



An Overview of the Hybrid Illinois Device for Research and Applications Material Analysis Test-stand (HIDRA-MAT)

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Abstract

The Hybrid Illinois Device for Research and Applications (HIDRA) at the University of Illinois at Urbana-Champaign is a toroidal plasma device that enables fusion plasma-material interaction testing with both stellarator and tokamak plasmas. HIDRA's long-pulse steady state stellarator plasmas provide a testbed for plasma facing component (PFC) plasma exposures. The HIDRA Material Analysis Test-stand (HIDRA-MAT) is a material characterization module attached to HIDRA that is being designed and fabricated to include thermal desorption spectroscopy and laser induced breakdown spectroscopy systems for *in-vacuo* PFC characterization. A specialized rotatable sample holder positions the sample for liquid metal droplet application from a liquid metal droplet injector on HIDRA-MAT. Early experiments look to investigate the effect liquid lithium has on porous tungsten samples' retention of H, D, and He after plasma exposure. Preliminary results from a dual residual gas analyzer system show the ability to differentiate D₂ and He in HIDRA-MAT. This work aims to advance the understanding of liquid metal PFCs and further the design and development of new fusion PFCs and technologies.

Introduction

Plasma facing components (PFCs) play a critical role in the development of fusion energy. The numerous underlying mechanisms responsible for plasma-material interactions (PMI) have a substantial effect on plasma performance and device design. Testing PFCs in fusion devices proves to be a considerably difficult and time-consuming task. These challenges arise from larger fusion devices (EAST, JET, etc.) having strict experimental schedules which collate data on several disciplines of fusion energy research, not just PFCs. Additionally, each device's unique design constraints can impede the feasibility of efficiently testing multiple PFC concepts and restrict experimental campaigns to focus on a single PFC. The development of an experimental device dedicated to testing a diverse set of PFC

materials in a time efficient manner would accelerate the development of fusion PFCs.

Fuel and reaction byproduct interactions with the wall have a significant impact on any fusion relevant PFC. Ion implantation leading to fuzz growth [1, 2] and retention of fuel [3] have highlighted some issues with using solid PFCs. The Center for Plasma-Material Interactions (CPMI) at the University of Illinois at Urbana-Champaign (UIUC) is one of the leading labs studying the use of liquid metals, specifically lithium, as a PFC in fusion devices [4–6]. Research at UIUC aims to understand the PMI associated with liquid lithium loops and the engineering required to make them a reality in fusion devices. One such area of PMI being investigated is the retention of D and He in lithium PFCs. The Hybrid Illinois Device for Research and Applications (HIDRA) provides an integral pathway for studies on D and He retention in lithium to be researched in an environment that can imitate some aspects of larger fusion devices.

HIDRA, located at CPMI, is the only tokamak stellarator hybrid dedicated to the study of PMI for fusion applications. HIDRA, formerly WEGA in Greifswald, Germany, has a major radius $R_0 = 0.72$ m and minor radius $a = 0.19$ m and is currently being run as a classical

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stellarator [7]. HIDRA can operate for ~ 60 min at low magnetic field (87.5 mT) and for tens of minutes in its high magnetic field regime (0.5 T). HIDRA's size and long-pulse plasmas equip it to act as a testbed for PMI research. Though HIDRA cannot reach the heat fluxes seen in larger devices, it can reproduce a similar magnetic field environment and particle flux that will produce relevant PMI edge mechanisms [8]. HIDRA, in combination with PMI diagnostics, can be used to conduct preliminary PMI experiments to provide useful data on PFCs to aid larger devices in determining what PFCs should be implemented in their experimental campaigns.

To efficiently utilize HIDRA for material testing, the HIDRA Material Analysis Test-stand (HIDRA-MAT) is being developed. HIDRA-MAT's design took inspiration from the Material Analysis Particle Probe (MAPP) used on NSTX [9]. Though both devices are used for PFC characterization, HIDRA-MAT differs from MAPP in size and shape due to space constraints around HIDRA and has different characterization capabilities as well. HIDRA-MAT is an extension to HIDRA that will be capable of surface characterization for a variety of materials before and after plasma exposure. HIDRA-MAT stands apart from other material analysis diagnostics due to its ability to expose liquid metal PFCs to long-pulse stellarator plasmas with periodic *in-vacuo* surface characterization. A sample can be heated with an ultra-high vacuum (UHV) heater sample stage and have liquid metal applied to it by using a liquid metal droplet injector (LMDI). After or in-between plasma exposures, materials can be characterized *in-vacuo* in the HIDRA-MAT main chamber using laser induced breakdown spectroscopy (LIBS) and thermal desorption spectroscopy (TDS).

