Material mixing of tungsten with low Z materials -Carbon and Helium-

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Outline of this talk

- Material mixing of C & W : introduction
- **C** deposition on W
- Effects of C & W mixing on retention/blistering
- Effects of simultaneous He bombardment to W on retention/blistering

Wall material selection in ITER

- CFC : T retention problem (associated with significant erosion) could greatly reduces DT shots number
 - J Tungsten : several concerns such as Melting, Cracking, Helium embrittlement, Core plasma contamination.

- In terms of T retention, a full W wall is a better choice.
 - But, several issues need to be settled for the use of full W
 - In H phase, W, C and Be are used to learn ITER plasma operation toward full W.
- ☐ W+C (CFC) system is still one option for DT operation.
- Material mixing of C and W is a very important subject.
- In addition, <u>He mixing effects</u> are significant in T retention.
- W-Be and C-Be mixing are also an important issues.
 - Be issues will be discussed in Prof. Tynan's talk.

Research on W & C material mixing

Many basic studies have been done in $C+D\rightarrow W$, but still quite a few remaining issues (deposition, effects on retention)

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Several complicated processes need to be considered.

Dynamic mixing process

- Mixed layer formation during ion irradiation (non-equilibrium state)
- Multiple ion irradiation (D/T, He, Ar(edge cooling), C(wall), O, etc.)
- Thermal processes of C (W, D) in mixed layer
- Chemical sputtering of C in mixed layer and deposition layer
 - Depending on chemical state and micro-structure
- Ion radiation enhanced processes
 - Radiation enhanced diffusion and segregation
- Necessary to consider actual conditions
 - Roughness(surface morphology)
 - Surface impurities (Ex. Oxide layer)

Effects of material mixing on T retention/permeation



Balance between C implantation and erosion

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□ C implanted

= <u>C injected</u> – <u>C reflected</u>

□ C erosion

- by <u>all ions</u>
 - Physical sputtering
 - Radiation enhanced sublimation
- by hydrogen isotope ions (oxygen)
 - Chemical sputtering
 - C in mixed layer
 - C in deposition layer
- Sublimation (at elevated temperatures)
- Flaking, exfoliation, or dust emission (for thick D deposition)
- C diffusion into the bulk

The simplest model for C balance (H+C ions) $\Gamma_i f_C \left(1 - R_{CC} \Delta_C - R_{CW} \left(1 - \Delta_C \right) \right) = \Gamma_i \left(\left(1 - f_C \right) Y_{HC} \Delta_C + f_C Y_{CC} \Delta_C \right)$ C reflection by W (2) C sputtered by H(3) C sputtered by C Injected C (4)C reflection by C (1) Δ_{c} : <u>Surface coverage</u> of C (Δ_{c} =1 : fully covered by C) Γ_{i} : lon influx $f_{\rm C}$: C concentration in injected ions R_{CC} : C reflection coefficient on C, R_{CW} : C reflection coefficient on W Y_{HC} : Sputtering yield of C by H, Y_{CC} : Sputtering yield of C by C (self-sputtering yield) More complicated for real system How to determine the thickness of layers for Δ_c Actually, for (1), (2), (3), and (4), thickness is different. Sputtering and reflection are not simple linear function of Δ_c . How does thermal effect play roles?

□ Surface segregation, diffusion



Reflection and phys. sputtering of C on W

Reflection coefficient is lower than that on W

- R~0.6 (50eV C to W)
 - R~**10**-4(50 eV C to C)
- Carbon mono-layer is easily re-sputtered by reflected H from W substrate.
- Carbon deposition is more pronounced on graphite.



