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1st MPEX Users Research Forum (MURF) summary report: specifications for the Surface Analysis Station (SAS)

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April 13, 2020

OAK RIDGE NATIONAL LABORATORY
MANAGED BY UT-BATTELLE FOR THE US DEPARTMENT OF ENERGY

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Material Plasma Exposure eXperiment (MPEX) program; Fusion Energy Division

1ST MPEX USERS RESEARCH FORUM (MURF) SUMMARY REPORT: SPECIFICATIONS FOR THE SURFACE ANALYSIS STATION (SAS)

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ABSTRACT

The Material Plasma Exposure eXperiment (MPEX) Team has reached out to the community of potential users of the MPEX facility to assess the high-priority capabilities which should be considered for inclusion in the design of the MPEX Surface Analysis Station (SAS). This activity is being carried out during the "advanced conceptual design" period of MPEX, i.e. post Conceptual Design Review (CDR) but prior to Preliminary Design (PD), to respond to a recommendation by the U.S. D.O.E. following the CDR. The MPEX Team will summarize the community recommendations into a guidance document (i.e. this report), which will influence the Surface Analysis Station during the Preliminary Design phase of the project.

1. INTRODUCTION

Many community reports addressing compelling research & development (R&D) issues for magnetically confined fusion (MCF) plasmas have identified a "plasma facing component (PFC) gap": an engineered material surface as part of the "first wall" of a fusion device is needed to enable successful, commercial production of energy, but such a material/structure is currently lacking. The ideal solution to the PFC gap is a PFC with surface material that will survive (i.e. not suffer degraded performance) during sustained exposure to fusion-relevant plasmas. Candidate engineered materials need to be identified and verified prior to reactor PFC design, since proven material characteristics are required to justify design choices. The confidence of fusion reactor designers that the PFC gap has been closed will arise from a comprehensive R&D program that provides an engineered material and PFCs that meet verified experimental test conditions. Since comprehensive diagnosing of the first wall of a fully integrated test is unlikely, such as in a fusion reactor, test beds are required where specific aspects of the "PFC gap" can be examined through directed R&D. MPEX (Figure 1) represents a test bed capable of performing such R&D, wherein the facility will be designed to provide good diagnostic access to make measurement of plasma material interaction (PMI) effects for conditions relevant to magnetic confinement fusion reactors.

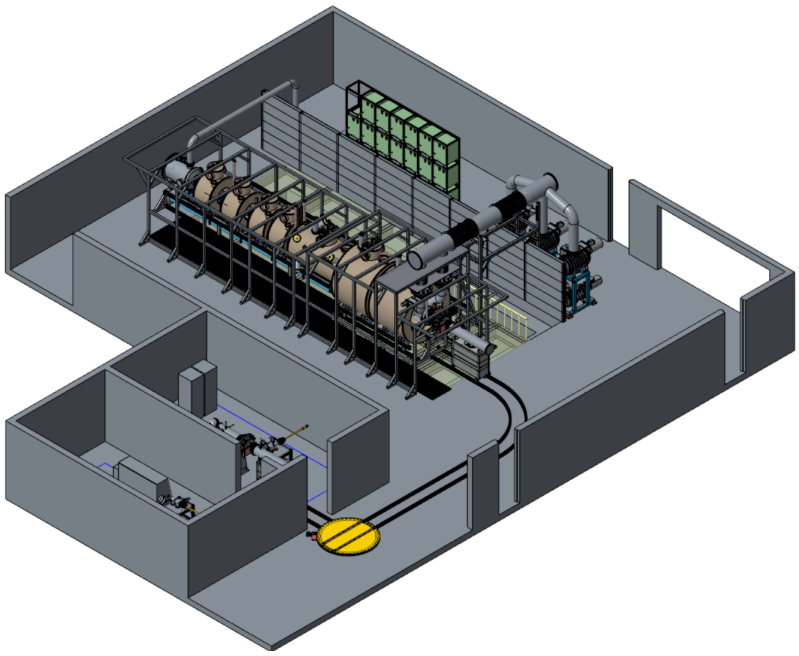

Figure 1: The conceptual design of the MPEX facility in Building 7625 at ORNL.

The structure of this report is as follows: Section 1.1 will give background information on the logic that motivates the conceptual design of the MPEX Surface Analysis Station. Section 1.2 will describe the MPEX Users Research Forum (MURF), and the process by which community input was solicited as input to the preliminary design of the initial MPEX SAS, which included requests in 5 "specification categories." Section 2 will summarize the information that was received for each Specification Category through the MURF process, which will be distilled into inferred "recommendations." Section 3 will list selected Recommendations for easy reference.

1.1 MPEX SAS Conceptual Design Drivers

Ideally, knowledge of the surface evolution of materials during high-heat flux plasma exposure would be readily available and easily accessible (at high spatial and temporal resolution), including information on the evolution of fundamental properties (for example electrical resistivity, thermal conductance, tensile strength, ductility, tritium retention, etc.) of the PFC material in response to the plasma-material interaction. Real-time *in situ* (a.k.a. *in operando*) measurement is the aspirational goal of diagnostic systems. However, the environmental conditions present in plasma generating devices pose constraints on the viability of many commonly available material measurement techniques. For example, the confining magnetic field present in the plasma device is fundamentally incompatible with inspection techniques such as scanning electron microscopy. And the presence of ~ MW/m^2 electron and ion (i.e. plasma) flux onto the surface makes *in operando* measurement of structural properties extremely difficult. Controlled experiments can nevertheless be devised (with commensurate caveats on the data interpretation), where exposed materials are removed further and further from real-time *in situ* conditions, to enable measurements with higher and higher fidelity or detail. This leads to a kind of aspirational hierarchy of measurements, which is driven by the specific data that is sought: 1) *in situ* real-time, 2) *in vacuo*, post exposure, 3) *ex situ*, post exposure and 4) *ex situ, postmortem*.

1.1.1 *In situ,* real-time data

The MPEX Team strives to incorporate real-time *in situ* (i.e. during plasma exposure) diagnostics where possible and where practical. In general, real-time *in situ* techniques (a.k.a. *in operando*) for material characterization are an emerging technology, of which there are only a few demonstrated techniques that address specific material attributes. Some of these (such as Laser Induced Ablation Spectroscopy (LIAS)) inflict micro- to macro-scopic damage to the surface to achieve the measurement (which leads to additional caveats when designing experiments that have an on-going plasma exposure). Other techniques, such as Digital Holographic imaging are non-destructive to surface features. Without going into exhaustive detail on the merits of these and other techniques, the MPEX conceptual design nevertheless has incorporated an array of access ports in the PMI chamber to enable the implementation of real-time *in situ* measurements as they are developed. These systems can be designed at ORNL or in collaboration with outside institutions, commensurate with the level of expertise and the funding provided to support the effort. Many of these advanced systems will not be present in the commissioning phase of MPEX but represent an expansion of capability in the operational phase of MPEX. However, there is an expressed desire by the U.S. D.O.E. ("the sponsor") to have some initial capability in the early stages of MPEX, and some real-time *in situ* measurements (e.g. Digital Holography) have been currently included in the baseline MPEX design. Similarly, while surface analysis is not one of the key performance parameters (KPP) needed to successfully commission MPEX, the ability to soon-thereafter exercise MPEX for scientific experiments means that an initial SAS is included in the diagnostic package for the project.

