## Materials and Innovations for Fusion Energy
## Room 321 - Session FUS-WeA

### Materials for Fusion Fuel Containment

**Moderators: Dr. Eric Dombrowski**, Commonwealth Fusion Systems, **Dr. Matthew Sharpe**, The Laboratory for Laser Energetics, The University of Rochester

2:15pm **FUS-WeA-1 MAX Phase Materials for Hydrogen Permeation Barriers***, **Eric Vogel**, Moses Nnaji,* Georgia Institute of Technology*; Dale Hitchcock,* Savannah River National Lab **INVITED**

Hydrogen isotope permeation is a critical materials challenge for fusion and other nuclear technologies, where tritium loss, inventory control, and degradation of structural alloys must be managed under high temperature and radiation. Conventional ceramic barriers such as $Al_2O_3$ can provide large permeation reduction factors, but brittleness, thermal-expansion mismatch, radiation sensitivity, and high processing temperatures limit integration with engineering alloys. This talk will present $Ti_2AlN$ MAX phase thin films as radiation-tolerant hydrogen isotope permeation barriers. MAX phases combine ceramic stability with metallic transport, radiation damage tolerance, oxidation resistance, and the ability to form protective alumina scales.

The presentation will focus on low-temperature vapor deposition routes for MAX phase Ti-Al-N coatings, especially two-stage reactive magnetron sputtering. Ti-Al-N precursor films are deposited near ambient temperature and then annealed to drive solid-state reactions and $Ti_2AlN$ crystallization. By controlling precursor architecture, composition, and modulation period, the formation pathway can be shifted from conventional high-temperature synthesis toward substrate-compatible processing. XRD, DSC-TGA, STEM show that Ti/AlN and TiN/TiAl multilayers follow distinct reaction pathways, with Ti/AlN enabling $Ti_2AlN$ formation at lower temperatures. Trace $Ti_2AlN$ was observed after extended annealing at 475 °C, an unusually low synthesis temperature for Ti-based MAX phase films by sputtering, while higher-temperature anneals produced crystalline $Ti_2AlN$ coatings for permeation testing.

Deuterium permeation measurements on coated Grade 91 steel demonstrate substantial flux reductions relative to uncoated steel. The best $Ti_2AlN$ coatings produced permeation reduction factors from tens to more than 1000, depending on temperature, pressure, film texture, and surface condition. Strong basal texture, improved crystallinity, and reduced high-angle grain-boundary content were correlated with improved barrier performance, consistent with anisotropic diffusion in layered MAX phase structures. Non-ideal behavior, including surface-limited dissociation, trapping, residual Ti-rich layers, oxide/carbon-rich surfaces, and pressure-induced flux changes, will be discussed. Together, these results identify processing-structure-property relationships that can guide radiation-hard, substrate-compatible MAX phase permeation barriers.

2:45pm **FUS-WeA-3 Atomic Layer Deposition on Novel Fusion-Relevant Substrates***, **Zachary Robinson**,* The Laboratory for Laser Energetics, The University of Rochester*; Soren Bentley,* UKAEA, UK*; Jeffrey Woodward,* NRL*; Alexander Kozen,* University of Vermont*; Thomas Cochran, Rashad Ahmadov, Joshua Ruby, Mark Wittman, Matthew Sharpe,* The Laboratory for Laser Energetics, The University of Rochester

Atomic layer deposition (ALD) is an ideal technique for deposition of films on arbitrarily-shaped containers such as those used to contain tritium for fusion applications. The substrates required for such containers generally differ from common ALD substrates, which have historically been primarily semiconductors. In this talk, I will give an overview of a custom ALD system built to deposit permeation barrier films on substrates and components relevant for various fusion and fusion fuel-cycle applications.

The primary benefit of our ALD system is the ability to deposit films on arbitrarily-shaped surfaces, such as the interior walls of tubing and canisters used to contain tritium. Initially, we deposited thermal ALD alumina films on both silicon wafers and planar copper foil substrates. Characterization with ellipsometry yielded ALD growth rates of ~1.1 Å/cycle for temperatures between 100 °C and 210 °C on Si witness samples. X-ray photoelectron spectroscopy (XPS) on both the Cu foils and Si substrates indicates stoichiometric $Al_2O_3$. To quantify the permeation reduction factor (PRF) of the of the ALD films, the permeability of deuterium through 25µm-thick Cu-foils coated with 10 nm of alumina was measured. We found that thin ALD films have a PRF of ~25 at permeation temperatures between 275 °C and 350 °C.

Finally, we discuss the difficulty of achieving a high quality ALD-stainless steel 316 (SS316) interface, which is complicated by the complex surface chemistry and the interactions with vapor-phase metalorganic species. The native oxide on SS316 substrates is reactive with TMA, and forms an AlOH layer, as evidenced by XPS measurements. To prevent this interfacial hydroxide formation, substrates were polished and conditioned in the ALD reactor. Following these steps, linear growth rates of 1.2Å/cycle were achieved. The ALD films on steel substrates resulted in orders of magnitude reduction in hydrogen permeation.

Current efforts include developing new materials in our ALD reactor, including a fundamental study of ALD bilayer and nanolaminate performance as a hydrogen-permeation barrier.

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3:00pm **FUS-WeA-4 Passivation of Stainless Steel for Tritium Service***, **Dale Hitchcock**,* Savannah River National Lab

Austenitic steels are generally preferred for hydrogen/tritium service due to their low hydrogen isotope permeability and relative resistance to hydrogen-isotope embrittlement. In applications where high purity gas is needed the surfaces in contact with hydrogen isotopes must be passivated to maintain gas composition. Passivation serves to minimize the surface features that catalyze isotopic exchange and to form a barrier against the ingress of impurities from the bulk. The passivation process generally includes an electropolishing step designed to minimize surface area by removing surface roughness and control surface chemistry followed by vacuum firing to both remove solubilized hydrogen from the bulk and also further modify the surface chemistry. Previously multiple vendors have supplied passivated components to the DOE, however, procuring "well" passivated components has become more difficult. This presentation will outline current efforts at SRNL to both evaluate the performance of vendor passivated material and also develop a robust passivation process inhouse.

3:15pm **FUS-WeA-5 Measurement of Hydrogen Isotopologues and Contaminant Species Within a Canister Coated with Alumina by Atomic Layer Deposition***, **Matthew Sharpe**, Thomas Cochran, Zachary Robinson,* University of Rochester

The interaction of tritium with pristine metallic surfaces catalyzes the formation of various hydrogen molecular isotopologues (HD, HT, DT, etc.). Additionally, because these surfaces have not been exposed to tritium, protium (H) native to the surface will enter the gas phase by isotope exchange with tritium. For many Fusion applications, this dilutes the gaseous tritium concentration, thereby contaminating the gaseous tritium with undesired isotopes. Furthermore, protium present within the bulk structure of the metal can act as a source of additional contamination. Finally, any trace organics present on the surface may also enter the gas phase as tritiated compounds. These organic molecules may poison other tritium technologies or become a health or environmental risk. Mitigating this contamination may be possible by use of atomic layer deposition (ALD) of low permeable materials.

In this work, we show the results of an aging study of a stainless-steel (type 316) canister coated with an alumina layer deposited on the interior surface by ALD. The canister was mounted in a custom ALD system designed for deposition on arbitrarily-shaped and fusion-relevant components. In a series of measurements performed to evaluate the deposition in a canister similar to those used in this study, a 10% variation in film thickness was observed between the inlet and outlet of the canister. Films of > 10 nm were used for this study, deposited at a temperature of 150°C.

Once the ALD-coated canisters were prepared, several similar canisters were prepared with pre-treatments including solvent surface cleaning and vacuum baking. After pre-treatment or ALD coating, the canisters are exposed to tritium gas. Assessment of the effectiveness of various treatments is done by periodically sampling the gas phase and measuring with gas chromatography (GC). The GC method utilized allows for simultaneous injection of a sample into two columns. One column allows for measurement of common impurities ($CH_4$, $CO_2$, CO, $O_2$, $N_2$, Ar), while the second allows for discrimination of hydrogen isotopologues ($H_2$, HD, HT, $D_2$, DT, $T_2$).

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3:30pm **FUS-WeA-6 Native Protium Content in Stainless Steel Pipework**, ***James Pittard***, *Kyle Shipman, Zhiheng Wu, Tshepo Mahafa, Rosemary Brown, Gowri Karajgikar, Iryna Bennett,* UKAEA, UK

Stainless steel pipework is used extensively for transporting gases, ranging from laboratory-scale experiments up to larger scale facilities. Such pipework is known to contain non-negligible amounts of native protium [J. Vac. Sci. Technol. A 21(1) (2002) 167–174], outgassing of which can limit the achievable vacuum and contaminate gas streams. For fusion applications, protium contamination could add additional requirements to an isotope separation system with the potential to change both the concentrations and speciation of isotopologues within gas flows. To assess the impact of native protium outgassing, an understanding of both the total inventory and the desorption energetics is required. This information can also be used to indicate potential tritium retention and transport through stainless steel components. Therefore, a series of thermal desorption spectroscopy (TDS) measurements have been carried out on samples taken directly from various pieces of SS316 pipework to give a value for inventory and an estimate for the variation between different pieces of pipe. TDS spectra consisted of a single broad peak which gave total inventory values of the order of 1018 H atoms g-1 (101 mol H atoms m-3). Spectra were fitted with a simple Fickian diffusion model using FESTIM [Int. J. Hydrogen Energy 63 (2024) 786–802] giving an activation energy for diffusion of 0.55 eV which aligns well with expected values. Some samples were measured twice, with the first measurement at a lower temperature meaning a desorption peak in the second measurement could still be seen. The model was able to reproduce desorption spectra for both initial and secondary measurements, whilst its simplicity would aid integration into larger, system scale models.

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