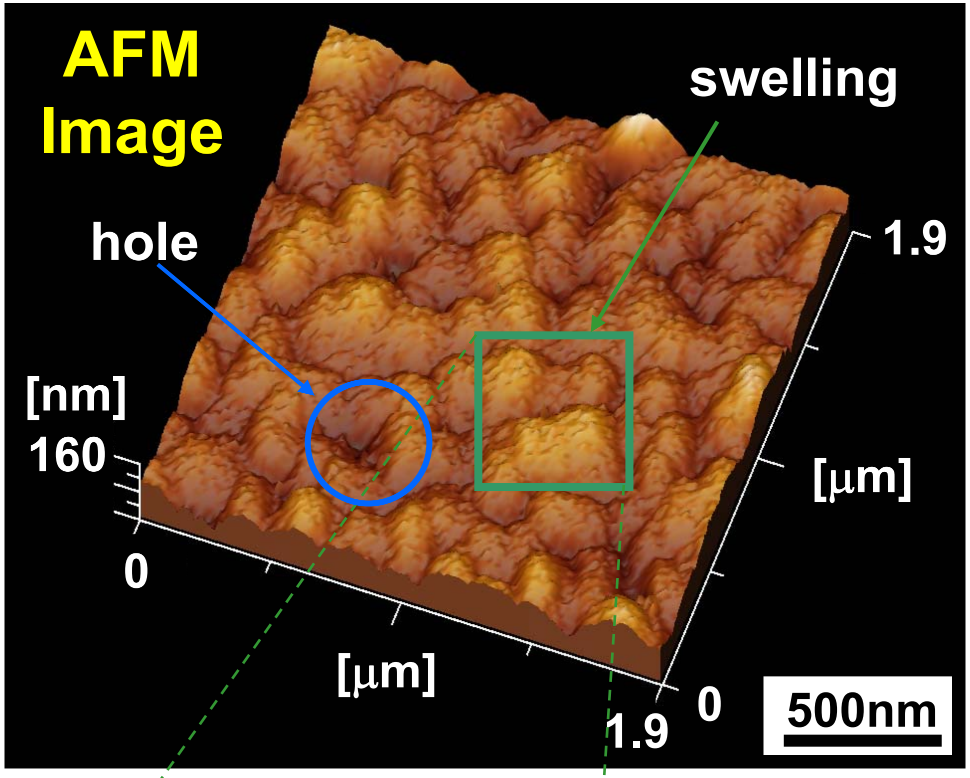
**PM-W@1273K, 8 keV-$He^+$, In *situ* obs. under $He^+$ irradiation**




- **Development of large bubbles by growth and coalescence.**
- **Holes and groove are formed at the surface once the large bubbles arrive at the surface.**

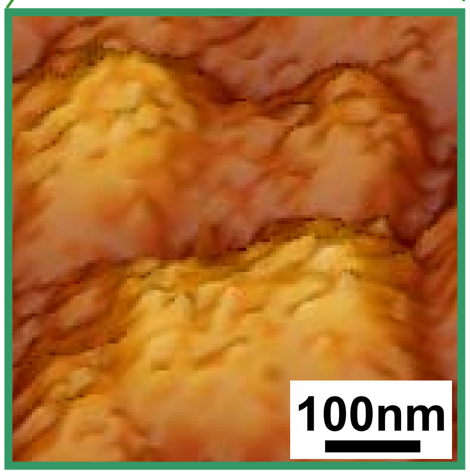
# Surface Morphology of He Irr. W

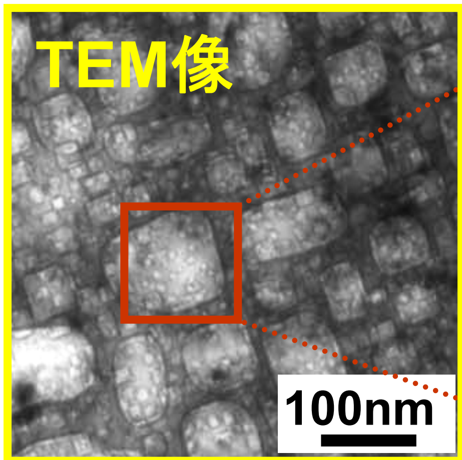
Iwakiri et al.

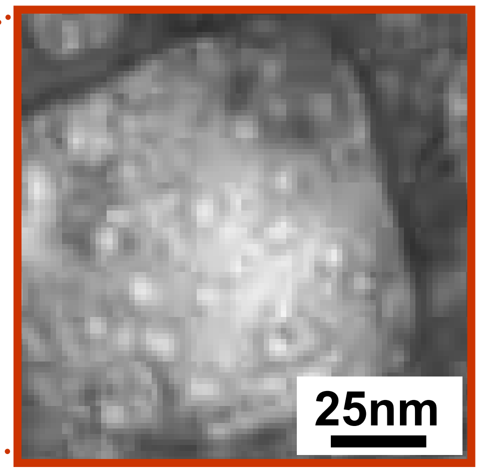



**Sample : PM-W**
**Ion energy : 8 keV**
**flux : $1.5x10^{22} He^{+}/m^{2}$**
**Irradiation temp. : 1273 K**

- **Large bubbles cause local surface swelling ➜surface roughing**
- **Holes are formed by arrival of bubbles at the surface.**



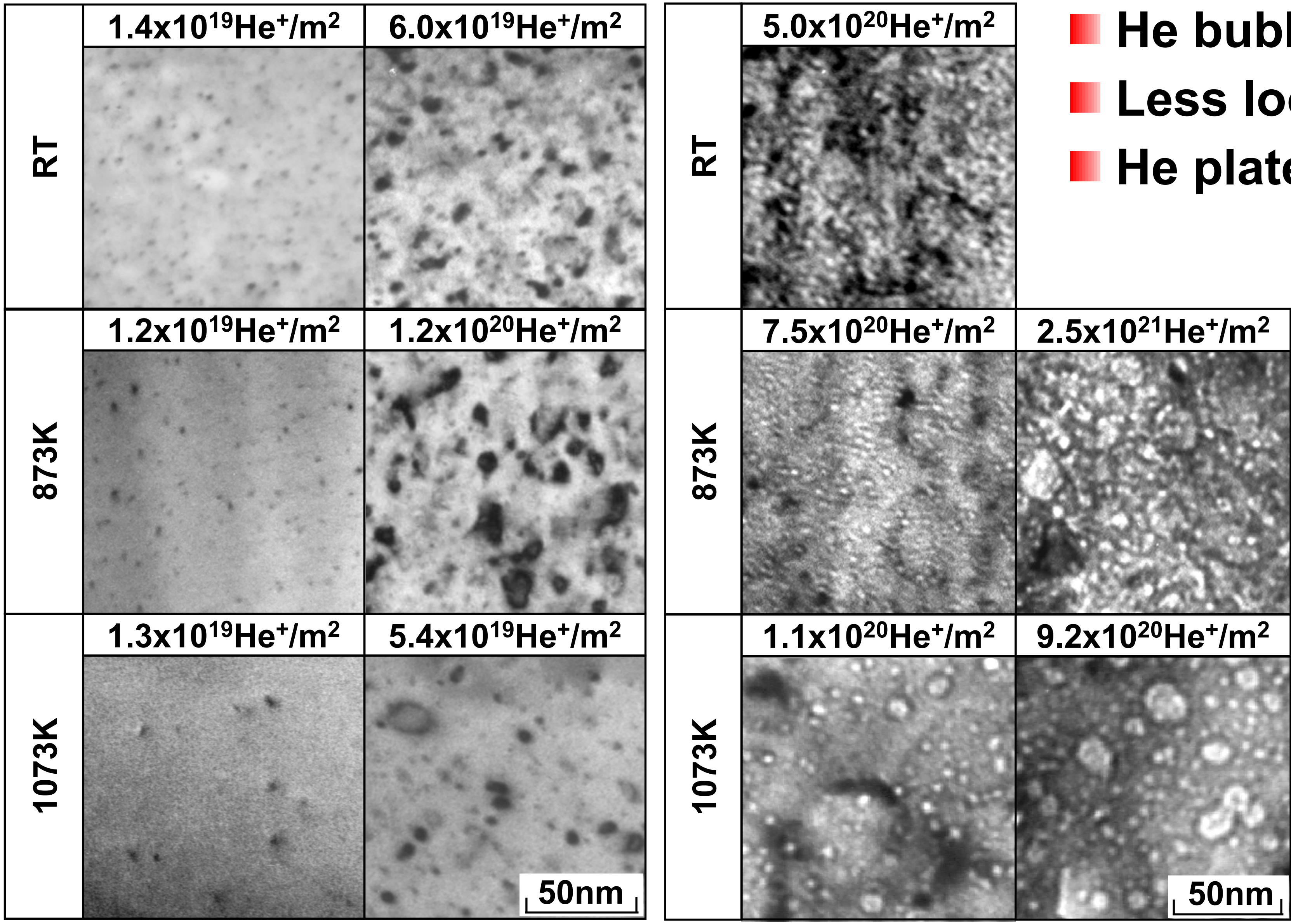



# Damage by $He^+$ with sub-$E_d$ Energy

Iwakiri et al.

## W, 0.25keV-$He^+$

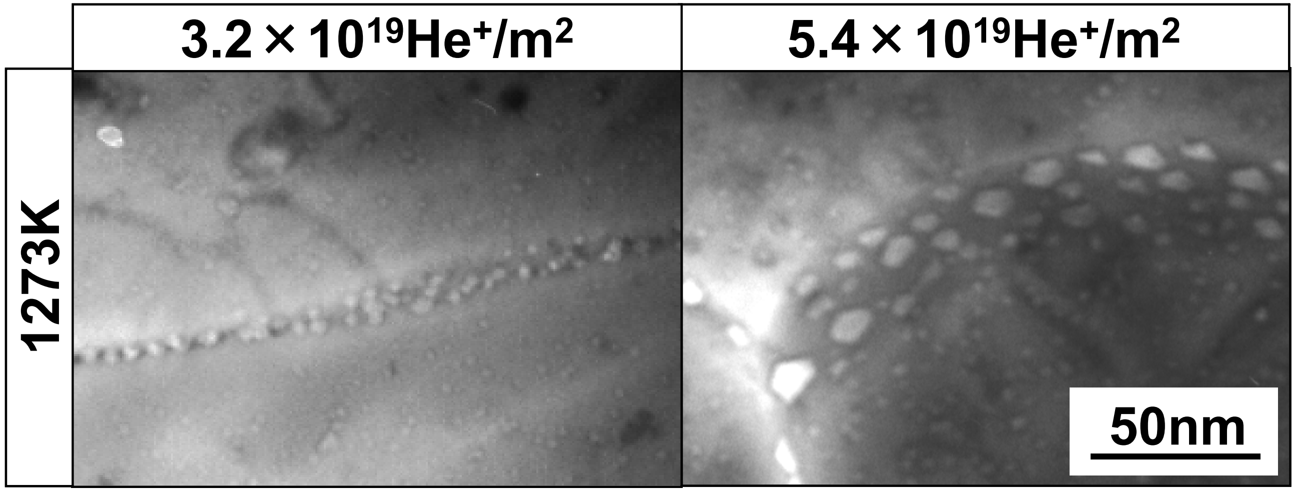



- He bubbles
- Less loops
- He platelets

# Form. of He Bubbles at Grain Boundary

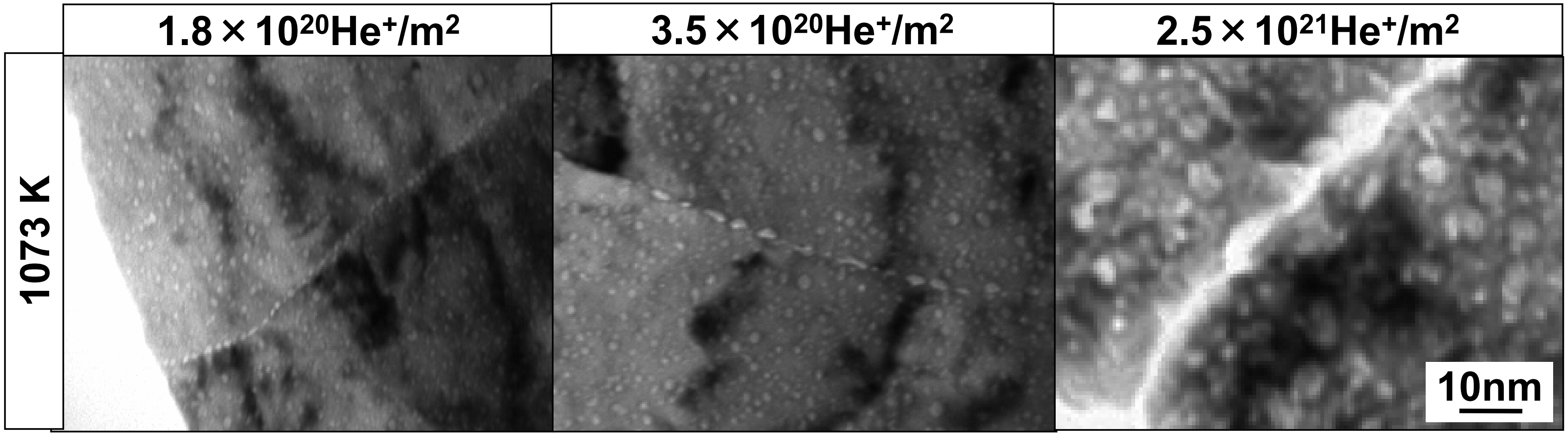
Iwakiri et al.

## $E>E_d$ case: 8keV-$He^+$ ⇛ PM–W@1273 K




**Preferential formation of He bubbles along GB causes GB embrittlement.**

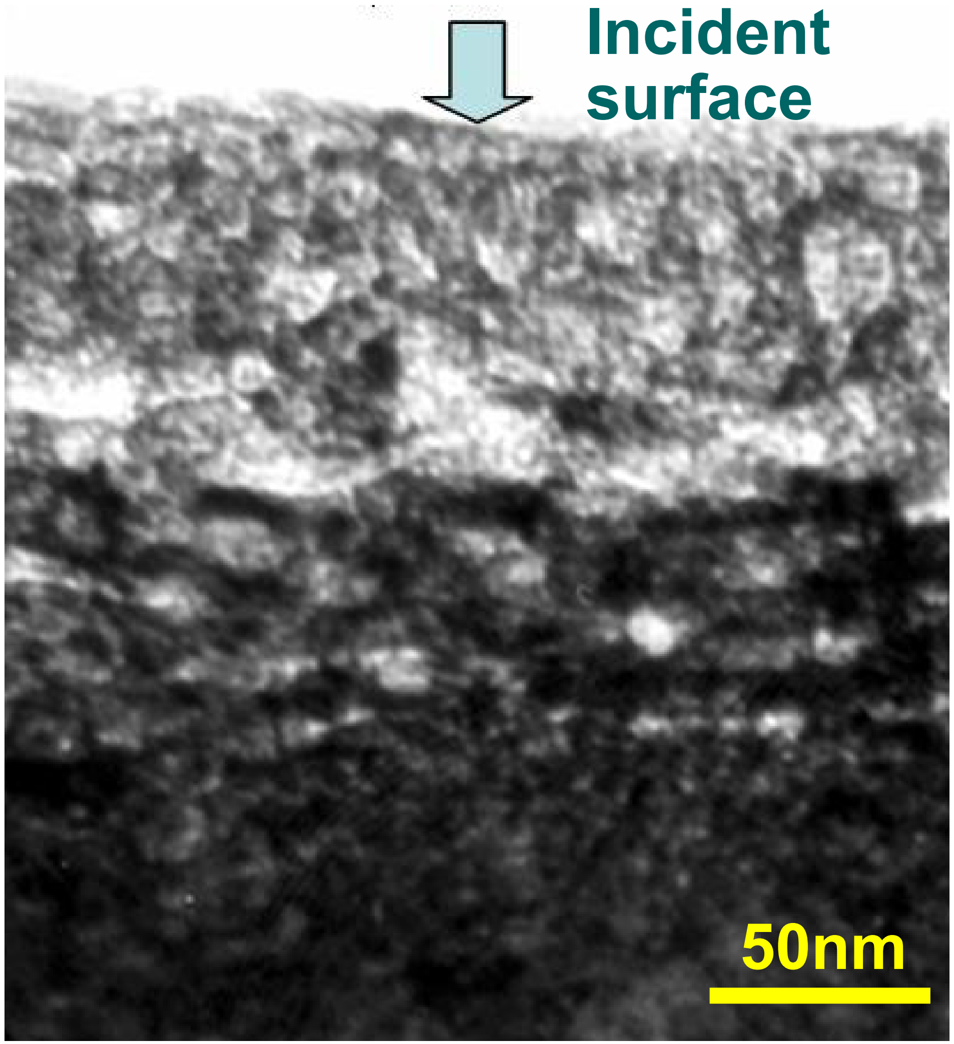
## $E<E_d$ case: 0.25keV-$He^+$ ⇛ PM–W@1073 K



# Cross-Sectional View of Sub-Surface Damage in He Irradiated Mo

Tokitani et al.