1.1.2 *In vacuo*, post exposure data

It is straightforward to utilize *in situ* real-time diagnostics to make "*in situ* post exposure" measurements. The measurement geometry is unchanged, but the restrictive presence of the high-heat flux plasma is removed. Without the plasma, the further step of reducing or eliminating the confining magnetic field can also be enacted (if procedurally prudent). For devices with superconducting magnets, it may not be permitted to fully ramp the B-field up/down cyclically above a certain imposed periodicity. In these circumstances though, additional diagnostic techniques can become viable, however access ports may still be limiting. For that reason, many facilities also take the step to move (without breaking vacuum) the plasma-exposed target to a separate analysis chamber with no/low magnetic field and greater diagnostic access. Consequently, *in situ* post exposure measurements can either be made in place, or in a nearby analysis chamber. This principle is employed on linear plasma devices, such as Magnum-PSI, PSI-2, and PISCES-B, and for MAPP experimentation on tokamaks. Generally, these measurements are referred to in this report as "*in vacuo*, post exposure." The vacuum conditions of all regions of the device thus becomes an important variable that must be monitored and controlled when planning experiments. For the MPEX conceptual design, the exposed target will be transported (in vacuum) to a nearby Surface Analysis Station, which is outside the MPEX device environment for *in vacuo*, post exposure analysis.

There are several considerations for transporting (via the target exchange chamber (TEC)) the exposed target from MPEX to the SAS for assessment. 1) The MPEX magnets do not need to be ramped down/up, which reduces the stress on the (high cost, long delivery time) magnets, and thus reduces the lifetime risk over the project. The residual B-field at the SAS is a subsequent design constraint on the instrumentation that can be implemented on the SAS. 2) The instrumentation package of the SAS can be modified without impacting MPEX operations. 3) The SAS can be vibrationally isolated from MPEX. 4) The instrumentation package located away from MPEX can be more extensive (with higher fidelity) than that implemented for *in situ* (real-time or post exposure) measurements. Similarly, the initial SAS could be expanded to consist of multiple analysis stations. 5) In the scenario that multiple target exchange chambers are available, MPEX could continue target exposures independent of surface analysis or target mounting activities.

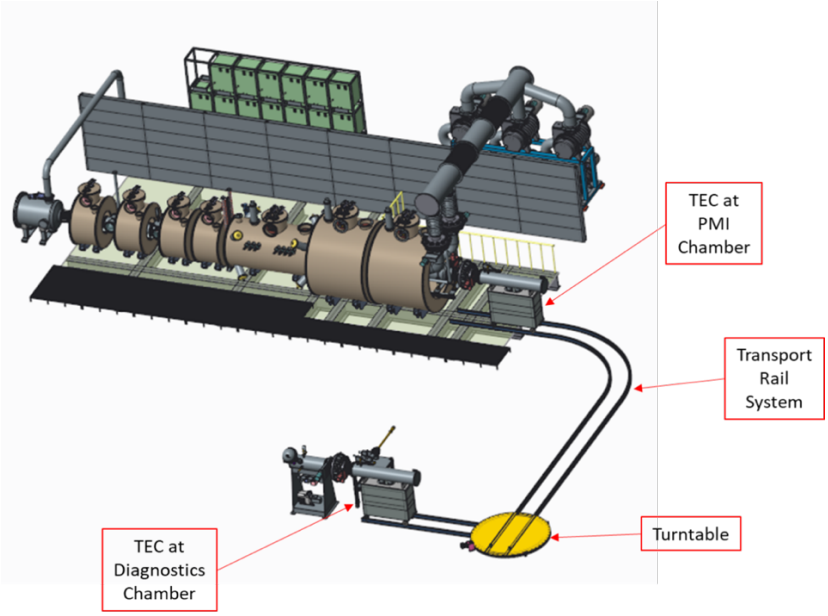

Figure 2: Conceptual design layout of the interface between the MPEX device, the Target Exchange Chamber, and the Surface Analysis Station (here called "Diagnostics Chamber").

The conceptual design of MPEX and the SAS are interfaced through the Target Exchange Chamber (as shown in Figure 2), which is a type of "vacuum suitcase." Not only does the TEC transport the exposed target, it also provides temperature stability to the target (via embedded cooling-water channels) during plasma exposure. It is a principle of the MPEX system design that: once mounted in the TEC, the thermal "boundary condition" on the target should not be changed. This allows experiments to be planned which involve cycles of expose-analyze-expose, without introducing additional unknowns on the thermal bonding of the target. For pre-irradiated targets, which are of extremely high value, this MPEX-TEC-SAS strategy gives the MPEX Team the ability to perform crucial "combined effects" plasma-exposed irradiated-material analysis with a minimum amount of irradiated material.

1.1.3 *Ex situ*, post exposure and *ex situ, postmortem* data

The initial SAS is being designed to provide high value surface analysis capability *in vacuo*, post exposure from "day 1" operation of MPEX, but represents a reduced capability compared to the data that can be gleaned *ex situ*. During the MPEX operational period, additional SAS instrumentation capability should be planned with input from ORNL scientists and from external collaborators. ORNL has extensive, state-of-the-art material analysis capability, and world leading expertise, for example the LAMDA and HTML facilities. *Ex situ*, post exposure analysis of targets from MPEX can be carried out, leveraging this capability and expertise. The fidelity, resolution, and functionality of these greater-ORNL instruments can be naturally expected to be much better than what can be implemented (on budget) in the SAS for *in vacuo*, post exposure analysis. In principle, these targets could be returned to MPEX for further plasma exposure, however there is a recognized concern about the reproducibility of the thermal bonding on the TEC manipulator arm, specifically the "target payload." Moreover, *ex situ postmortem* analysis can yield information on exposed material properties that are simply impossible to get by any other means. For example, destructive sampling and segmenting of the exposed target can yield information on changes to material properties due to plasma exposure. ORNL has the capability and expertise to perform detailed *postmortem* analysis of both irradiated and non-irradiated samples.

When taken in aggregate, these factors have driven the integrated design decisions for the conceptual design of MPEX, the TEC, and the instrumentation planned for the SAS.

1.2 MPEX Users Research Forum Process

Closing the "PFC gap" to enable magnetically confined fusion energy is a key issue facing the world fusion community. Consequently, there are many researchers pursuing this goal, at domestic and international facilities. Their collective experience should be leveraged as "lessons learned" to inform the MPEX design, particularly in the instrumentation that is necessary to address key scientific objectives. These facts were pointed out to the MPEX Team during several project review meetings, including the MPEX CDR. This prompted the organization of a one-day workshop that was satellite to the 7th International Workshop on Plasma Material Interaction Facilities for Fusion Research (PMIF 2019, https://sites.google.com/ucsd.edu/pmif2019/home).