Reactive materials such as lithium pose a challenge in predicting behavior across different device environments. HIDRA-MAT will be utilized to provide preliminary data on retention behavior associated with high particle fluxes. HIDRA's particle fluxes are on the order of $10^{22} \text{ m}^{-2} \text{ s}^{-1}$ at low magnetic field operation making it comparable to particle fluxes seen on larger devices. A particle flux on an order of magnitude higher than HIDRA's would mean HIDRA's pulse length would be run for an order of magnitude longer. The increase in time does have an effect on overall passivation of the PFC surface but retention should not be affected because of the fast diffusion of hydrogen isotopes in lithium [10] assuming that a monolayer of impurities forms every second at 10^{-6} Torr. Thermal effects will play a role in understanding PFC behavior, but HIDRA and HIDRA-MAT can still provide a basis for obtaining useful data to be used in the development of future liquid metal PFCs. In any device using liquid metals, passivation will be present and through experimentation

HIDRA-MAT can also provide data related to passivation effects.

HIDRA-MAT's characterization techniques enable it to gather valuable PMI information. LIBS's short characterization time, on the order of a few seconds, paired with HIDRA's stellarator plasma pulse lengths permit multiple sequences of plasma exposures and surface characterizations in a single pulse. This is advantageous because a temporal evolution of the material surface between exposures can be discerned. TDS data will show what molecules are desorbing from the material surface. A dual residual gas analyzer (RGA) setup used for TDS can also distinguish between D_2 and He. These diagnostics will yield data detailing the dominating PMI edge mechanisms' effect on the PFC material including particle retention, impurity segregation, and impurity/complex molecule formation. This paper presents HIDRA-MAT's main components' design and functions.

Sample Preparation Systems

High Linear Shift Mechanism Transfer Arm

A sample in HIDRA-MAT is required to move linearly and be rotatable for plasma exposure and characterization. A transfer arm, seen in Fig. 1, provides high accuracy linear sample positioning for diagnostics and plasma exposure. A rotatable sample stage is attached to an actuated high linear shift mechanism (HLSM) with 800 mm of linear travel. Figure 2a depicts how samples can be transferred from the HIDRA-MAT main chamber through 17.6 cm of the HIDRA vacuum chamber midplane. Experiments will show how far into the HIDRA plasma the sample can be placed before the heater elements disrupt the plasma completely. Figure 2b shows how initial experiments will expose the sample at the plasma edge which has been determined from magnetic field mapping and plasma shape results from previous experiments [11]. Following exposure, samples are retracted into HIDRA-MAT for surface characterization. The HLSM's micron position resolution can be taken advantage of for LIBS measurements at various sample surface locations.

Sample Heater and Rotation Mechanism

Sample heating is required for favorable wetting conditions of the sample [12] and TDS [13]. A HeatWave Labs Model 101491-01 UHV Heater with sample clips secures and heats the sample. The UHV Heater can hold samples as large as 25 mm in diameter and 6 mm in height. Sample heating under vacuum conditions is expected to reach up to 1000 °C and heater temperatures are monitored by a built-

Fig. 1 HIDRA-MAT's high linear shift mechanism attached to the HIDRA-MAT main chamber. The transfer arm is responsible for linear positioning of the sample, moving between plasma sample installation, characterization, and plasma exposure positions

