2003 US-Japan Workshop on
High Heat Flux Components and Plasma Surface Interactions for Next Fusion Devices
& High Power Density Devices and Designs
Manresa Hotel, Port Townsend, Washington, USA, July 28-31, 2003

Workshop Agenda

Irradiation Effects of Metals by He Plasma Bombardment in LHD

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Collaboration

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Background and Objective

- In the case of LHD, He glow discharge is mainly used for the cleaning of the wall surface due to the limitation of wall heating.
- However, leakage of He retained in the wall materials due to the He-GDC often disturb plasma discharge experiments. Control of He retention is very important for good performance of plasma.

Present study:

- **Microscopic damage of metal exposed to the He-GDC was examined by TEM, in order to understand the mechanism of the retention and desorption of He and other gas.**
  (This is very useful not only for understanding of GDC but also for understanding of damage in fusion environment where He particles are produced.)
- **Low energy He ion irradiation experiments were also carried out to understand damage at elevated temperature around 1000ºC.**

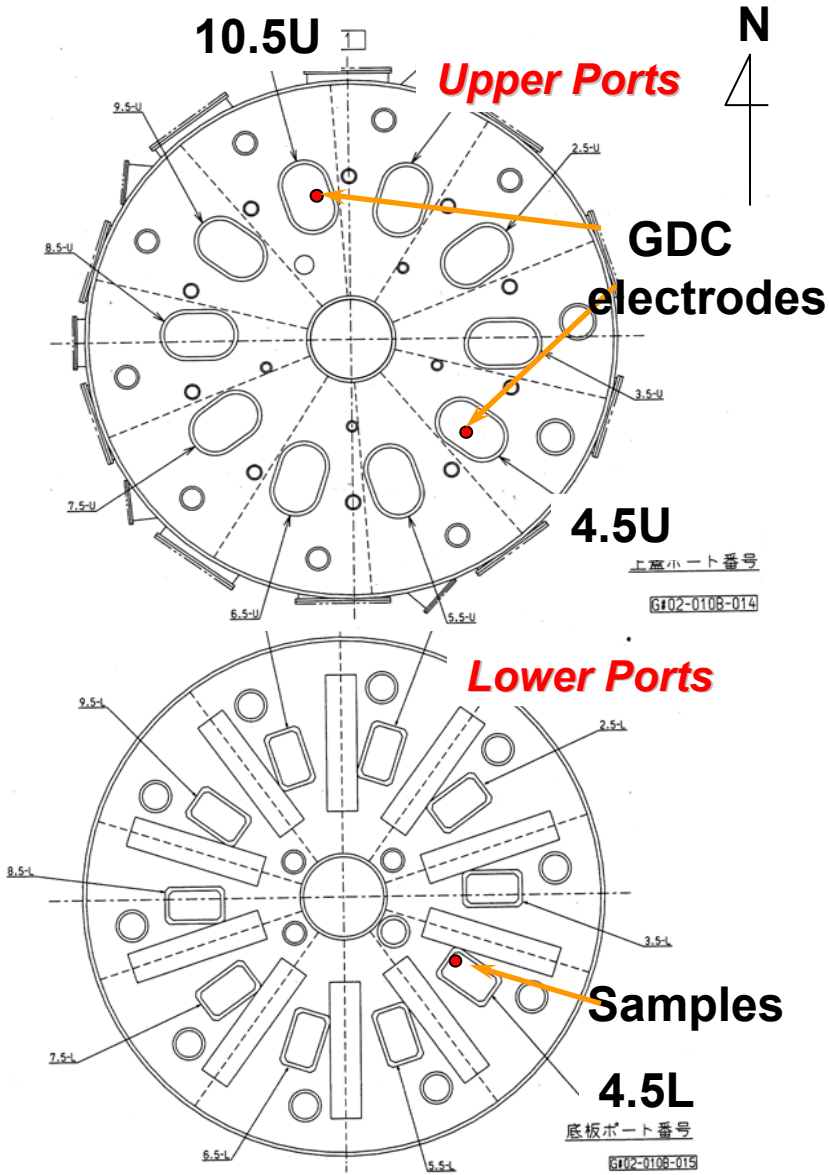
Outline of Glow Discharge Cleaning

- **LHD**
  - Major radius： 3.9m
  - Total area of VV： 780m^2
  - Wall material： SUS316L

- **Conditions of GDC**
  - He：200V, 20A(10Ax2), 65h
    3.7x10^{22} [He/m^2] (average)
  - H：300V, 20A(10Ax2), 71.5h
    4.1x10^{22} [H/m^2] (average)
  - Ne：200V, 20A(10Ax2), 55h
    3.2x10^{22} [Ne/m^2] (average)

- actual fluence > average value
- specimen temp. ~ room temp.

Position of electrodes and samples

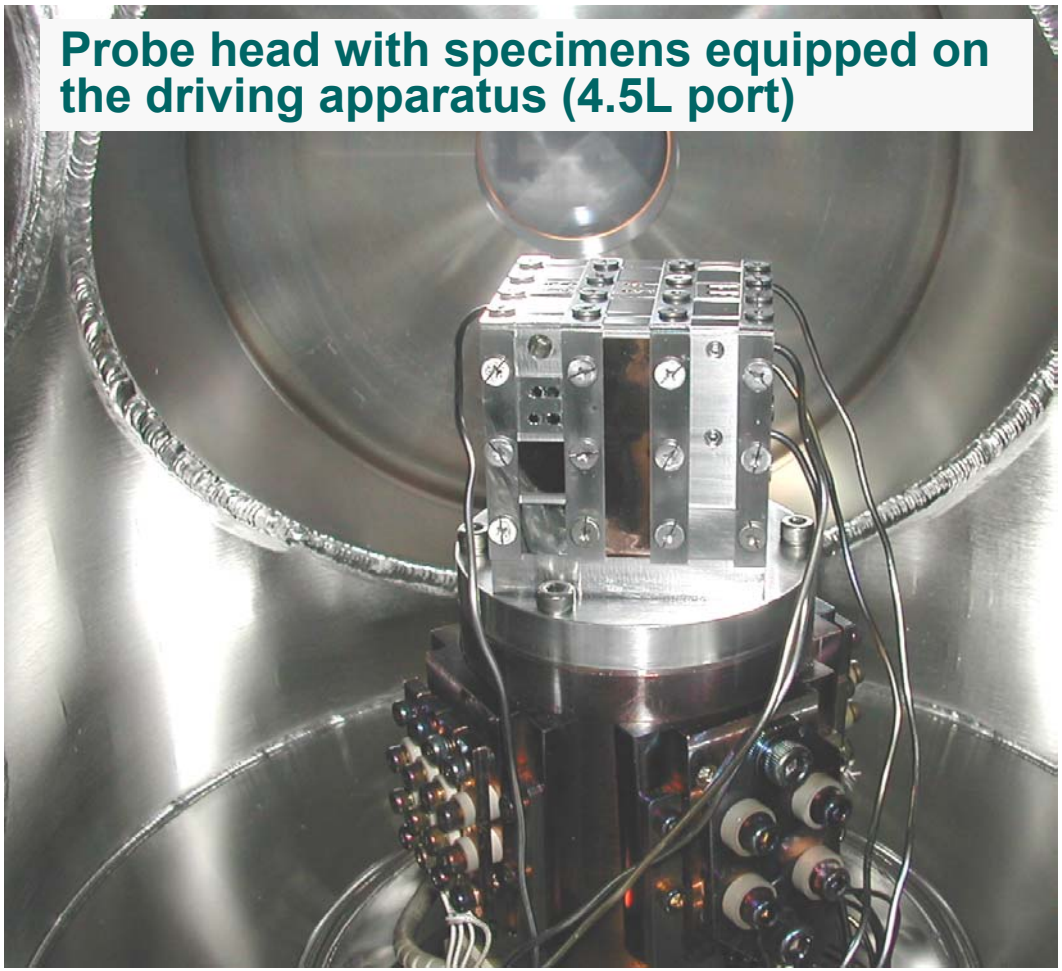


Retractable Material Probe of LHD

Specimens

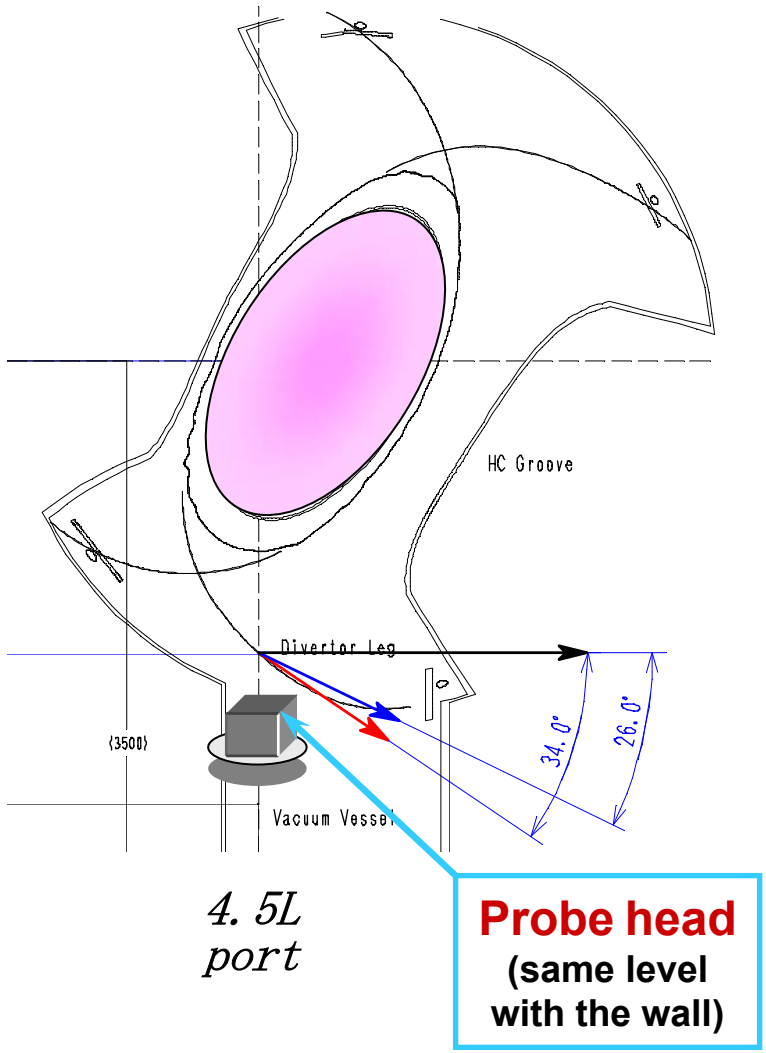
SUS316L, SUS304, Mo, W, Cu

(Pre-thinned TEM discs, thick plate)



Probe head with specimens equipped on the driving apparatus (4.5L port)

