"First meeting of CRP on irradiated tungsten"
IAEA, VIC, 2013.11.27

Microstructure and hydrogen isotope retention in tungsten with radiation induced defects

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Contents

Main Objective

To understand the effect of neutron and surrogate irradiation upon microstructure of tungsten and

through that effect of the damage of tungsten on hydrogen isotope retention

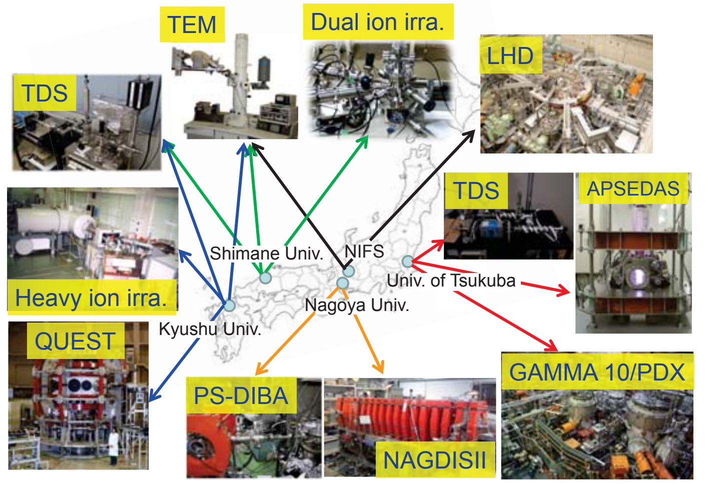
by using charged particle beams (H, D, He, Cu and W), transmission electron microscopy (TEM) and thermal desorption spectroscopy (TDS) after divertor-like plasma exposure.

Project Members

Specialty area of the project members covers from material to plasma-wall interaction.

M. Sakamoto University of Tsukuba	Plasma-wall interaction in the stady-state discharge. Hydrogen isotope retention in tungsten and surface modification analysis of tungsten using a divertor simulator.
H. Watanabe Kyushu University	Neutorn and high enenergy ion induced damage on plasma fascing materials and low activaton structural materials for DEMO. Snergistic effcts of ion (neutorn) and plasma on W.
N. Yoshida Kyushu University	Formation mechanisms of the radiation induced lattice defects such as dislocation loops and voids through TEM obserbation. Plasma surface interaction studies in large-sized fusion devices.
N. Ohno Nagoya University	In-site mesurement of hydrogen isotope retenstion with an ion beam analysis under plasma exposure. Surface modification of tungsten (fuzzy structure, bubbles and holes). ELM-like heat pulse irradiation to tungsten.
M. Miyamoto Shimane University	In-situ TEM observation of W samples irradiated with low energy He and D ions. Study on the effects of Be and He seeding to D plasma on D retention property and microstructure of tungsten.
M. Tokitani NIFS	Retention properties of helium and hydrogen isotopes in the materials exposed to the large sized-plasma confinement devices. TEM and TDS observations and ion beam analysis.

Facilities used in this project



Some of results achieved to date

1. Cu ion irradiation to W
    - Observation of microstructure
    - D retention in the damaged W
2. Low energy and high flux plasma irradiation to W (APSEDAS)
3. In situ measurement of hydrogen isotope retention in W using ion beam analysis under plasma exposure (PS-DIBA)

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1. Cu ion irradiation to W

EXAMINED MATERIALS

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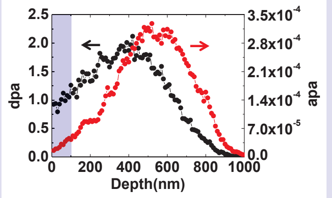
- Base Material: 0.1mm-thick tungsten sheets (99.95% pure, Nilaco Co.)
- Cu Ion Irradiation: 2.4MeV-Cu^{2+}, $1x10^{19}$ ions/m^2 (1.3 dpa at the peak), @ 300K.