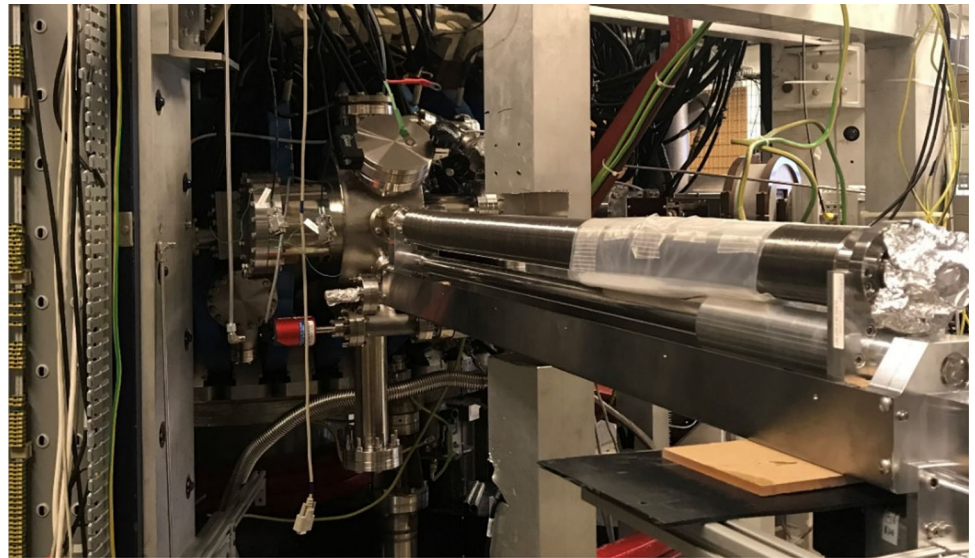
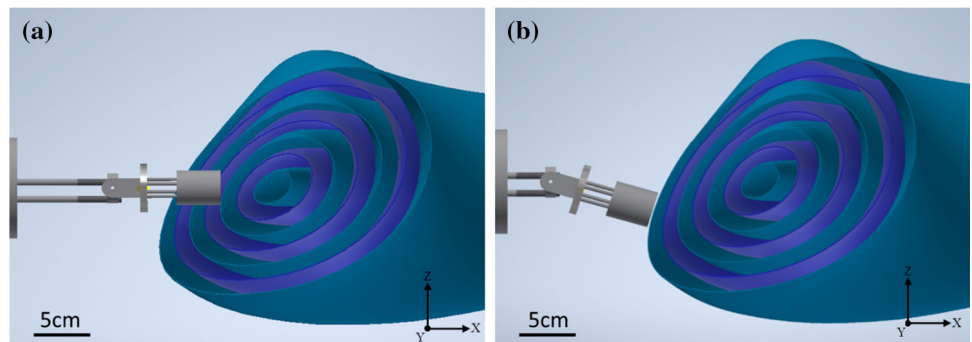


Fig. 2 Full extension of **a** HIDRA-MAT's transfer arm 17.6 cm into HIDRA's vacuum vessel while a plasma is present and **b** the sample heater positioned normal to the plasma by using the sample rotation mechanism



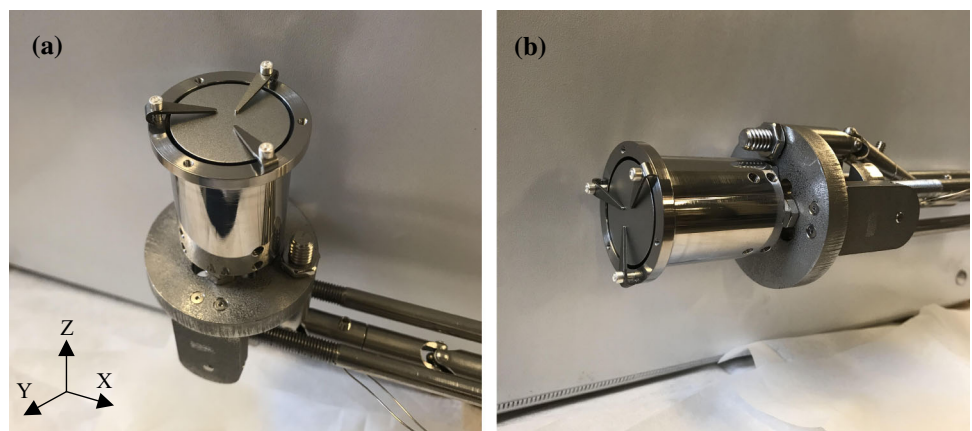
in thermocouple connection. The heater is mounted to a rotation mechanism that has 180° of sample rotation allowing the sample to be positioned normal to diagnostics and the plasma. Rotation is achieved by a connection of u-joints, a stainless-steel rod, and a linear bellows drive (LBD) with 50 mm of travel. The u-joints translate linear motion in the x-axis into rotation of the entire sample head shown in Fig. 3. Additionally, the sample head rotates

about a horseshoe support that is connected to the transfer arm, so the sample head moves linearly with the transfer arm motion.