8keV-$He^+$, $1x10^{22}$ $He/m^2$ ⇒ Mo @ 873K




(Corresponding 1111K of W)

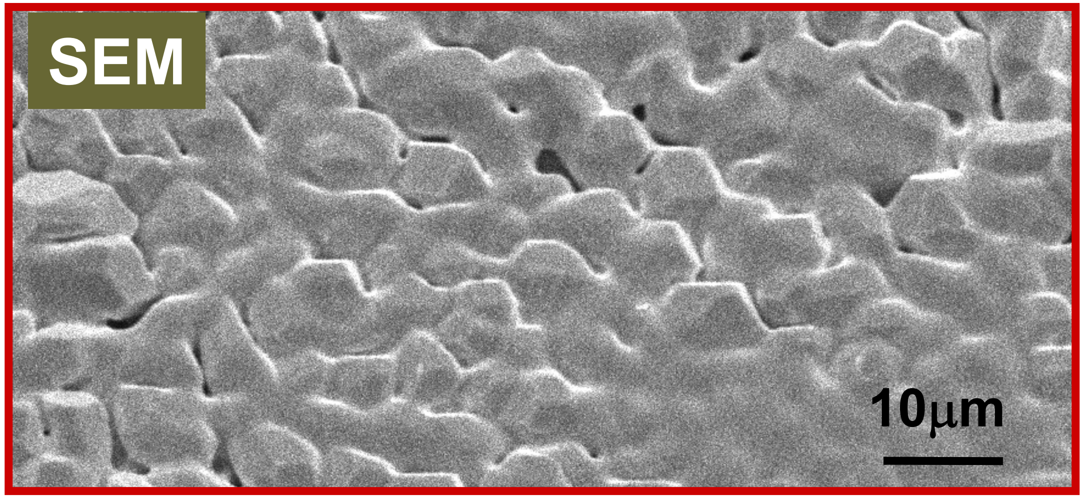
- Very dense fine He bubbles are formed in the sub-surface region of about 200nm-thick.

  **nano-size sponge-structure**

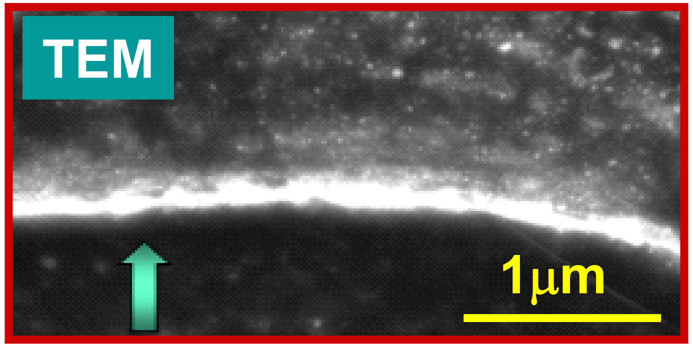
- The sponge layer will change the physical properties of the surface, such as thermal conductivity and optical properties.

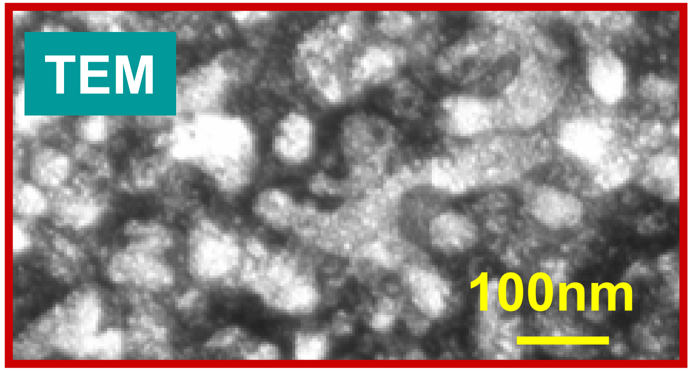
# Form. of Grooves at Surface by $He^+$ Irr.

**PM-W, 1273K, 0.25keV-He, $1\times10^{21} He^+/m^2$**

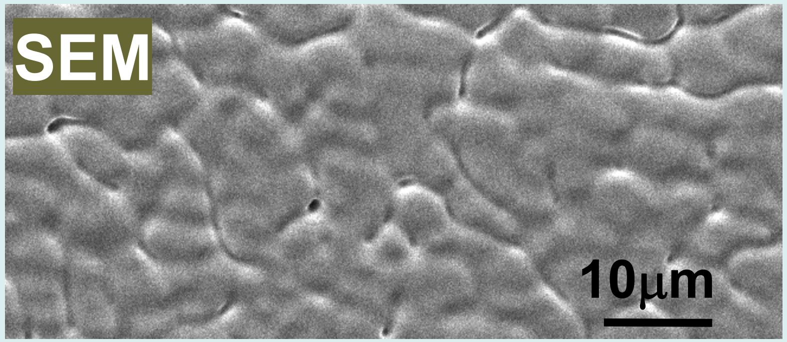



- Typical damage at high temperatures ( ≥1273K)
- Deep grooves are formed by the growth and aggregation of He bubbles.
- Role of thermal vacancies.
- Similar damage in LHD

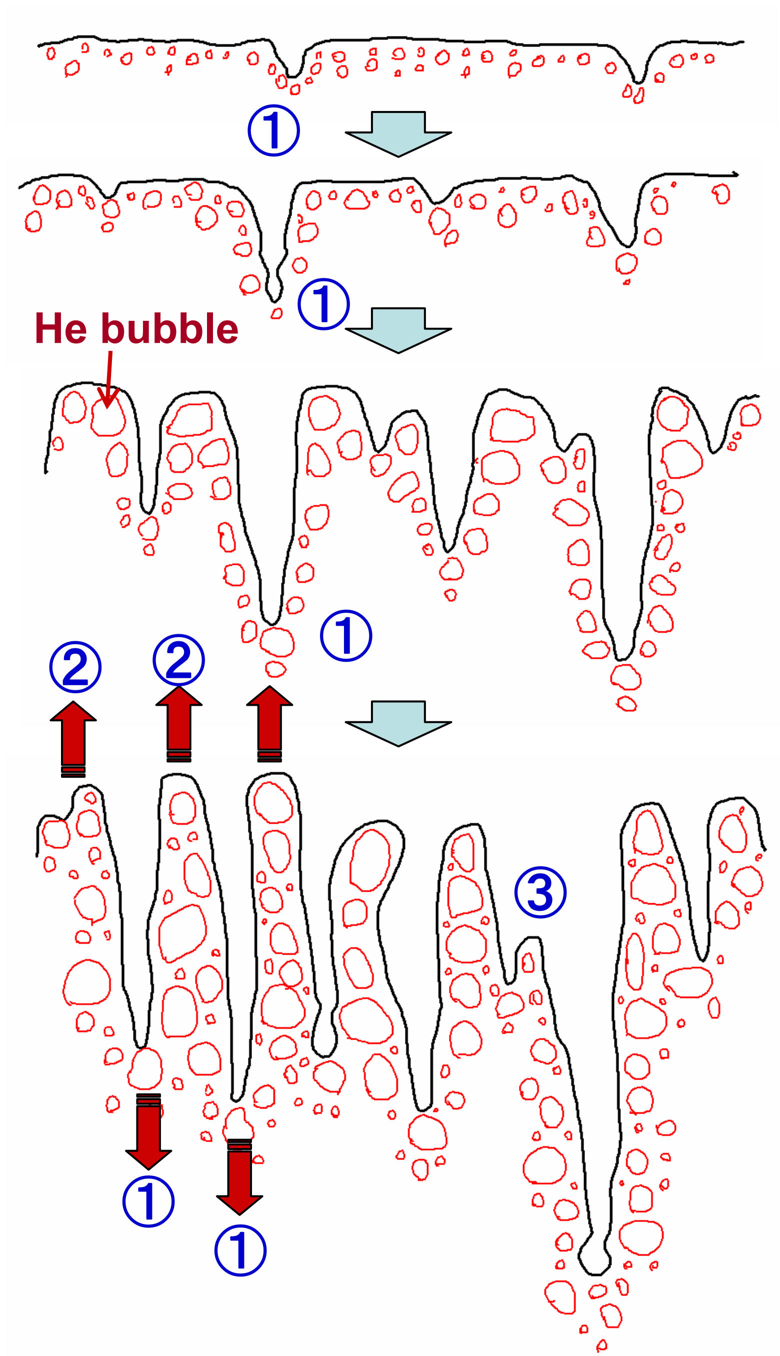



W at divertor position
LHD He plasma (1s)



# Formation Process of Sub-Micron Projections under $He^+$ Irradiation




## *Formation Processes*

① **Arrival of bubbles to the bottom surface of the grooves ➜ grooves become deeper and deeper by repeating the process.**

② **Projections are elongated by the swelling due to He bubbles.**

③ **Blanching.**

## *Conditions for Projection Formation*

1. **thermal growth and thermal migration of He bubbles,**
2. **supply of thermal vacancies**
3. **adequate surface diffusion**

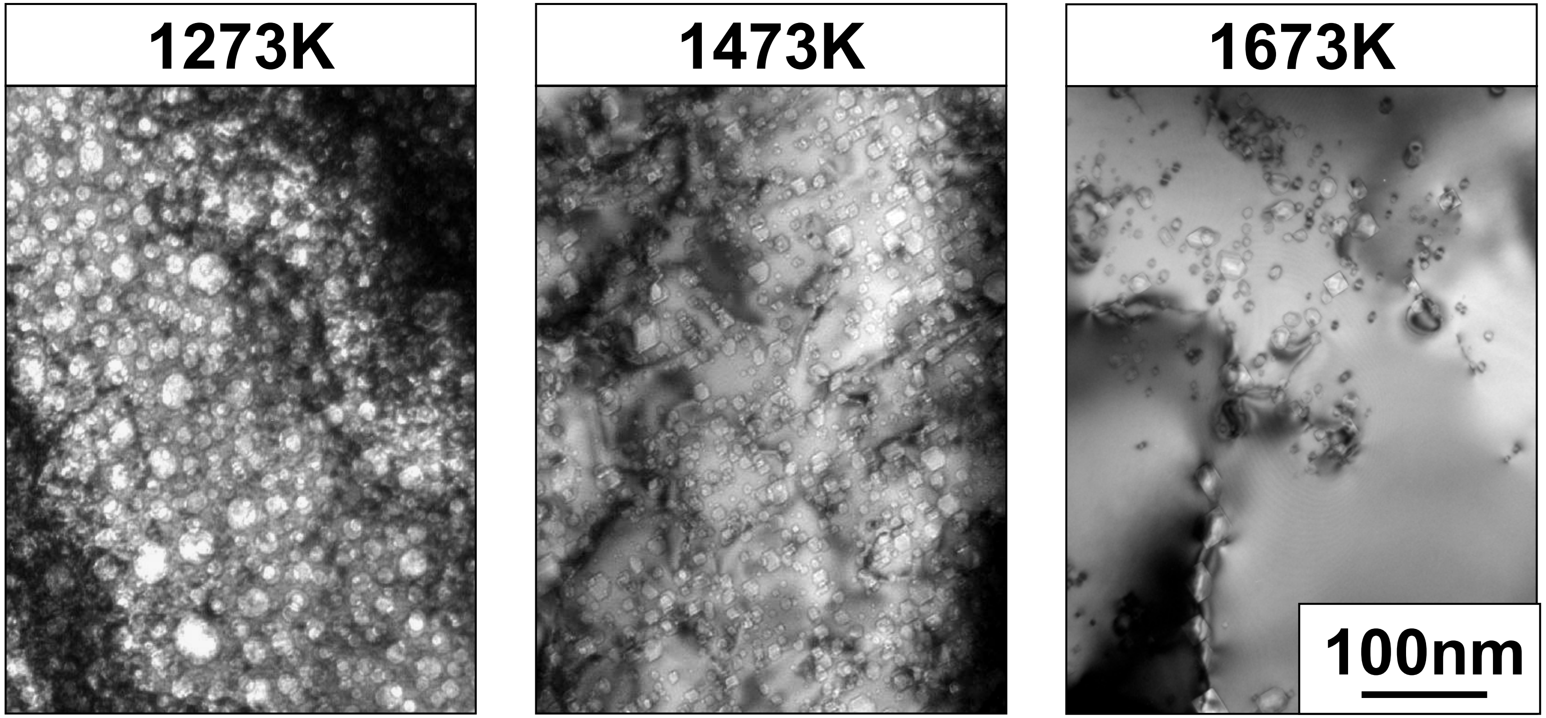
***Possible Temp. Range (W)***

**$0.3T_m \sim 0.5T_m$**

4. **Sputtering erosion may reduce elongation speed.**

# Role of Dislocations on Bubble Formation

**PM-W, 5keV-$He^+$,$1x10^{21}$ions/$m^2$** **Specimen thickness ～100nm**

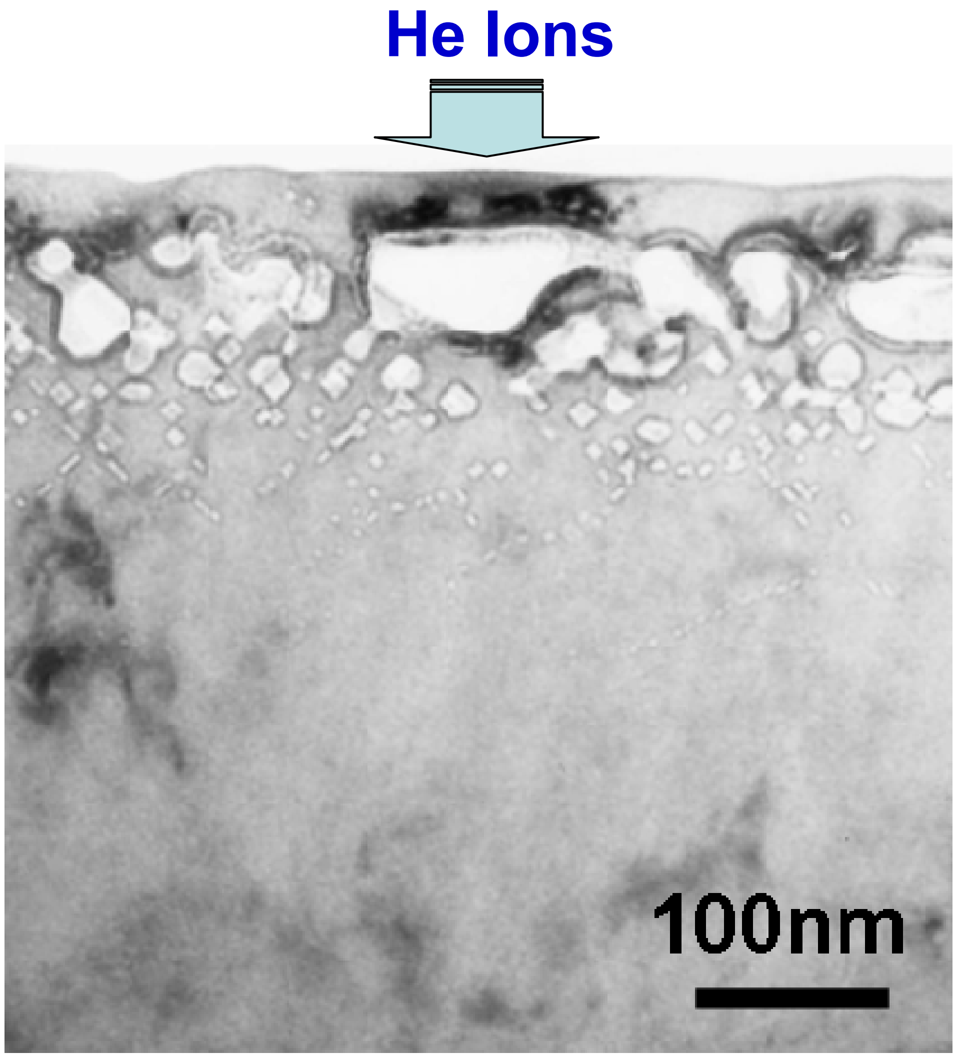



- **He bubbles exist along dislocations ⇒ mutual trapping ⇒ stabilization of bubbles**
- **At 1673K, bubble density decreases, because the dislocations annihilate actively at the surface.**
- **But, large bubbles with low migration rate exist in deeper region. ➜ see next view graph**