For comparison

- Heat Treatment:
    - 1173K/10min, 1173K/30min,
    - 1223K/30min,
    - 1273K/30min
    - 1200K/3h (stress-released (SR-W))
    - 2300K/20min (re-crystallized (RC-W))
- Plastic Deformation of RC-W: rolled 5%, 10%

Depth Distribution of Damage

2.4MeV-Cu^+ ($1x10^{19}Cu^{2+}/m^2$) ➔ W



EVALUATION OF IMPLANTED D

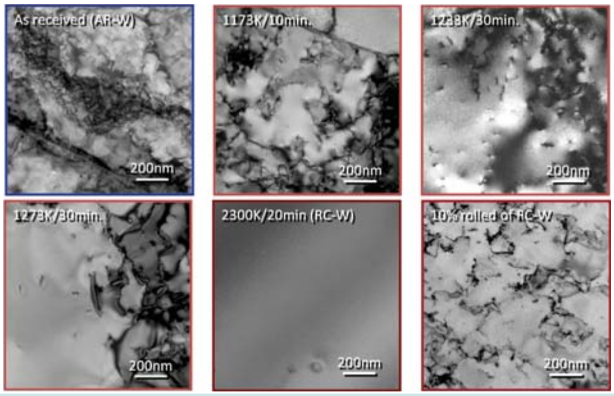
- D Ion Implantation: 2keV-D_2^+, $1x10^{21}D_2^+/m^2$ (TDS)
- Thermal Desorption Spectroscopy (TDS):
    - Ramping rate:1K/s
    - Measured gas: D_2(M=4), DH (M=3)

OBS. OF MICROSTRUCTURE

- TEM
    - Pre-thinned RC-W (thickness ≦ 100nm) were observed.

Results of Mechanically deformed W (no heavy ion irradiation)

Texture of the Examined Samples



- Highly accumulated strain and dislocations in the grains decrease by annealing above 1173K.

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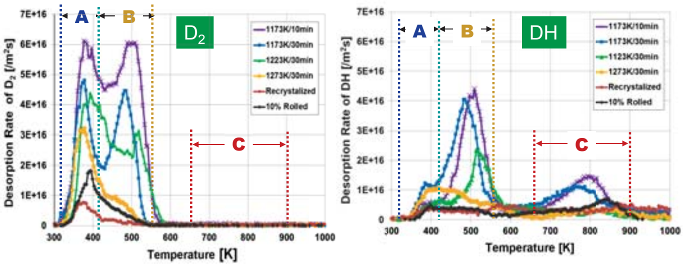
TDS of Imp. D from mechanically deformed W

Desorption of D_2 and DH after implantation of D ions (2keV-D_2^+, $1x10^{21}/m^2s$ @R.T.)



- Three large desorption peaks named A, B and C exist. Each peak may have sub-peaks. Peak A: 330~420K, B: 420~560K, C: 660~900K.
- Retention of D: Peak B > Peak A > Peak C
- Peak A: trapping increases by heavy deformation and shrinks by pre-annealing above 1173K. Most of D desorb as D_2.
- Peak B: appears in both D_2 and DH. Disappears by the annealing up to 1273K.
- Peak C: appears only in DH. Disappears by annealing above 1223K.

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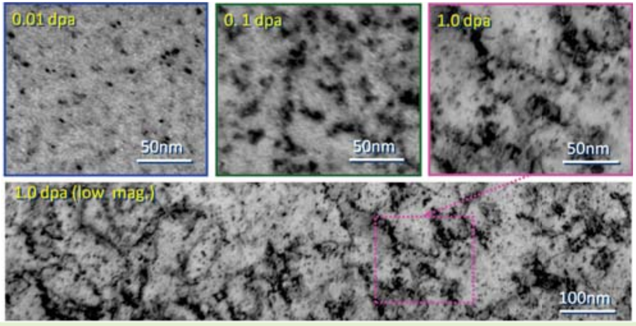
1.2 Damage evolution in W with Cu ion irradiation

300K , 2.4MeV-Cu^{2+}, 0.007~0.7dpa, about $1x10^{-4}$dpa/s



- Very dense defects are accumulated above 0.1dpa.
- Most of interstitial loops (IL) must be nucleated by cascade collisions.
- Each IL can not grow large individually but aligned ILs grow by coalescing. Concentration of ILs and interstitials accumulated in ILs exceeds $2x10^{-7}$ and $5x10^{-4}$ (≧0.1dpa), respectively. ➔ vacancy concentration (C_V) > $5x10^{-4}$
- Small ILs align and change to large IL by coalescing each other.