Liquid Metal Droplet Injector

An LMDI has been designed and fabricated to control droplet size before applying it to the sample. After the

Fig. 3 The UHV Sample heater in **a** a $+90^\circ$ vertical position used for liquid lithium application and **b** a 0° horizontal position used for plasma exposure. Rotation can continue to a -90° vertical position for 180° of rotation



UHV heater, rotation mechanism, and transfer arm have positioned and heated the sample to the correct wetting temperature, the LMDI applies the droplet to the sample. Figure 4 highlights the main components of the LMDI. The injector is loaded with solid lithium in an argon environment then transferred and installed on HIDRA-MAT which is also being pumped with argon. A TEMPCO Mightyband coil heater brings the reservoir temperature to ~ 220 °C ($T_{melt,Li} = 180.5$ °C). Injection is performed by driving a piston with an actuated LBD through the reservoir and out of the reservoir nozzle. The system is designed such that the piston, when fully inserted, reaches the end of the reservoir to maximize the usable volume of loaded lithium. An actuated linear shift mechanism (LSM) with a 50 mm stroke length provides controllable application of droplets to the sample. Droplets formed stay attached to the nozzle due to the high surface tension of the liquid metals used [14, 15] and then are lowered and transferred to the heated sample. When the weight of the droplet exceeds the surface tension forces, a droplet will detach from the nozzle and fall to the sample. Programming the LBD controller for shorter and slower movement of the piston results in greater control over droplet size. A DN160CF viewport on the side of HIDRA-MAT provides line of sight to monitor droplet size and position relative to the sample.

Material Characterization Systems

Laser Induced Breakdown Spectroscopy System

LIBS will be utilized to collect data on the temporal evolution of the PFC's surface *in-vacuo*. Simulations have shown that in a 26 kW heating scenario operating at low field (87.5 mT) HIDRA's poor plasma confinement gives rise to large particle fluxes to the sample on the order of $10^{22} \text{ m}^{-2} \text{ s}^{-1}$. The stellarator pulse is on a much larger

time scale than other devices [5], so many particles are interacting with the wall in one pulse. HIDRA-MAT studies the effects of these plasma characteristics by performing LIBS on exposed PFC materials. Since the plasma is still present in HIDRA when LIBS is being done in HIDRA-MAT, the material can once again be exposed and then characterized. Combining separate LIBS measurements will elucidate the temporal evolution of how the PMI mechanisms are affecting the PFC.

With the sample being heated and the high surface tension of lithium, lithium will remain a liquid and stay on the sample surface during the plasma exposure and characterization. LIBS can be performed on solids, liquids, or gases, making it advantageous for this application. In HIDRA-MAT's first experimental campaign its dual-pulsed LIBS system will attempt to produce a strong signal depicting the retention of H, D, and He on the surface of lithiated porous tungsten that was exposed to a D-He plasma. Once *in-vacuo* characterization is complete, samples can be transferred out of HIDRA-MAT to other characterization diagnostics. The transfer will cause the sample to be exposed to atmosphere immediately causing the surface to oxidize. Conclusions reliant on data from these secondary characterization techniques must take oxidation effects into account. The LIBS laser pulse will form a crater on the surface from ablated material, which will be measured with atomic force microscopy. Knowing crater depth opens the possibility of using multiple laser pulses, and the signal obtained from each, at the same sample location to produce a depth profile. Scanning electron microscopy imaging can be utilized to show crater location on the sample surface and 3D profilometry can be used to characterize the morphology of the crater to obtain metrics that will be useful to determine surface/volumetric retention values.

In-vacuo measurement is crucial to successfully identifying liquid metal PFC behavior in fusion devices when it

Fig. 4 CAD of HIDRA-MAT's a LMDI for applying liquid metals to samples in HIDRA-MAT and b cross section view of the LMDI's reservoir assembly