10 Computer simulation by EDDY Acknowledgment (K. Ohya) 200 $D^+ + C^{4+}$ mixed ion irradiation to Te_10eV Te_15eV Te_20eV Te_25eV Thickness change (nm) $T_{\rm e} = 10 {\rm eV}$ tungsten 100 Simulated by EDDY code 20 Te_40eV Te 80eV 0 D:96%, C:4% 25 No chemical sputtering -100 40 Thickness change As deposition proceeds, Y_c and R_c 80 drastically decrease. -200 2 2 8 6 6 - 8 10²⁰ 10¹⁹ 10¹⁸ Fluence (cm⁻²) 0.1 0.5 10eV Reflection of C : R_c 15eV Sputtering Yield of C 100 C reflection coefficient 0.4 20eV 25e\ 40e\ 0.3 T_80eV Te_80eV 0.2 Te_40eV Te^{25eV} Te 20eV 0.1 Te 15eV 20 T_e = 10 eV 2 Te¹0eV Sputtering of C:Y_C 0.001 0.0 2 3 4 5 6 2 3 4 5 2 3 4 5 6 4 6 8 68 2 2 2 10²⁰ 10¹⁹ 10²⁰ 10¹⁹ 10¹⁸ 10¹⁸

Fluence (m⁻²)

Fluence (cm⁻²)

Deposition layer : quite different from solid C

- Different structure depending on Temperature, flux, D ratio, etc.
- C deposition layer is not dense (0.91 g/cm³ on JT-60U tiles (2.23 g/cm³ for graphite crystal)).
 - Y. Ishimoto et al., J. Nucl. Mater. 350 (2006) 301.



Structure of C deposition layer (JT-60U)

Some comments on erosion

Chemical erosion of C deposition layer

- Depends on bulk properties (Soft C-H film (H/C~1), Hard C/H film (H/C~0.4))
 - □ W. Jacob, J. Nucl. Mater. 337-339 (2005) 839.
- Local ¹³C deposition experiments and their simulations suggest enhanced re-erosion of C deposition layer.

□ A. Kirschner et al., J. Nucl. Mater. 328 (2004) 62.

Chemical erosion of C in mixed layer with W

- In general, C in mixed layer has lower chemical sputtering yields than graphite.
- Temperature dependent C-selfsputtering was reported. But mechanism is not well known.

□ H. T. Lee, K. Krieger, J. Nucl. Mater. 390–391 (2009) 971.

Carbon deposition on W (TEXTOR test limiter experiments)

TEXTOR – a test bed for power exhaust concepts ...









International Workshop on TEXTOR and PWI 12/10/2007

Forschungszentrum Jülich in der Helmholtz-Gemeinschaft





Experimental conditions for TEXTOR experiments

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Effects of surface roughness on C deposition

- Tungsten
 - **D** Roughness $R_a = 9 \sim 180$ nm
- Graphite (fine grained graphite)
 - **D** Roughness $R_a = 70 \sim 700$ nm
- He plasma pre-exposed W
 - Nano-structure formed
- C deposition on tungsten at elevated temperatures

Temperature range

- ☐ ~300 °C : ~ITER wall
- □ ~550 °C : ~Chemical Sputtering peak
- ~850 °C : Thermal diffusion + RES



Setup for study on surface roughness effects

Pure W samples

- *R*_a~9 nm, ~22 nm, ~180 nm
- Difference in surface polishing
- □ Graphite (fine grained)
 - *R*_a~70 nm, ~350 nm, ~700 nm
- Deposition mechanism
 - Lower T_e deeper into SOL
 - Higher carbon density deeper into SOL





C deposition and D retention on W

C deposition

- Roughness enhances C deposition
- R_a~180 nm : Long tail
- Sharpe boundary between erosion and deposition
- **D** retention

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- similar to C deposition
- no surface retention in erosion zone
- D/C = 0.1~0.15



W Graphite



D/C ratio in C deposition layer

For the roughest case (R_a = 180 mm), the region of D/C
 ~ 0.1-0.15 extends to r ~ 46.5 cm, suggesting thin C
 deposition layer exists over wide area of the sample.



D retention (C deposition) on graphite

C deposition on graphite

- D retention was mainly in C deposition layer
- D/C ~ const in deposition layer
 - D retention ~ C deposition
- Characteristics of
 C deposition on graphite
 - Roughness enhanced C deposition also on graphite
 - No sharp transition between erosion and deposition



Sharp C deposition- erosion boundary on W

- C deposition rate is much higher on C than W.
- Once C deposition layer is formed, the deposition rate increases.