# Cross Sectional TEM Image of He$^+$ Irradiated Fe-9Cr Alloy

**8keV-He$^+$, 8x10$^{21}$ He/m$^2$ ⇒ Fe-9Cr Alloy @ 873K**

**(Corresponding 1800K of W)**




- **Almost no bubble near the surface.**
- **Large bubbles are formed in the area beyond the projection range of He ions (40nm).**

**Diffusion Coef. of Bubbles**

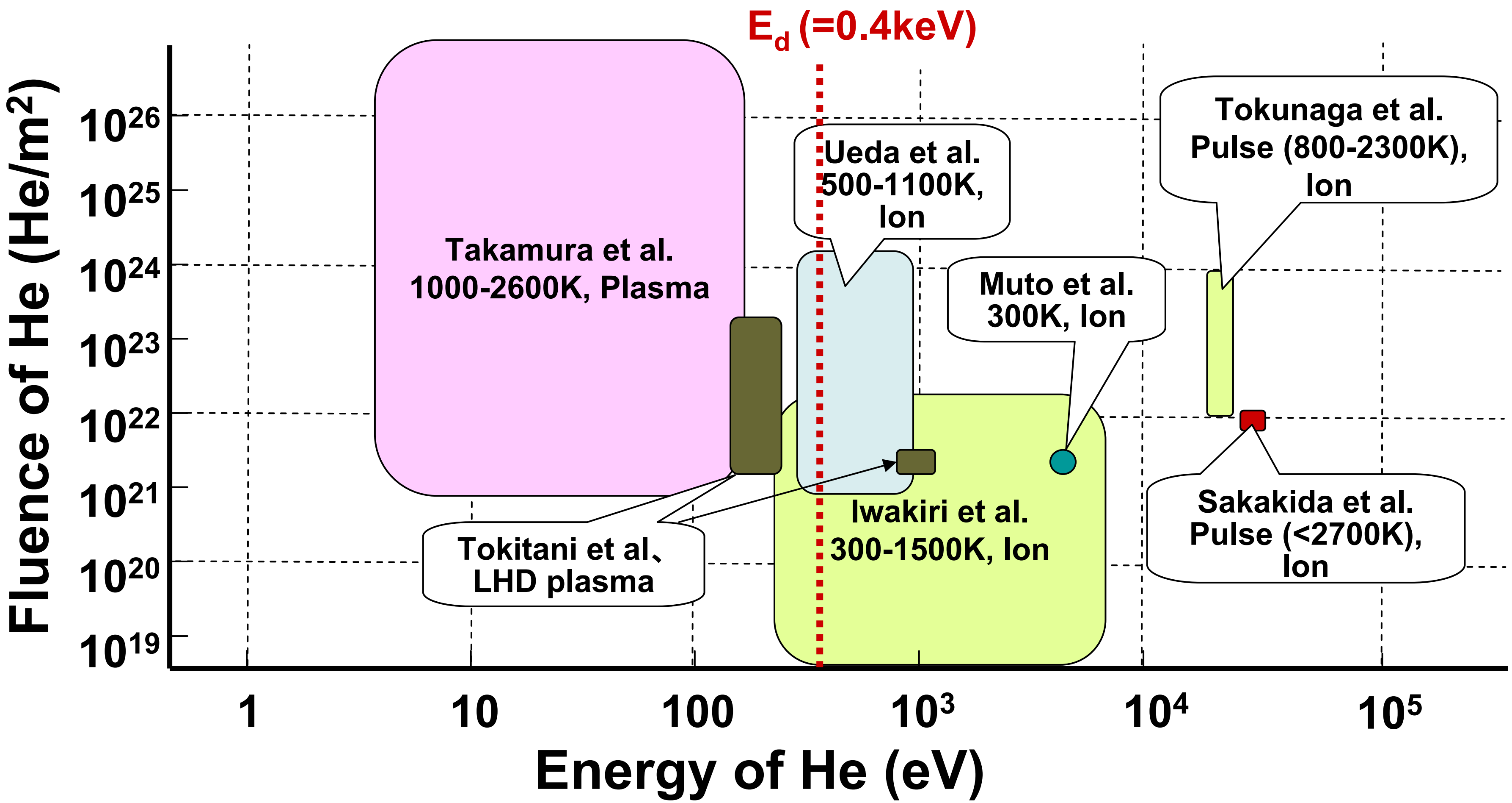
$$D_b = D_s (3\Omega^{4/3}) / (2\pi r^4)$$

Ω: atomic volume
*r*: bubble radius
$D_s$: coefficient of surface diffusion

# Studies on He Irr. Effects in Japan

**Materials**: **PM-W, VPS-W, UFG W-TiC, etc**

**Subjects**: **surface and internal damage, He retention, effects on H retention, thermal shock resistance, mechanical properties, optical reflectivity etc.**



# Summary (1/2)

- In case of He ion irradiation of W above 1273K (1000°C), which is divertor relevant temperatures, remarkable surface roughening occurs. At high dose, dense nano-size projections are formed. It leads to serious reduction of heat load resistance.
- The first step of the projection formation is the formation grooves at the surface due to the arrival of grown up He bubbles to the surface.
- With increasing He dose, grooves are getting deeper and deeper, and the tops elevate due to formation of He bubbles inside (swelling). This results in the formation of nano-size long projection at high dose.

# Summary (2/2)

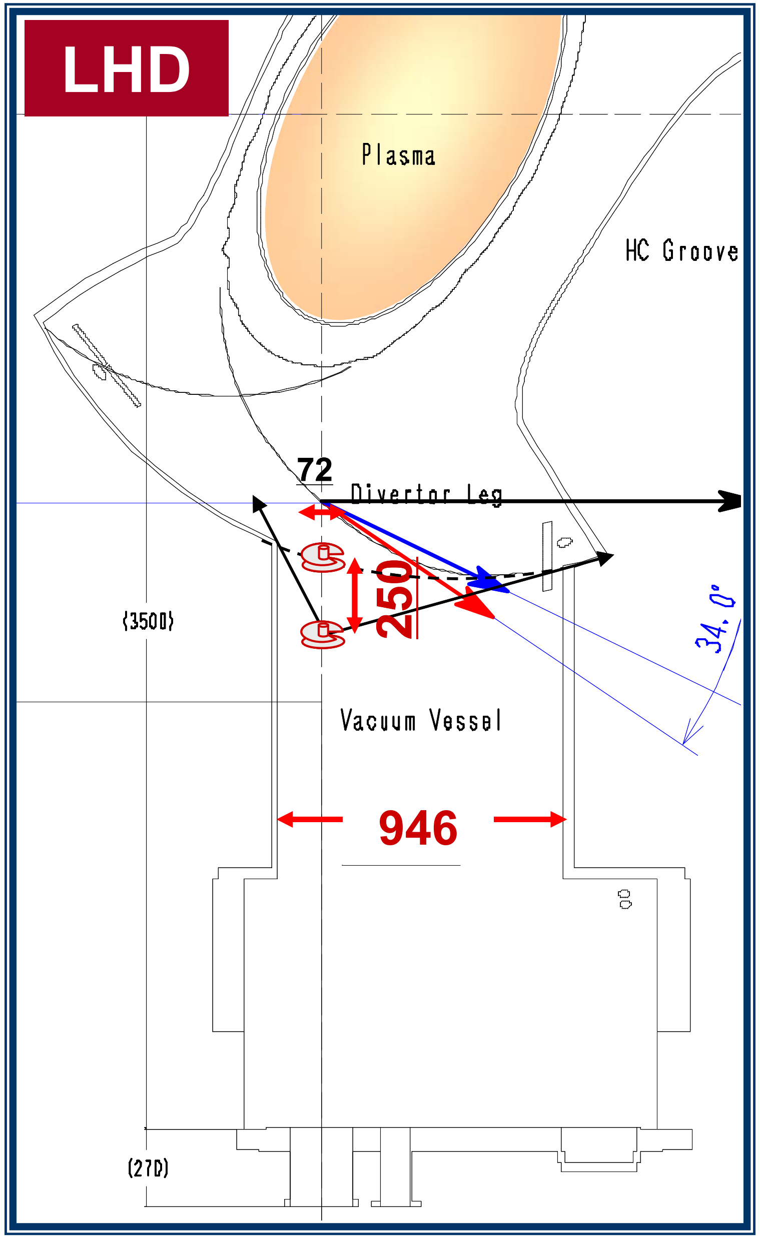
- For the formation of the projection, appropriate migration of bubbles and surface atoms are necessary. In case of W, these condition are satisfied above 1000°C.
- Upper limit of the temperature is strongly depend on the energy and flux of the helium ions.
- In case of keV range He ion irradiation, dislocations formed by the irradiation will shift the formation temperature range to the higher temperature side.

以下予備

# Concerning Damage in Ports of LHD

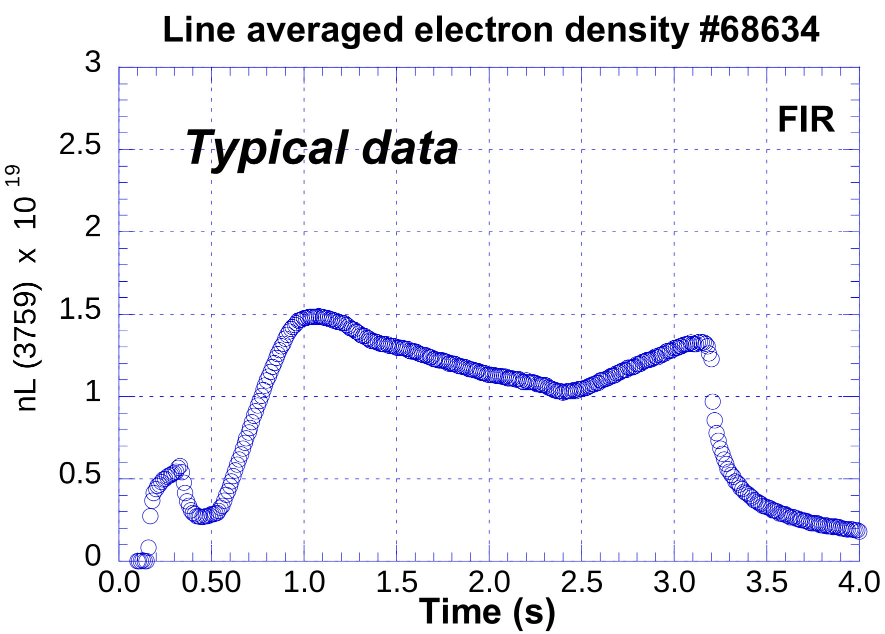
- In order to estimate the **damage in ports** by plasma particles, **Mo** and **SUS** were exposed to **He plasmas** heated by **ICRF** or **NBI** at the position of **0cm** (wall position), **-5cm** and **-25cm** by using **retractable material probe**.
- Internal damage and optical reflectivity were observed.

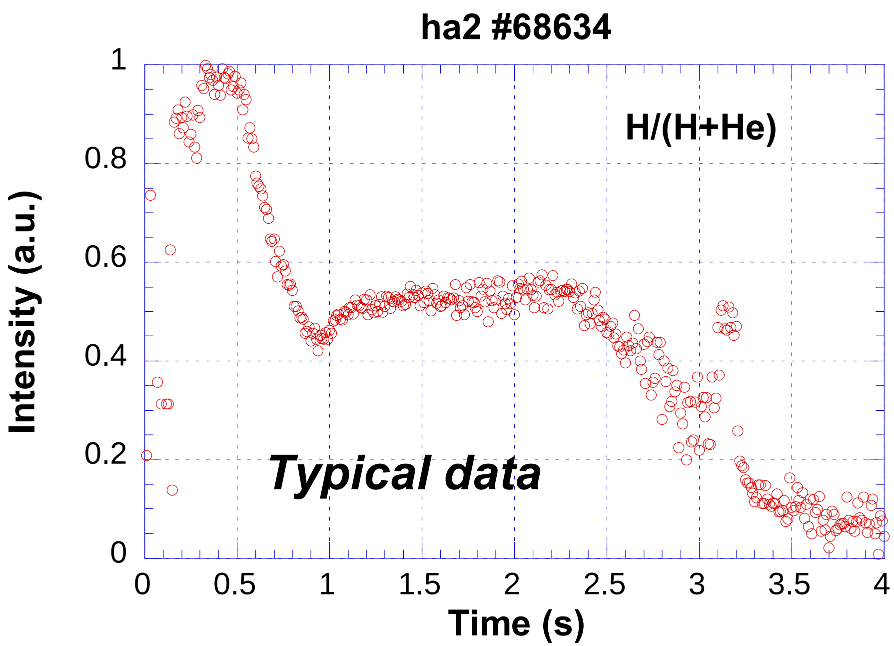
Sample holder of the material probe



# Discharge Conditions (ICRF)

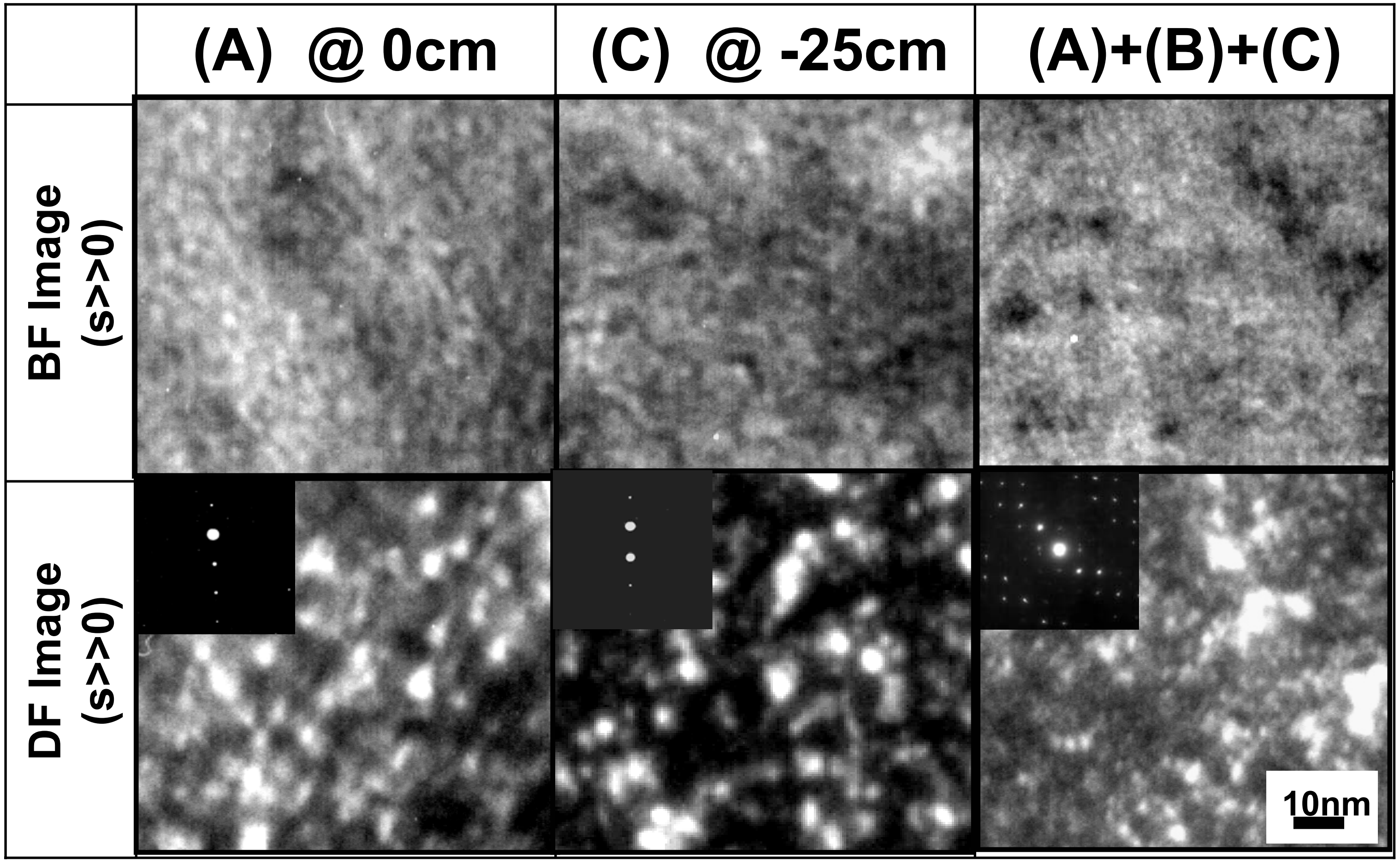
| | position | No. of shots | Discharge time (s) | Ion temp. (keV) | e density ($\times 10^{19} m^{-3}$) | Shot # |
|---|---|---|---|---|---|---|
| (A) | 0cm | 13 | 408 | 0.5～2.0 | 0.25～3.6 | *No.68705～68717* |
| (B) | -5cm | 81 | 349.1 | 0.2～2.0 | 0.25～2.25 | *No.68299～68379* |
| (C) | -25cm | 86 | 351.5 | 0.5～2.0 | 0.25～3.6 | *No.68618～68703* |
| (A)+(B)+(C) | | 185 | 1138.2 | 0.1～2.5 | 0.23～3.85 | *No.68299～68383, No.68618～68718* |