The 1st MPEX Users Research Forum (MURF) was organized specifically to gather community input to assess the needs of the Surface Analysis Station. The agenda for the MURF can be found in Appendix A. There was no explicit charge to the participants, but it was noted in recommendations from the MPEX Critical Decision 1 (CD-1) "Directors Review" written out-brief (May 2019), that the MPEX Team should "solicit input from the user community (PMI science, PFMC engineering) for essential and preferred capabilities for *in situ*, *in vacuo* (intermittent and incremental), and *post situ* characterizations." And to "identify instrument needs for the on-site material characterizations (Surface Analysis Station) based on user community input and assess adequacy of the current plan in terms of EMI and other interferences from MPEX operation." This input is summarized in this report. Moreover, the Team

should “reflect the results in designs of the target and backend facilities,” which will be done in preliminary design. This guidance is commensurate with the general motivation given in Section 1.1 above, and further details are given below.

Invited speakers to the MURF were asked in advance to make presentations on 5 specification categories (SC):

SC1: Instrumentation capabilities of the SAS

SC2: Required range of motion for surface analysis

SC3: Environmental control requirements before/during/after exposure

SC4: Conditions for transfer of samples from/to MPEX and to/from SAS

SC5: Material handling considerations

Further descriptive details on the type of information solicited is given below.

1.2.1 SC1: Instrumentation capabilities of the SAS

Participants were asked to comment on the priority of incorporation of instrumentation on the SAS (Figure 3), for example:

Focused Ion Beam Scanning Electron Microscopy (FIB-SEM)

X-ray Photoelectron Spectroscopy (XPS)

Secondary Ion Mass Spectrometry (SIMS)

Laser Induced Break-down Spectroscopy (LIBS)

Confocal imaging

Atomic Force Microscopy (AFM)

And others …

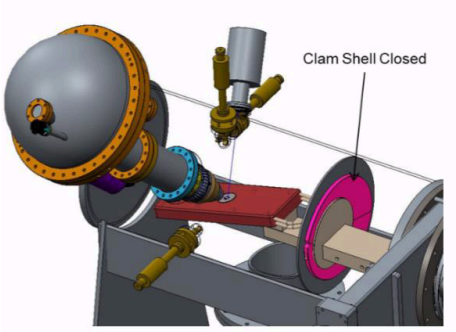

Figure 3: Conceptual design of the SAS with 15-degree target holding pre-irradiated material puck, oriented for XPS.

1.2.2 SC2: Required range of motion for surface analysis

The conceptual design of the SAS has been designed for 2-axis movement. (We presume that cost and capability increase with each additional degree of freedom.) How many axis movements would be recommended? Is lateral movement of the target necessary? E.g. 5-axis table movement.

What are the requirements on the tilting angle, α? Is there a recommended minimum α? Should the angles be discrete or continuously adjustable?

What is the fidelity of motion of the target that is needed for analysis? (e.g. range of motion, resolution, accuracy of repositionability)

1.2.3 SC3: Environmental control requirements before/during/after exposure

Can you provide recommendations on typical environmental parameters that MPEX should strive for? E.g. MPEX baseline pressure, MPEX neutral pressure during plasma exposure, TEC pressure during transport, SAS pressure during analysis.

Do you typically bake targets after loading? Is it important to maintain an elevated target temperature during transport?

1.2.4 SC4: Conditions for transfer of samples from/to MPEX and to/from SAS

The MPEX approach is to transport (exposed) targets via the TEC to the SAS; it has been estimated that a one-way exchange could require ~30 minutes. Please comment on the experience of your team with mono-layer deposition, oxidization, etc. on these time scales, and the impacts on the quality of exposure data. Is cleaning of the surface before analysis an acceptable option?

1.2.5 SC5: Material handling considerations

The MPEX approach is to be able to handle and expose pre-irradiated material targets. Please comment on the suggestion that the MPEX project should consider establishing and maintaining both a “clean” and “contaminated” SAS.

2. MURF SPECIFICATION CATEGORY INPUT

2.1 SC1: Instrumentation capabilities of the SAS

A significant amount of the discussion period of the MURF revolved around the instrumentation package of the SAS, addressing recommendations that were introduced in the presentation period. Salient aspects of that discussion will be summarized here, but the consensus (among those in attendance) that emerged by the end of the MURF can essentially be distilled to this recommendation:

The first-generation SAS has been relatively well-defined (without a cost constraint); the prioritized instrumentation package should include: a scanning electron microscope (SEM) to image surface morphology with energy-dispersive X-ray (EDX) analysis to examine surface composition, a high-energy, compact light ion beam for Nuclear Reaction Analysis (NRA) to study presence/retention of light isotopes, and laser induced break-down spectroscopy (LIBS) to enable moderate depth profiling of static retained gases. As a corollary to this recommendation, it was agreed that X-ray Photoelectron Spectroscopy (XPS) is not a priority, and that the locally destructive nature of nanoindentation should be performed as *postmortem* at an *ex situ* facility (perhaps LAMDA Lab at ORNL). Another corollary is that FIB/SEM capability inclusion warrants further discussion.

This consensus conclusion was reached after much discussion: Several people stressed the enhanced value of *in situ* real time material data, particularly the UCSD (PISCES) group and the Nagoya Univ. (NAGDIS) group. ITER representatives advocated likewise that the diagnostic community needs to move beyond "taking pictures," and strive to provide real-time measurements of the (potential) modification of material properties that may be occurring during plasma exposure (Figure 4). This report recognizes the value of *in situ* real time measurement capability (as stated in section 1.1.1), and MPEX will strive to incorporate such systems. However, it was noted by the sponsor that the charge of this MURF was specifically to address the instrumentation planned for the SAS, which is inherently an *in vacuo* post exposure configuration.

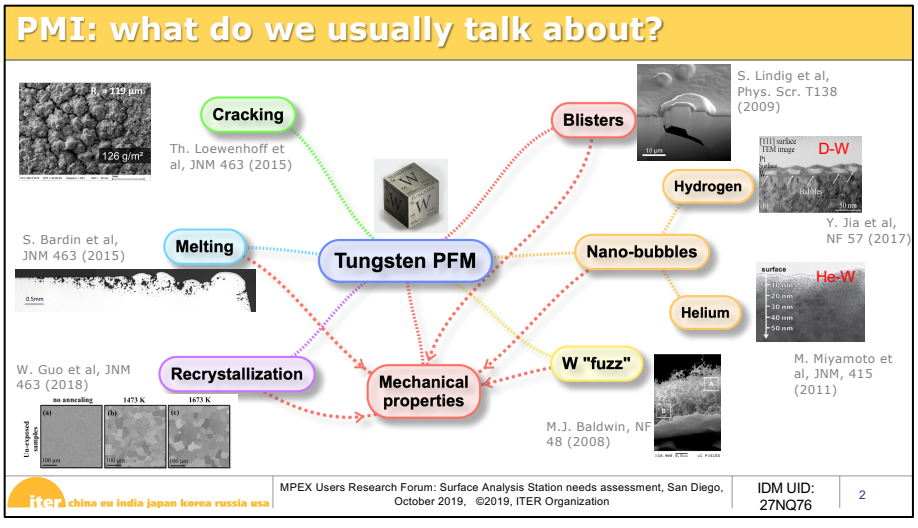

Figure 4: Slide extracted from G. De Temmerman (ITER IO) presentation on "ITER research needs to capture PMI science of evolving surfaces/materials".

