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Boronization System for the NSTX-Upgrade Fusions Device

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BORONIZATION SYSTEM FOR THE NSTX-UPGRADE FUSION DEVICE

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Boronization has been widely used as a key vacuum interior wall conditioning method to improve plasma performance in fusion devices. In this paper we report on a new boronization system designed and built for the NSTX-Upgrade fusion device (NSTX-U). Deuterated trimethylboron (C_3D_9B) is used to produce a thin boron containing coating on plasma-facing components (PFCs) via a glow discharge with vessel pressure maintained in the 1.5-2.0 millitorr range. This hard protective coating reacts with active molecules and reduces the amount of impurities observed in the plasma. Deuterated trimethylboron (dTMB) is a toxic and pyrophoric gas and great care must be used in the handling of this hazardous gas. Details of this new system are described which include enhanced equipment and operational safety, programmable logic controller (PLC) control of the process, improved equipment maintenance and serviceability, and increased operating pressure range and process flexibility. Results from boronization using this newly developed system will also be covered at the end of this paper.

Key words: Boronization, Fusion Device, Deuterated Trimethylboron

I. INTRODUCTION

The National Spherical Torus eXperiment (NSTX) has recently undergone a major upgrade to NSTX-U at Princeton Plasma Physics Laboratory (PPPL). NSTX-U will double the toroidal field, plasma current, and neutral beam injection heating power, as well as significantly increase the pulse duration [1]. Control of the influx of impurity gases and fuel recycling from plasma facing components (PFCs) are critical for NSTX-U plasma performance. Wall conditioning technologies such as baking and glow discharge cleaning (GDC) are typically used to remove water, hydrogen and other impurities trapped on PFCs [2]. After baking and GDC, other conditioning technologies have been used to deposit a thin layer of reactive material, i.e. a getter, on the plasma facing surfaces to further reduce the plasma impurities such as carbon, oxygen and metals, as well as to decrease the chemical and physical erosion of the walls by deuterium atoms and ions. Boron and lithium are among the most common reactive coating materials used for wall conditioning. Boron or boron/carbon coatings are typically applied onto PFCs through plasma enhanced chemical vapor deposition (PECVD)[3-10], during which boron precursors, typically diborane or trimethylboron, are dissociated in helium plasma and the ionized boron containing radicals then impinge onto the PFC walls with high energy to form a hard, reactive and corrosion resistant coating.

In this paper, we report on the new boronization system built for NSTX-U. Designed for enhanced operational safety and fully automated process control, this new system has been successfully used to condition the PFCs for NSTX-U during the FY16 plasma campaign. Deuterated trimethylboron (dTMB) was selected as the precursor gas since it is much less hazardous than diborane, and our previous boronization results from NSTX using dTMB[2,5] have showed similar beneficial effects compared with the boronization results of TFTR in which diborane was used as the precursor gas[4]. Deuterated TMB is also used to minimize the hydrogen inventory in the vacuum vessel, since hydrogen can cause parasitic resonance effects during RF experiments [5]. With an increase in the number of dTMB injections ports on NSTX-U vacuum vessel and reduced deposition pressure, the coating thickness and uniformity were improved.

II. SYSTEM SETUP

II.A. Gas Management System

Figure 1 shows the dTMB gas management system. It is designed to be capable of conducting both helium GDC and boronization. Five bottles of dTMB gas are placed inside a purged gas cabinet, with one bottle connected online and the remaining four strapped on to the supporting shelves. The dTMB bottle is filled with about 380 PSIG of 5% dTMB in 95% He, so each bottle contains slightly less than 10 grams of dTMB. In addition one 50-liter helium bottle is placed outside of the gas cabinet and secured on the right side. A helium gas control panel is mounted on the right side of the gas cabinet above the helium bottle, and a mass flow controller (MFC) cabinet is mounted on the left side. Both pure helium and dTMB/helium mixture can be delivered through the same MFC and the operation can be switched between helium GDC and dTMB boronization without interruption. The electrical control box with PLC modules is on top of the gas and MFC cabinet, with an emergency gas shutoff switch and a system emergency shutoff switch on the front panel. The vacuum pumping assembly is capable of pumping on any section of the gas delivery system and is located under the MFC cabinet. This dry mechanical pump also serves as the backing pump for the turbomolecular pump (TMP) on NSTX-U vessel during He GDC and dTMB boronization. The dTMB gas delivery consists of a single coax line between the gas management system and the vacuum vessel where it is then split into separate lines to the three gas delivery ports.