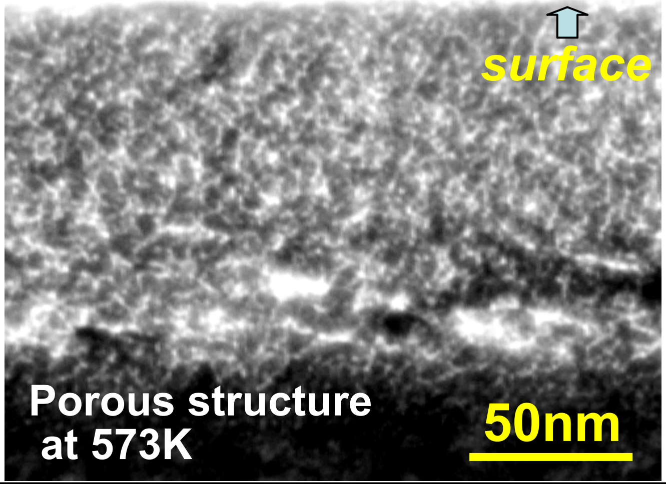
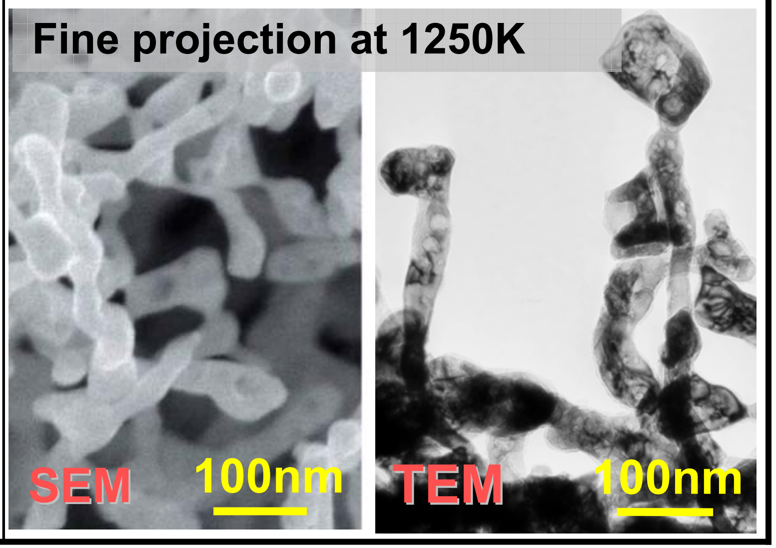



# Damage in Mo Exposed to ICRF-He Plasma

- **Even at the position of -25cm, serious damage (He bubbles, dislocation loops) occurred like wall position. (exposure time~350s, $T_i$~2keV, $n_L$~$2x10^{19}m^{-3}$). Similar damage was also observed in NBI case.**
- **This is the damage caused by CX-He with energy of about 1keV. The flux is estimated to be $10^{18}$~$10^{19}He^0/m^2s$.**



# Studies on He Irr. Effects on Optical Reflec.

| | 1st Wall Relevant Conditions | Divertor Relevant Conditions |
|---|---|---|
| Research G. | Yoshida Lab. (Kyushu U.) | Takamura Lab. (Nagoya Univ.) |
| Material | Mo | W |
| Irr. Temps. | R.Temp.~873K | 1250K~3000K |
| Ion Energy | 1.2keV, 8keV, 14keV | 10eV~100eV |
| Ion Fluence | ≦ $3x10^{22}He^{+}/m^{2}$ | ≦ $4x10^{27}He^{+}/m^{2}$ |
| Mechanism of Blacking | •Blistering<br>•Porous structure by nm-size He bubbles | •Fine projections (a few 10nmϕ) at 1250K<br>•Projections (a few 100nmϕ) and pin holes (~1μmϕ) above 1500K |
| Micro-structure | Cross sectional view   |   |

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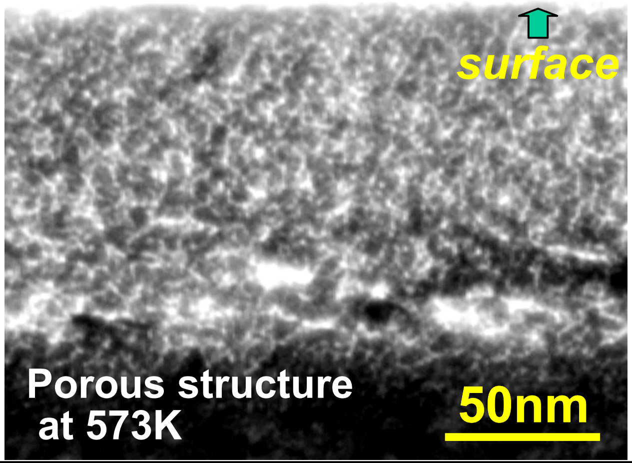
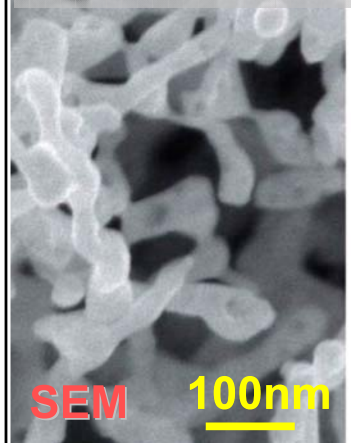
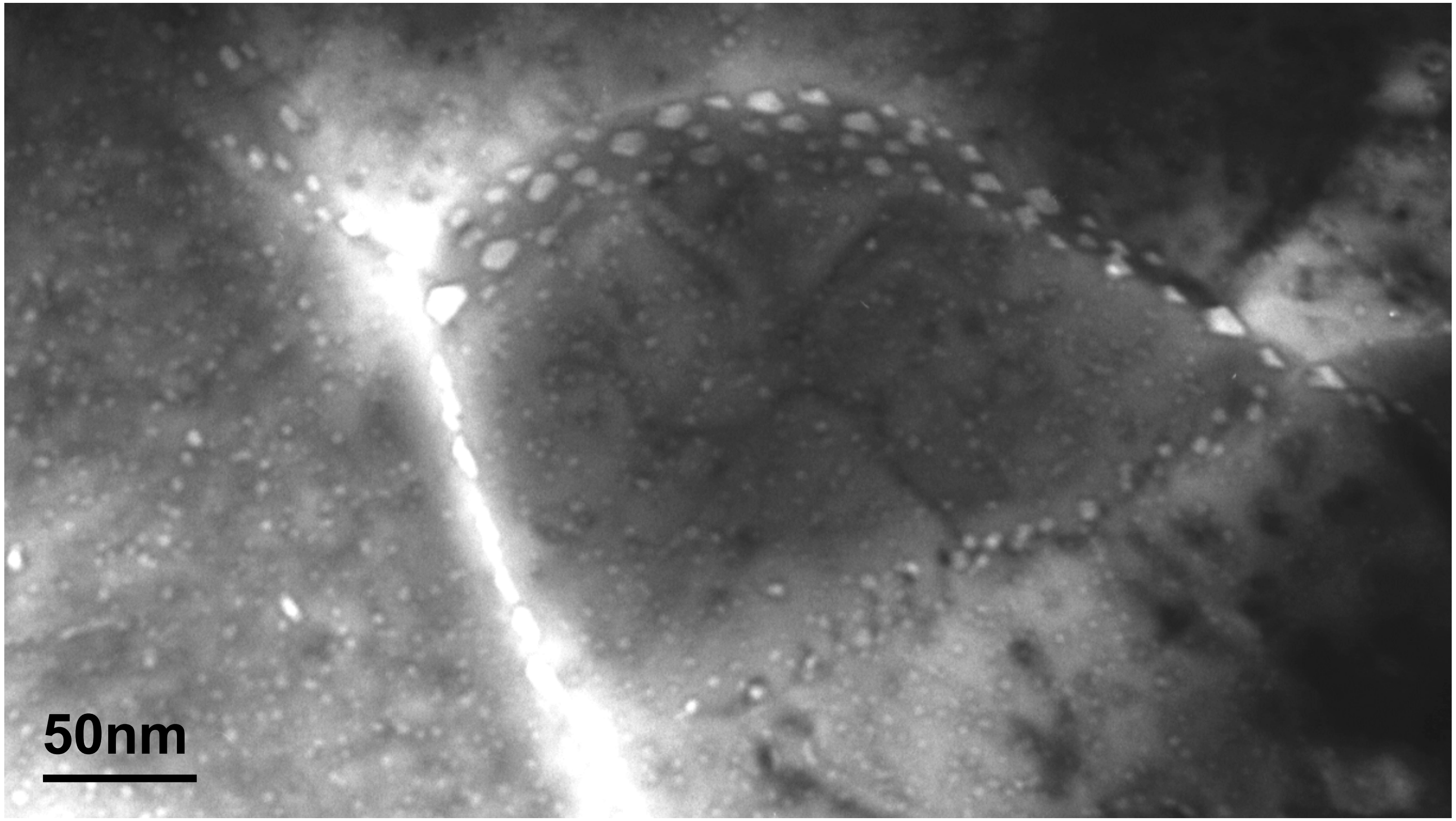
| Ion Energy | 1.2keV, 8keV, 14keV | 10eV~100eV |
| Ion Fluence | ≤ $3 \times 10^{22} He^{+}/m^{2}$ | ≤ $4 \times 10^{27} He^{+}/m^{2}$ |
| Mechanism of Blacking | • Blistering<br>• Porous structure by nm-size He bubbles | • Fine projections (a few 10nm$\phi$) at 1250K<br>• Projections (a few 100nm$\phi$) and pin holes (~1$\mu$m$\phi$) above 1500K |
| Micro-structure | Cross sectional view   | Fine projection at 1250K     |

**Behavior of He bubbles determines these variety of microstructures and resulting optical properties.**

# Preferential Growth of Bubbles at G.B.

Iwakiri et al.

**PM-W@1273K、8 keV-$He^+$、$5.4 \times 10^{19}$ $He^+/m^2$**

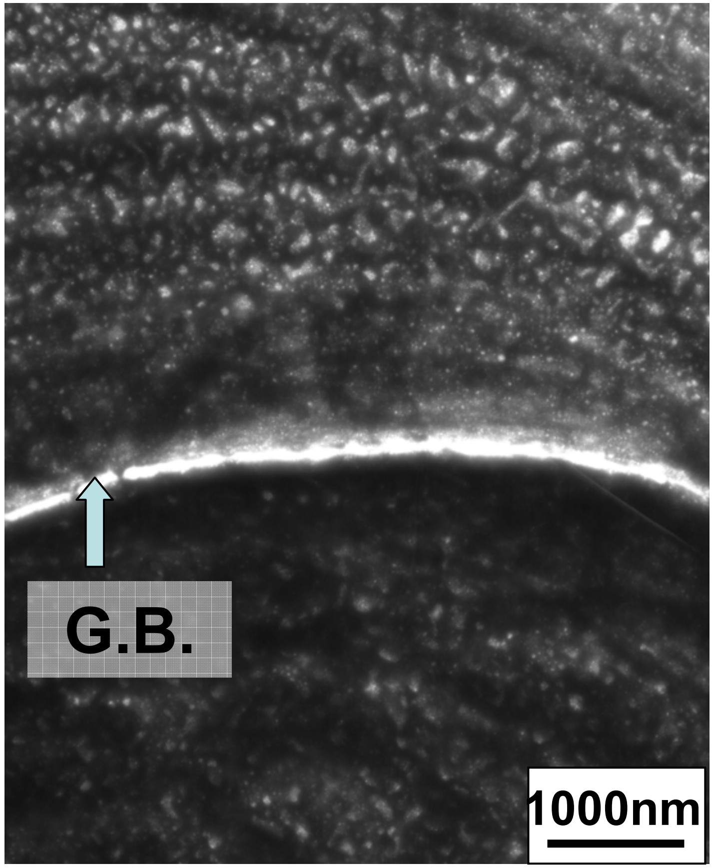


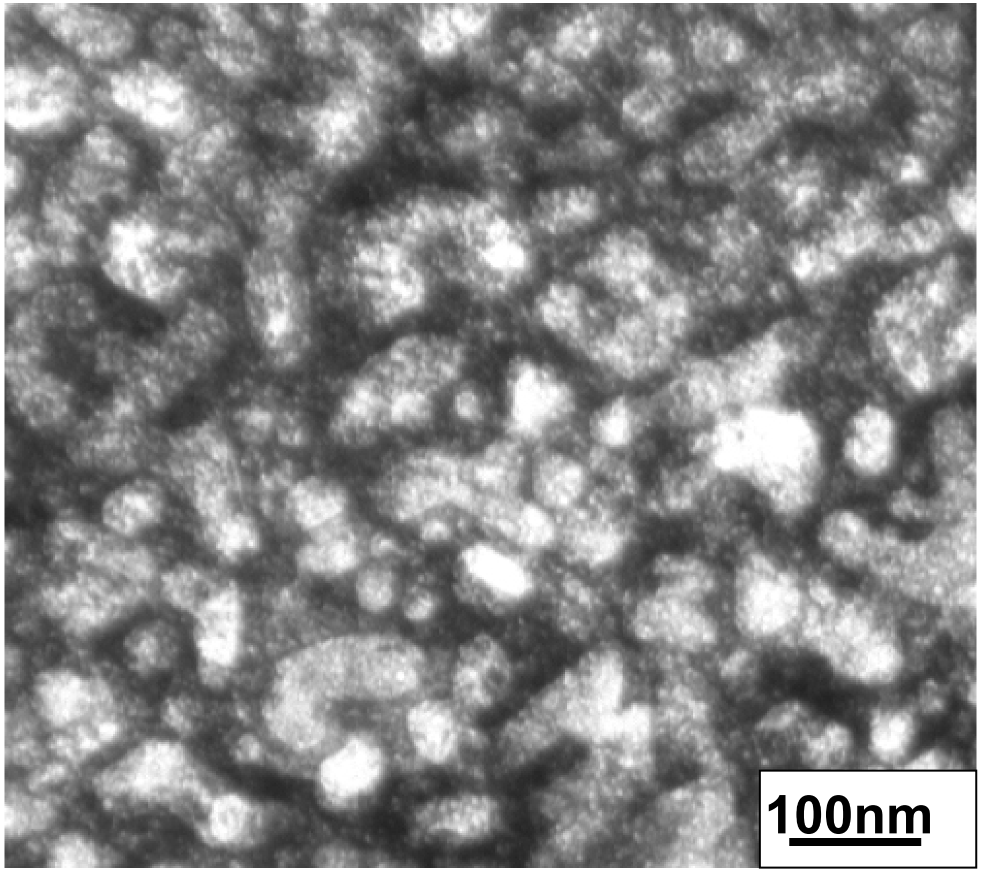

- **Aggregation of He atoms and vacancies at grain boundary ➜preferential formation of He bubbles at G.B.**
- **Aggregation of He bubbles ➜ deep groves along GB**

# Heavy Irr. with $He^+$ with sub-$E_d$ Energy

Iwakiri et al.