The discussion emphasized that the SAS instrumentation package should be determined by science drivers, i.e. the science questions that MPEX is being positioned to answer. It was recognized that MPEX will have a ~20-year operational lifespan, and that not all the science questions MPEX will face can be currently stated with authority. MPEX is being designed to provide fusion relevant plasma flux onto material targets, with the ability to present surfaces at shallow magnetic field angles (as will be present in the Chodura sheath geometry of fusion reactor divertors), and to handle pre-irradiated material samples in this configuration. The material presented by several groups (e.g. Figure 5) was useful to highlight the immediate issues that fusion materials will need to address to close the "PFC gap," such as: T retention in the surface, T migration into the bulk material, surface material erosion and redeposition, material properties of plasma-reformed surfaces (such as thermal conductivity), and especially all of these in the context of neutron damaged materials (pre-irradiated targets). All these effects are potentially dependent on the accumulating plasma fluence; hence they need to be studied "to end of life" of the material so that accurate projections of the PFC lifetime can be made, which impacts the economic viability of fusion reactors. Moreover, ITER representatives pointed out that high-heat flux testing done with electron or ion beams may not be representative of the structural material effects that occur from high-heat flux plasmas. And that devices like MPEX and Magnum-PSI provide a more representative experience for fusion materials testing. MPEX is expected to provide steady-state, high flux plasma onto material surfaces for ~10^6 s, garnering "end of life" fluence material data that is currently unobtainable with other devices.

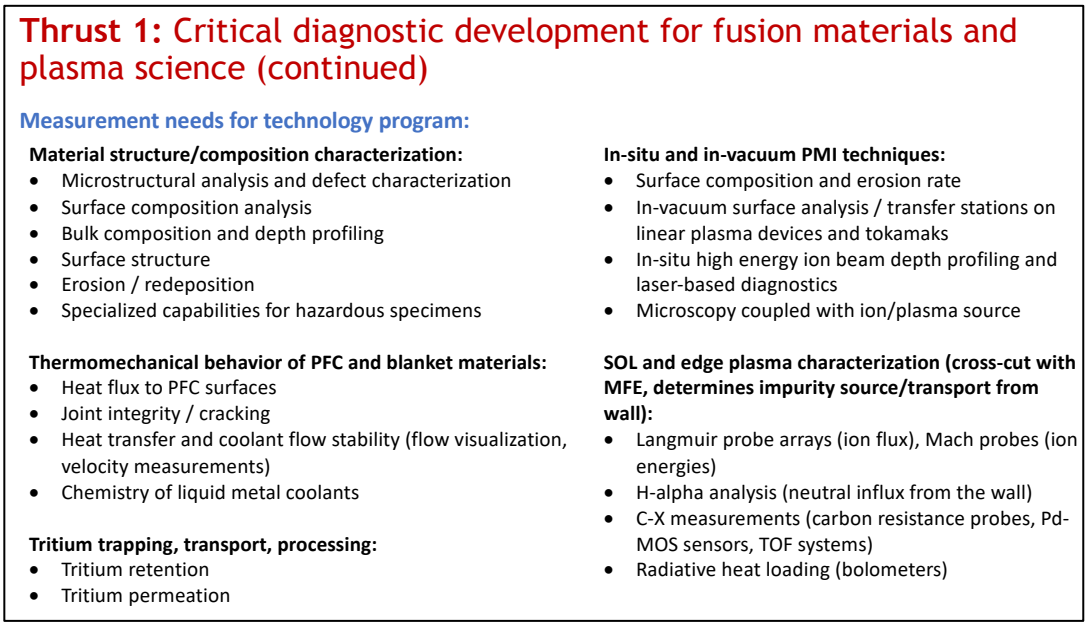

Figure 5: Slide extracted from R. Kolasinski (Sandia Nat. Lab.) presentation on the APS DPP Community Planning Process assessment of research needs for fusion materials development to enable a Fusion Pilot Plant (FPP).

The design strategy of the MPEX facility in this context includes an interfacing between the SAS (*in vacuo* post exposure analysis) via the TEC with the MPEX device (with *in situ* real-time and *in situ* post exposure capabilities). It was recognized that this strategy does not preclude the incorporation of multiple surface analysis stations. Hence, **a single "do all" SAS design is not required**. Over the MPEX operational lifetime, several SAS upgrades or additions could be envisioned to address emerging science drivers. The sponsor expressed agreement but stressed that the outcome of this MURF process should be to **arrive at a reasonable instrumentation package for a "first generation (single) SAS" that will be incorporated into the MPEX facility baseline project design and cost**. Moreover, *ex situ* post exposure capabilities at ORNL were extensively described in a presentation and represented during the discussion. Given the industrial environment adjacent to the MPEX device, it is unlikely that the SAS

capability can be made (within reasonable budget) to be competitive with *ex situ* instrumentation at ORNL. **Judicious planning of experiments in MPEX should strive to take advantage of the range of measurement capabilities: *in situ*, *in vacuo*, and *ex situ* at ORNL**.

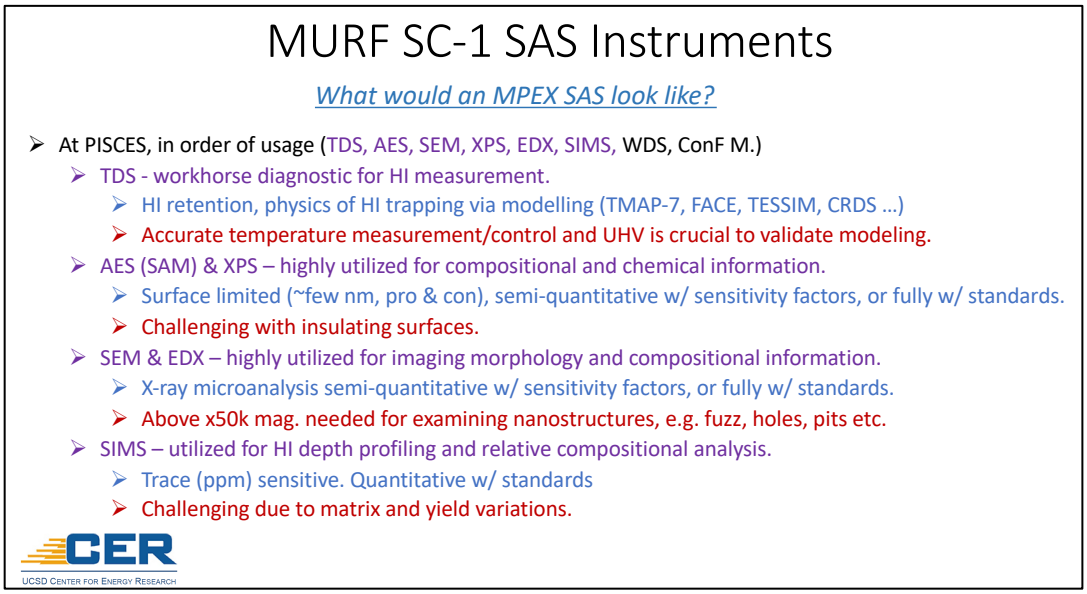

Figure 6: Slide extracted from M. Baldwin (UCSD) presentation, outlining the SC1 recommendations from PISCES experience.