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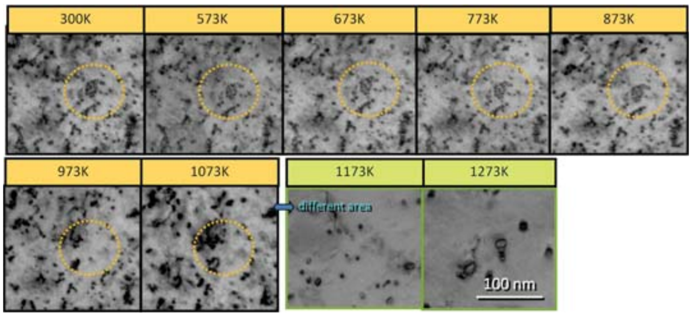
1.2 Thermal stability of interstitial loops

300K , 2.4MeV-Cu^{2+}, 1.0dpa, about $1x10^{-4}$dpa/s, isochronally annealed 25min/100K



- No remarkable change up to 873K but ILs start to change their shape and annihilate gradually above 973K.
- This indicates that fine vacancy clusters accumulated in the matrix become mobile or thermally unstable (break-up) above 973K.
- Remarkable annihilation of ILs occurs above 1173K by absorbing the vacancies.

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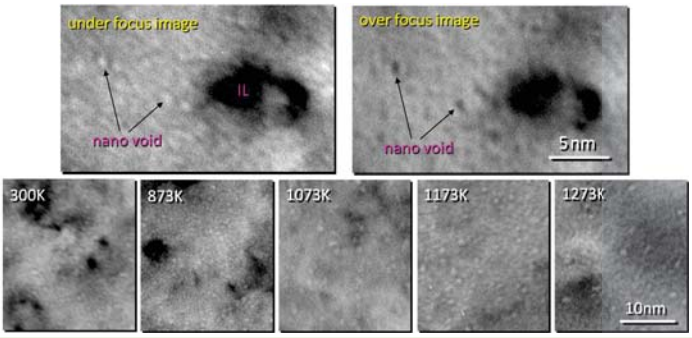
1.3 Formation of Voids and their Thermal Stability

300K , 2.4MeV-Cu^{2+}, 1.0dpa, about $1x10^{-4}$dpa/s, isochronally annealed 25min/100K



- Nano-voids (d<1nm) are formed densely in a thin part of the TEM sample. 🡐 effect of cascade collisions and radiation induced diffusion.
- Nano voids start to grow above 1073K but their density decreases remarkably above 1273K. ➔Highly accumulated vacancy clusters become mobile above 873K-1073K and even nano-voids becomes unstable above 1273K.

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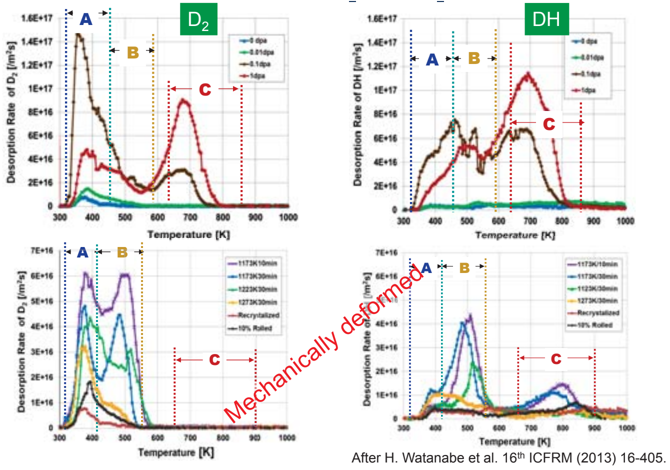
1.4 TDS of Imp. D from Cu ion Irradiated W (RC-W)

2.4MeV-Cu^{2+}, 0.01~1.0dpa @300K + 2keV-D_2^+, $1x10^{21}D_2^+/m^2$ @300K



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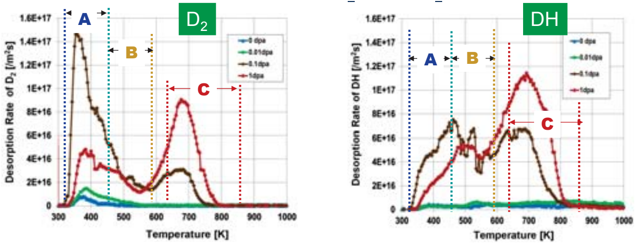
1.4 TDS of Imp. D from Cu^+ Irradiated W

2.4MeV-Cu^{2+}, 0.01~1.0dpa @300K + 2keV-D_2^+, $1x10^{21}D_2^+/m^2$ @300K



- With increasing irradiation dose of Cu ions, trapping of A, B and C increases.
- Especially trapping of A at 0.1dpa and C at 1dpa are remarkable.
- Retention of D (1dpa): Peak C > Peak A > Peak B
- Judging from the TEM observation and the computer simulation, trapping sites of A and C are vacancies (+ their very small clusters?) and nano voids, respectively.