**PM-W@1273K, 0.25keV-$He^+$, $2.4x10^{21} He^+/m^2$**

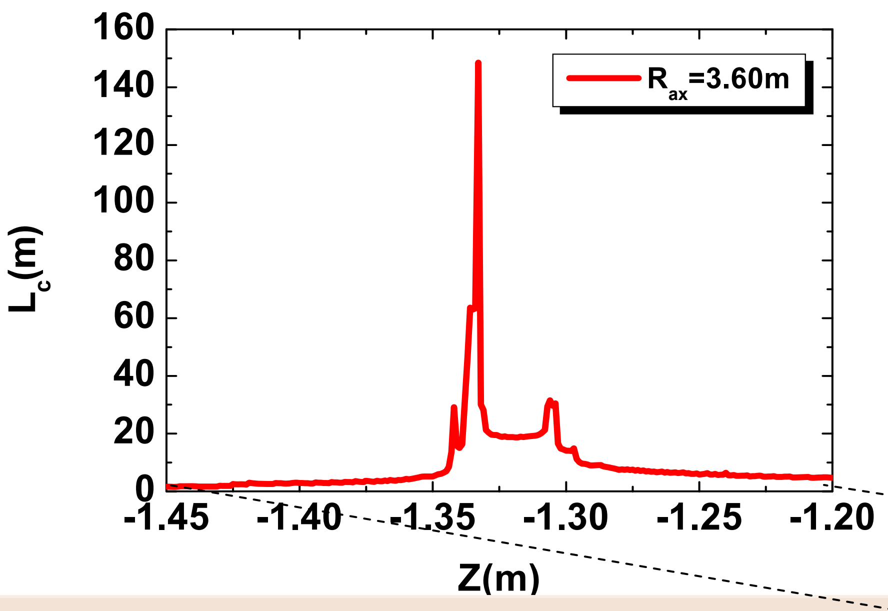


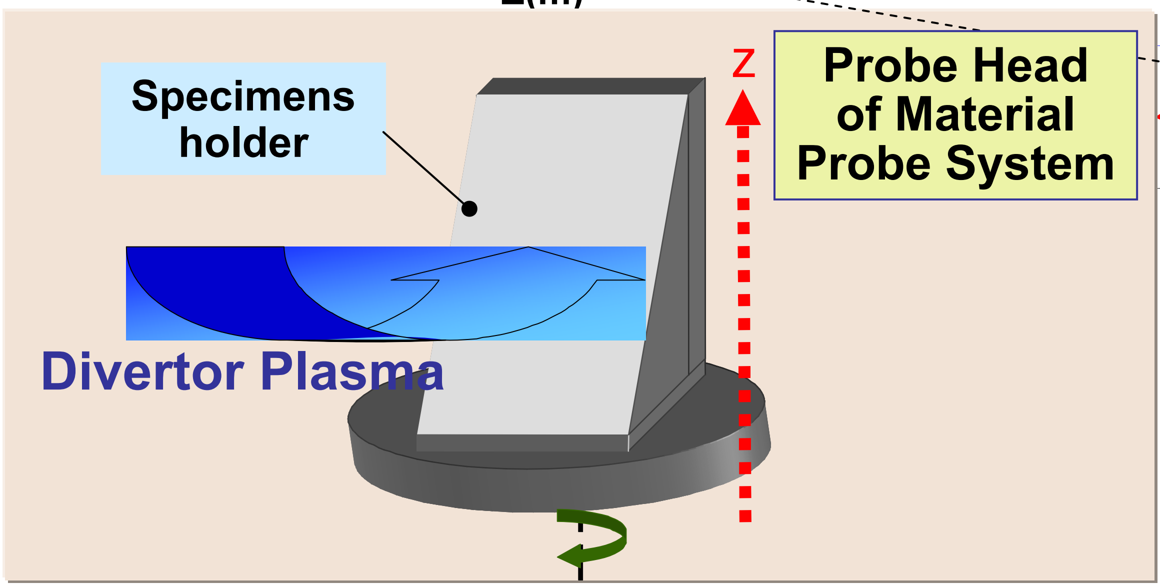



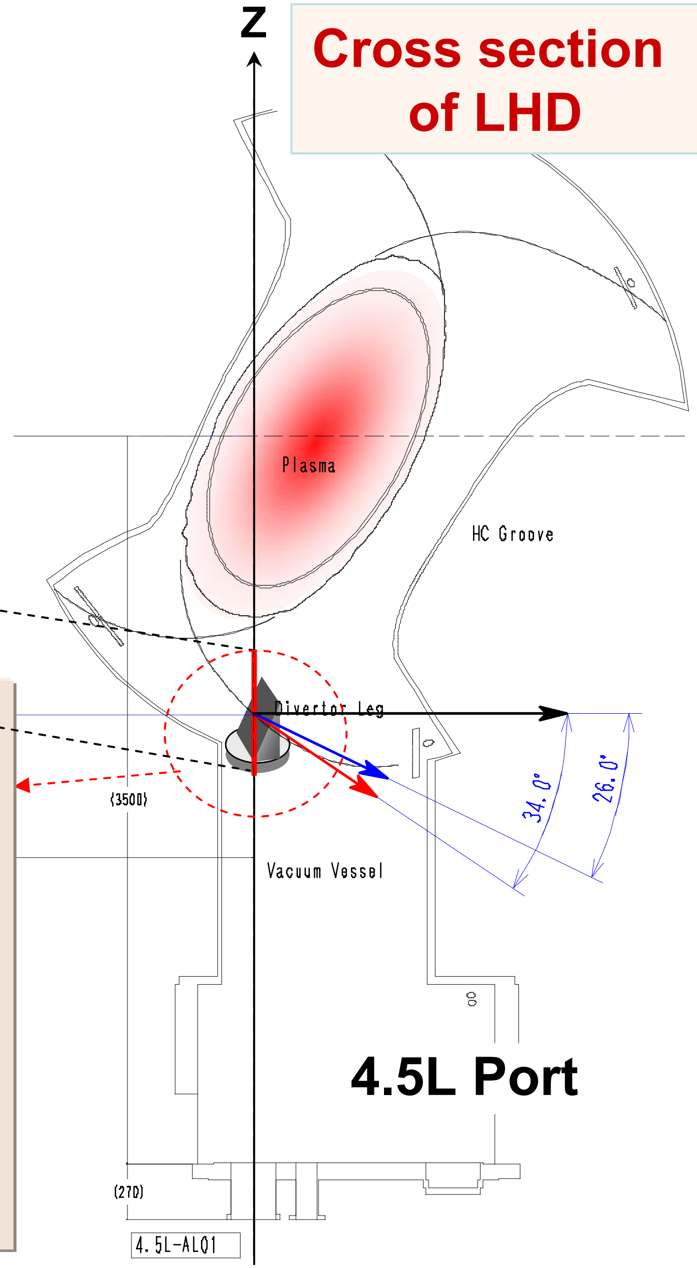

- **Deep groves along G.B. , Surface holes**
- **Spherical bubbles ➜ very high gas pressure ⇐ less supply of vacancies.**

# Plasma Exp. at Divertor Eq. Position

Connection length $L_c$ (Z-axis)



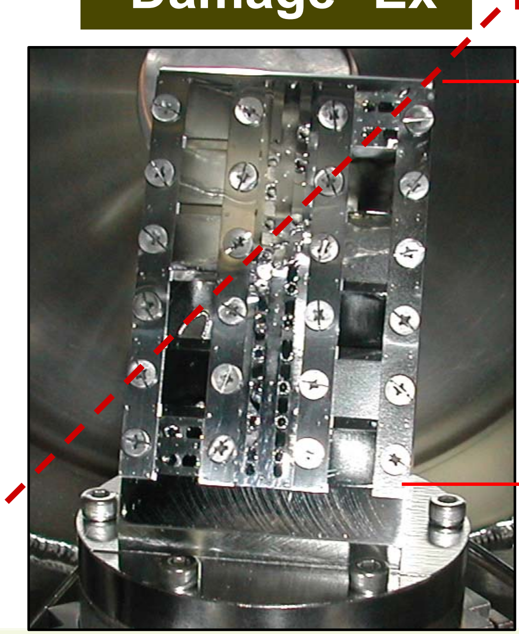
# Effects of He Plasma Exposure

## Irradiation Conditions (at divertor equivalent position)

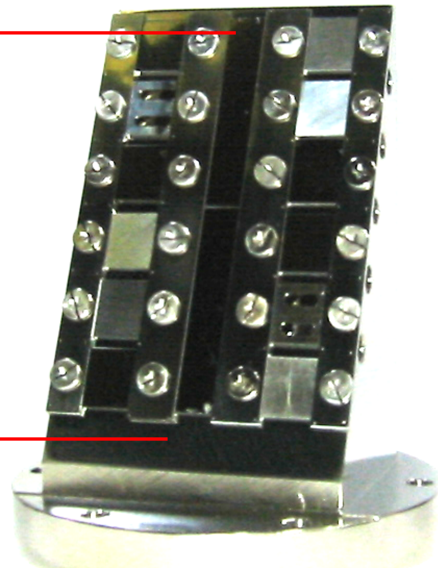
| | *Energy$_{(div)}$ (Maxwell Dis.)* | *Irr. Time* | *Heat & gas injection* | *Heating Power* | *$n_{e\ (core)}$* |
|---|---|---|---|---|---|
| **Damage** | **(20~50 eV)** | **1 s (1 shot)** | **NBI & gas-puff** | **1.6 MW** | **$3.0 \times 10^{19}\ m^{-3}$** |
| **He retention** | **(20~50 eV)** | **30 s (1 shot)** | **NBI & gas-puff** | **0.5 MW** | **$2.0 \times 10^{19}\ m^{-3}$** |

**Damage Ex**

**Foot print**



**He retention Ex**



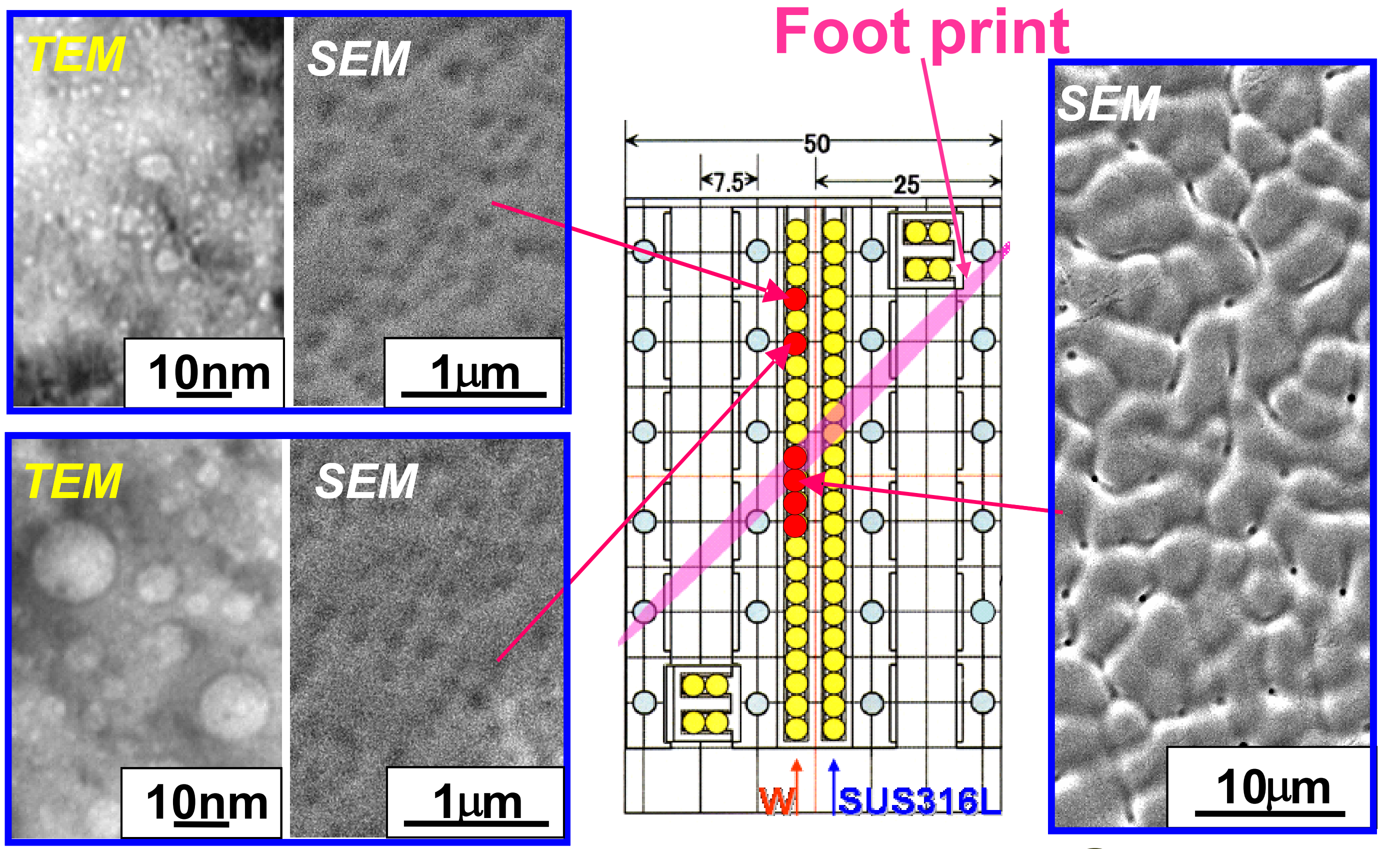
**70mm**

**0mm**
**Private side**

- **Surface: SEM and AFM, Internal damage: TEM**
- **Metallic Impurity: RBS, He: ERD**

# He Plasma Irradiation Damage of W at Divertor Position in LHD

Discharge time = 1 s



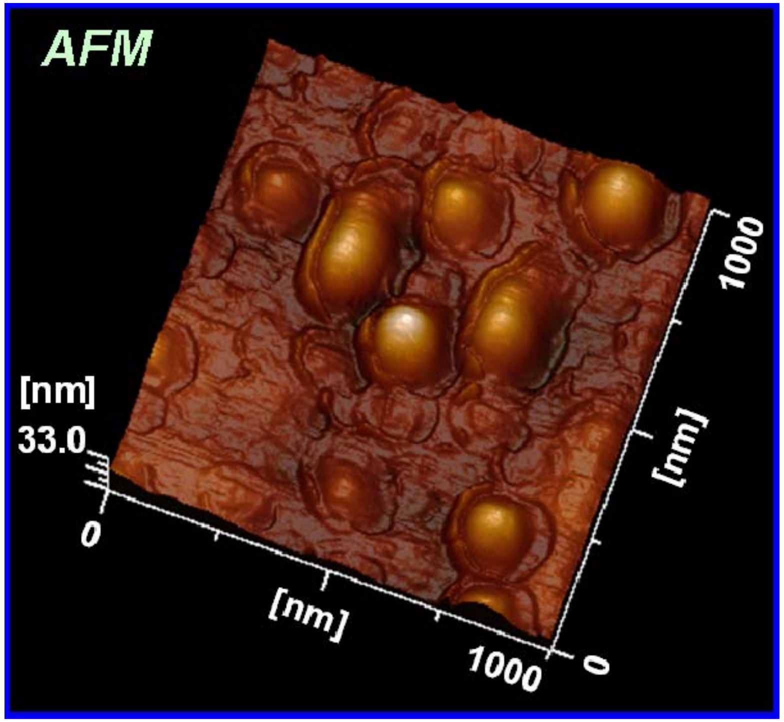

He bubbles    Blisters    Grooves, cracks

- These are damage at erosion dominant area.
- We should know the phenomena at deposition dominant area.