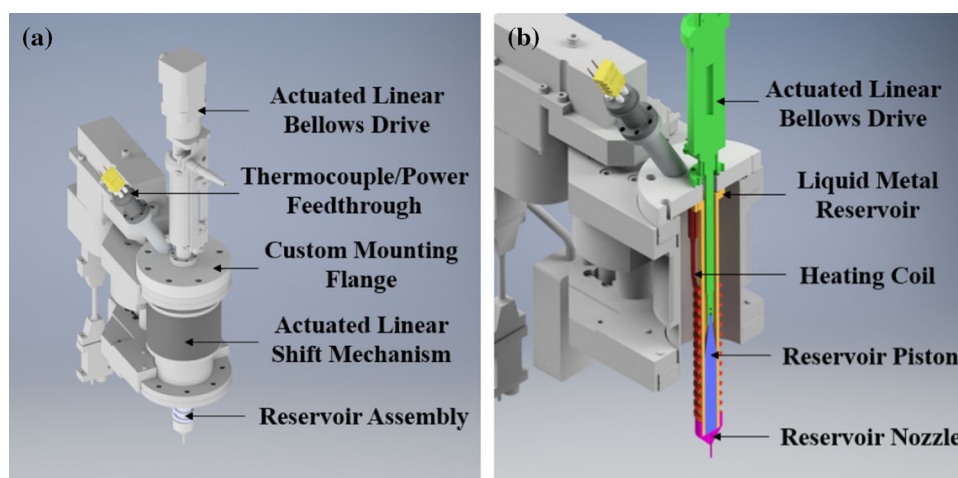
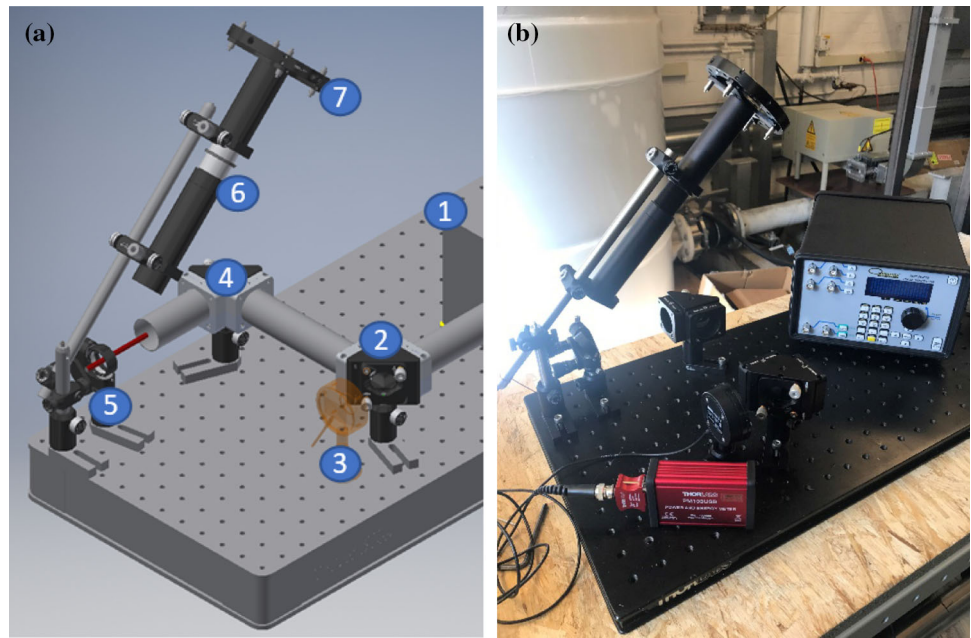


Fig. 5 The **a** CAD of a fully assembled LIBS system with the following labeled components: (1) 532 nm Nd:YAG laser (2) 90:10 (R:T) UVFS plate beamsplitter (3) ES220C pyroelectric energy sensor (4) Nd:YAG mirror (5) gimbal mount, 360° adjustable with Nd:YAG mirror (6) adjustable lens tube and lens ($f = 500$ mm) and (7) transmission viewport. Progress on **b** assembled LIBS optics along with pyroelectric sensor and pulse generator



comes to H, D, and He retention. Characterization requiring samples to be taken out of vacuum immediately introduces impurities in the form of oxidation and other atmospheric contaminants [16]. Lithium oxidizes quickly in atmospheric conditions, so *in-vacuo* characterization is a necessity. HIDRA-MAT and LIBS can gather the essential preliminary data on how these ions will react with liquid lithium PFCs. *In-vacuo* characterization produces data that more accurately imitates realistic reactor conditions by removing external surface degradation.