Cross section of LHD at 4.5L port



Microstructure Formed by He-GDC （W）

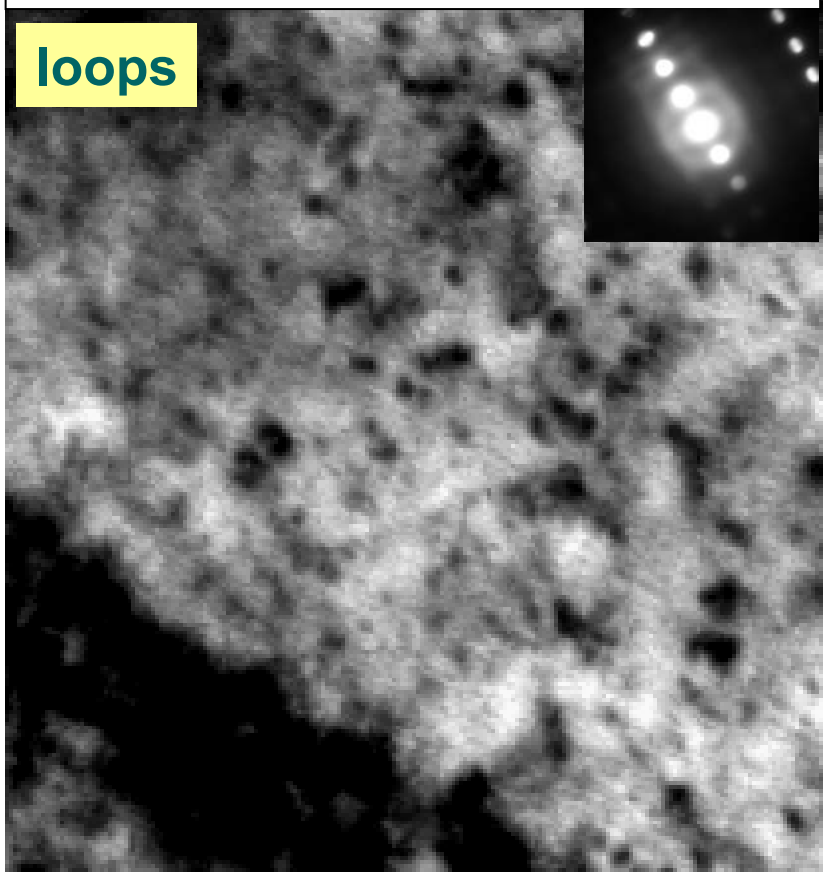
LHD@NIFS

He energy：150~200eV

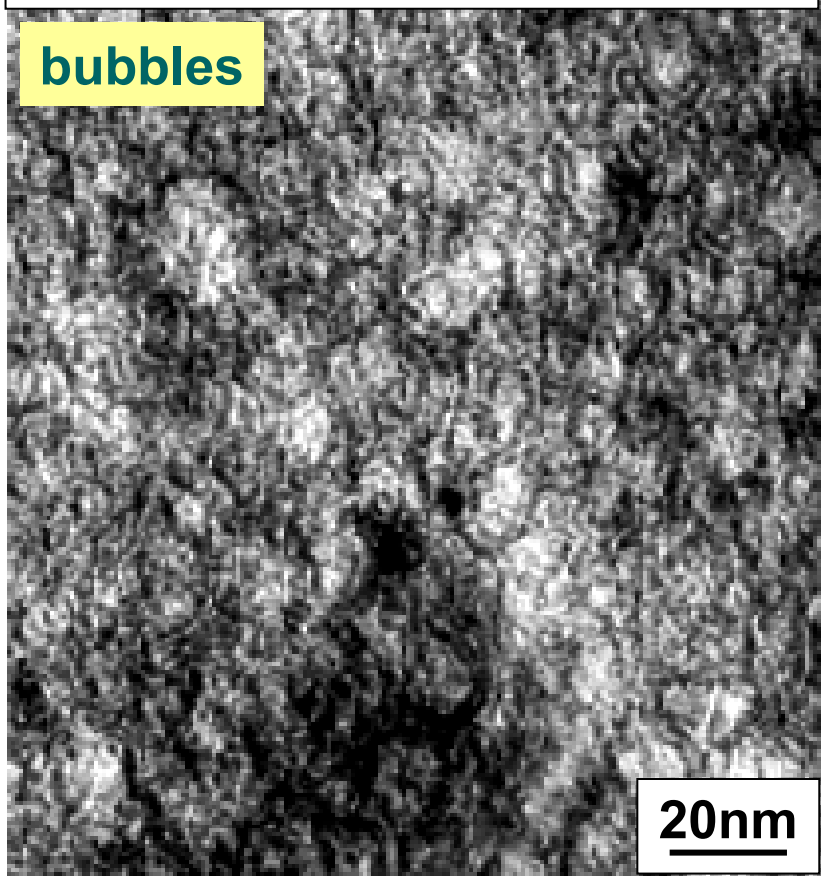
He flux：1.6x10^{17}ions/m^{2}s, fluence：3.8x10^{22}ions/m^2（about 65 hours)

Very heavy radiation damage (bubbles、dislocation loops, etc.)

"Dislocation contrast" (s~0)

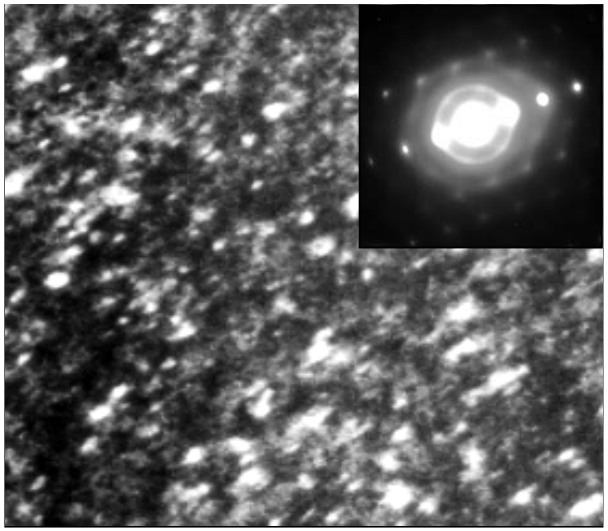


"Void contrast"(s>>0)

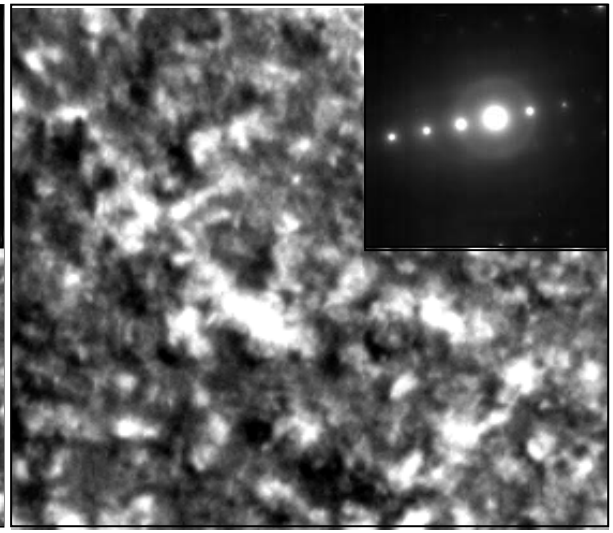


Dislocation Loops Formed by He-GDC

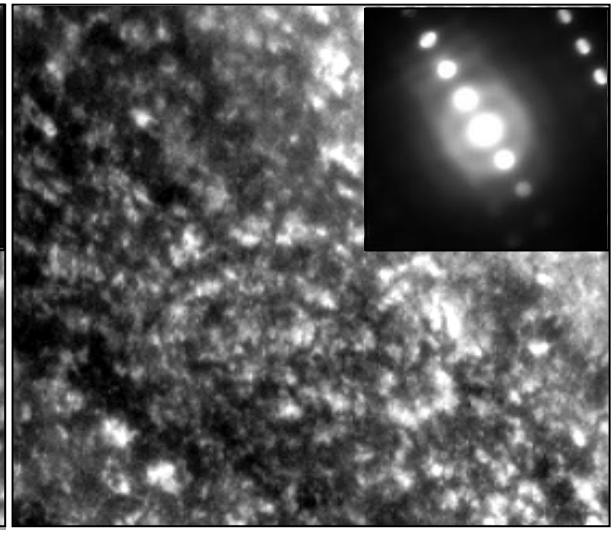
SUS316L



Mo



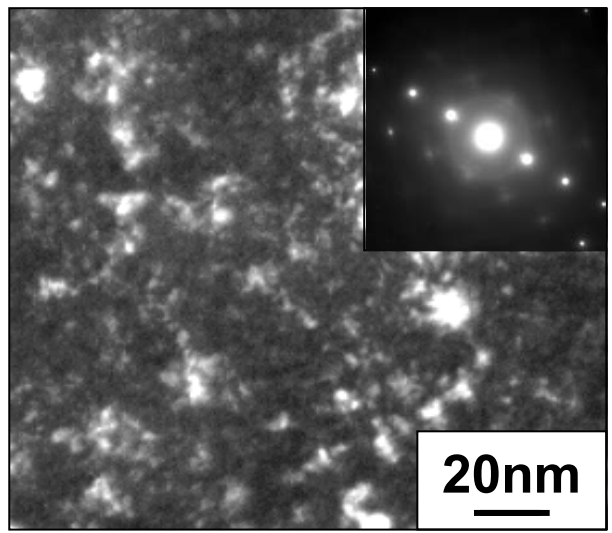
W



Dark field images

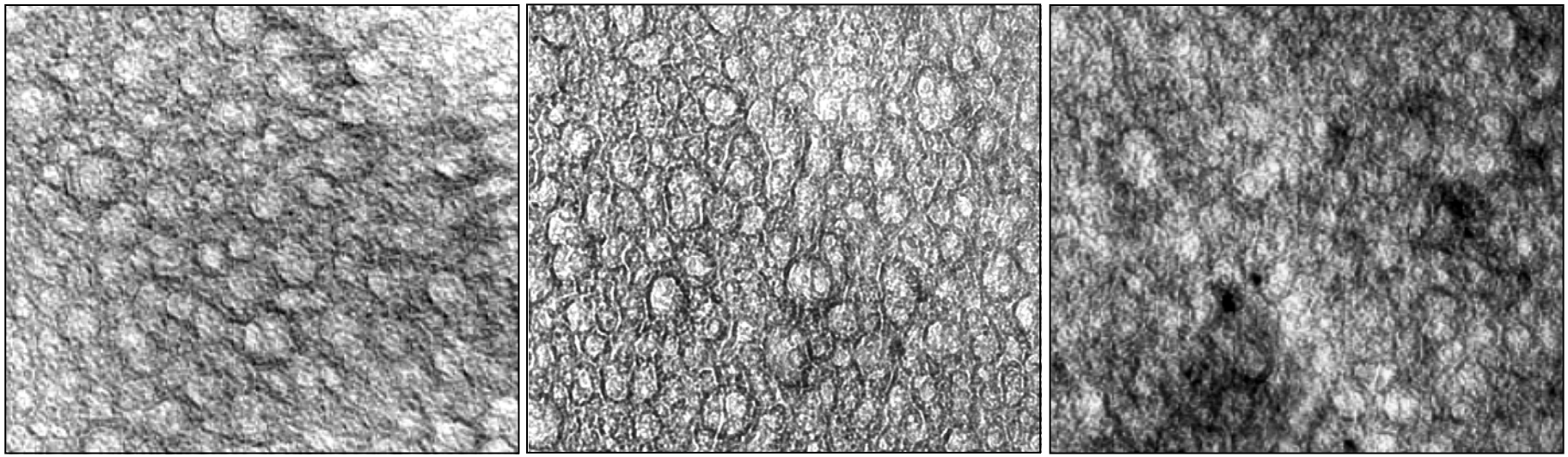
- **Formation of interstitial-loops**
- **Common phenomena in metals**

Cu

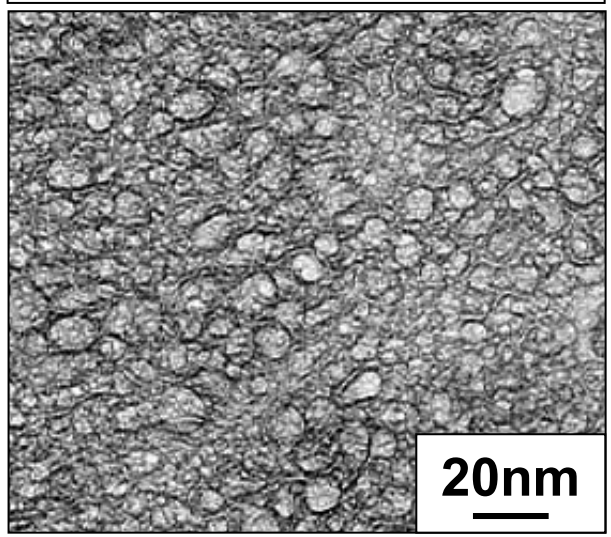


He Bubbles Formed by He-GDC

SUS316L	Mo	W



Cu

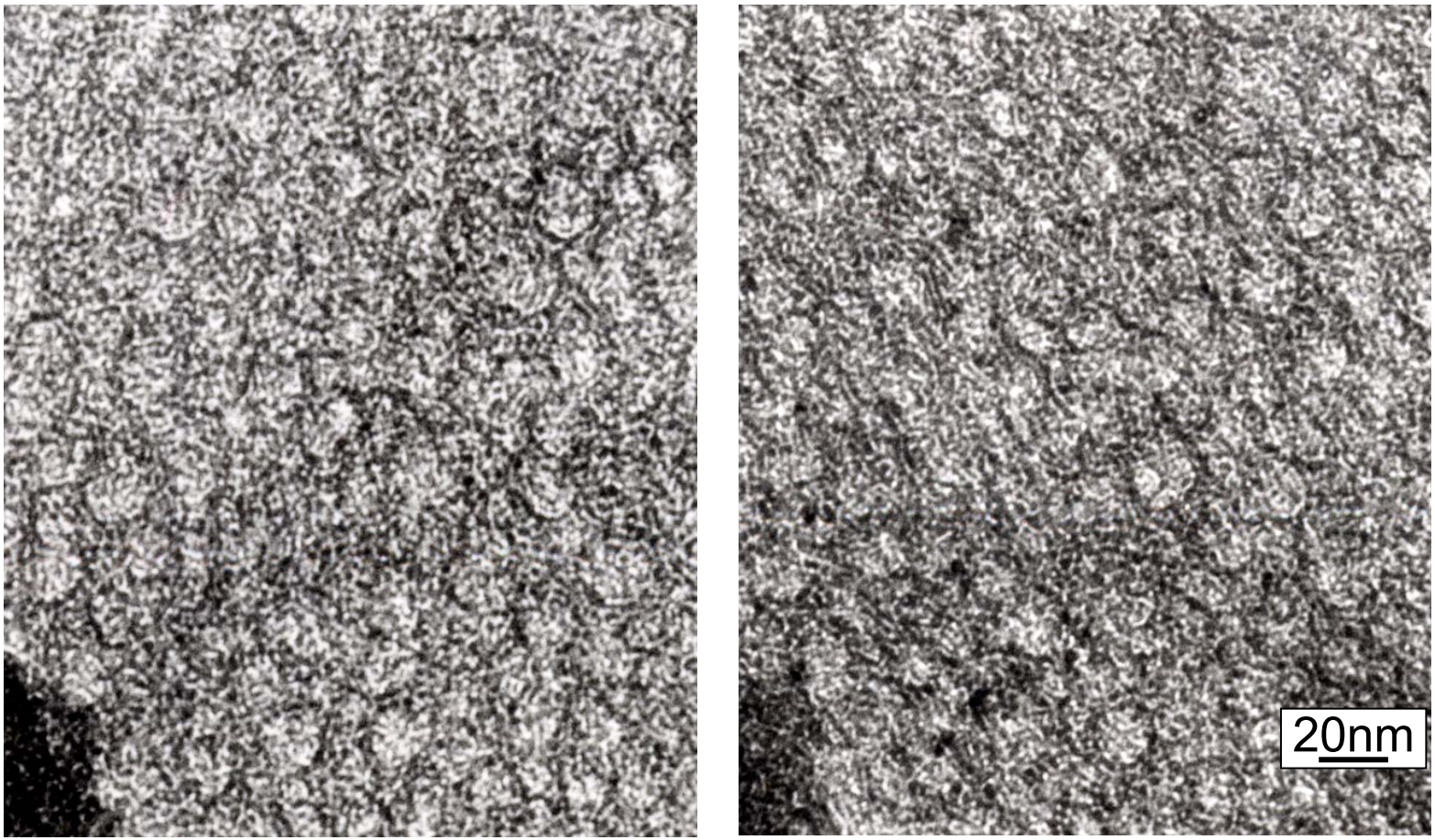


Bright field images

- **Formation of dense bubbles.**
- **Common phenomena in metals**

Depth Analysis of Bubbles (3D-Obs.)