# Hydrogen Plasma Irradiation Damage of W at Divertor Position in LHD

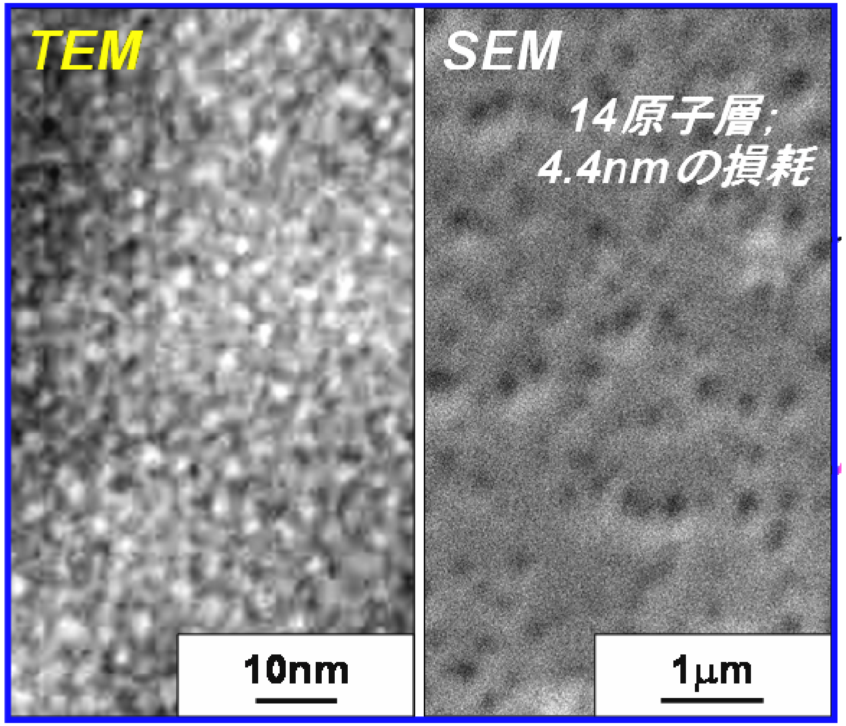
**30s Irra.（1shot）**

***Formation of blisters***




**Repeated irr.（26s, 19 shots）**

***Exfoliation of blisters, formation of bubbles inside.***

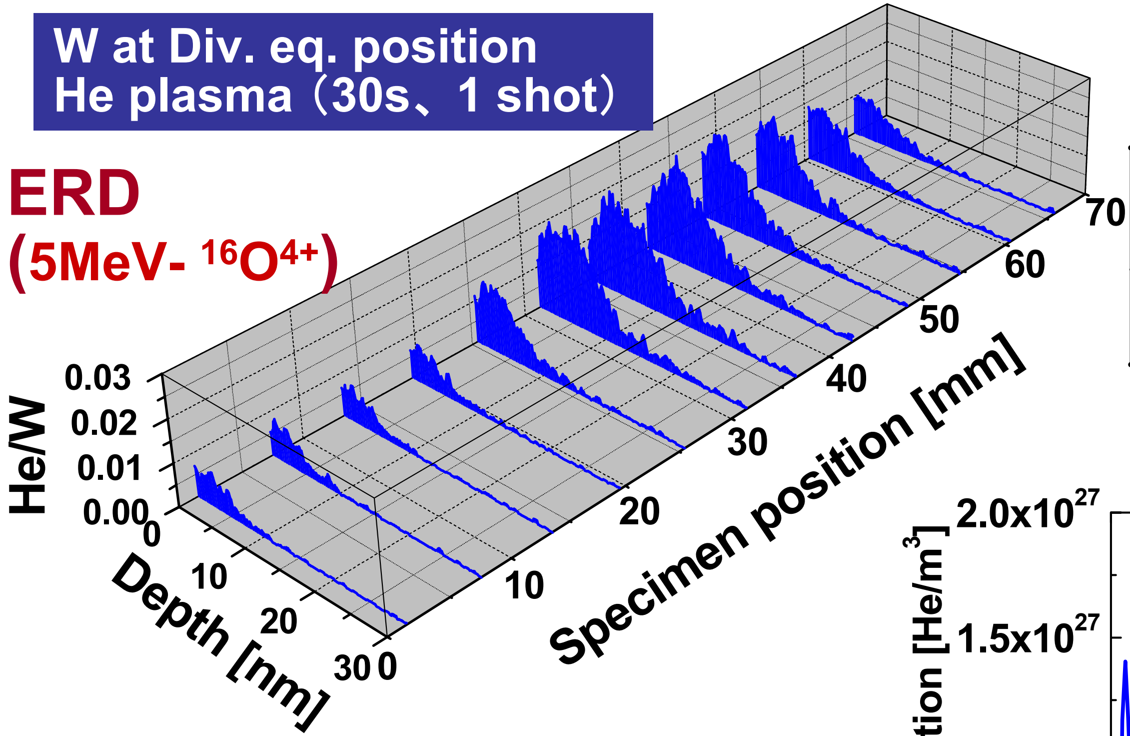



**Fine blister formation is new phenomenon**

**➔ Surface erosion, dust formation, retention of H**

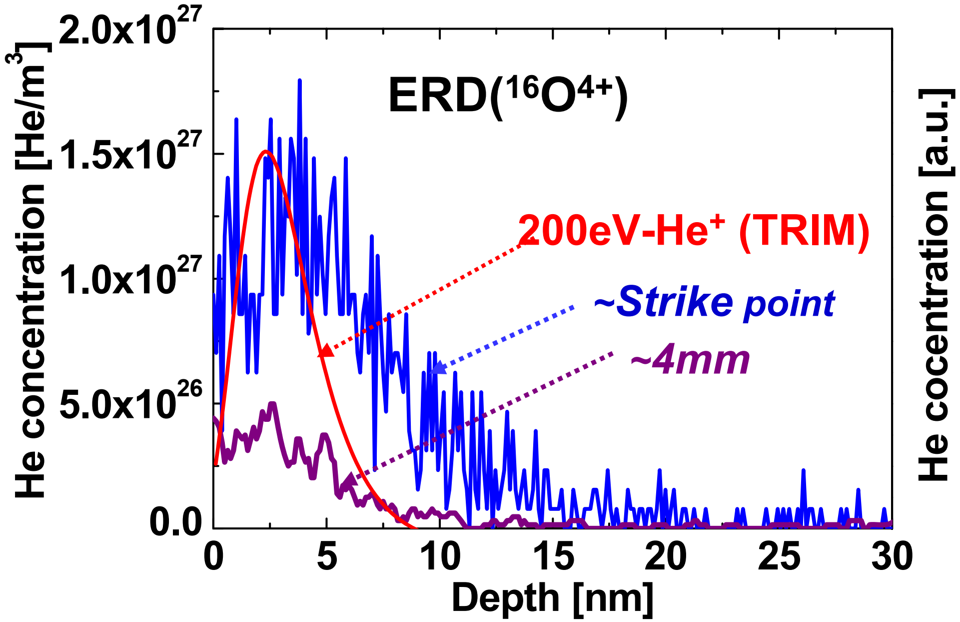
# Depth Distribution of He in W at Divertor Equivalent Position

W at Div. eq. position
He plasma（30s、1 shot）




**Parameters of core plasma**

| Electron density ($n_e$) | Ion temp. ($T_i$) | Input power |
|---|---|---|
| $1.3 \times 10^{19}$ m$^{-3}$ | 1.6 keV | 0.5 MW |



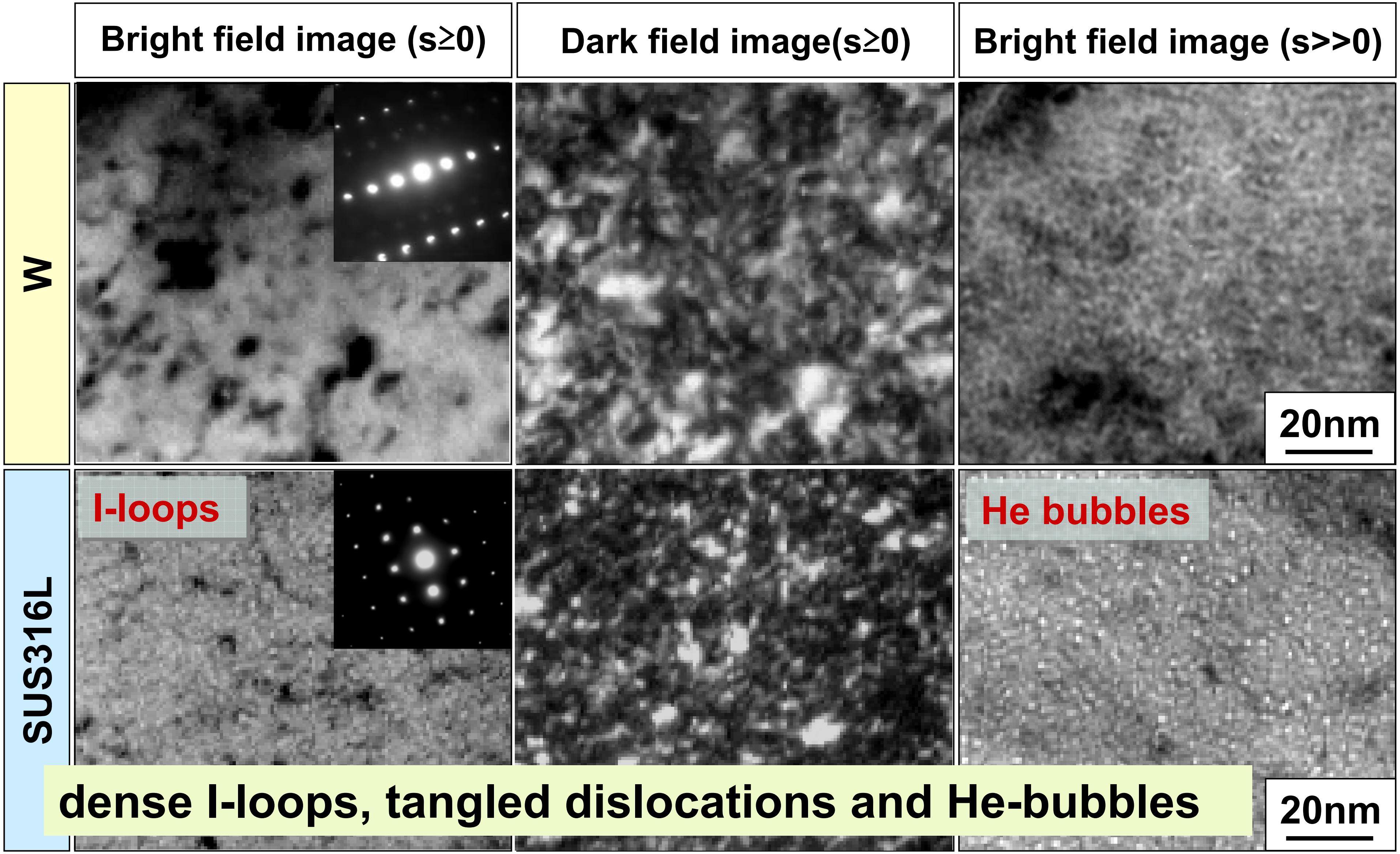

**Peak of He: ~3nm**

⇒ He ion energy ≥200eV
⇒ accelerated by sheath potential

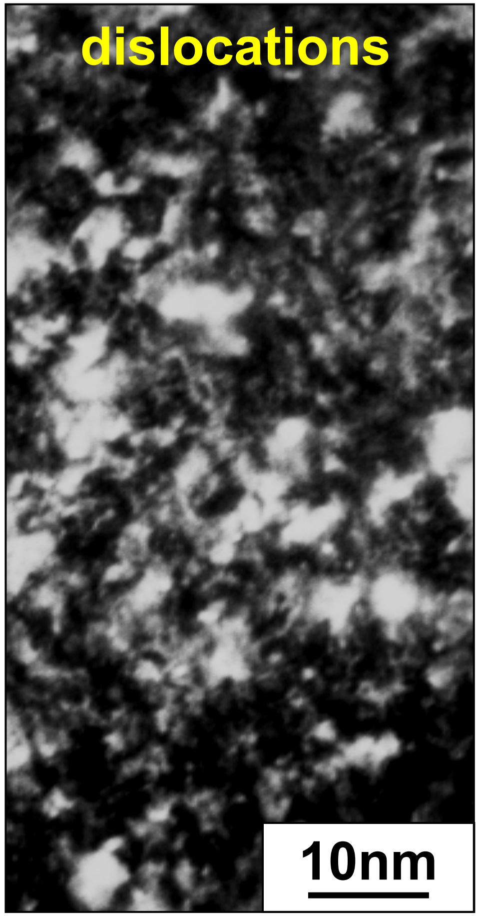
*($T_i$, $T_e$ at divertor region=20~50eV)*

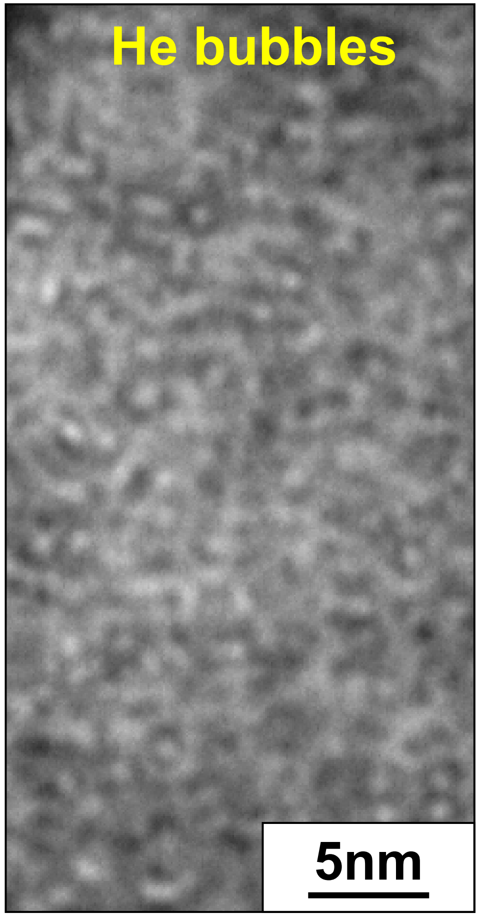
# Damage by He Plasma at the Wall Posi.

**Irradiation time = 87 s (total), Temp. ~ R. Temp**



# Characters of He Causing Wall Damage



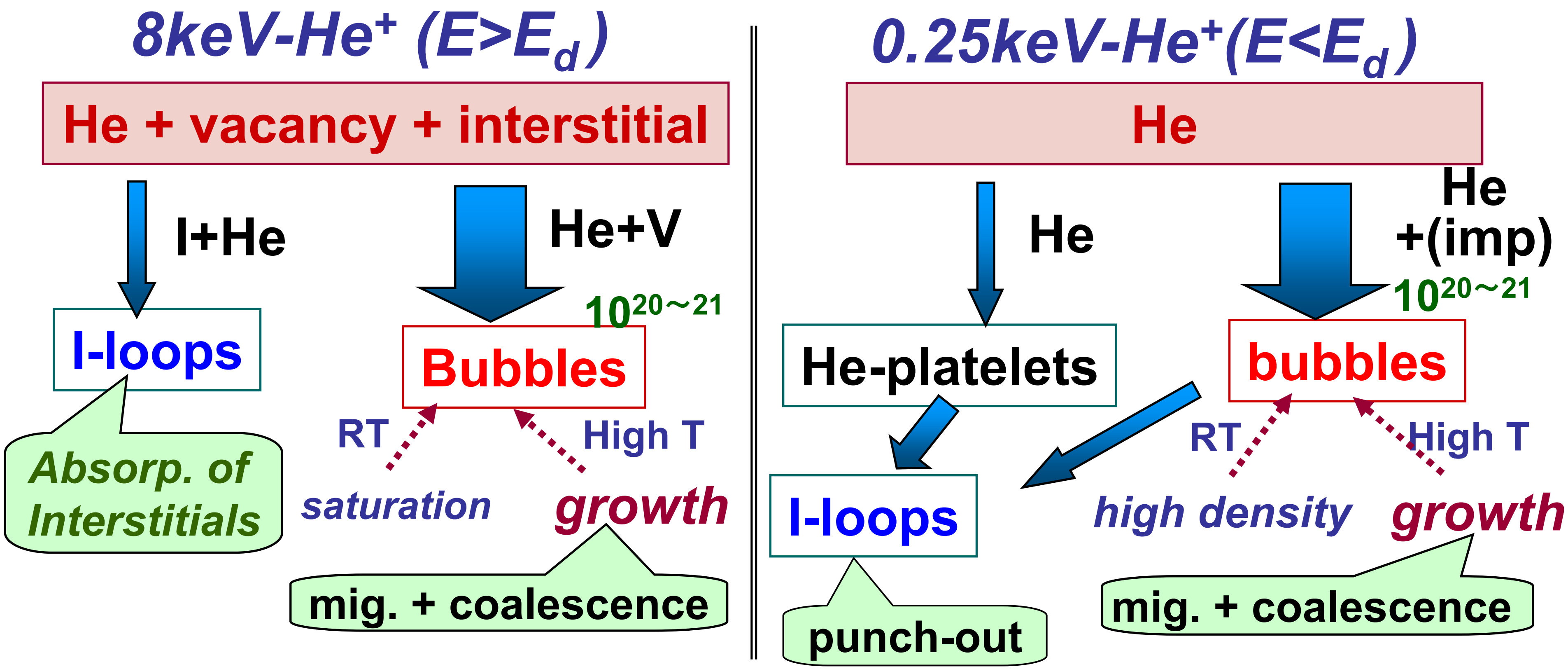

$T_i$: 0.37~1.71keV
$n_e$: $0.28 \times 10^{19}$~$8.7 \times 10^{19}/m^3$
Irr. Time: 87 s
Shot. No: 40048~40092, 40101~40119
Specimen: W

From the detailed He ion irradiation experiments (functions of ion energy, fluence, temperature) followings are concluded

## Characters of He Particles Causing Damage at the Wall Surface

- charge: neutral
- energy*: keV order
- Fluence: ~$10^{21}$He/$m^2$
- Flux: ~$10^{19}$He/$m^2$s

# Formation of Defects under $He^+$ Irra.

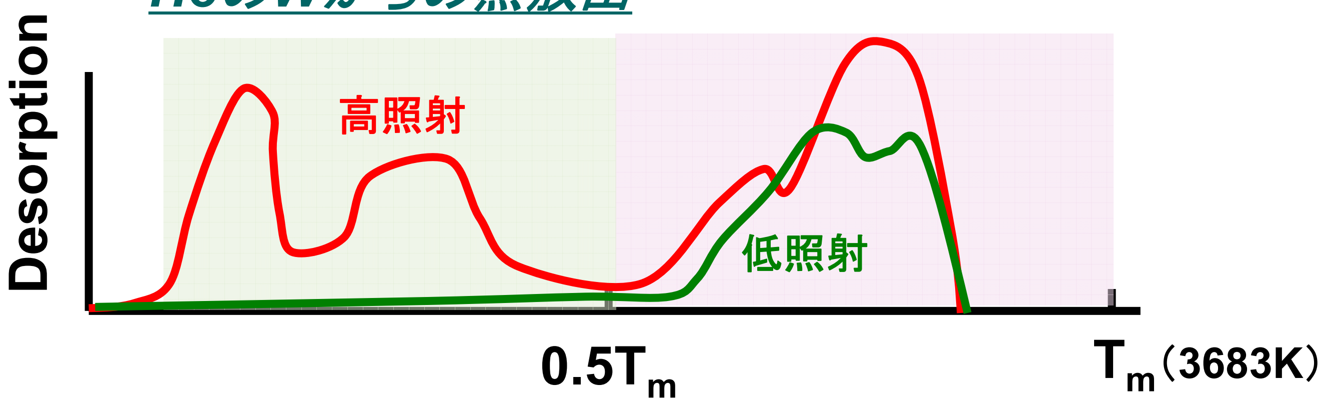



- Due to strong binding of He with vacancies and bubbles, He bubbles are formed even if the energy is very low (10eV range) and at very high temperatures (>2500K).
- At high temperatures (>$0.3T_m$), thermal vacancies may enhance the formation of bubbles.