The ordering of experiments on MPEX will likely effect the evolution of the instrumentation package present in the SAS (or in subsequent, additional SAS's). In particular, the chemical bonding present in plasma reformed surfaces does not (seemingly) play a large role for W targets, but is a critical process that must be understood for liquid metal (particularly liquid Li) surfaces. Liquid metal studies are being considered for the lifetime science mission of MPEX, but are not likely to be among the set of first experiments. Hence, a subsequent SAS (Mark II) could be envisioned that is tailored with instrumentation for chemical bonding analysis. **Consequently, an XPS system (which gives information on chemical bond energies) could be deferred to the Mark II SAS**. The capabilities of the 1st generation SAS should then be directed by other science drivers, like surface erosion/redeposition and morphology changes (which needs a SEM or confocal imaging) and T retention/migration (which would benefit from the incorporation of an ion beam). EDX can be used to identify elemental composition. A compact, high-energy, light ion beam enables a host of analysis techniques, such as Nuclear Reaction Analysis (NRA), Rutherford Back-scattering (RBS) analysis, Secondary Ion Mass Spectroscopy (SIMS) and Secondary Neutral Mass Spectroscopy (SNMS). The damage footprint of these techniques is small (~20 micron diameter), and hence does not "consume" a larger area of the target, which in the MPEX strategy can/will be returned for additional exposure to plasma for end-of-life fluence studies. Similarly, LIBS damage craters can be highly localized, allowing elemental depth profile information. **FIB/SEM capability inclusion warrants further discussion**; the damage done to the surface may not warrant inclusion in the first-generation SAS, but FIB capability is a typical feature with modern SEM set-ups.

Some of the instrumentation, particularly the SEM, planned for the SAS is sensitive to vibration and to residual magnetic field. Hence, the instrumentation package of the SAS as defined in preliminary design and final design, will influence the design requirements on the MPEX facility. I.e. the magnetic shield wall planned for the MPEX facility must be sufficient to reduce the residual magnetic field at the location of the SAS to a value that is compatible with operation of the instrumentation. The "Site Preparation

Guide" for a MICROLAB 310-F High Performance Field Emission Auger electron microprobe (Figure 7) specifies that the following environmental requirements be met:

Temperature
For normal operation the temperature should be 20°C ± 5°C with a stability of ±1°C (essential for the instrument's drift specification).

Heat Dissipation (nominal) is 3.5kW under normal operating conditions and 9.5 kW during bake-out

Humidity
Humidity has an effect on the instruments performance and stability. We recommend a relative humidity of less than 65% during normal operation.

Ground Vibration
Although the instrument has its own vibration isolation device, low frequency vibration from the room floor can be a problem. Other machines in neighbouring rooms, mechanical pumps, water recirculators and air conditioners can be sources of such noise. Some rooms have an intrinsic vibration (especially if the room is not a ground floor room with a solid connection to the buildings foundations).

The floor vibration should be measured and must be less than the specifications quoted below if the installation is to be completely successful.

Less than 5µm ptp in range greater than 10Hz .
Less than 1µm ptp in the range up to 10Hz

Measurement
Bandwidth : 23% or 1/3 octave

Room Noise and Airborne Vibration
RMS sound pressure level should be less than 40dB (A) over the frequency range DC to 250Hz, unweighted, with a reference level of 20µPa RMS.

Magnetic Fields
Stray magnetic fields in the vicinity of the electron gun and analyser can affect the operation of the Microlab. These fields should be less than the following values:-

Static fields : <1 Gauss in any orientation
Time varying fields : <2m Gauss peak to peak

Figure 7: Page extracted from "Site preparation guide" for an VG Scientific, MICROLAB 310-F, High Performance Field Emission Auger electron microscope.

2.2 SC2: Required range of motion for surface analysis

There was broad consensus in the discussion of SC2 that the MPEX design should **strive for the highest possible fidelity in the range of motion, and in the number of axis degrees-of-freedom**. This principle results in the greatest device flexibility, and the ability to meet unanticipated future science drivers. Specific guidance on the fidelity of range of motions was not given but is ultimately driven by the requirements of the instrumentation (as described in SC1).

Amongst the existing linear plasma facilities there was discussion of the present capabilities and "lessons learned." The range of motion needs to be engineered into the concept from an early stage of design to maximize the ability to control the target/sample positioning. The "SAS" of PISCES-B was added to the facility as an upgrade and was not part of the initial design. Consequently, only 1-axis motion was achieved. This results in "one shot" analysis scenarios, which impacts the planning of experimental sequences. Their recommendation is to invest resources early to **enable "at least 3-axis" range of**

motion for the MPEX SAS/TEC. Since it is possible to raster scan electron and ion beams, this can be used to explore the target surface, effectively adding surrogate range of motion to the sample positioning.

The PSI-2 group did extensive engineering of their target holder, allowing 5-axis range of motion (Figure 8). This enables the target to be exposed to plasma at a continuously adjustable range of angles. Moreover, when the target is retracted from the plasma exposure region for *in vacuo*, post exposure analysis, the sample can be oriented in 3D to a variety of instrumentation ports. This allows for multiple measurement techniques on a single vacuum chamber.