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1.5 Summary of Cu ion irradiation

- Three remarkable desorption peak of D (D_2, DH), named Peak A, Peak B and Peak C, appear commonly in W with different treatments.
- Trapping sites corresponding to each peak are speculated as following:
  - Peak A (330~420K) : In addition of adsorption at the surface, single vacancies and their very fine clusters act as trapping for D^+.
  - Peak B (420~560K) : Kinks of dislocations is one of the candidate of the corresponding trapping sites.
  - Peak C (660~900K) : Very fine vacancy clusters and nano-voids for D_2. Trapping energy was estimated to be 1.8eV.
- In the case of high energy Cu^+ irradiation, very dense defects are accumulated above 0.1dpa and they act as effective trapping site for D.
- Especially highly accumulated vacancies, their clusters, interstitial loops near the incident surface act as good trapping site.
- Formation of fine vacancy clusters and nano-voids even at low temperatures where vacancies can not migrate thermally, is very important from the standpoint of T retention. In the case of irradiation with high energy heavy ions and neutrons, in which cascade collisions occur often, fine vacancy clusters are formed even at low fluence and at low temperature.
- Mechanically deformed W has also Peak C trapping because the vacancies and their fine clusters are formed by the crossing of dislocations. To diminish the strong trapping of H isotope at Peak C, the mechanically deformed W should be annealed well above 1223K.

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Some of results achieved to date

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2. Low energy and high flux plasma irradiation to W
3. In situ measurement of hydrogen isotope retention using ion beam analysis

Exposed to the low energy and high flux plasma

Compact PSI Simulator APSEDAS

- Cylindrical plasma (ϕ~50mm) is produced by RF wave of 13.56 MHz.

Typical parameters of D plasma exposure

n_e ~ 2.4 x 10^{18} m^{-3} , T_e ~ 8 eV ,
Ei ~ 30 eV, flux ~ 3 x 10^{21} $m^{-2}s^{-1}$
Ts ~ 500K (P_{RF} = 0.8 kW)

Achieved D Plasma parameters (pulsed)

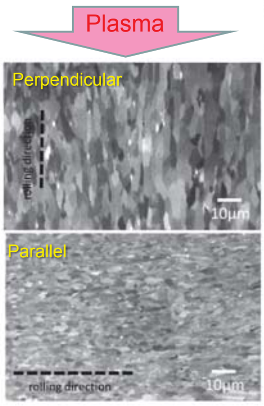
n_e ~ 3.0 x 10^{18} m^{-3} , T_e ~ 16 eV ,
flux ~ 4.1 x 10^{22} $m^{-2}s^{-1}$
(P_{RF} = 3.5 kW, P = 20mTorr)

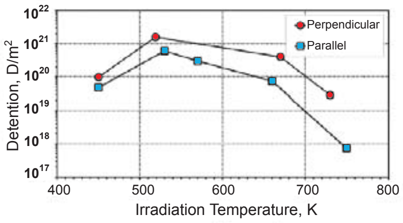
- Fluence dependence
- Surface temperature dependence

Magnetic field < 0.05 T

Exposed to the low energy and high flux plasma

Fluence: 2 × 10^{25} D/m^2
Flux: 3 × 10^{21} D/m^{2}s

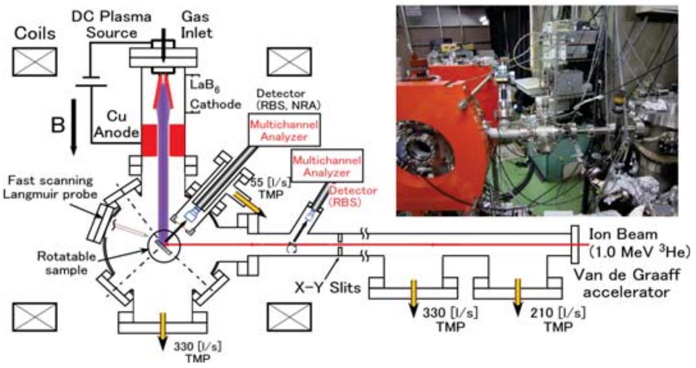




- Dependence has a maximum at around *T*irr ~ 550K.
- The retention in the perpendicular sample is larger than that in the parallel one for all irradiation temperatures.
- It was suggested that the difference in the retention can be caused by different migration ability or effective diffusion coefficient of deuterium atoms along and across to the grain elongation.