# W中のヘリウムと格子欠陥の挙動

| 現　　　象 | 温度領域（定常状態） |
|---|---|
| 格子間原子の熱的移動 | > 20 K |
| He原子の熱的移動 | > 120 K |
| 注入Heの放出（高照射時） | > 300 K |
| 原子空孔の熱的移動 | > 700 K |
| He量の少ないHe-V 複合体の熱的移動 | > 700-800 K |
| $He_nV_1$ からのHeの解離 | 1000-1500 K |
| He量の多いHe-V複合体の熱的移動 | > 1500-1700 K |
| 再結晶 | > 1600 K |
| 大きな $He_nV_m$ 複合体（バブル）からのHeの解離 | > 2100 K |

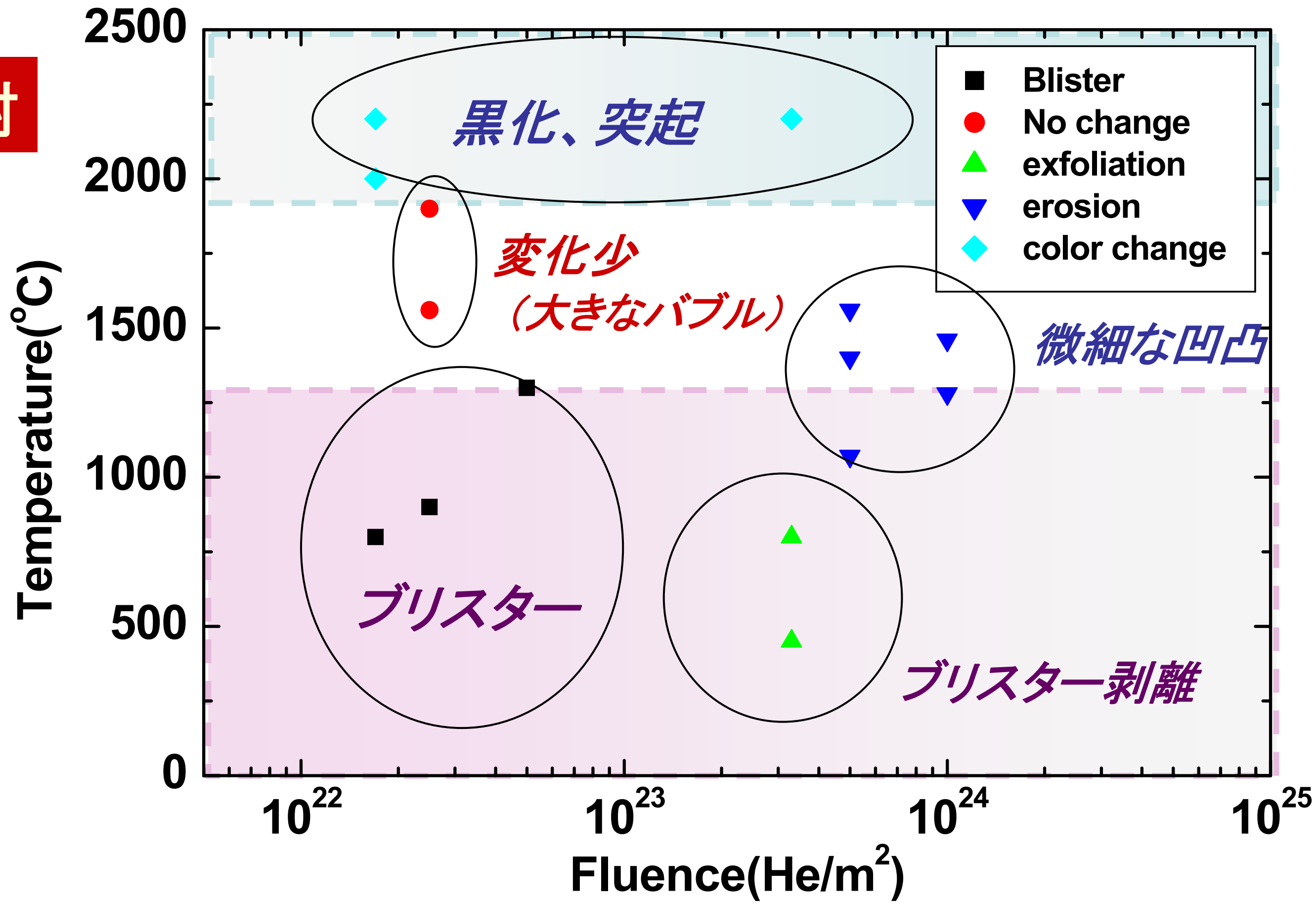
*HeのWからの熱放出*




$E_{mi}$= 0.054 eV
$E_{mHe}$= 0.3 eV
$E_{mv}$= 1.7 eV
$E_{fv}$= 3.6 eV

# 表面形状の照射量／最高到達温度依存性

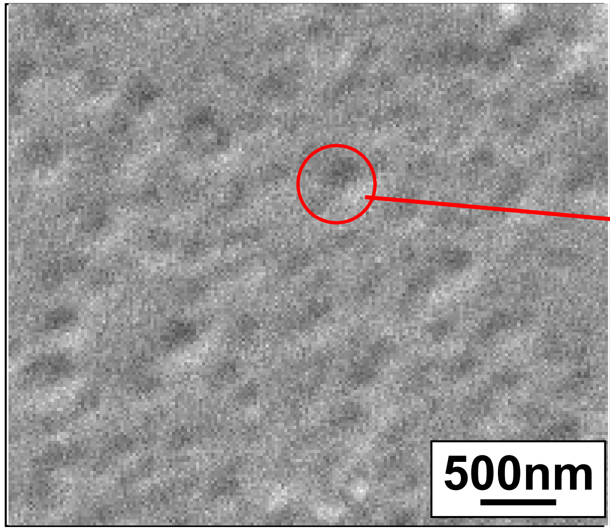
**パルス照射**



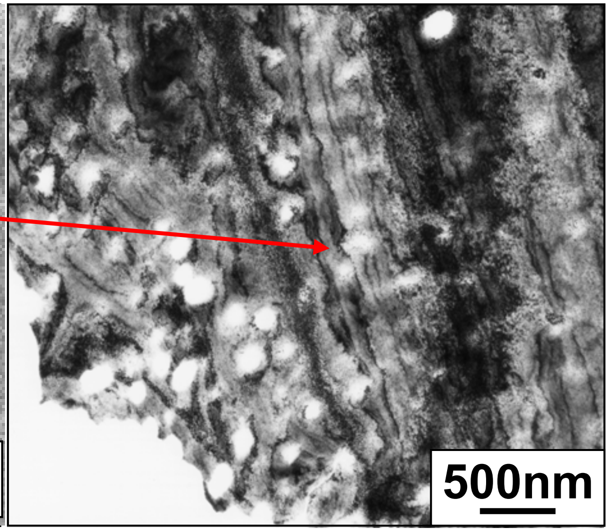

- Heのバブルへの極めて強いトラップ、バブルの熱的移動によって低温から極めて高温まで様々な表面形態が出現。
- ヘリウム照射特有の現象。

# Heプラズマに曝したW（LHD、ダイバータ･レグ）

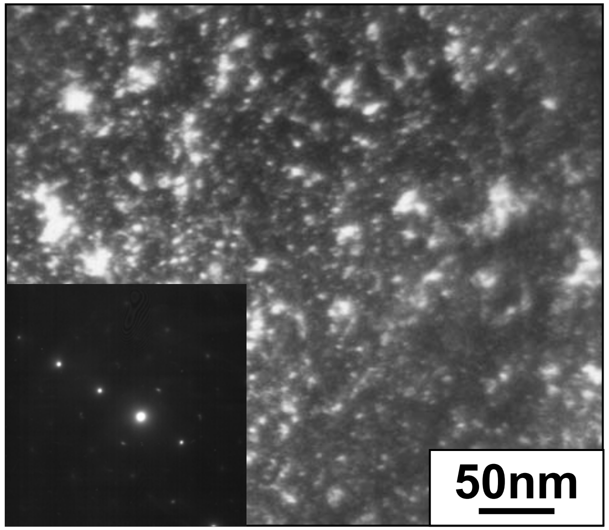
**表面 (SEM)**

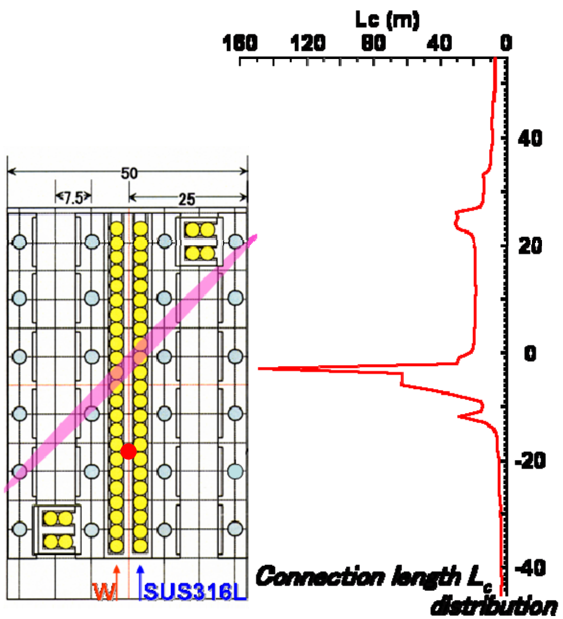



**明視野像（低倍TEM）**

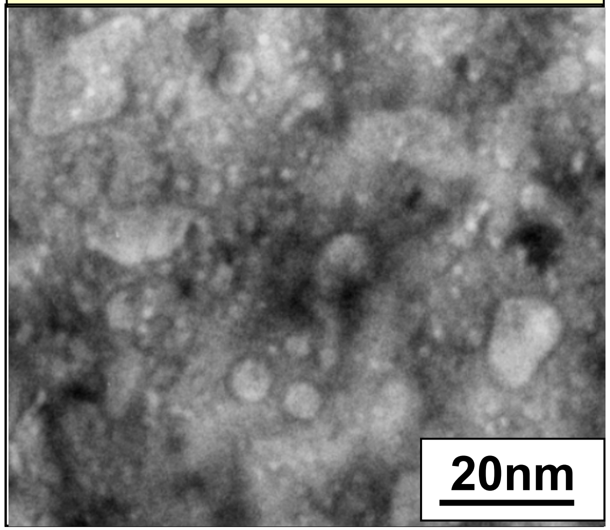



**暗視野像（低倍TEM）**




**明視野像（低倍TEM）**

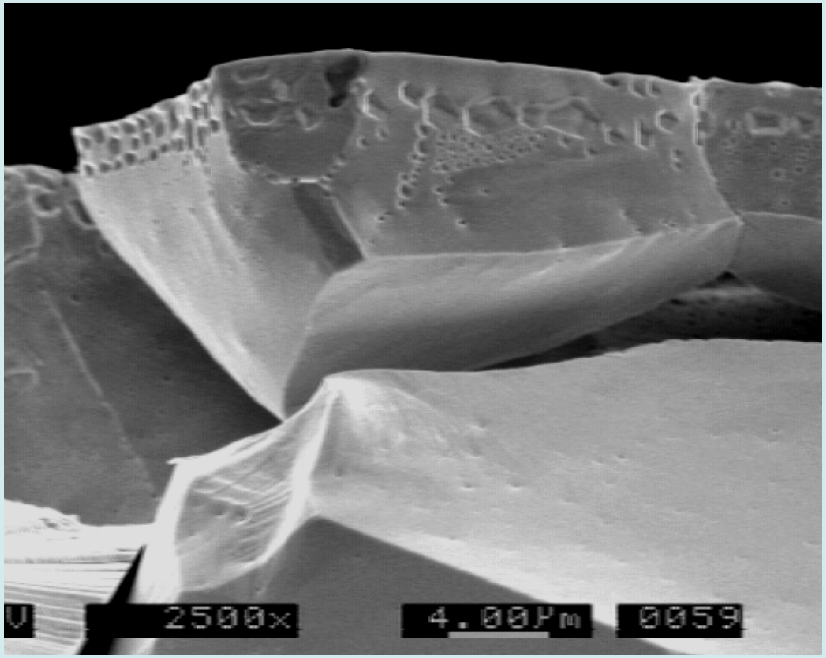



- 1秒間の照射にもかかわらずブリスタリングによる損耗が発生。
- 損耗量＝6.6nm/shot
- 瞬間的な温度上昇により形成。

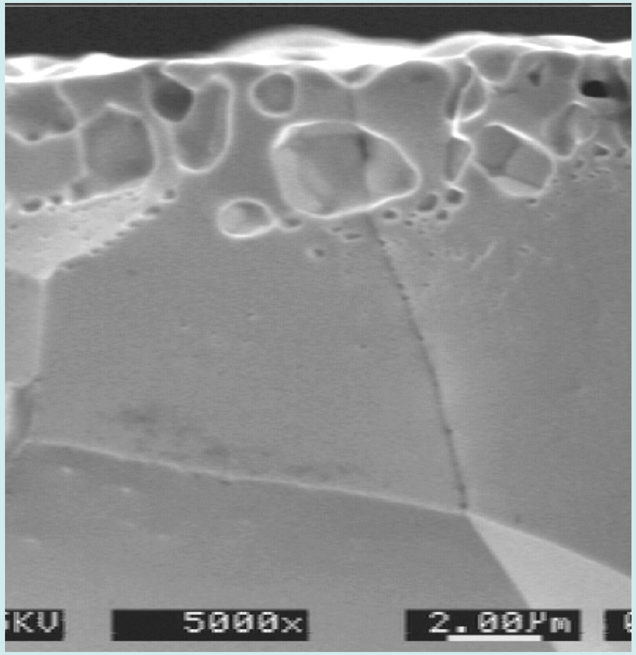
# Very Low Energy He Plasma Bombardment

- He form bubbles once they get in the martial due to high migration rate and strong binding with cavities (E > ~5eV).
- Migration of bubbles and supply of thermal vacancies may assist the growth of the bubbles.