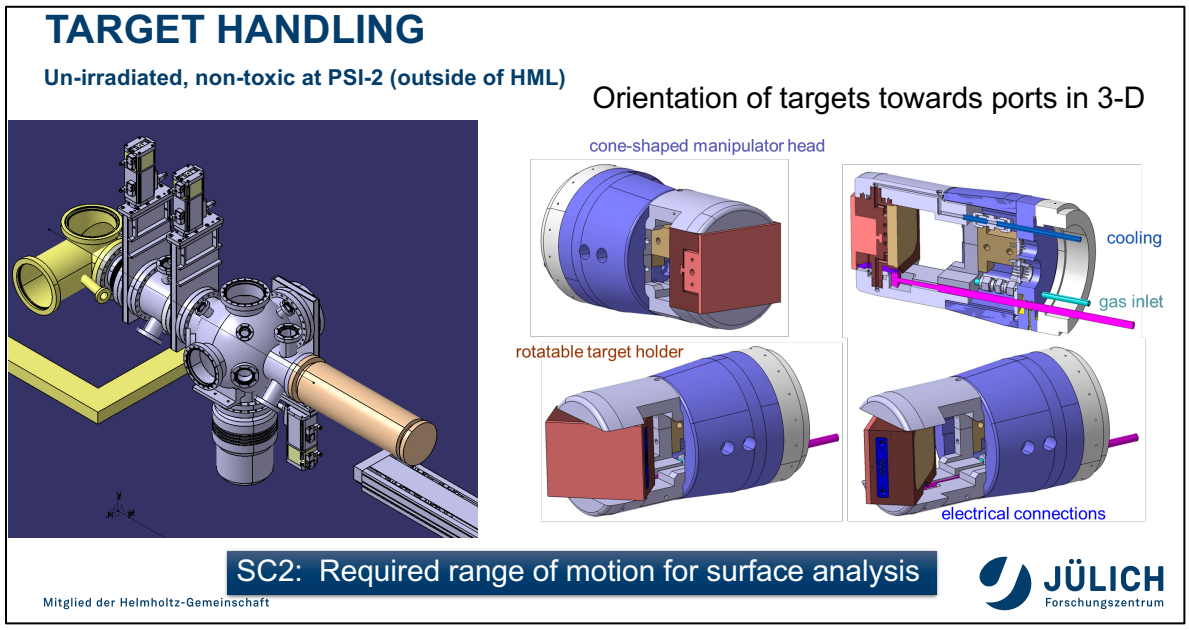

Figure 8: Slide extracted from B. Unterberg (FZ Juelich), detailing the 5-axis range of motion engineering for range of motion in PSI-2.

In both facilities, since the effective- "TEC" arm is occupied during analysis, it precludes the ability of the facilities to do (on separate targets) both plasma exposure and analysis. It was noted that the MPEX/TEC/SAS design does not in principle suffer from this limitation. Even if there is (initially) only 1 SAS, **if there are two (or more) TECs then the MPEX facility could run plasma exposures simultaneously to sample analysis (and loading, removal, etc.)**. Alternating the two TECs (and targets) could enable a greater "duty cycle" of facility utilization.

2.3 SC3: Environmental control requirements before/during/after exposure

Many presenters at the MURF expressed confusion about the distinction between SC3 and SC4 and pointed out that they are interconnected. This suggests that the input requested for these specification criteria was not well communicated by the MPEX Team. Nevertheless, input on these topics was given in the presentations and covered in the discussion, though often in a mixed or combined way. Consequently, the summarizing of SC3 and SC4 input into this report will be oftentimes similarly mixed.

The TEC (Figure 9) has several roles in the MPEX facility. It is the vessel that physically transports the target between MPEX and the SAS. Hence the vacuum conditions of the TEC are important to the accumulating history of the sample surface pre- and post- exposure; vacuum conditions will be discussed in section 2.4 in the context of SC4. Moreover, the target payload is mounted to the TEC arm, which positions the target both inside MPEX and inside the SAS. Hence the TEC arm must be engineered to

provide the location and orientation fidelity that was discussed in section 2.2 in the context of SC2. Additionally, the TEC arm provides the (in vacuum) water cooling connection to the target payload, that is necessary to maintain temperature stability of the target during MW/m^2 plasma heat flux exposure. The conceptual design of the MPEX TEC and target payloads does not include a separate heating mechanism beyond the water loop. Active heating of the target is available in some international devices. PSI-2 has the ability to control the target temperature between 150 and 1500 C. Heating is applied, for example to initially out-gas (or "bake") the target and chamber after installation from air or another controlled atmosphere. And heating of the sample may be used as part of a diagnostic technique, specifically Thermal Desorption Spectroscopy (TDS) and its variants. TDS is a commonly used diagnostic technique, especially on PISCES-B and other devices. As was discussed in section 2.1 in the context of SC1, it was decided that TDS would not be advocated for in the first-generation SAS. Hence, **it is unnecessary to include a heating element in the MPEX TEC design for the SAS**. The MPEX Project Requirements Document (conceptual design stage, in section 3.2.4) indicates that the TEC should be able to be baked up to 150 C, and that MPEX device should be capable of being baked to 90 C.

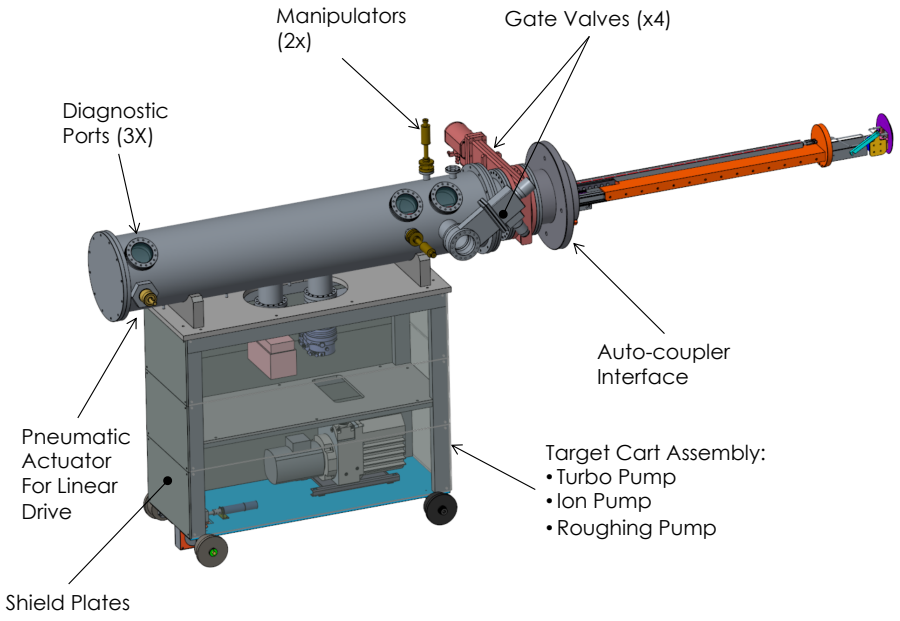

Figure 9: Conceptual design of the MPEX Target Exchange Chamber (TEC).

2.4 SC4: Conditions for transfer of samples from/to MPEX and to/from SAS

The UCSD Group gave a nice summary (M. Baldwin presentation, Figure 10) on the role that the vacuum conditions play in monolayer growth subsequent to plasma exposure. In "high vacuum" conditions (baseline pressure ~10^{-6} Torr (~10^{-4} Pa)) monolayer grown is largely due to the presence of residual water vapor. On a W substrate (typical for MPEX, though Li coatings or substrates are also possible) one monolayer of O will grow in ~0.4 minutes. In "ultra-high vacuum" conditions (baseline pressure ~10^{-8} Torr (~10^{-6} Pa)), this process is dominated by the presence of CO molecules and will occur in ~72 minutes. In dedicated surface science laboratory ultra-high vacuum (~10^{-10} Torr (~10^{-8} Pa)) monolayer growth due to CO will occur in ~120 hours. For these reasons, the PISCES-B Group recommended **sputter cleaning of the surface if vacuum conditions in the SAS and TEC cannot be lowered to 10^{-10} Torr**. In subsequent discussion Bruce Koel (Princeton University) suggested that this could be achieved

by **incorporating a cryo-pump into the design of the TEC**. PISCES-B typical vacuum baseline is ~10^{-8} Torr, and PSI-2 is ~10^{-7} Torr. The baseline vacuum pressure in Proto-MPEX is ~10^{-6} Torr, though it should be noted that Proto-MPEX is not intended to perform extensive PMI experimentation.