The LIBS system includes a Litron B-PIV 532 nm nanosecond pulsed laser with a pulse energy of 145 mJ at 15 Hz. Optics redirect and focus the beam to a spot size of ~ 1 mm resulting in a calculated power density of 4.15×10^{12} W/m², which is consistent with similar LIBS experiments on tungsten PFCs [17]. The laser will separate the two pulses on the order of tens to hundreds of nanoseconds. The first pulse will produce the ablation of the sample material and the second one can be triggered after an optimized delay that maximizes the eventual plasma plume formation and the concomitant spectral emission from the ablated and excited material. After the dual pulse, a Quantum Composers 9520 Series pulse generator will trigger two Ocean Insight HR2000 + series spectrometers that will collect and analyze the light signal. The delay between laser and spectrometer trigger is sensitive to each specific LIBS setup and will range from hundreds of nanoseconds to microseconds. The first spectrometer ranges from 200 to 1100 nm and will scan a range of wavelengths that include Li, W, and any other contaminants' peaks with a 0.1 nm resolution. The second spectrometer ranges from 630 to 680 nm and is able to

resolve the H α and D α peaks that are present with a resolution of 0.2 Å. Future experimental campaigns will strive to quantify retention in samples.

Figure 5 shows the LIBS optics setup and Fig. 6 shows a CAD of the final assembly on HIDRA-MAT. The beam first interacts with a ThorLabs 90R:10T BSX10 beam-splitter. The transmitted beam hits an ES220C pyroelectric sensor and the registered value is used in the calculation of the pulse power density at the sample. The reflected beam interacts with two NB1-K13 flat mirrors before being redirected at a 45° angle through a connection of lens tubes. An adjustable lens tube houses a 25.4 mm LA1908-YAG lens with a focal length of 500 mm. The lens position can be adjusted to change the spot size on the sample. Following the lens, the focused beam is transmitted through a VPZL-275LYAGD1 transmission viewport before hitting the sample.

Thermal Desorption Spectroscopy

TDS is a surface characterization technique that will be used to confirm LIBS measurements and provide quantitative data that will describe the overall retention behavior of the exposed sample surface. A HeatWaveLabs Model 101303 power supply is connected to the UHV Heater and controls the heater temperature up to 1000 °C. Temperature is monitored by two thermocouples. One is placed inside the heater and the other is at the surface of the sample. The sample surface can be heated with programmed ramp rates and a dual RGA setup identifies the desorbed molecules traveling through a sniffer probe.

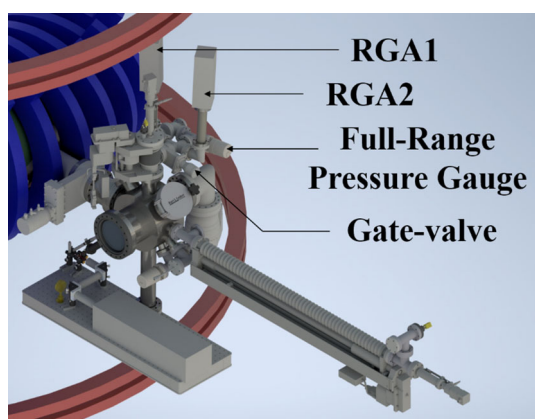


Fig. 6 CAD representation of the final HIDRA MAT assembly with sniffer section components labeled. The sniffer probes location is between the gate valve and main chamber

The sniffer probe has a simplistic design to execute a specific function. It consists of a solid blank copper gasket with a 1 mm sized orifice in the center. This gasket restricts flow between the main chamber and the sniffer section. The sniffer section refers to the section attached to the main chamber of HIDRA-MAT that has a gate valve, two RGAs, and a full-range gauge as seen in Fig. 6. Differential pumping can be achieved because of the size of the orifice and turbomolecular pumps on both sides of the sniffer probe. Differential pumping ensures that any rise in pressure from desorption of molecules from the sample surface will not raise the pressure in the sniffer section to a pressure that damages the RGA filaments.

The dual RGA system on HIDRA-MAT serves two purposes. The first is for data verification. Having two RGAs allows for data signals to be compared and establish confidence that the data is correct. The second reason is for

He and D₂ differentiation. He and D₂ have extremely similar masses and the resolution of the SRS RGA100 cannot distinguish between them. To overcome this issue the electron energy of one RGA is set to 25 eV (the lowest setting) and the other has it set to 50 eV. Ionization potentials of He and D₂ are 24.58 eV and 15.46 eV respectively. Though the electron energy of the 25 eV RGA is above the ionization potential of He, experimentation showed the mass 4 signal is negligible when flowing He into the chamber as seen in Fig. 7a. This approach is similar to another experiment using an RGA to differentiate He and D₂ in EAST [18].