Fig. 1. dTMB gas management layout for the boronization of NSTX-U

II.B. NSTX-U Setup

The NSTX-U vacuum vessel has twelve bays (A-L) evenly distributed toroidally, with a total internal volume about 28 m^3 and approximately 41 m^2 of plasma facing surface. Previous boronization results for NSTX [2] showed that the boron deposition thickness on the PFCs was very non-uniform. For NSTX-U the number of dTMB injection ports was increased from one at Bay L midplane in NSTX to three at bay C lower dome, at bay D upper center stack, and at bay F midplane, respectively. To initiate and maintain the plasma for dTMB dissociation, two fixed filaments and two electrodes are installed in vacuum vessel. The two filaments are biased to 500 volts to create emission electrons that initiate the breakdown of the glow discharge. The two electrodes located at bay G and Bay K mid-plane are connected to 1000 VDC power supplies. Four quartz crystal microbalance (QCM) sensors are used to monitor the deposition rate and deposition uniformity during the boronization process, and are located on the internal vessel wall at bay E top, bay B midplane, bay I midplane and bay F bottom [11]. The approximate physical layout of the dTMB gas injection ports, the high voltage electrodes and the QCMs are shown in Figure 2. A residual gas analyzer (RGA) is used to monitor the residual vacuum species content and partial pressure before, during and after boronization. It has its own high vacuum pumping system and during boronization an orifice is inserted to maintain a pressure differential between the GDC and RGA.

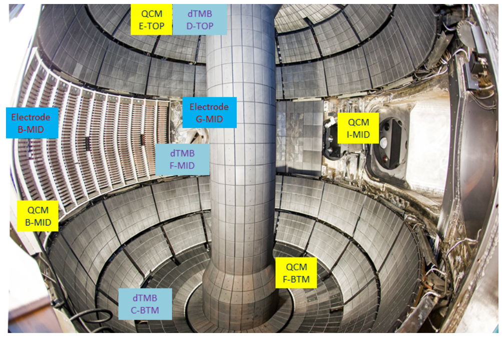

Fig. 2. Physical location of the dTMB injection ports, the glow discharge electrodes and the QCMs on the inner wall of NSTX-U vessel. Picture is taken from the Bay A neutral beam port.

II.C. Vacuum Pumping System

The torus vacuum pumping system consists of two 2800 L/s TMPs backed by a roots/mechanical pump package. For NSTX-U boronization, only one of the TMPs is used and the dTMB system dry mechanical pump, with a helium pumping speed of 68 L/s, is used to back the selected TMP. This dry pump is placed under the MFC cabinet and the distance between this pump and the vacuum vessel is about 50 feet. A detailed calculation was conducted to determine the vacuum pumping line size to ensure the overall effective pumping speed matches the required gas load, which is approximately 2.5 torr*L/s.

III. SAFETY DESIGN AND ANALYSIS

dTMB (C_3D_9B) gas is toxic and pyrophoric. It is colorless with a repulsive odor. The NFPA 704 health rating of pure dTMB is 4, and, therefore, to reduce toxic level and overall risk of storage and handling, 5% dTMB in 95% helium is used, which has a NFPA 704 health rating of 1.

Equipment and operational safety design of the dTMB boronization system were centered on the safe use of dTMB gas. Both the dTMB gas and the MFC cabinets are kept at negative pressure to prevent any potential gas leakage to the test cell. Pressure switches were used to ensure pressure at the inlet of cabinet top exhaust duct was more than 0.5 inch water (1 Torr) lower than the ambient pressure. The wire reinforced safety glass viewing window on the dTMB cabinet allows us to turn on/off dTMB gas bottle cylinder valves or connect/disconnect the CGA 350 fitting on dTMB gas bottle through the window area when it is open. Fixed gas detectors, calibrated with specific TMB gas mixtures (3 to 500 PPM), were used to detect any dTMB gas leakage inside the cabinet. A 24VDC stand-alone horn and signal light module is mounted on cabinet top to indicate the status of the dTMB gas cabinet through the combination of audible and visual signals. This module produces about 75 dBA @10' and has green for normal, yellow for warning and red for alarm based on the dTMB gas concentration detected in the cabinet. The suggested time weighted average (TWA) personnel exposure limit for dTMB is 7 PPM [5]. Therefore the dTMB warning level and alarming level were set to 3.5 PPM and 7 PPM respectively.