SC-3, SC-4 Pressures, MPEX, TEC

To what extent will impurity and oxidation effects play a role in the TEC and SAS ?

- AES, XPS, probes surface MLs.
- ML coverage is given by kinetic theory:

$$t_{MLc} \cong \frac{10^{19}\sqrt{2\pi mkT}}{\sigma P}$$

- Coverage time depends on target 'reactiveness' to H_20 at HV & CO at UHV.

Vaccum condition	P (Torr)	ML cov. of Be, Li in (σ=1)	ML cov. of W in (σ=0.5)
High (H_2O)	10^{-6}	0.2 m	0.4 min
Ultra-high (CO)	10^{-8}	36 m	72 min
Surf. Sci. Ultra-High (CO)	10^{-10}	60 h	120 h

- PB 5 x10^{-9} – 2 x10^{-8} Torr.
- PB target to SAS is ~10 mins at high priority
- AES on plasma exposed 'clean' Be shows surface coverage in C & O at ~40 mins.
- Sputter cleaning is useful above 10^{-10} Torr.
- Active ion beam in depth profiling mitigates impurity effects but can give false carburization signatures in XPS.
- In-situ TDS can produce unwanted desorption products.

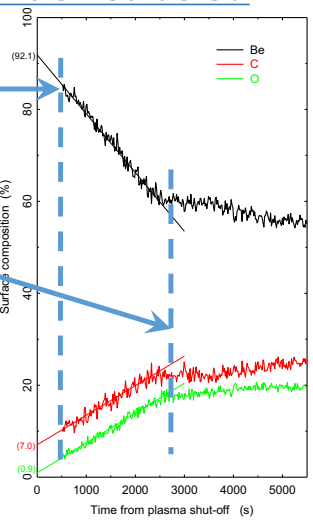

Figure 10: Slide extracted from M. Baldwin (UCSD) presentation, outlining the SC3-4 recommendations from PISCES-B experience.

During the operation of MPEX, the pressure in the PMI chamber at the target is expected to be 1 to 10 Pa (~10^{-2} to ~10^{-1} Torr), commensurate with the neutral pressure expected to be found in the ITER divertor during operation. During 1 s Proto-MPEX pulses, the neutral pressure at the target is measured to reach ~10^{-3} Torr. The MPEX Project Requirements Document specifies (conceptual design, in section 8.2) that the baseline pressures in the MPEX device should be <~10^{-7} Torr (<10^{-5} Pa), in the TEC should be ~10^{-8} Torr (~10^{-6} Pa in Table 16), and in the SAS should be ~10^{-9} Torr (~10^{-7} Pa section 8.2.2). It is estimated that the transfer time (post exposure) between MPEX and the SAS could be 20 to 30 minutes (including various procedural checks). Most of this time would be at TEC baseline pressure, i.e. ~10^{-8} Torr. Hence, for W target substrates, only a fraction (~1/3) of a monolayer would form during this time. The TEC (conceptual) design includes both a turbo- and an ion- pump to achieve these pressures. The base pressure of the SAS is specified to be an order of magnitude lower, which suggests that ~1 day of analysis could be performed before significant (additional) monolayer growth occurs.

2.5 SC5: Material handling considerations

There was discussion on material handling concerns presented by the PISCES Group (derived from Be handling experience) and from the TPE Group (derived from T handling experience). The ORNL Material Science (LAMDA) Group, which handles nuclear irradiated samples including nuclear fuel, also relayed their experience in the discussion. The groups reported that cross-contamination of irradiated material to surrounding surfaces is common. In the INL TPE experience (Figure 11) this occurred both as a result of plasma exposure and sample heating. So, it is likely that erosion/redeposition in MPEX of pre-irradiated samples will spread irradiated material to the PMI chamber of the MPEX device. But, furthermore, if TDS is incorporated into the instrumentation package of the SAS, then irradiated material will migrate during analysis in the SAS to cause cross-contamination to surfaces within the chamber.

However, there is little/no evidence to suggest that contamination on the (non-heated) SAS chamber would migrate back onto non-irradiated targets in subsequent exposures. This sentiment was similarly reflected in statements by the ORNL Material Science Group members that were present; there is no evidence of back-contamination occurring from the irradiated instrumentation to samples. But **the instrumentation should be treated as a consumable**, since once contaminated it cannot be refurbished by the manufacturer. At ORNL this is costed into the rate charged to perform (*ex situ*, post exposure) analysis of samples. Similarly, the PISCES-B Group reported that Be smears tested positive both in their "SAS" chamber and in their *ex situ*, Be compatible SEM. They attribute this to highly mobile Be dust, which is produced when sputter cleaning of samples post exposure, prior to analysis. **Diagnostic techniques which require the sample surface to be sputter-cleaned could potentially result in irradiated dust** from other materials**.**

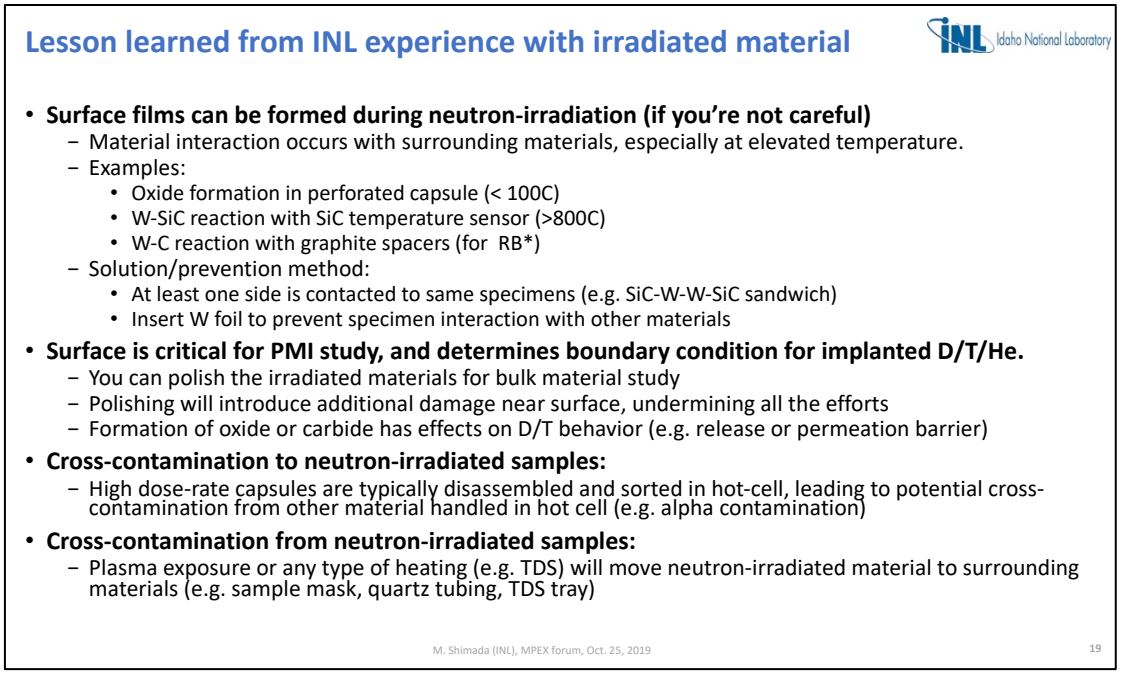

Figure 11: Slide extracted from M. Shimada (Idaho Nat. Lab.) presentation on TPE experience with irradiated material.