Ultimately, the goal is to use the RGA data and pressure readings to quantify the amount of D₂ and He that is desorbing from the sample surface during TDS. A temperature of ~ 550 °C is needed to desorb most of the deuterium from W-Li films [19] which is well within the heater's capability. This data will enhance the understanding of PFC PMI by providing quantitative data about retention in the PFC immediately after plasma exposure. To quantify He and D₂ concentration in a mixed gas scenario, each RGA will be calibrated for He and D₂ signals at their respective electron energies like the graphs seen in Fig. 7 but with more data points to provide a complete and accurate data set. Using calibrated pressure readings, the 25 eV signal can be related to a physical pressure change because it is strictly dependent upon D₂. Figure 7b shows the relationship between the D₂ signal at each RGA electron energy so a relationship can be formed between the two. Converting the 25 eV signal and subtracting it from the measured 50 eV signal will give a value that can be converted into a physical pressure change due to He. A percentage He to D₂ can then be calculated because each gas' pressure contribution is known. Furthermore, the

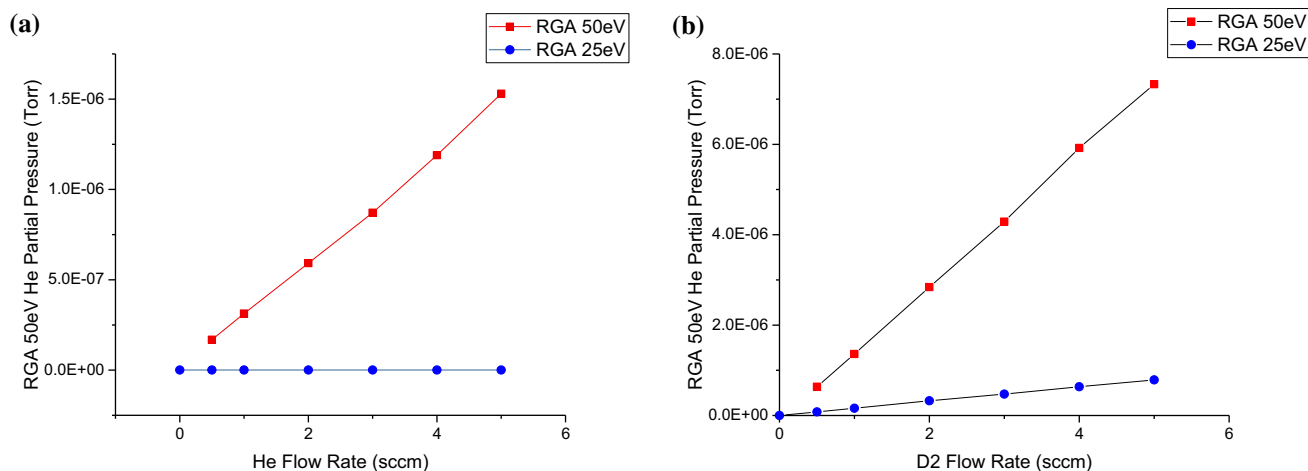


Fig. 7 Raw RGA data for **a** mass 4 partial pressure versus He flow rate and **b** mass 4 partial pressure versus D₂ flow rate. Data showing the RGA set at 25 eV shows no mass 4 signal when He is introduced

into the HIDRA-MAT chamber. The same 25 eV RGA shows a mass 4 signal when D₂ gas is introduced into the HIDRA-MAT chamber

pressure in the main chamber compared to the sniffer section will be different due to differential pumping. A CMR 365 baratron gauge attached to the main chamber will give a true pressure reading that can be used in combination with the calculated He and D₂ sniffer section pressures to determine the amount of He and D₂ desorbing from the sample surface.

Conclusions and Future Work

The optimization of next generation PFCs being employed in long pulse fusion devices will require a deeper understanding of different crucial topics such as chemical alteration, material mixing, and surface erosion produced following long term plasma exposure. HIDRA-MAT's design and development creates a unique material characterization test-stand that allows for a variety of *in-vacuo* fusion PFC testing. After the completion of fabricating and installing all HIDRA-MAT's components, many experiments are being planned. Preliminary experiments will include calibrating each of the characterization systems. This includes RGA calibration, laser tuning and calibration, and UHV sample heater testing. Following the preliminary experiments, an experimental campaign to demonstrate sample plasma exposure and *in-vacuo* surface characterization will take place. Such endeavors will provide experimental data that will help in the design, selection, and qualification of next generation PFCs.

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