A fire sprinkler with a fuse rating of 68 °C was installed at the top of the dTMB cabinet to cool the dTMB gas cylinders in case of fire. All the fittings were sealed with metal gasket and leak checked to a level better than 1.0×10^{-9} SCC/sec. The gas delivery lines consist of a 1/4" SS316 line for the process gas enclosed in a coaxial ½" SS316 line for containment. The outer jacket area is pumped and backfilled with 1100 torr helium and the pressure is monitored for a leak. Helium is used so that if the inner process tube, which is typically under vacuum during process, ever leaks, there will be no process contamination for NSTX-U vessel. All the gas lines can be pumped by the dry mechanical pump to millitorr range and back filled with helium. Critical pressure, temperature and flow rate readings are monitored by the programmable logic controller (PLC) for proper system interlocks. The dTMB PLC also communicates directly with the NSTX-U main PLC systems. Figure 3 shows the detail of the PLC page for the dTMB system. The status of all the gas components from gas bottle to pump exhaust are shown on the PLC page, as well as the important process values and system safety interlocks.

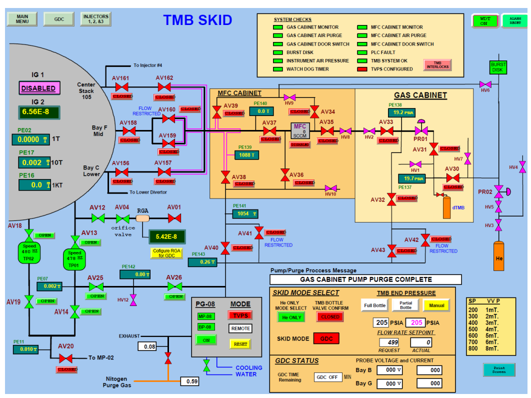

Fig. 3 The PLC page of the fully automated dTMB boronization process for NSTX-U

A detailed calculation was conducted to ensure the internal cabinet purge flow rate was sufficient to prevent any leaked dTMB concentration in gas cabinet reaching the low flammable limit, which is about 0.5% in air. The calculation showed that, with a dilution air flow of 300 SCFM, a full online bottle of dTMB needs to be released into the gas cabinet in less than 5 seconds to reach 0.5% of dTMB in cabinet, which is very unlikely to occur. Nitrogen is used to dilute the exhaust dTMB residual gas so that the boron containing residual gas concentration in the sealed exhaust line after the dTMB mechanical vacuum pump is lower than the TWA 7 PPM. The flow rate is based on a conservative estimate that 10% of the supplied dTMB will reach the vessel exhaust as boron containing residual gas. The actual nitrogen dilution flow rate is monitored and PLC interlocked to ensure required flow rate is always above the set value. PPPL has set an administrative limit of 50 g maximum allowed inventory of dTMB at any one time in the NSTX-U test cell. Each dTMB bottle contains slightly less than 10 grams of pure dTMB, and the gas cabinet is designed to hold a maximum of 5 bottles.

The design was also extensively reviewed and approved by PPPL physics and engineering, as well as staff from the safety division. Comprehensive Failure Modes and Effects Analysis (FMEA) was also conducted to examine possible accident scenarios and the mitigation plan. Emergency Service Unit (ESU) personnel were trained to respond to a dTMB leak or fire.

IV. SYSTEM OPERATION

Before boronization, a preoperational test procedure (PTP) and a dry run using helium via the new dTMB system were successfully conducted. A team of NSTX-U technicians and engineers were trained on the system and operational procedures. Prior to each operation, a pre-job brief was held to review the hazards and operations.

Two detailed operation procedures were developed to ensure safe dTMB operation. One of the procedures deals with the dTMB gas cylinder changeouts. Prior to removing a depleted cylinder and replacing it with a full dTMB cylinder, the cylinder valve is verified closed and then the line is pump purged with the helium system via an automated program in the PLC. The CGA350 fitting for dTMB gas bottle is then loosened through the opened window of the cabinet while a purge flow of air away from the operator is maintained. After the CGA350 fitting is removed from the depleted gas cylinder and capped off, the cabinet door is opened to remove the cylinder and secure a new full bottle in the cabinet. The new cylinder is then connected to the CGA350 fitting through the opened window. For consistency the CGA fitting is tightened with a pre-set toque wrench. The lines exposed to air during bottle change are pumped and back filled with helium three times, and a leak check is conducted to ensure the CGA connection is leak tight using an automated program in the PLC.

The second procedure is for dTMB boronization and the follow up helium GDC. This procedure allows the deposition of various amounts of boron with chosen injection ports and vessel pressure. A helium plasma is initiated with filament biased at 500 V. Before switching to dTMB, filaments are turned off and allowed to cool to minimize the formation of tungsten boride, which causes filament embrittlement. The GDC electrodes are typically maintained around 550 V during boronization. At a boronization pressure of 1.7 millitorr, it takes about 6 hours to deplete one full bottle of dTMB gas. As with the bottle changeout, this process is fully automated using the PLC in order to minimize the possibility of human error. RGA readings and key process parameters are recorded periodically by the operators during boronization. Up to two hours of helium only GDC was routinely conducted after boronization to deplete the plasma facing surface of excess deuterium.

V. RESULTS AND DISCUSSIONS

Fifteen bottles of dTMB gas were used during NSTX-U FY16 campaign. Significant reduction of impurities such as oxygen was achieved with boronization [12]. Experiments were also conducted to measure the effect of using different injection ports, boronization pressure and electrodes on the boron coating thickness and deposition uniformity.

Figure 4 shows the RGA spectrum of the vessel exhaust during dTMB boronization at a vessel pressure of 1.7 milli-torr. dTMB (amu= 65) was *not* observed at vessel exhaust, indicating that dTMB was fully dissociated in vessel. RGA peaks at 46 and 47 amu were observed, corresponding to a fragment of deuterated di-methyl boron, $B(CD_3)_2$, as shown in Fig.4. However, it is estimated that the volume percentage of the boron residual is less than 2% of the supplied dTMB, which indicates very high deposition efficiency, and that the specified dilution nitrogen gas at pump exhaust can drop the dTMB residual concentration to about 1.4 PPM.

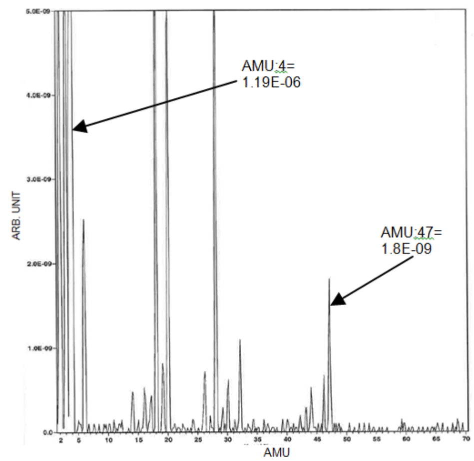

Fig. 4. RGA reading of the vessel exhaust during boronization

Figure 5 shows the relative coating thickness at different vessel pressure and location. For each case a total of 1.5 grams of dTMB was injected from the Bay C bottom injection port only. It is shown that at a boronization pressure of 4 millitorr, coating thickness achieved at Bay F bottom QCM is about 5 times of at Bay E top QCM. When glow discharge pressure is lowered to 2 millitorr, the overall coating thickness at Bay F bottom QCM is almost doubled, while the thickness at Bay E top QCM is increased by 5 times. This indicates that lowering the boronization pressure not only increased overall coating thickness at vessel top and bottom, but also significantly improved the deposition uniformity.

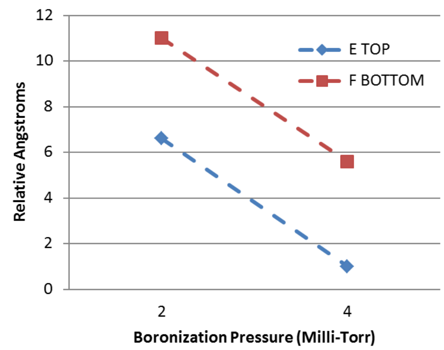

Fig 4. Coating thickness at Bay F bottom and Bay E top under boronization pressure of 2 and 4 millitorr

Experiments were also conducted to find out the effect of dTMB injection location on coating thickness and uniformity[12]. Injection from bottom resulted in about 20% more deposition at the lower vessel QCMs, while injection from the top showed enhanced top deposition. However, the deposition was largely dominated by the location of the electrodes. The Bay B midplane QCM was 0.65 m away from the Bay B GDC electrode and experienced about 10 times more deposition than the top or bottom QCMs, regardless of the deposition pressure and injection location. The anode has a much lower surface area compared to the cathode (vessel wall) and this leads to the formation of an anode glow and an order-of-magnitude higher ion flux in the vicinity of the anode.

Previous boronization results for NSTX revealed that coating thickness at midplane is over 30 times higher than at top or bottom of the vessel. The uniformity increase for using the new dTMB system is mainly due to lower boronization pressure and added injection ports. To further improve deposition uniformity, electrodes should be added at top and bottom of the vessel. Also to achieve profiled deposition, such as more deposition at top or low divertor area, it is important to have independent control of the different sets of electrodes. Boronization pressure has been set at 1.7 milli-torr currently. Reducing the plasma pressure to 1 milli-torr is possible for the new dTMB system, which should further increase the deposition efficiency, as well as deposition uniformity. However the deposition time will be significantly increased also by dropping the vessel pressure. Use of 10% dTMB mixture would halve the boronization duration time and still comply with safety standards.

VI. ACKNOWLEDGMENT

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