- **SUS304 ; LHD, He glow, 65 hours**
  - **Bubbles distribute even in the deeper region**

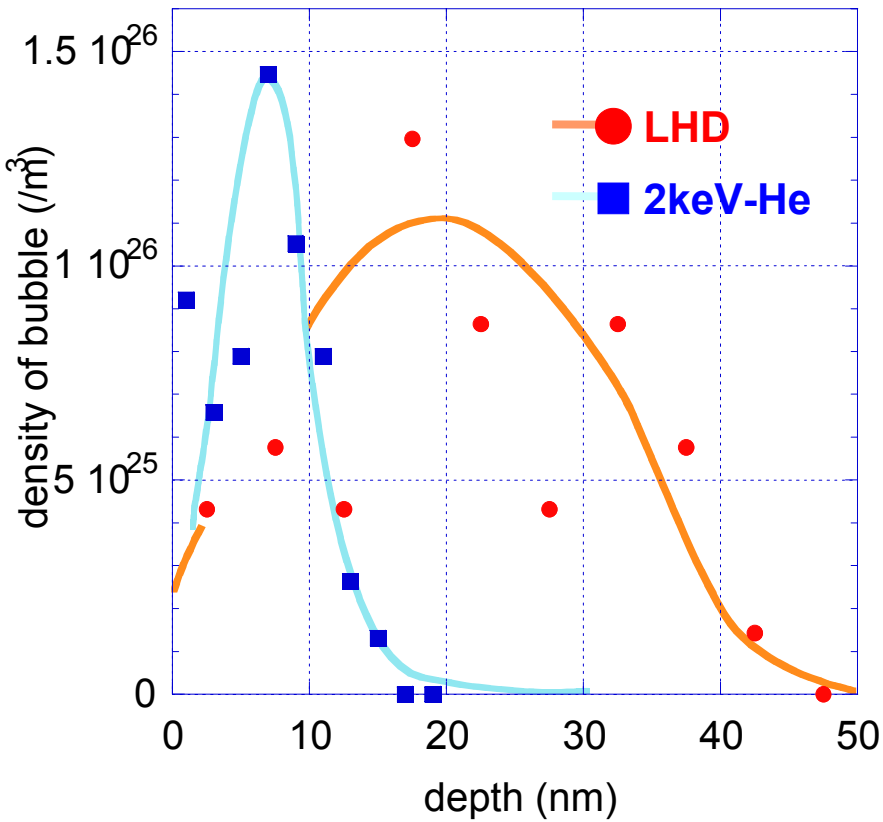


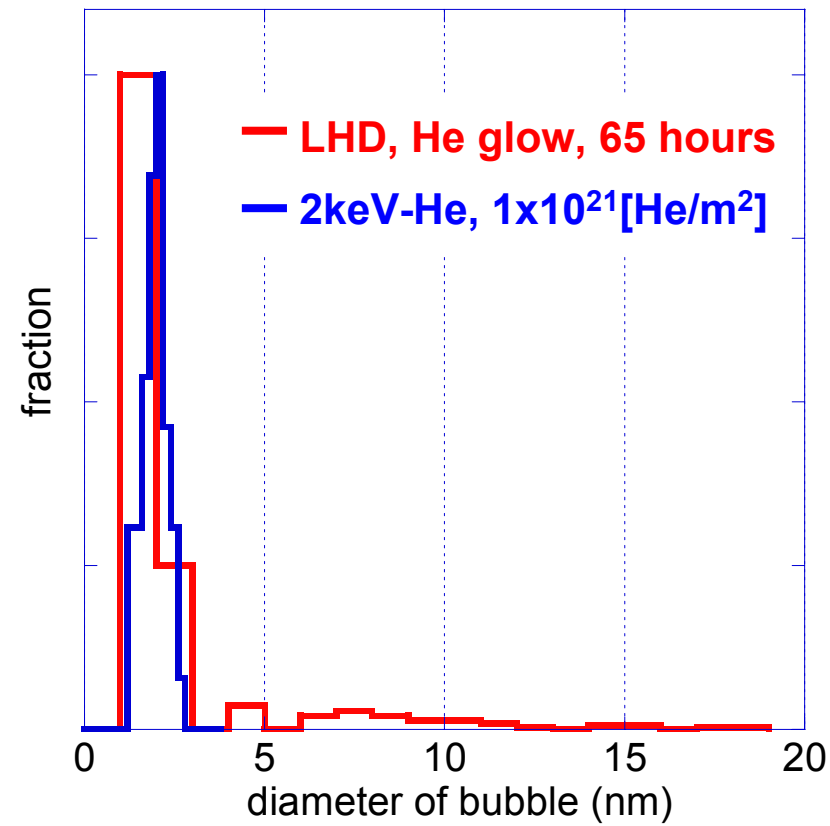
Tilting angle θ =10°

Depth and Size of He Bubbles (SUS304)

LHD:150~200 eV, 3.8×10^{22} ions/m^2, (2keV-He: 2 keV, 1.0×10^{21} ions/m^2)

- **Bubbles distribute far beyond projected range (~7nm)←*diffusion of He***



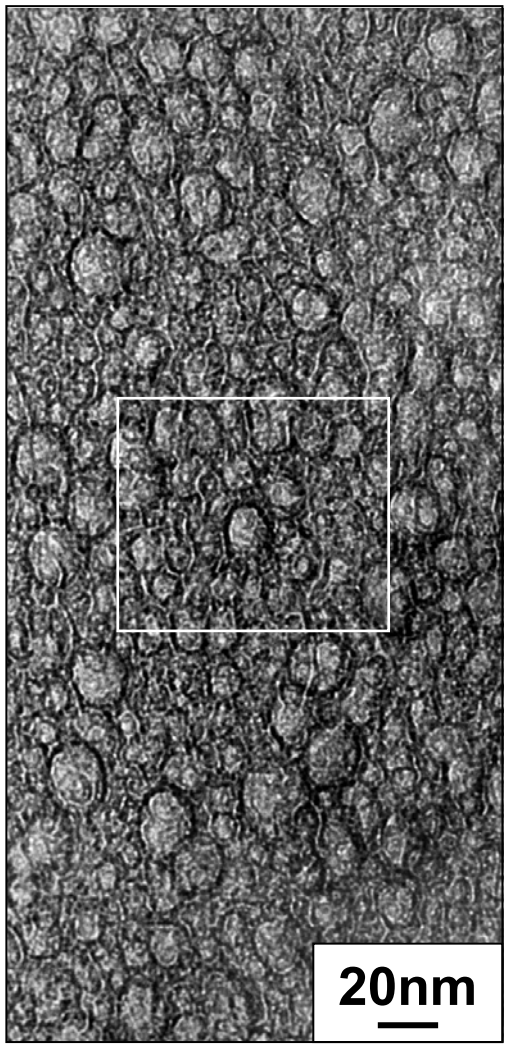


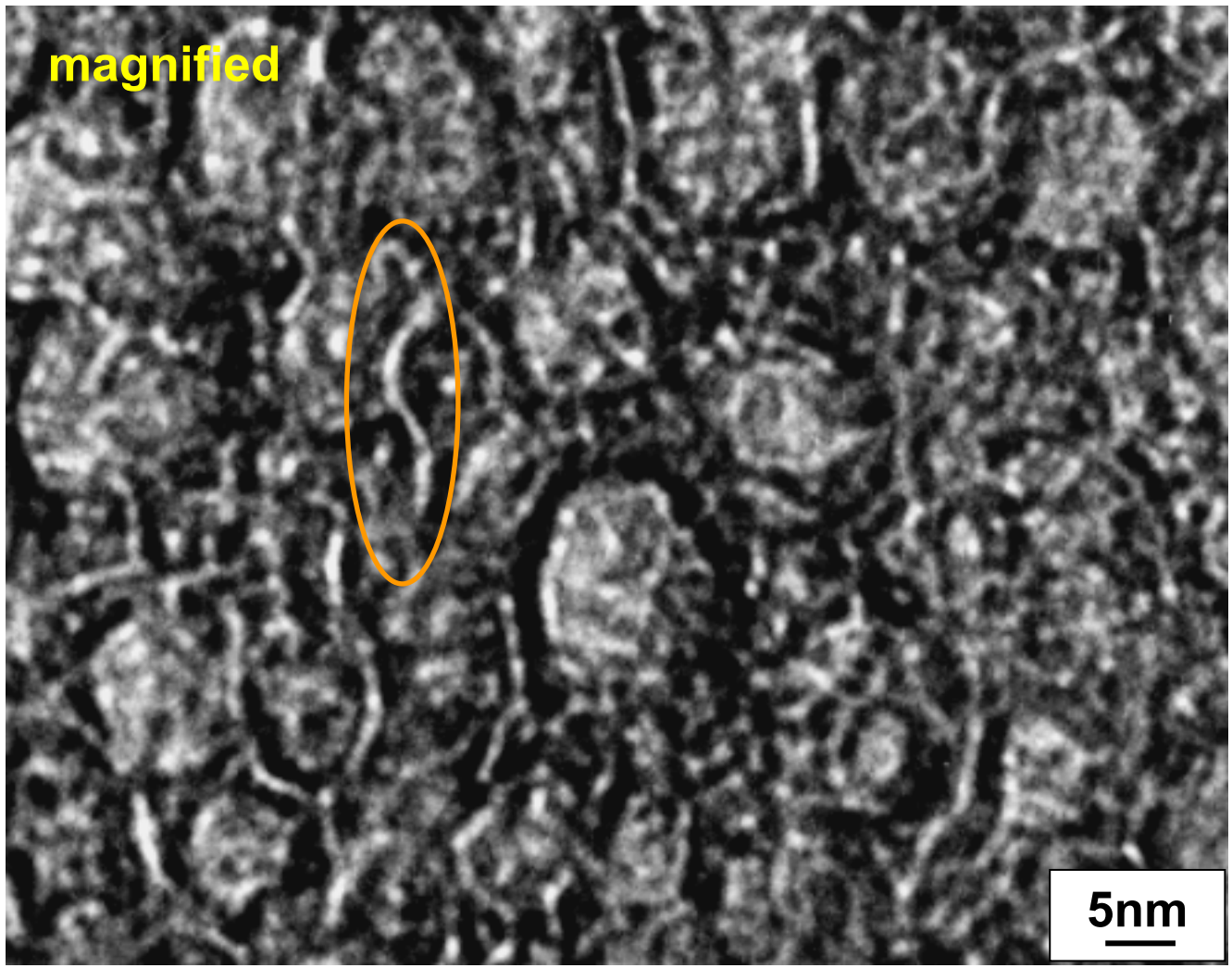
	Av. Diameter (m)	Av. Volume (m^3)	Arial density (m^{-2})	Crt. Pre., He atoms
LHD	2.5×10^{-9}	5.5×10^{-26}	2.8×10^{18}	
2keV-He	1.9×10^{-9}	4.1×10^{-27}	1.2×10^{18}	2.2GP, 326He/200sites

Nano-Cracks in Mo by He-GDC

- In addition to the dense fine bubbles (1 ~10 nm), nano-size cracks which connect bubbles are formed.

→ Pass of gas leakage?