23 ¹³CH₄ puff exp. with graphite limiter (TEXTOR) C deposition on graphite test (d)limiter (TEXTOR exp.) Deposition Efficiency α Deposited ¹³C /injected ¹³CH₄ C on unpolished C ($R_a \sim 1 \mu m$) Unpolished $\Box \alpha \sim 9\%$ *α* ~9% $R_{\rm a}$ ~1 µm C on polished C ($R_a \sim 0.1 \mu m$) film deposited near injection aperture background deposition $\Box \alpha \sim 1.7\%$ (e) Surface roughness seems to affect C deposition toroidal direction 130 mm Similar or larger than substrate Poloidal direction 80 mm effects (W or graphite) Polished $\alpha \sim 1.7\%$ *R*_a ~0.1 μm Ohmic discharge

A. Kreter, et al., Plasma Phys. Control. Fusion (2008)

C deposition on He pre-exposed W

He plasma pre-exposure

- High density pure He plasma exposure in NAGDIS-II (Nagoya U.)
- Black surface after ~1h exposure at 1300 °C (flux ~10²³ m⁻²s⁻¹)
 - He bubble and nanostructure formation
- Surface structure removed before TEXTOR plasma exposure
 - Loosely bound nano-structure was wiped out mechanically

Roughness of He exposed W

- Roughness ~15 nm (after exp.)
 - □Small pits could be missing due to stylus type measurement



M. Baldwin et al., I-20, PSI18



W surface in this work

C deposition on He pre-exposed W

- He pre-exposed W
 - Enhancement of C deposition
 - C profile : long tail
 - increase in deposition area
 - large enhancement of deposition despite small roughness (~15 nm)
- H+C pre-irradiated W
 - C deposition speed relates to surface C concentration
 - only 10% initial C affects deposition
 - No deposition on pure W (0%C)
 - $R_{\rm a} \sim 10 \text{ nm}$ for each W



46 shots (Ohmic plasma) r = 46 cm (same as LCFS)



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After

Explanation of roughness effect on deposition

C Roughness (0.01-1 μ m) << Ion Lamor radius (0.1-1mm)

- D ion flux and C ion flux did not change locally
- Local shading effect of D ions may not occur
- Some of sputtered or reflected particles redeposited immediately.
 - Trapping rate depends on the morphology
 - He roughened surface was very fine and complicated structure
 - □ He induced roughness could have high trapping rate (C deposition)



M. Kunster et al., Nucl. Instrum. Meth.B145 (1998)320.

He roughened W surface

Experimental conditions for TEXTOR experiments

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Effects of surface roughness on C deposition

Tungsten

- **Roughness** $R_a = 9 \sim 180 \text{ nm}$
- Graphite (fine grained graphite)
 - Roughness $R_a = 70 \sim 700$ nm
- High density He plasma pre-exposed W
 - Nano-structure formed

C deposition on tungsten at elevated temperature

Temperature range

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Comparison with previous C deposition data



Partially heated limiter exp. for C deposition on W



1 mm

A-A' cross section

Graphite bond

Thermòcouple

Deposition by edge plasma exposure Deposition due to "gas puff" (CO)

non-Heated

280 →340 °C

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No deposition on the heated sample.

CO gas : desorbed above ~700 °C







Retention/blistering by simultaneous C/He/D exposure



can enhance blister formation on W.

Experimental conditions <u>Beam Energy</u>: 1keV H₃⁺, <u>Flux</u> : (3-4)x10²⁰ Hm⁻²s⁻¹ <u>Temperature</u> : 653 K <u>Sample</u> : pure W with mirror polished

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Blisters without gaps

Blisters without gaps (or small gaps) are reported lately.

- Formation mechanism is not known.
 - Abnormal diffusion of W?
 - Giant swelling due to high flux D/H irradiation?



W and C mixing layer reduced desorption

C depth distribution

- Absolutely calibrated by NRA
- broader than ion implantation range
- Recoil implantation by H
- □ High C (~0.9% in the beam) case
 - WC layer reduced desorption of H
 - Enhance bulk diffusion of H
 - Enhance blister formation

- Low C (~0.1% in the beam) case
 - Low surface C concentration
 - no significant reduction of recombination







D & C mixed plasma exposure to W

Planar DC magnetron plasma

- Energy : $\sim 200 \text{ eV} (D_2^+ \text{ mainly})$
- Flux : 1 x 10²¹ m⁻²s⁻¹
- C plate on cathode surface to provides C into plasma
- For D+C, D retention near surface (a) and bulk (b) increased at elevated temp.
- For D+C, fraction of C on W surface is higher.
- Possibly, surface C+W mixed layer (C existed as carbidic and graphitic phases) reduces release of D from surface.



From 300-700 K, thin and thick layers of Be suppresses blister formation. M. Baldwin et al. PSI 18(2008)

- Blistering & exfoliation of blister caps is a concern for certain varieties of W.
- Increased retention is associated with the trapping of hydrogen in blisters.

E.g. *K Tokunaga et al. J. Nucl. Mater.* (2004) <u>337–339</u>, 887.

- At 550 K a blistered surface is / prevalent after exposure to D₂ plasma.
- A thin layer of Be as little as a few 10's of nm, or thicker, is found to suppress blister formation.



CODE Aerospace Engineering

He effects on W

□ High temperature (> ~1,600 K)

- Large He bubbles formation with recrystallization
- Degradation of mechanical and thermal properties
- Dust formation (enhanced erosion)
- □ Medium temperature (> ~1,100 K)
 - Nano-structure formation
 - Dust formation (enhanced erosion)
 - Initiation of arcing
- □ Low temperature (< ~900 K)
 - Small He bubble formation (a few nm)
 - Significantly affects D/T retention and diffusion



RN09272005 (d) 9000 s

T ~ 1,600 K



Submicron structure on W (T~1250 K)



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From Prof. Takamura presentation at ITPA sol./div meeting, Toronto, Nov. 2006.

Basic Behavior of He in W

Y. Yoshida (Kyushu U) 18th PSI (2008)

- Very low solubility.
- Very fast thermal migration via interstitial sites (very high mobility even at R. Temp.)
- Very deep trapping in a vacancy (Large E_V^B)
- He enhances the formation of voids (bubbles) and dislocation loops even above 1000°C →hardening, embitterment
- He atoms can aggregate by themselves → He atoms can form clusters once get in the lattice (E>E^s_I) → no need displacement damage



He effect on retention

Sequential irradiation of He and D.

Formation of He bubbles enhances D retention very much.

- He bubbles become traps of D.
 - (H. Iwakiri et al., J. Nucl. Mater. 307-311 (2002) 135-138)







Depth (nm)

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TEM observation of near surface structure





M. Miyamoto et al., Nucl. Fusion 49 (2009) 065035 (7pp).

Blisters disappeared above 5% He.

D retention



TDS after plasma exposure

✓ High resolution Q-Mass (D_2 and He can be separated).

✓ Heating rate : 0.59 K/s

D.L. (Detection Limit) ~10¹⁸ m⁻²





Is He bubble layer a diffusion barrier?

For low energy He implantation (less than recoil threshold), He bubble layer could extend deeper and become the barrier.







Possible mechanisms

- Narrowing diffusion channels by He bubbles
- Release of D² through He created pores
- Reduction of diffusion through stress-induced W



Summary

There still remain unsolved problems in C & W mixing.

- C erosion from C&W mixed layer and C deposition layer
- Effects of surface morphology (roughness) on C deposition
- C atom behavior at elevated temperatures (> 800 °C)
- C & W mixed layer strongly affects D behavior
 - W&C mixed surface layer reduces recombination of D atoms
 - Diffusion of D in W&C mixed layer is reduced compared to pure W
- He bubble layer strongly affects D retention
 - Initially increase retention by increasing trapping sites
 - But, under high fluence condition, He bubbles greatly reduce retention as they work as diffusion barrier.
- We do not have enough knowledge on material mixing to correctly evaluate T retention in ITER.