10eV He plasma,
about $10^{27}He^{+}/m^{2}$, 2600K




32eV He plasma,
about $10^{27}He^{+}/m^{2}$, 2300K

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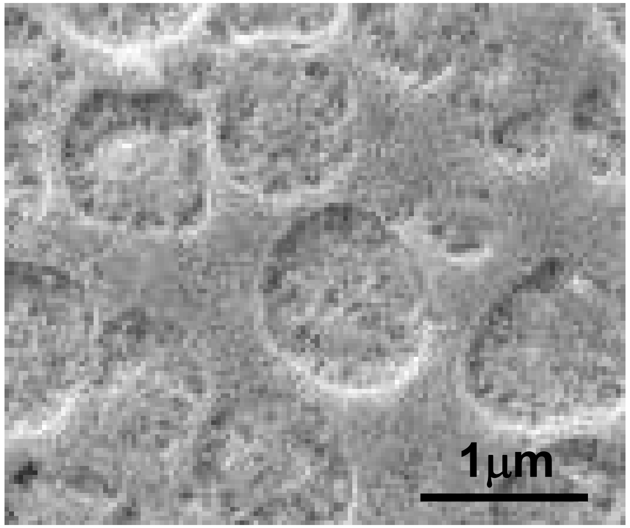
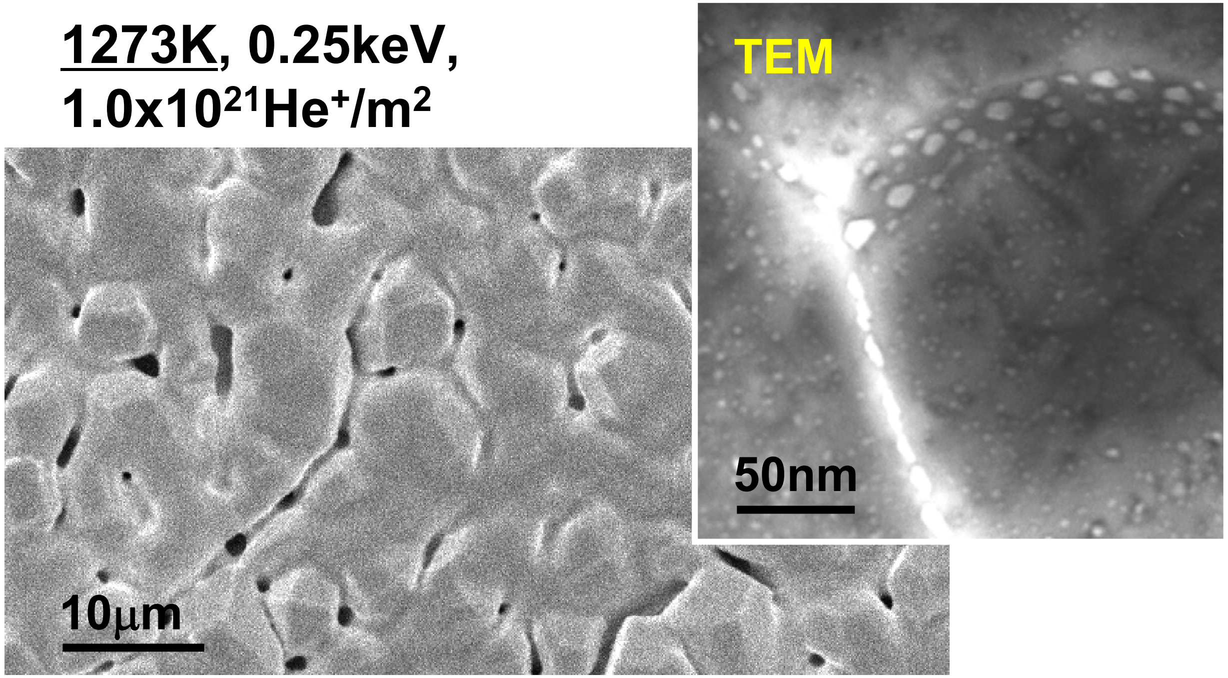
# Irr. Temp. Depend. of Damage at High Dose

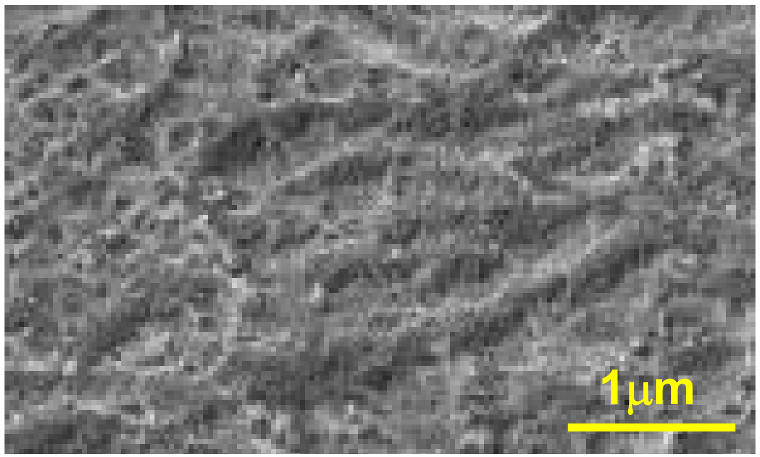
1073K, 14keV, $3.3\times10^{23} He^+/m^2$



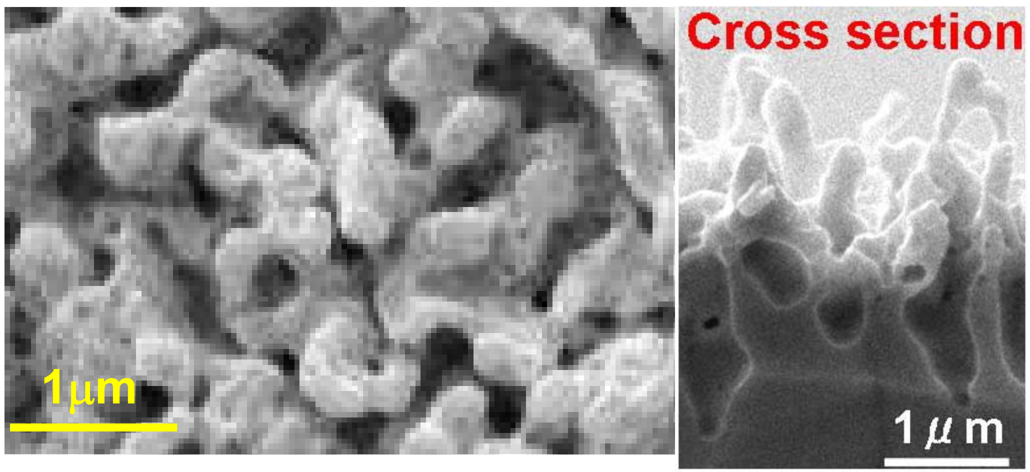

1273K, 0.25keV, $1.0\times10^{21} He^+/m^2$



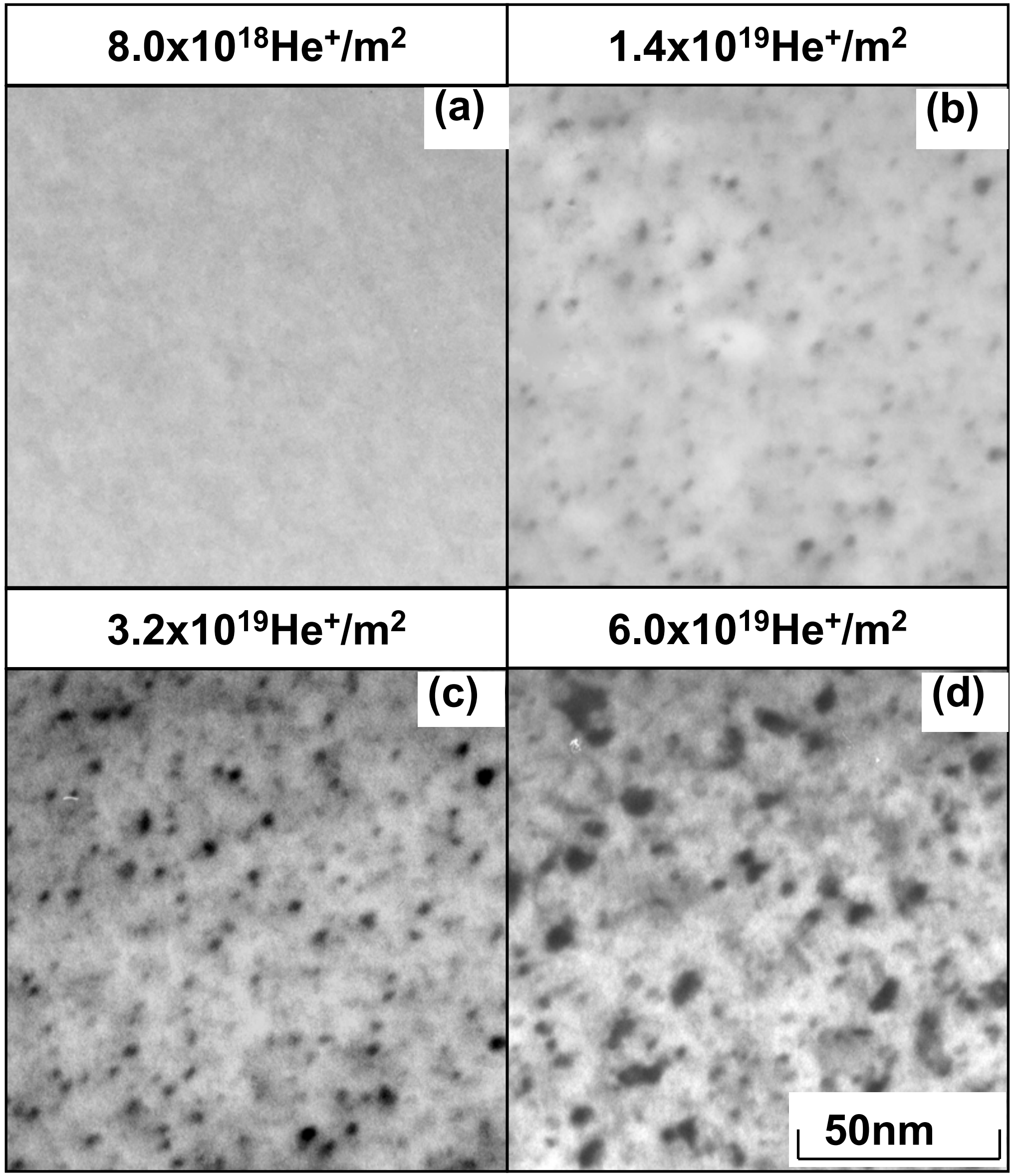

1673K, 14keV, $5.0\times10^{23} He^+/m^2$




2873K, 14keV, $3.3\times10^{23} He^+/m^2$



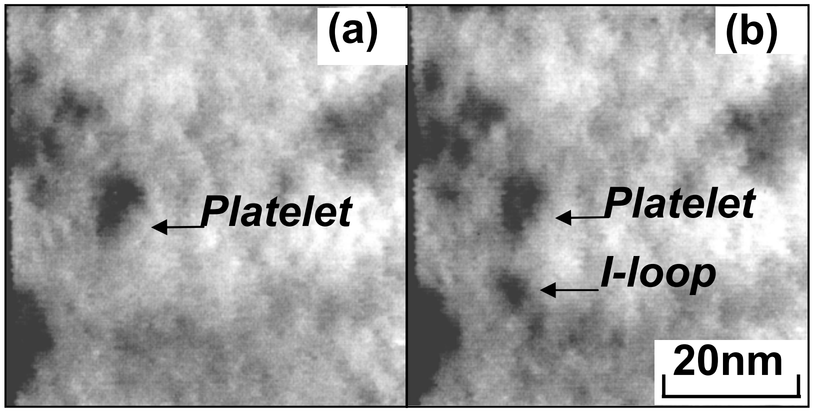
# sub-$E_d$ Heイオンによる損傷（低照射域）




**W、0.25keV He$^+$ イオン、室温照射**

(原子はじき出し損傷を起こすための閾イオンエネルギー($E_d$= 0.4keV )以下の照射)

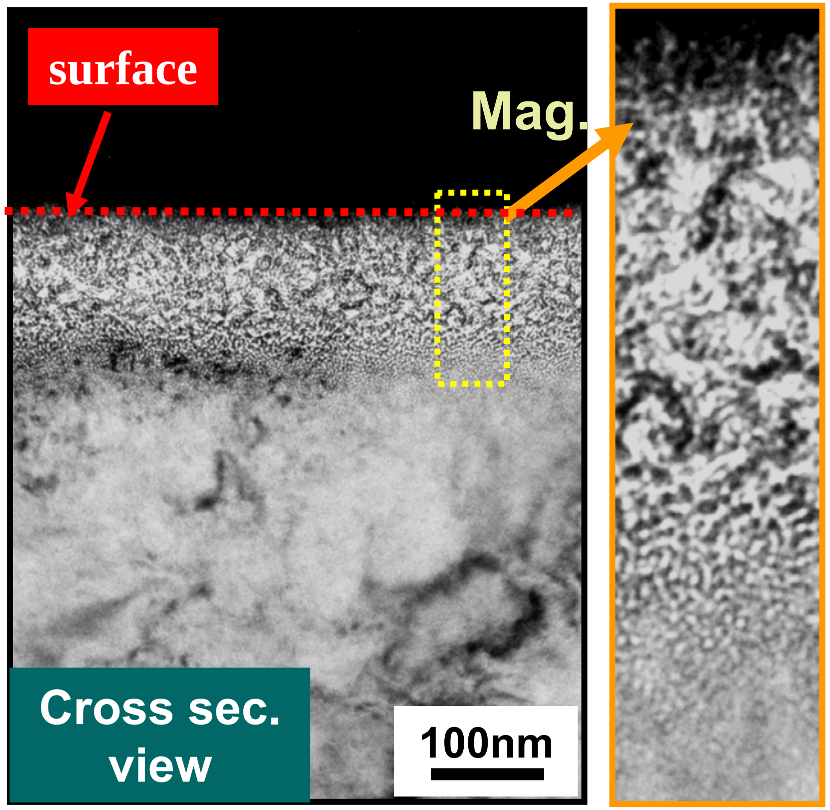
- まず、板状He集合体が作られ、パンチアウトによる I-型転位ループの形成



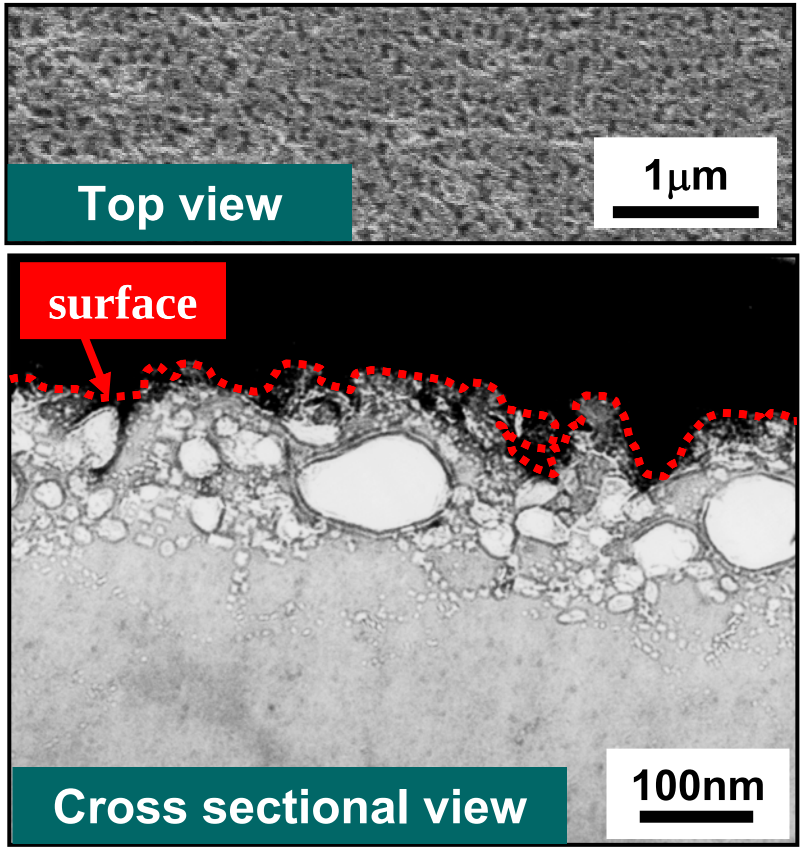
# Sub-Surface Structure in Fe-9Cr Alloy

**8keV $He^{+}$ ⇛Fe-9Cr Alloy**

**300K、$3.0 \times 10^{22}$ $He^{+}/m^{2}$**




**873K (=$0.5T_m$), $1 \times 10^{22} He^{+}/m^{2}$**




- **Sponge-structure, deep distribution of bubbles along grain boundaries and dislocations**
- **⇛reduction of thermal conductivity**