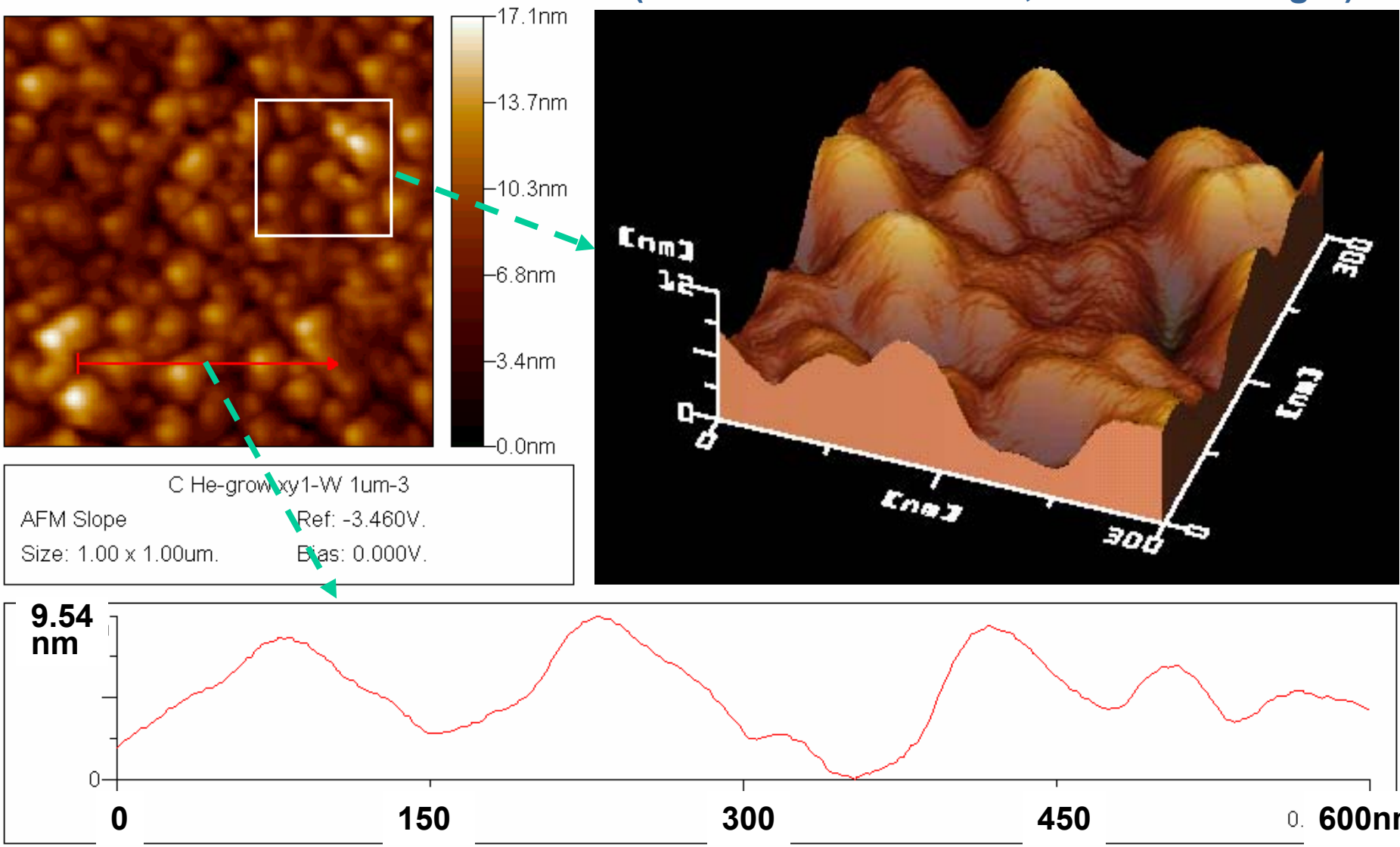




Blistering by He-GDC (W)

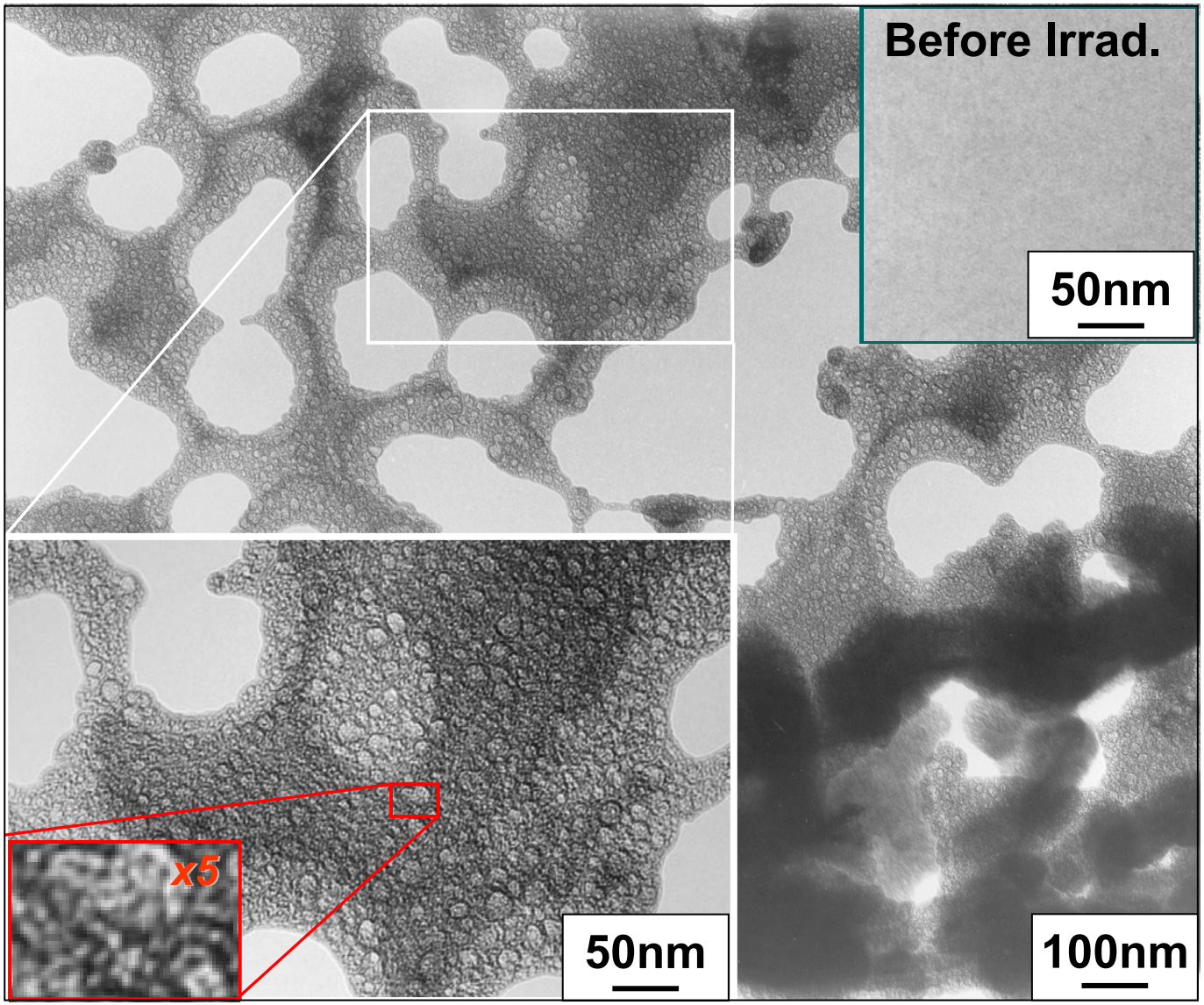
Observation by AFM

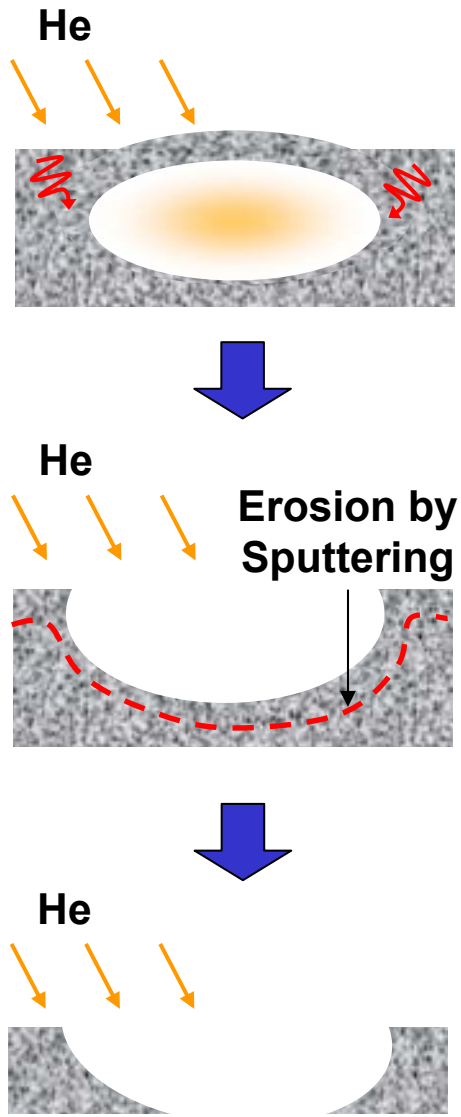
- Irradiated surface is covered by fine blisters.
  (50~150nm in diameter, 5~10nm in height)



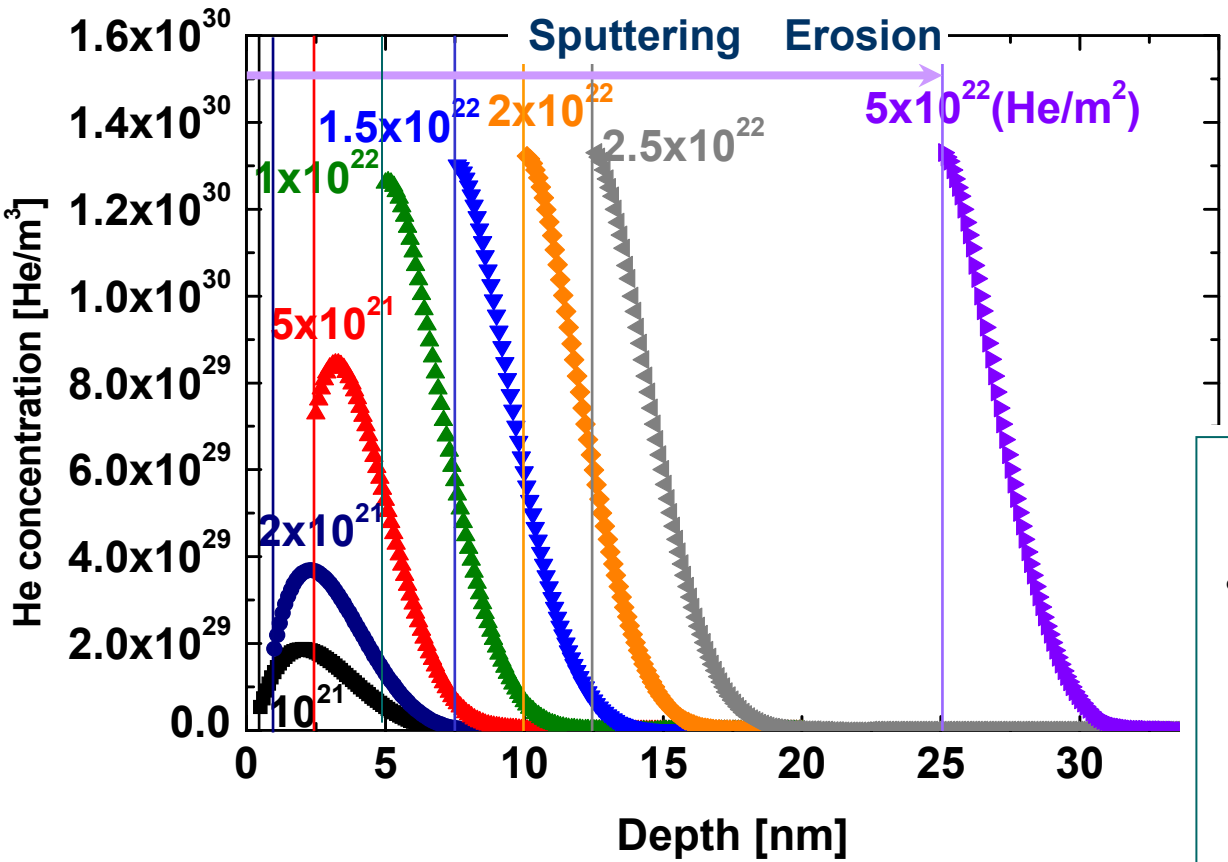
Erosion of Blisters by He-GDC(SUS316L)

- ***Holes and bubbles are formed at a thin part.***





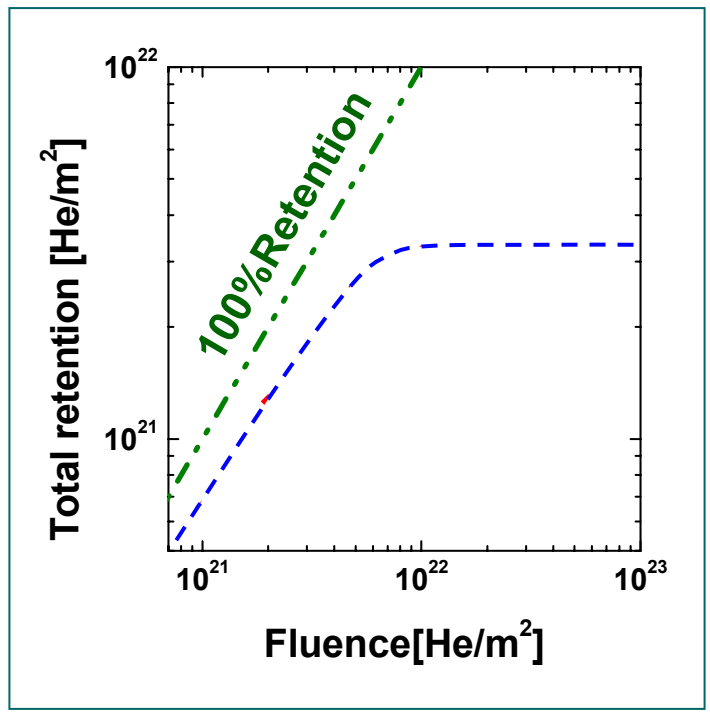
Sputtering Erosion and He Distribution



200eV-He⁺

⇒ SUS316L

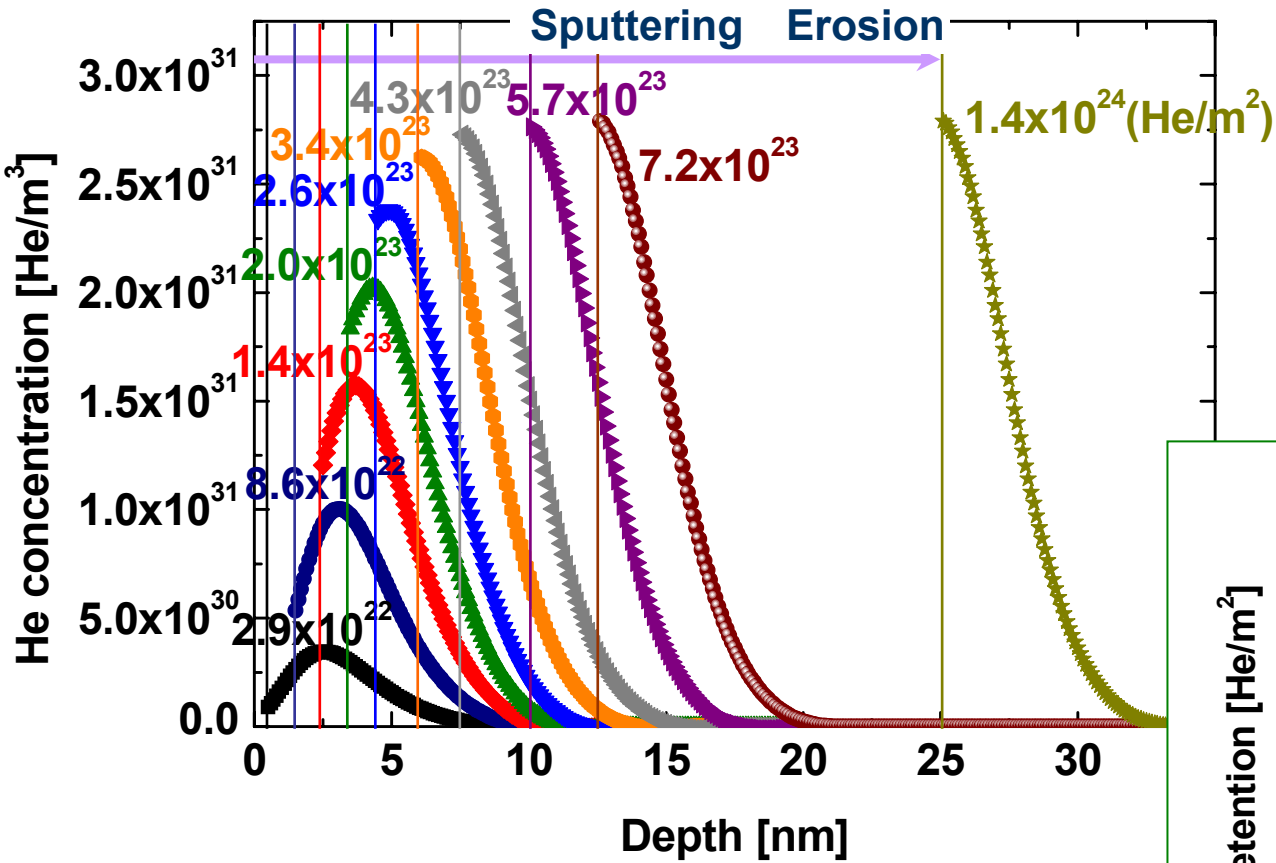
(TRIM91)



backscattered ion : 37%

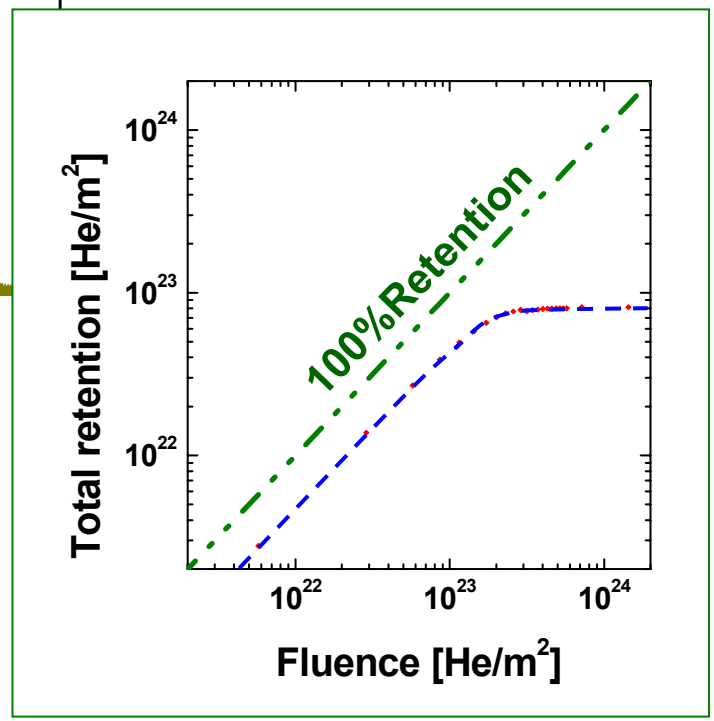
- **By the balance of sputtering erosion and He injection, distribution of He near the surface reaches steady state above $1x10^{22}$He/m^2.**
- **He distribute up to 7nm. Max at the surface (He/lattice atom ~ 13).*(Diffusion of He must be considered in actual)***

Sputtering Erosion and He Distribution



200eV-He⁺ ⇒ W

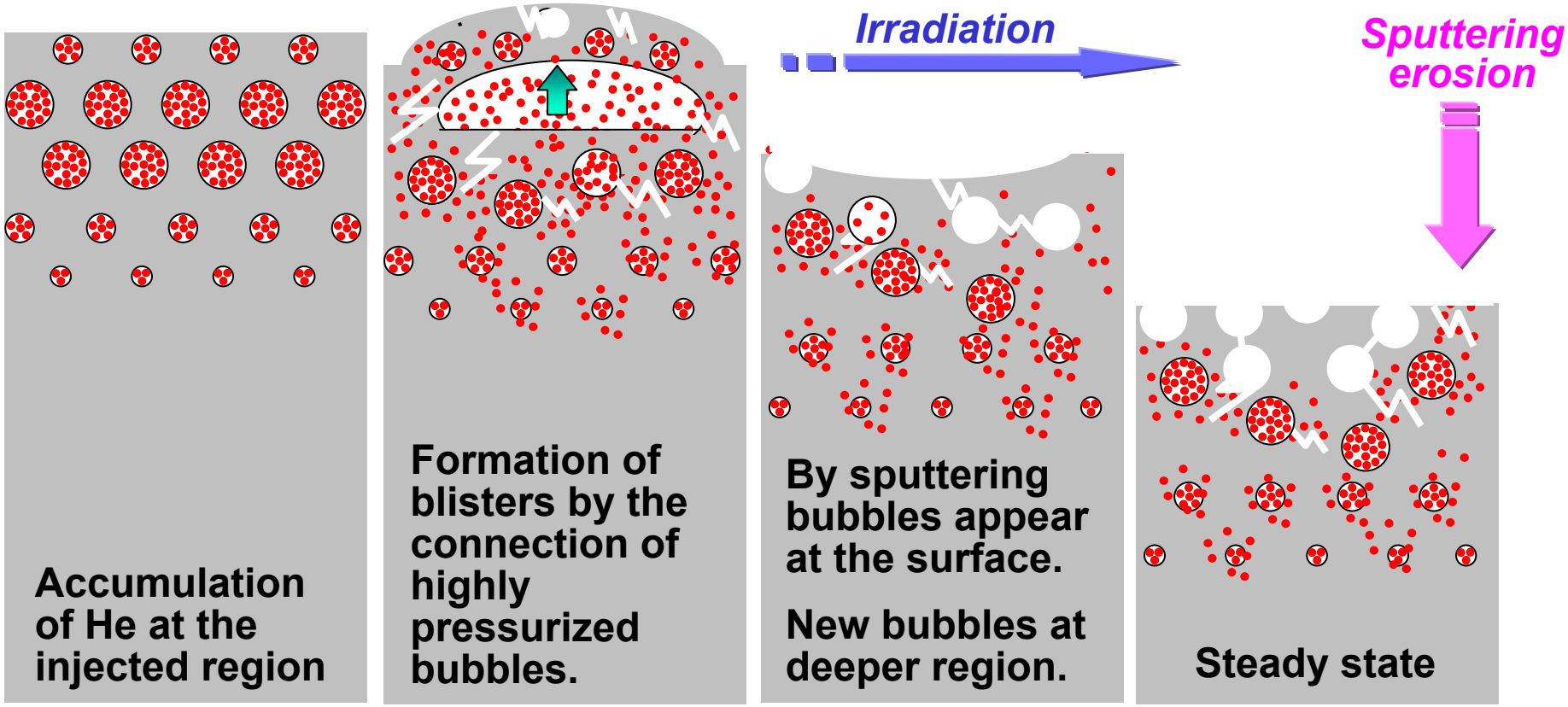
(TRIM91)



backscattered ion : 51%

- By the balance of sputtering erosion and He injection, distribution of He near the surface reaches steady state above 4×10^{23}He/m^2.
- He distribute up to 7nm.

Change of Surface Structure by Heavy Low Energy He Irradiation



By the balance of injection and loss by erosion of the matrix, accumulation of He reaches "steady state".

- **Effective surface area must increases by the cavities connecting to the surface.**
- **He: strongly trapped in bubbles, weakly trapped around bubbles and other defects.**

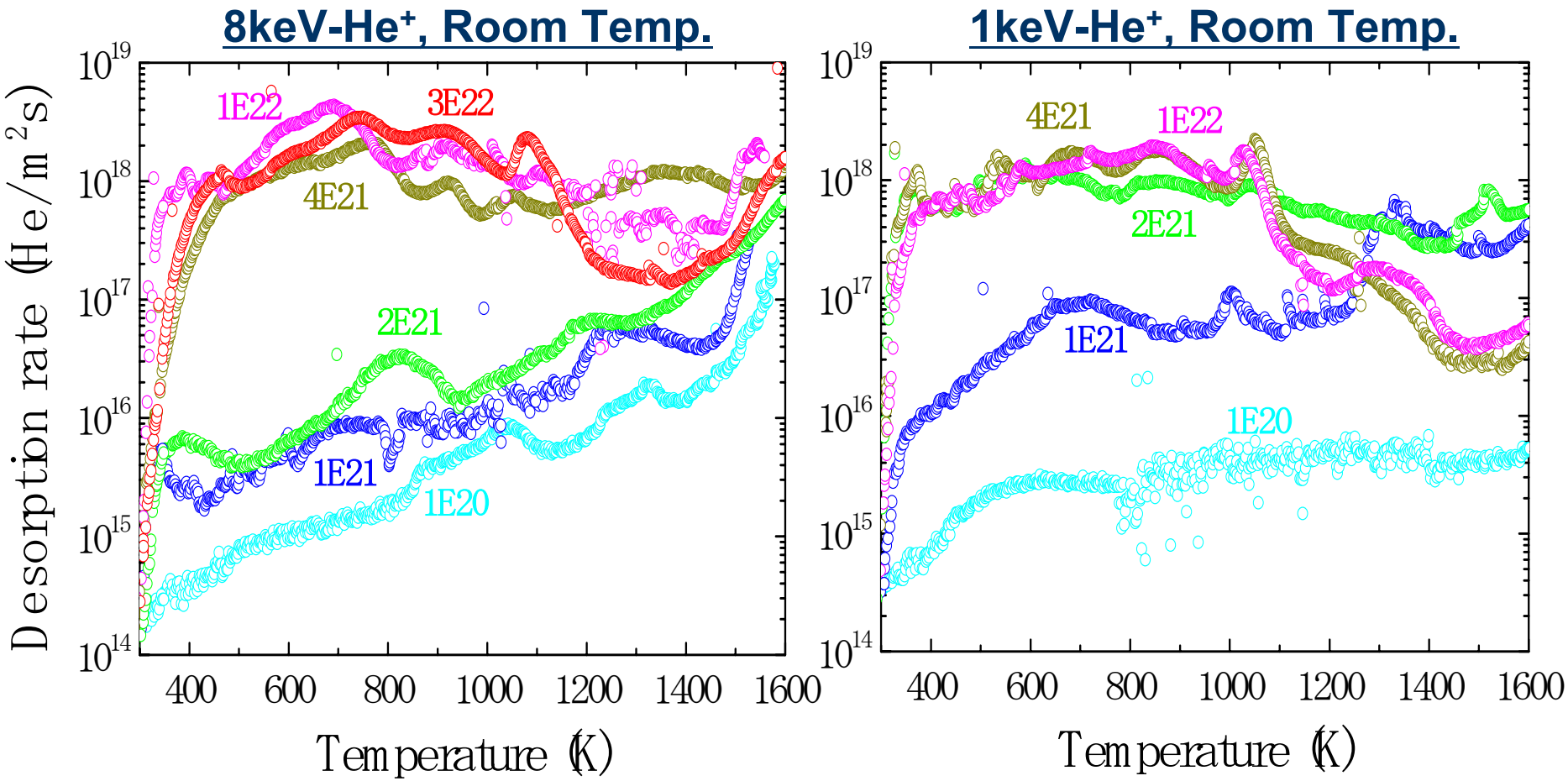
Big Change of PWI

Adsorption & absorption of gas

Thermal Desorption of Injected He (W)

- **Desorption of He at lower temperature region increases drastically above the critical dose.**

  critical dose : 4×10^{21} He^+/m^2(8keV) , 1×10^{21} He^+/m^2 (1keV)

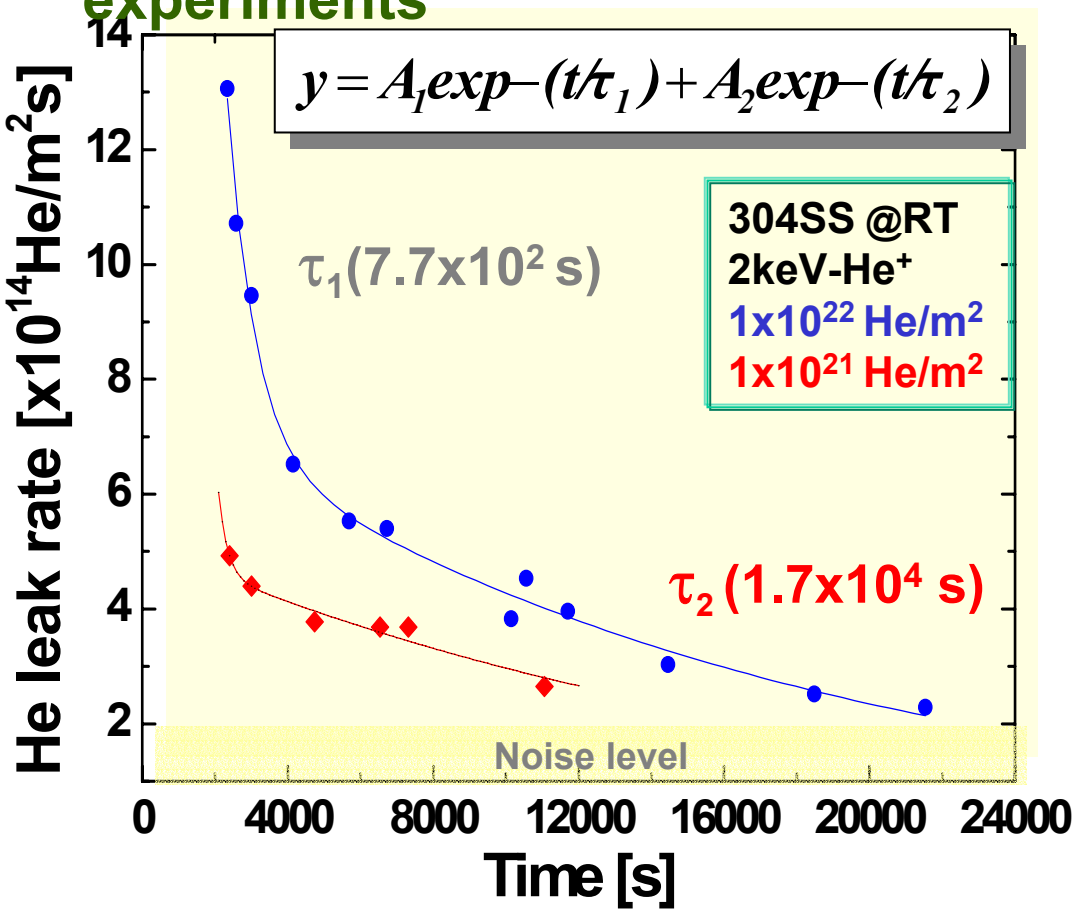


(Large desorption stages exist above 1600K)

Desorption of He at R. Temp. (SUS304)

- Two desorption stages with different time constant
- Long duration of He leakage at R. Temp.

⇒ Bad influence on successive H discharge experiments



Example:

LHD

(vacuum vessel wall:~780m^2)

- Leak of He after 24 h:

  ~4.8x10^{12} He/m^{2}s

- Total of leak after 24 h:

  ~3.7x10^{15} He/s

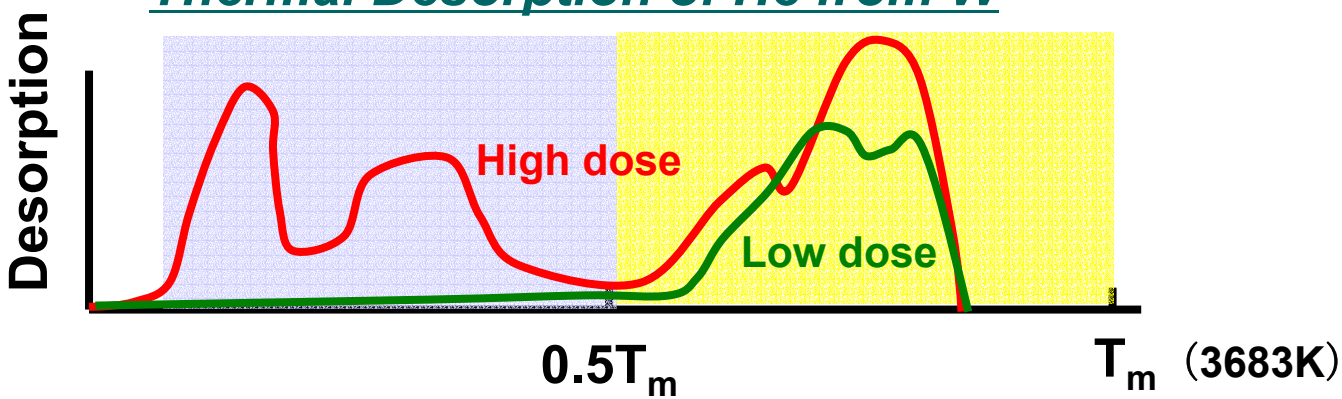
  ↓

  1.4x10^{-5}(Pa m^3/s)

Phenomena at High Temperatures

Phenomena	Temp. region
Migration of Interstitial atoms	> 20 K
Migration of He atoms	> 120 K
Loss of He (at high very He dose)	> 300 K
Migration of Vacancies	> 700 K
Migration of He-V complex with low number of He	> 700-800 K
Loss of He from He_nV_1	1000-1500 K
Migration of He-V complex with high number of He	> 1500-1700 K
Re-crystallization	> 1600 K
Loss of He from large He_nV_m complex	> 2100 K

Thermal Desorption of He from W

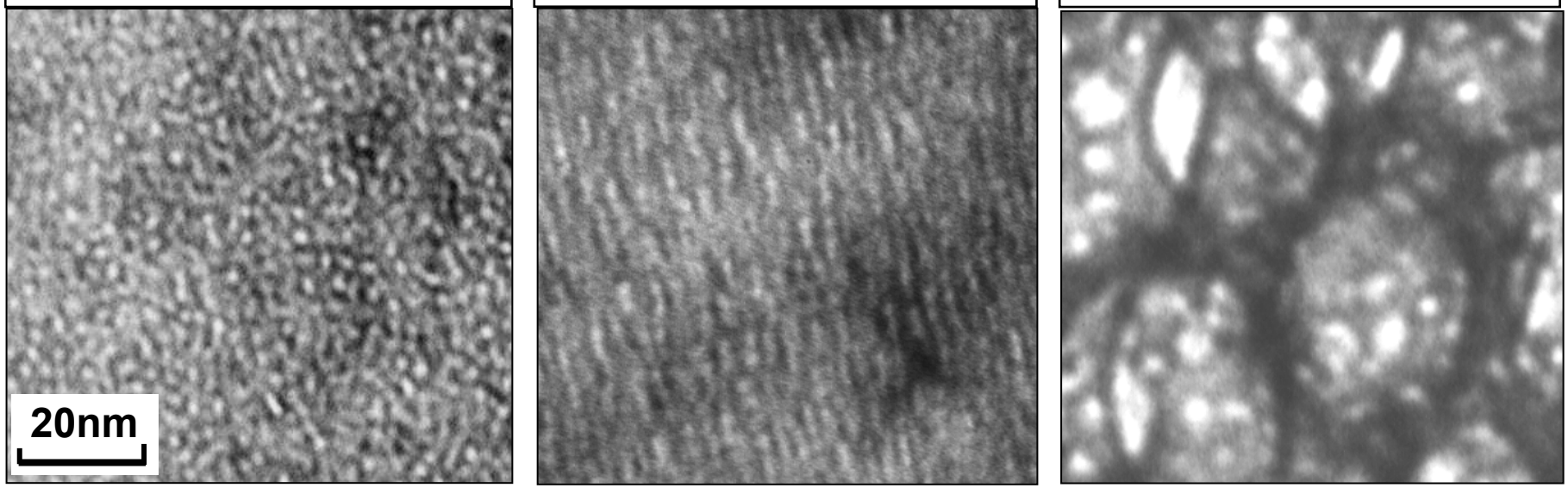


E_{mi}= 0.054 eV
E_{mHe}= 0.3 eV
E_{mv}= 1.7 eV
E_{fv}= 3.6 eV

Temp. Dependence of Damage

W, 8 keV He^+ Irradiation

R.T., 2.0×10^{21} He^+/m^2	873K, 1.5×10^{21} He^+/m^2	1073K, 3.6×10^{20} He^+/m^2



Room Temp: very high density, nano-size

← *dissociation and migration of He-V complex: very difficult*

873K: very high density, nano-size but some of them have grown.

← *low dissociation, slow migration of He-V complex*

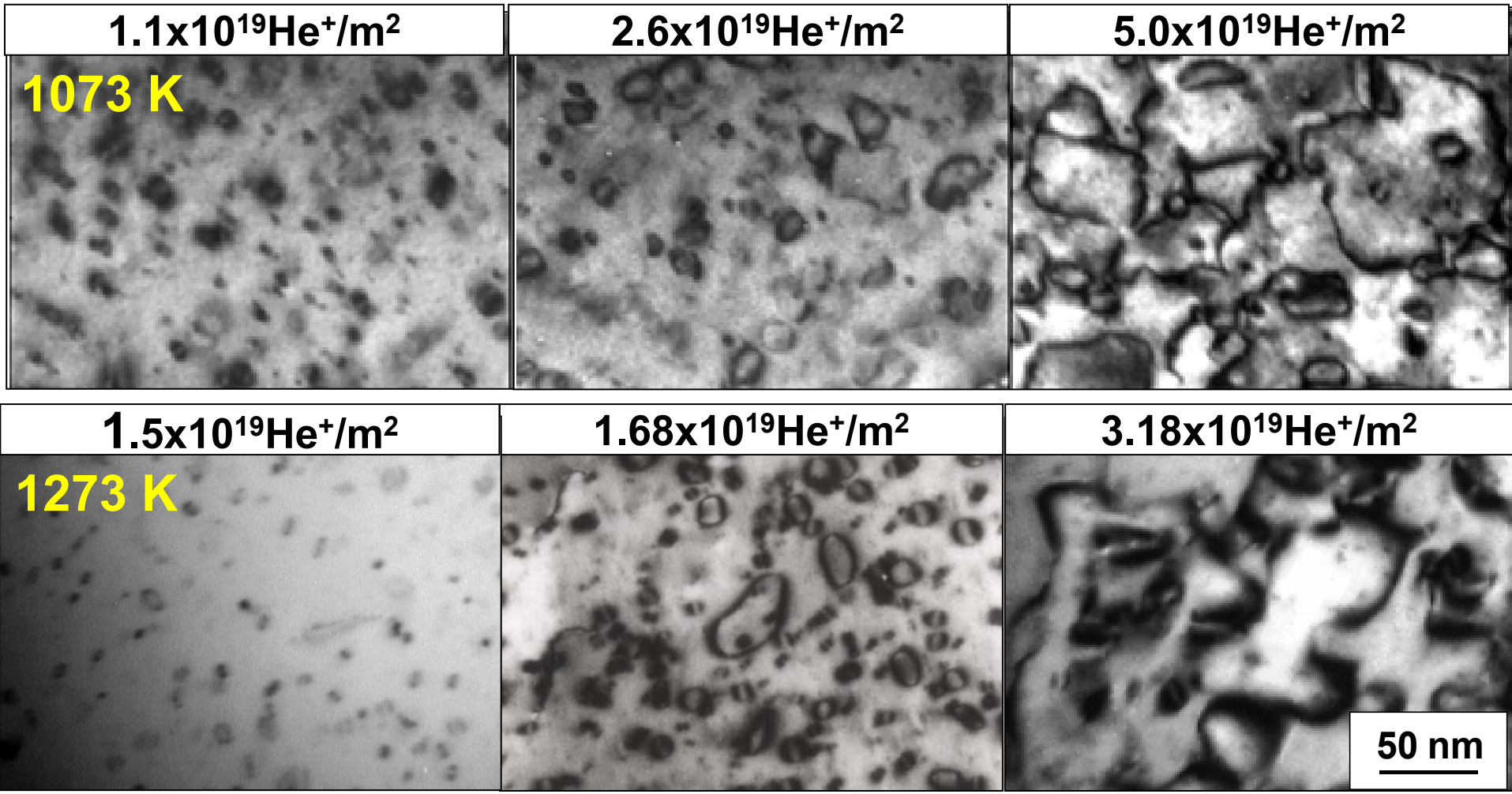
1073K: decrease of density, growth of bubbles

← *instability of the nucleus (He_nV_1) ,*

migration of He-V complex with low number of He.

Formation of I-Loop at 1073K, 1273K

W, 8 keV He Ions Irradiation

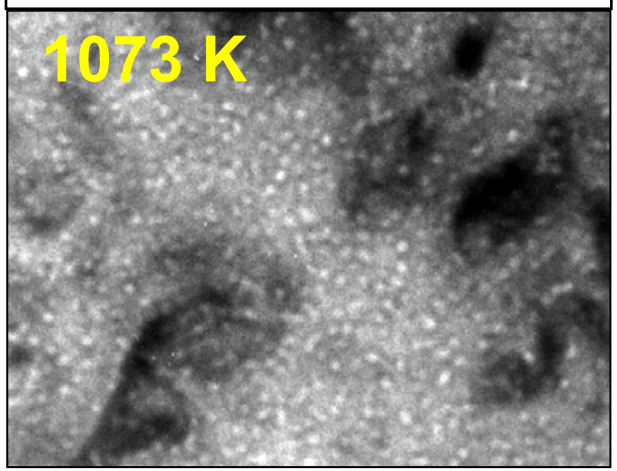


- I-loops appear at low dose and form tangled dislocation structure.
- I-loops grow for a while even after stopping irradiation (1273K).

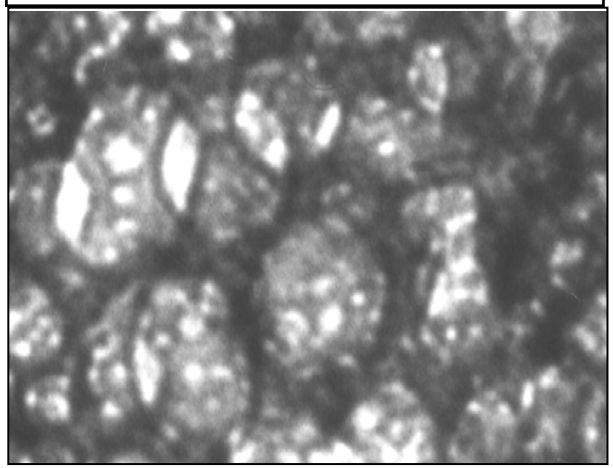
Formation of Bubble at 1027K, 1273K

W, 8 keV He Ions Irradiation

5.0×10^{19} He^+/m^2



3.6×10^{20} He^+/m^2



- Spherical at 1073K
- Polygonal at 1273K

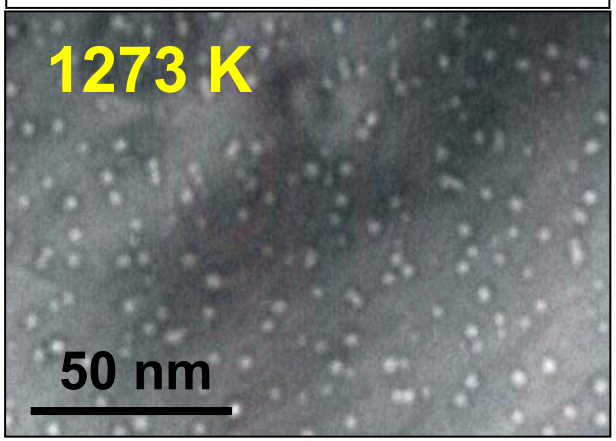
⇧

supply of vacancies

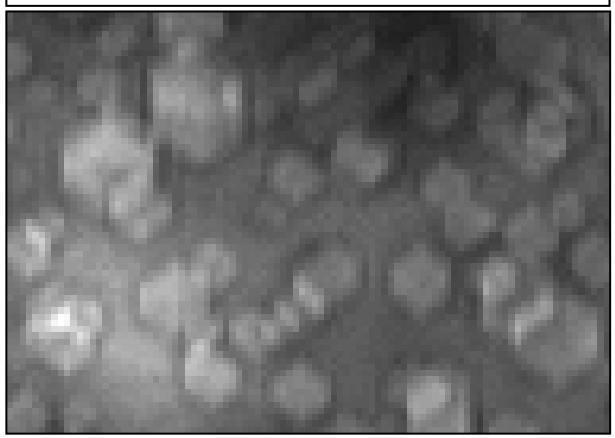
thermal vacancies?

(cf. E_{fv}=3.6 eV)

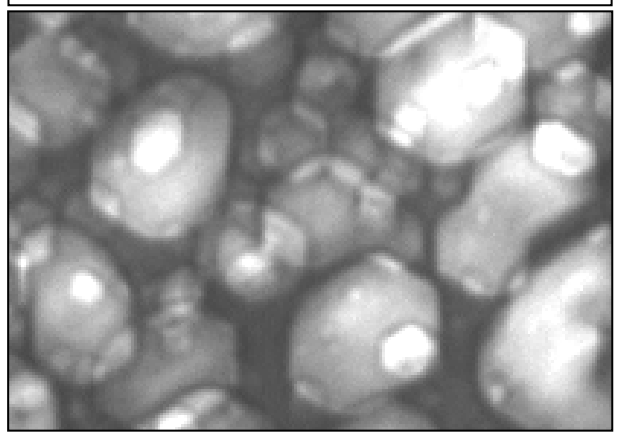
3.2×10^{19} He^+/m^2



4.0×10^{20} He^+/m^2

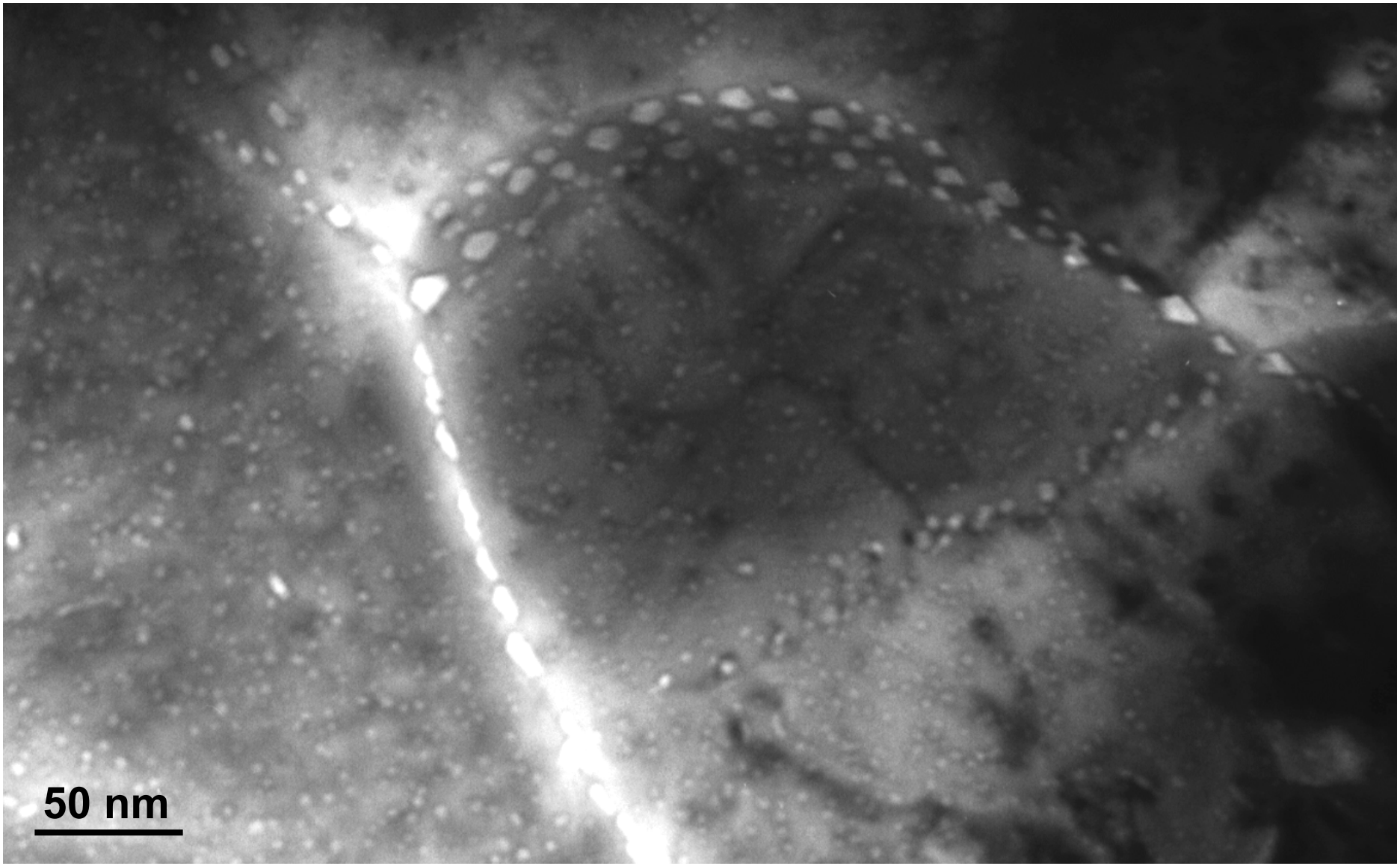


6.7×10^{20} He^+/m^2



Preferential Bubble Formation at G.B.

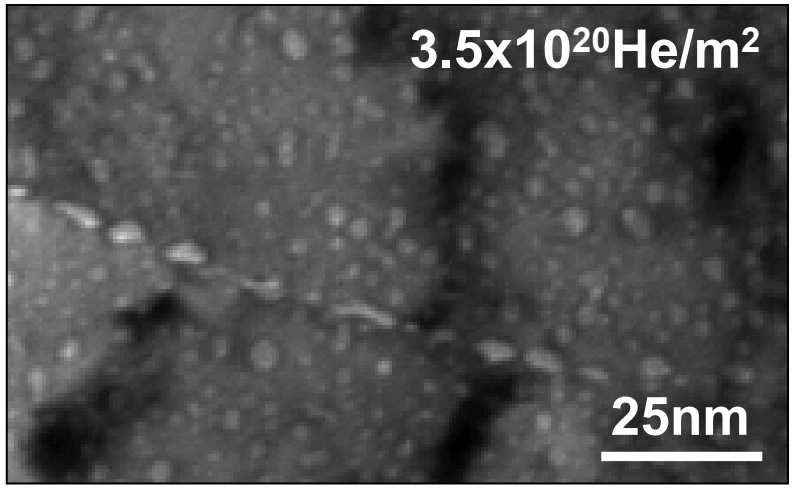
W, 1273K, 8 keV He Ions Irradiation, 5.4×10^{19} He^{+}/m^{2}

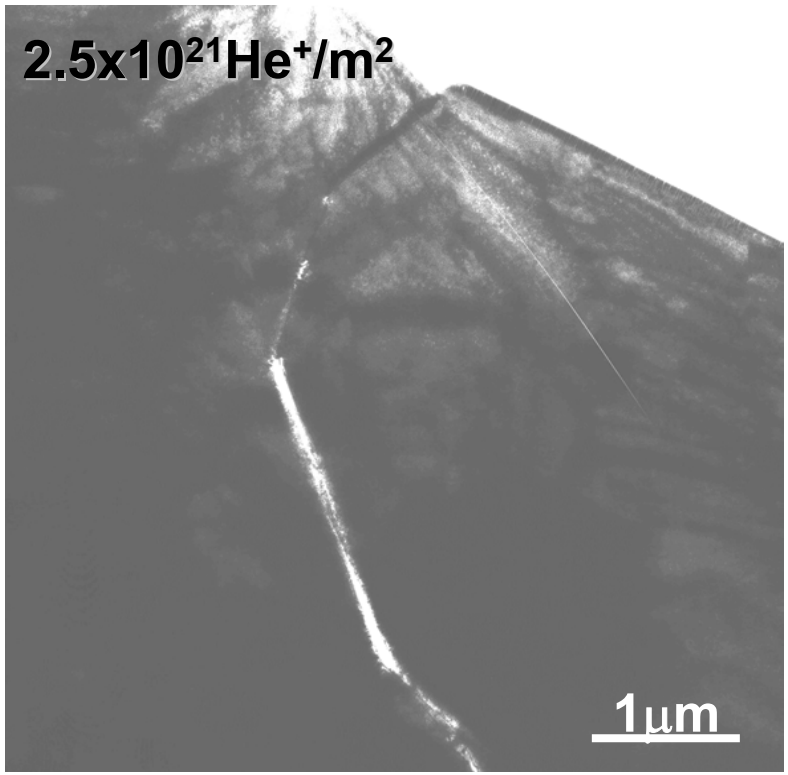


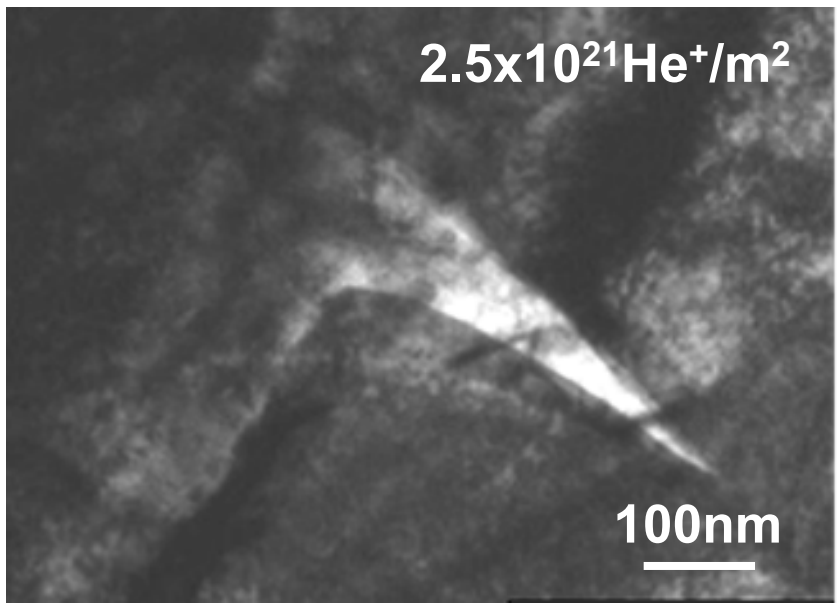
Grain Boundary Embrittlement

W，1073K、0.25keV He^+

- He diffuses preferentially along the G.B. and results in strong G.B. embrittlement by forming bubbles.
- Affect on bulk properties (bulk phenomena)







Summary (1)

Damage by He-GDC

- Microscopic damage of metal exposed to the He-GDC was examined by TEM, in order to understand the mechanism of the retention and desorption of He and other gas.
- Though the energy of He ions is too low for the displacement damage, fine bubbles and cracks are densely formed.
- By the balance of He injection and sputtering erosion of the matrix atoms, damaged layer with the bubbles, cavities (de-gassed bubbles) and cracks always exist at the surface at high dose. The thickness of the layer is about 35 nm for SUS.
- Cavities directly appearing to the surface and connecting to the surface by the cracks must increase effective surface area very much. It seems that this surface structure enhance the adsorption of gas molecules.
- By the formation of the damaged layer, retention of He increase drastically and some part of the He release slowly even at room temperature.

Summary (2)

Damage at Elevated Temperatures

R. Temp ~ 873K

- Formation of fine He bubbles

1073K

- Increase of bubble size, decrease of bubble density.
  ⇒ instability of nucleus of bubbles (He_n-V_1)
- Spherical shape
  ⇒ very high pressure is kept
- Preferential formation of bubbles at the grain boundaries
  ⇒ cracking along the grain boundaries, embrittlement

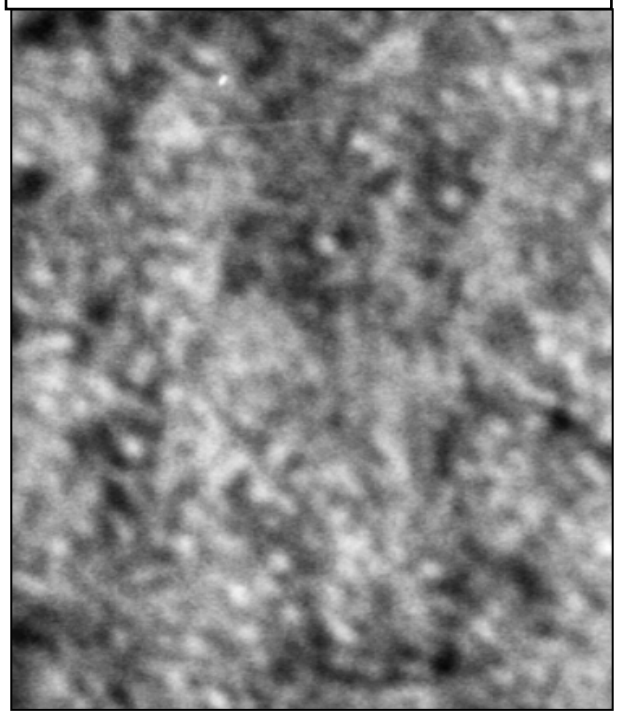
1273K

- Increase of bubble size, decrease of bubble density, polygonal shape, migration and coalescence of the large bubbles
  ⇒ supply of thermal vacancies, surface diffusion control the phenomena

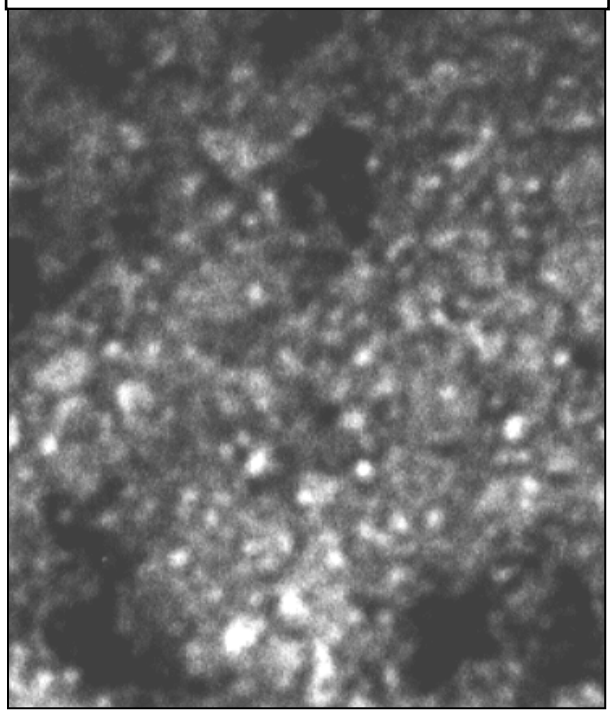
Damage by Sub-E_d He ions

W、 0.25keV He^+ Irradiation

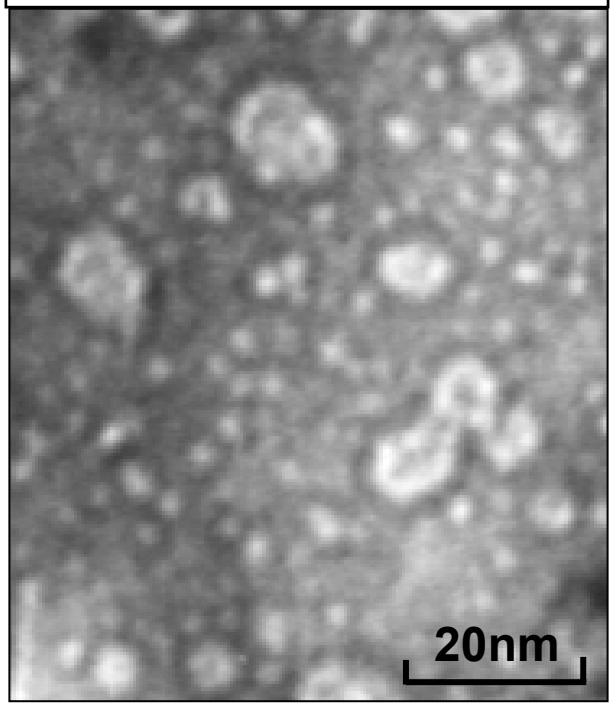
R.T., $5.0 \times 10^{21} He^+/m^2$



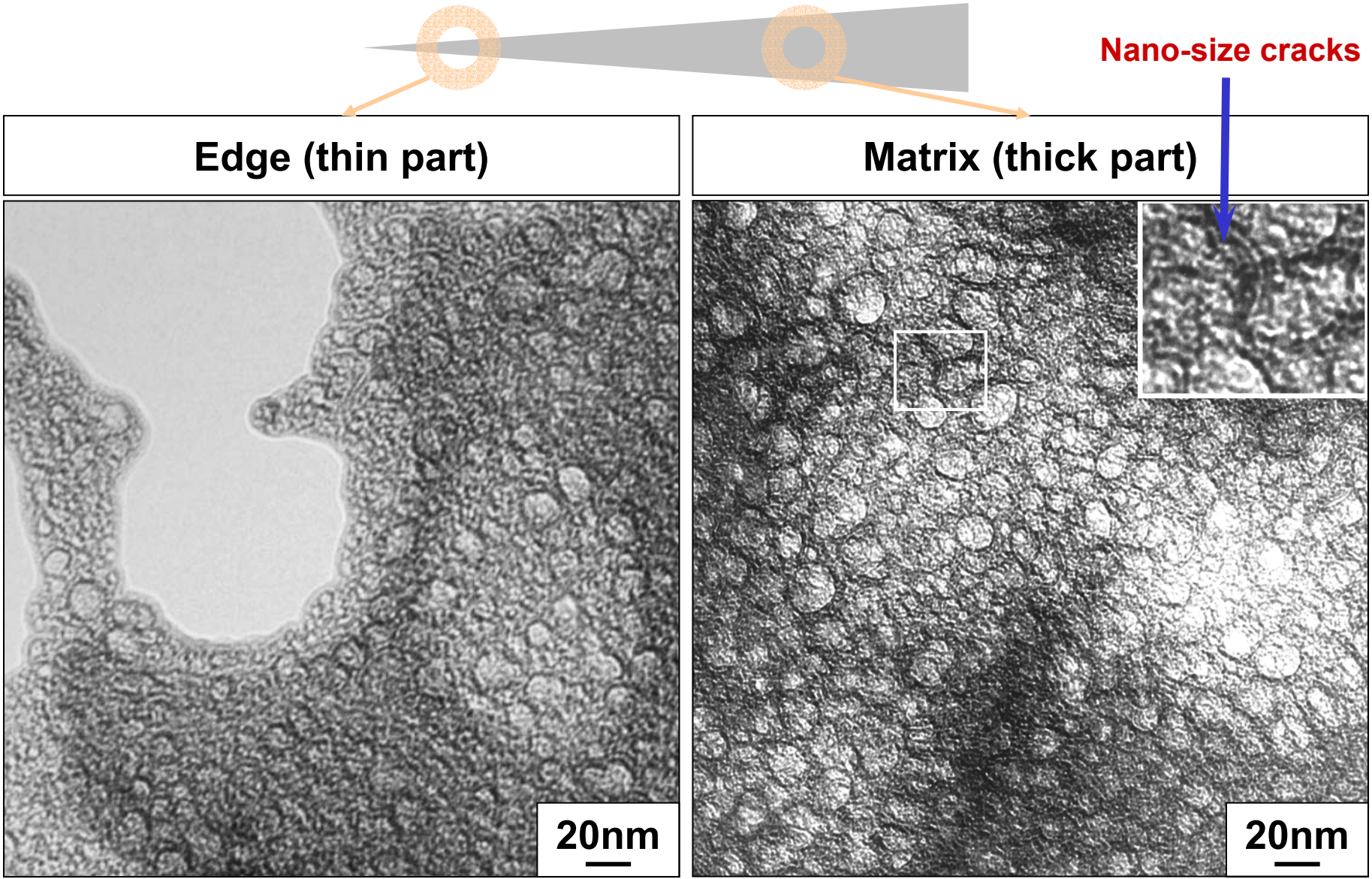
873K, $2.5 \times 10^{21} He^+/m^2$



1073K, $9.2 \times 10^{20} He^+/m^2$



Damage in SUS316L by He-GDC

Workshop Agenda

Holes appear at very thin parts