A. Rusinov, M. Sakamoto et al. Plasma and Fusion Research Vol.7 (2012) 1405105.

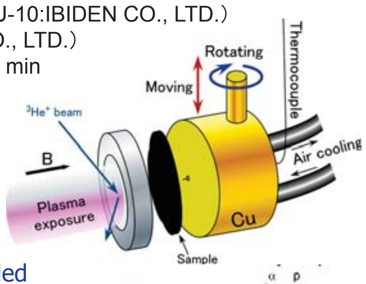
Plasma Surface Dynamics with Ion Beam Analysis (PS-DIBA)



i) Compact and powerful plasma source
ii) Differential pumping to protect detectors and Van de Graaff accelerator
iii) Ion beam monitoring system during plasma exposure
iv) Sample temperature was controlled by air cooling

Experimental Setup

Specimen：Isotropic Graphite（ETU-10:IBIDEN CO., LTD.）
Tungsten（A.L.M.T. CO., LTD.）
Annealing 900℃ 10 min



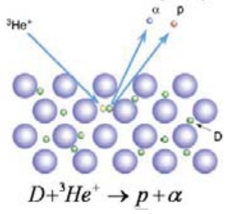
・Deuterium Plasma

Incident Ion Energy E_i :30 eV
Flux Γ_D :~10^{22} m^{-2}s^{-1}
Surface Temp.T_s :350 - 550 K

・Surface temperature is controlled
by air cooling during plasma exposure
by electron beam heating after plasma exposure

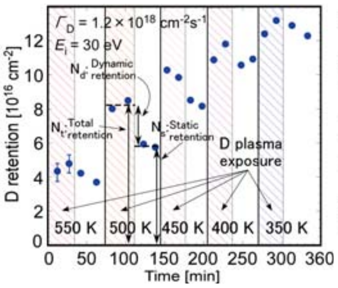
・Deuterium retention analyzed by NRA

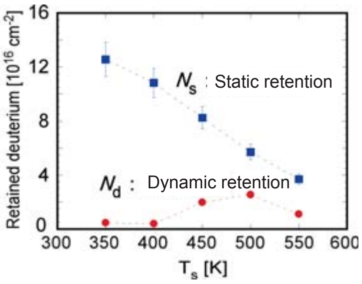
Detection depth 2.5μm: Isotropic Graphite
1.2μm：Tungsten



In-situ Measurement of Deuterium Retention in W

Tungsten（A.L.M.T. CO., LTD.）Annealing 900℃ 10 min





Static and dynamic retention in W can successfully be evaluated as well as C.

Static retention decreases with surface temperature. More experiments are necessary.

"Study of hydrogen isotopes behavior in carbon based materials with in situ ion beam analysis under plasma exposure", Y. Nakamura, M. Yamagiwa, T. Kaneko, N. Matsunami, N. Ohno et al., Journal of nuclear materials, Vol. 438 (2013) S1036-S1039 .

"In situ measurement of hydrogen isotope retention using a high heat flux plasma generator with ion beam analysis", M. Yamagiwa, Y. Nakamura, N. Matsunami, N. Ohno, et al., Physica Scripta, Vol. 2011 (2011) 014032 .

Work plan

- At first, Cu ion beam is used as a surrogate for neutron irradiation. By changing the dpa level up to 10 dpa, relation between the size and density of the radiation damage of tungsten and the dpa level is investigated.
- The damaged tungsten sample is exposed to the high flux D plasma in APSEDAS and then the hydrogen isotope retention in the sample is examined by TDS.
- The dynamic retention properties of the damaged tungsten is also studied in PS-DIBA by using in situ measurement of hydrogen isotope retention during high-density plasma exposure with ion beam analysis.

Work plan (Continued)

- Preparation and tune up of the 1 MeV tandem accelerator is made to obtain the W ion beam. The same experiments mentioned above are carried out using W ion beam.
- Simultaneous irradiation of W ion beam and D or He ion beam is done to investigate the synergistic effect on the microstructure of W and hydrogen isotope retention.
- Hydrogen isotope exchange experiments are also carried out by D plasma and H plasma exposures in sequence to study the effect of tritium removal from the damaged W sample.

Work plan (Continued)

- In addition, the tungsten samples exposed to the plasma confined in LHD, GAMMA 10 and QUEST are irradiated by the W ion beam and then hydrogen isotope retention property is measured by means of D plasma exposure and TDS to investigate the effect of complex circumstances in the actual confined plasma on the hydrogen isotope retention in the damaged tungsten.