The notion that the MPEX facility should maintain both a "clean" and a "contaminated" SAS is largely a matter of procedures and support funding. **It is not strictly necessary to maintain a "clean" SAS, since even a "contaminated" SAS would be unlikely to back-contaminate a clean sample**.

3. RECOMMENDATIONS

3.1 General Recommendations:

Strive for *in situ* real-time PMI measurements.

Experiments in MPEX should strive to take advantage of the range of measurement capabilities: *in situ, in vacuo*, and *ex situ* at ORNL.

3.2 Recommendations for SC1: Instrumentation capabilities of the SAS

The prioritized instrumentation package should include: a scanning electron microscope (SEM) to image surface morphology with energy-dispersive X-ray (EDX) analysis to examine surface composition, a compact high-energy, light ion beam for Nuclear Reaction Analysis (NRA) to study presence/retention of light isotopes, and laser induced break-down spectroscopy (LIBS) to enable moderate depth profiling of static retained gases.

As a corollary to this recommendation, it was agreed that X-ray Photoelectron Spectroscopy (XPS) is not a priority, and that the locally destructive nature of nanoindentation should be performed as *postmortem* at an *ex situ* facility (perhaps LAMDA Lab at ORNL).

Another corollary is that FIB/SEM capability inclusion warrants further discussion.

A single "do all" SAS design is not required.

Arrive at a reasonable instrumentation package for a "first generation (single) SAS" that will be incorporated into the MPEX facility baseline project design and cost.

3.3 Recommendations for SC2: Required range of motion for surface analysis

Strive for the highest possible fidelity in the range of motion, and in the number of axis degrees-of-freedom.

Enable "at least 3-axis" range of motion for the MPEX SAS/TEC.

3.4 Recommendations for SC3: Environmental control requirements before/during/after exposure

It is unnecessary to include a heating element in the MPEX TEC design for the SAS.

3.5 Recommendations for SC4: Conditions for transfer of samples from/to MPEX and to/from SAS

Incorporate a cryo-pump into the design of the TEC.

3.6 Recommendations for SC5: Material handling considerations

The instrumentation should be treated as a consumable.

It is not strictly necessary to maintain a “clean” SAS, since even a “contaminated” SAS would be unlikely to back-contaminate a clean sample.

APPENDIX A. 1st MURF AGENDA

APPENDIX A. MURF AGENDA

MPEX Users Research Forum (MURF): Surface Analysis Station needs assessment

Goal: The MPEX Team is reaching out to the community of potential users of the MPEX facility to make an assessment of the high-priority capabilities which should be considered for inclusion in the design of the MPEX Surface Analysis Station (SAS). This activity is being carried out during the "advanced conceptual design" period of MPEX, i.e. post Conceptual Design Review but prior to Preliminary Design, to respond to a recommendation by the DOE following CDR. The MPEX Team will summarize the community recommendations into a guidance document, which will influence the Surface Analysis Station during the Preliminary Design phase of the project.

Format: 1-day workshop organized as follows:

8:00-11:20	International and Domestic experts will deliver **"lessons learned" presentations** from their facilities, and recommendations for surface analysis capabilities for the MPEX SAS, **with brief discussion**
11:20-12:20	Working lunch: **CDR-level design concept for SAS** from MPEX Team
12:20-2:30	Group discussion to identify measurement wants/needs for MPEX SAS, design features to enable Science Mission of MPEX
3:00-4:30	Group discussion to prioritize measurement systems for MPEX SAS
5:00	MPEX Team concludes with Summary/Restatement of high priority measurements for MPEX SAS, and describes Further Actions

Date: Friday, October 25, 2019
Following the PMIF workshop (https://sites.google.com/ucsd.edu/pmif2019)
Preceding the ICFRM workshop (https://icfrm-19.com)

Venue: La Jolla Shores Hotel "Acapulco Room" in San Diego, CA area (location of PMIF and ICFRM workshops) (https://www.ljshoreshotel.com/meetings/venues)

Confirmed attendees (25):
Allain (PSU, remote), Kolasinski (SNL), Shimada (INL), De Temmerman (ITER IO), Sakamoto (Tsukuba), Unterberg (FZJ), Van Eck (Differ, afternoon), Guo (GA), Woller (MIT), Sinclair (GA), Clark (DOE), Andruczyk (Illinois, remote), Ohno (Nagoya), Thomas (GA), Abrams (GA)
ORNL/UTK: Rapp, Biewer, Parish, Garrison, Echols, Lau, Fergusson
UCSD: Doerner, Tynan, Baldwin, Thakur

Agenda:

Time:	Name	Duration	Title
7:30		30 min	Breakfast (continental)
8:00	Rapp	5 min	Welcome
8:05	Biewer	15 min	Intro, Statement of Goal, Expected deliverables
8:20	De Temmerman	15+5	ITER's perspective on surface analysis needs to capture PMI science of evolving surfaces/materials
8:40	Kolasinski	15+5	Surface analysis needs/Initiatives identified by APS DPP Community Planning Process
9:00	Parish	15+5	State-of-the-art surface and near-surface analysis capabilities at ORNL apart from MPEX facility
9:20	Unterberg	15+5	Plans for Jule PSI and PSI2 experience
9:40		20	Coffee break
10:00	Allain	15+5	Recommendations for surface analysis capabilities from MAPP experience
10:20	Ohno	15+5	Recommendations for surface analysis capabilities from NAGDIS experience
10:40	Baldwin	15+5	Recommendations for surface analysis capabilities from PISCES experience
11:00	Shimada	15+5	Recommendations for surface analysis capabilities from TPE experience
11:20		60	Start of lunch
11:40	Biewer	15+5	Overview of the Conceptual Design of the MPEX Surface Analysis Station
12:20		100	Group discussion to identify measurement wants/needs for MPEX SAS
2:00		30	Coffee break
2:30		120	Group discussion to prioritize measurement systems for MPEX SAS
4:30	MPEX Team	30	Summary/Restatement of high priority measurements for MPEX SAS, Further actions

APPENDIX B. MURF COMMENT FORM

MPEX Users Research Forum (MURF): Surface Analysis Station needs assessment
October 25, 2019
The MPEX Team is soliciting your input to help refine the requirements on the Surface Analysis Station (SAS), based on "lessons learned" from your experience.

Send to: Ted Biewer (biewertm@ornl.gov)

Submitted by:

Specification Categories:

- SC1: Instrumentation capabilities of SAS
- SC2: Required range of motion for surface analysis
- SC3: Environmental control requirements before/during/after exposure
- SC4: Conditions for transfer of samples from/to MPEX to/from SAS
- SC5: Material handling considerations
- SC6: Other

Recommendation: