Plasma–Wall Interaction for Irradiated Tungsten and Tungsten Alloys in Fusion Devices

Summary and progress report regarding TPD/TDS round robin experiment held at PSI-22 Rome

The Pontifical Urbaniana University, Rome, Italy
Thursday, June 2nd 13:00~13:45

Report prepared by H.T. Lee[1], W. Jacob[2], and H.K. Chung[3]

[1] Graduate School of Engineering, Osaka University, Japan
[2] Max Planck Institut für Plasmaphysik, Garching, Germany
[3] International Atomic Energy Agency, Vienna, Austria

Executive Summary

Progress on the status of calibration samples to be used in the thermal desorption spectroscopy (TDS) round robin experiments were reported to participants. Technical details of the samples were presented by W. Jacob (IPP, Germany) and feedback from participants received. Issues with respect to sample size and D content were discussed (~50 nm thick W:D layer on W substrate). Sample distribution timeline was estimated to be within three months. Participants agreed in principle to complete the exercise prior to the last CRP meeting to be held in early 2017. A brief discussion of the experimental conditions (ramping rate of 0.1-1 K/s, delay time following sample distribution) was discussed with the finalization of such details to be performed via email coordinated by H.T. Lee (Osaka U, Japan). A brief update on the timeline of code comparison exercise was presented by M. Shimada (INL, USA). This meeting follows from the Second Research Coordination Meeting of the IAEA Coordinated Research Project on Plasma–Wall Interaction for Irradiated Tungsten and Tungsten Alloys in Fusion Devices held in Seoul, Republic of Korea, in September 2015. Fourteen projects participating in the CRP were represented as well as group from University of Toronto.

1. Introduction

A short meeting regarding the present status of calibration samples to be used in thermal desorption spectroscopy (TDS) round robin experiment was held at PSI-22, Thursday, June 2nd (13:00~13:45). The purpose of this exercise is to perform benchmark experiment in various TPD/TDS devices with a common sample prepared under controlled conditions. IPP, Garching has graciously volunteered to prepare such samples. A more in-depth description of the background and need of this round robin experiment can be found in the "Summary Report of the Second Research Coordination Meeting" prepared by H.K. Chung and B. J. Braams (2016).

The meeting featured progress report on sample preparation by W. Jacob (IPP) with the purpose of receiving feedback from participants and finalizing the technical details prior to mass production of samples for distribution. 14 of the 19 projects in the CRP and one guest (U of Toronto) were represented. In addition the meeting featured brief status update on the code comparison project for TPD/TDS by M. Shimada (INL).

A short summary of the proceedings and discussion of the meeting is provided in Section 2. The meeting agenda is given in Appendix 1, and the list of participants is in Appendix 2. Summary of the technical details of the sample provided as a handout at the meeting is in Appendix 3.

2. Proceeding and Discussion

The meeting was led by H.T. Lee and W. Jacob with input from M. Shimada, and organized by H.K. Chung according to the agenda in Appendix 1.

W. Jacob first provided a brief overview of the sample preparation method by deposited magnetron sputtered W layers on Si substrates and first characterization of D content using nuclear reaction analysis (NRA) and TDS. The technical details are found in Appendix 3. Afterwards, concerns and questions regarding the samples were solicited and the following discussion points were raised and where indicated *consensus* reached (*in italics*):

1. **Sample preparation method:** *All participants agreed that D-containing W films (DcW) would work*. W. Jacob clarified that in contrast to the technical details outlined in Appendix 3, the samples distributed will be DcW layers on W-substrates (not Si substrates). These W substrates are cut from well characterized W batch, polished mechanically to a mirror-like finish, and outgassed at 1 h at 1200 K for stress relief. Issues related to stability of the DcW were raised by A. Pisarev. W. Jacob replied that the films would be thin enough ~50 nm and deposited with sample bias to increase the adhesion.

2. **Sample size:** Sample size of 10×10×0.8 mm is planned for mass production by IPP. At least 3 groups voiced concerns and preferred smaller sizes. Where possible IPP will try to accommodate special requests but it is within their discretion. An EXCEL sheet will be sent out to participants to confirm they can handle 10×10×0.8 mm samples and if not, to clearly communicate their special needs.

3. **D content:** *All participants agreed that 1×10^{16} D-atoms would be measurable.*

4. **Special requests:**
   - **a.** U of Toronto, MEPhI, and CEA group requested samples with un-deposited area on the substrate, which would allow for spotwelding of a thermocouple.
   - **b.** CEA group requested one sample without DcW coating (i.e W substrate only)
   - **c.** Prof. Hatano requested 6 mm disc samples for TDS system used to measure neutron irradiated samples at Oarai, Japan.
   - **d.** Prof. Wirth requests 5~5.5 mm diameter or 5×5 mm square samples.

5. **Number of samples:** *All participants agreed on 2 samples/group.* This limit is set to reduce the workload on IPP for sample preparation but to check for reproducibility in each group's system.

6. **Outgassing issues and changes to D content:** Due to the nature of the preparation method, samples will outgas resulting in reduction of D content. To minimize such efforts it was agreed that experiments should be coordinated and performed within an agreed time window (e.g. one month) following sample distribution. However, due to the impracticality of enforcing such constraint it was agreed upon that all participants *clearly note the delay time between sample distribution and actual experiments*. It was suggested that IPP holds onto the sample for approximately one month following deposition prior to shipment to all participants [C .Taylor].

7. **Ramping rate:** *All participants agreed that a ramping rate in the range 0.1-1 K/s was possible.* This will be confirmed by survey email with an EXCEL sheet to confirm the operational parameters of each TDS setup.

8. **Miscellaneous:** B. Unterberg requested to be added to the email list.

Next, M. Shimada briefly described the status of the code comparison exercise and informed that the modeling exercise should proceed within three-month time window. Finally, H.T Lee summarized the meeting by informing everybody that a summary of the meeting will be emailed to all participants including the technical details of the sample and an EXCEL file to survey the TDS operational parameters.

Appendix 1

Agenda

The Pontifical Urbaniana University, Rome, Italy

Sala Seminari/Sala Colloqui V, Thursday, June 2nd 13:00~13:45

Introduction (H.T. Lee, Osaka U)– 5 min

- 14 groups represented in today's meeting.
- Update on number of participants: Jim W. Davis + Tamara Finlay (U of T)
- Purpose of the short meeting.

Sample details (W. Jacob, IPP) – 20 min

- Explanation of technical details: D content, size.
- Feedback regarding samples.
- Timeline of distribution.

Discussion/Feedback (H.T. Lee, Osaka U) – 5 min

- Timeline of TDS experiments following distribution.
- TDS conditions: ramping rate.

Code comparison exercise (M. Shimada, INL) – 10 min

- Present status and timeline.
- Possible data set(s) to model.

Summary – 5 min

- Distribution of meeting summary report, technical details, and EXCEL file for surveying TDS operational parameters following meeting.

Appendix 2

List of Participants

Alexander Pisarev, National Research Nuclear University "MEPhI", Moscow, Russian Federation

Brian D. Wirth, University of Tennessee, USA

Christian Grisolia, R. Bisson, E. Hodille, Commissariat a l"Energie Atomique, Cadarache, France/ Aix-Marseille University, France

Davide Curreli, University of Illinois at Urbana Champaign, Urbana IL, USA

Guang-Nan Luo, Institute of Plasma Physics, Chinese Academy of Sciences, Hefei, People's Republic of China

Heun Tae Lee, Osaka University, Osaka, Japan

Jim W. Davis, Tamara Finlay, University of Toronto, Canada

Jochen Linke, Bernard Unterberg, Forschungszentrum Jülich, Germany

Long Cheng, Beihang University, Beijing, People's Republic of China

Masashi Shimada, Chase Taylor, Idaho National Laboratory, USA

Mizuki Sakamoto, Plasma Research Center, University of Tsukuba, Japan

Sabina Markelj, Josef Stefan Institute, Ljubljana, Slovenia

Yuji Hatano, Toyama University, Japan

Yong-Gil Kim, Seoul National University, South Korea

Wolfgang Jacob, Thomas Schwarz-Selinger, Max-Planck-Institut für Plasmaphysiik Garching, Germany

Hyun-Kyung Chung, International Atomic Energy Agency, Vienna, Austria

Appendix 3

Status of the plans regarding a

TDS Round Robin Experiment (TDS-RRE) within the CRP on damaged tungsten (prepared by W.Jacob, IPP, Germany)

- During the meeting in Seoul (2 RCM of CRP on Irradiated Tungsten) it was decided to carry out a TDS Round Robin experiment.
- Heun Lee [HL] (heunlee@st.eie.eng.osaka-u.ac.jp) volunteered to coordinate the task and W. Jacob [WJ] (Wolfgang.Jacob@ipp.mpg.de) volunteered to produce reference samples.
- The basic aim is to compare TDS spectra measured with different TDS set-ups.
- For this a set of identical reference samples is necessary.
- Initially it was suggested by WJ to produce reference samples by plasma loading in the PlaQ decvice (see: *A. Manhard, T. Schwarz-Selinger, and W. Jacob: "Quantification of the Deuterium Ion Fluxes from a Plasma Source", Plasma Sources Science and Technology 20, 015010 (9pp) (2011). doi:10.1088/0963-0252/20/1/015010*). In PlaQ up to 5 samples can be exposed simultaneously to a well-quantified deuterium ECR plasma.
- However, the large number of participants in the TDS-RRE and the correspondingly large number of required samples makes this option not attractive.
- WJ and TSS (Thomas Schwarz-Selinger) started to think about alternative sample production routes that would allow producing a larger set of identical samples.
- They suggested depositing magnetron-sputtered W layers in a D-containing atmosphere.
- Finally first tests were made in March/April 2016. So far (status May 17, 2016) the following was achieved:
  - D-containing W films (DcW films) were deposited on silicon samples
  - 3 different types of DcW films were produced by magnetron sputtering in a mixture of Ar and D:
    Deposition time: 15 min / 30 min – no bias on substrate (approx. thicknesses 70 and 140 nm)
    Deposition time: 15 min – 135 V bias on substrate (approx. thicknesses 50 nm)
  - According to the De Temmerman scaling we anticipate a D concentration in the DcW-15min-135V films of about 0.5% (*G. De Temmerman, R.P. Doerner, "Deuterium retention and release in tungsten co-deposited layers", J. Nucl. Mat. 389 (2009) 479–483, doi: 10.1016/j.jnucmat.2009.03.028*).
  - The layers were analyzed by **^{3}He NRA** (nuclear reaction analysis) resulting in the following total D amounts:
    DcW-15min-0V = 4.6×10^{19} D-atoms per m^2
    DcW-30min-0V = 9.5×10^{19} D-atoms per m^2
    DcW-15min-135V = 12.6×10^{19} D-atoms per m^2

- First **TDS** spectra were recorded using samples: DcW-15min-0V and DcW-15min-135V (see Fig. 1). The peak height ratio is roughly 1210:2870 (1:2.4). This is reasonably close to the NRA ratio (1:2.7).
- A rough absolute calibration of the TDS signal (integration the 3 and 4 amu signal from start to the end of the T ramp, no background subtraction) results in:
  DcW-15min-0V = 1.1×10^{19} D-molecules per m^2 and 1.0×10^{19} HD per m^2
  DcW-15min-135V = 3.5×10^{19} D-molecules per m^2 and 2.2×10^{19} HD per m^2
  The sample area is approx. $1cm^2$, the corresponding total released D amounts are:
  DcW-15min-0V = 3.2×10^{15} D-atoms
  DcW-15min-135V = 9.2×10^{15} D-atoms
  Agreement between NRA and TDS is not very good, but ok for this rough evaluation.

- **We also thought about an alternative production route for reference samples:**
  We have a capacitively coupled RF plasma devices with a large substrate holder (20 cm in diameter). The plasma itself is not characterized, but we know from a-C:H film deposition that the plasma is very homogeneous across this large sample holder. On this holder we can easily accommodate 50 samples and more. Although we do not know the particle fluxes, flux composition and substrate temperature during deposition, we are convinced that all samples will finally receive the identical D fluence and should therefore have the identical D content. The remaining uncertainty in this experiment would be the sample temperature. Different samples could have different thermal contact to the substrate and by this have different temperatures during deposition. Since T during deposition has a huge influence on the total retained D amounts, this could lead to significant variations of the D content. A resort for this problem could be to measure all samples by NRA (one ^{3}He energy should be sufficient to check if they all have the same D content) prior to shipping them.

It would be good to meet with the participants in the TDS-RRE during the PSI-22 conference in Rome and discuss further details (samples, layer thickness, total anticipated D amount, timing, ….)

- We suggest to deposit DcW films on bulk W samples (MF reference W [Plansee] $10\times10\times0.8$ mm^3, polished to mirror-like finish, degassed for 1 h at 1200 K). If some parties would prefer Si samples we could also try that, but adhesion might be problematic.
- How much D (absolute amount) should be in the samples?

- Is the present amount of about 1×10^{16} D-atoms sufficient or should we aim for a higher amount? (This amount corresponds roughly to the total amount of D retained in W).
- Prior to "mass production" the adhesion of DcW on W substrates need to be checked.
- Are suggested sample sizes and pretreatments ok?
- How many samples are needed?

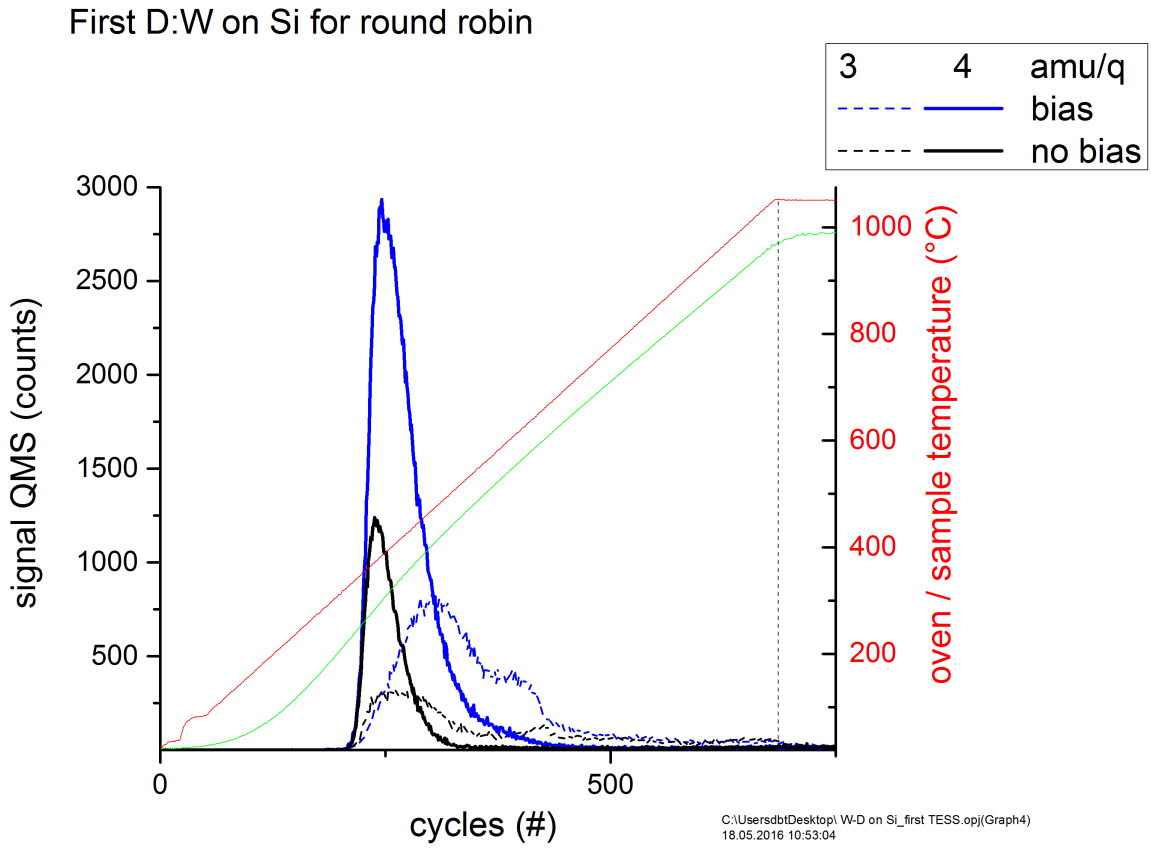

Fig 1: First TDS spectra of DcW-15min-0V and DcW-15min-135V. Please observe: The spectra are not calibrated. The x axis is the number of measurement cycles which is approximately time. The red line is the oven temperature and the green line the estimated sample temperature (This is a calibration curve from earlier measurements using Si samples and has to be checked for the current data). The peak maxima are: DcW-15min-0V = 1210 and DcW-15min-135V = 2870. The HD signals are also shown. Their peaks are shifted and they have a second peak at higher T. The estimated peak temperature of the 4 amu signal is 307 °C ≈ 580 K (±20 K).