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Characterization and conditioning of SSPX plasma facing surfaces

D.A. Buchenauer^{a,*}, B.E. Mills^a, R. Wood^b, S. Woodruff^b, D.N. Hill^b,
E.B. Hooper^b, D.F. Cowgill^a, M.W. Clift^a, N.Y. Yang^a

^a Sandia National Laboratories, MS-9042, P.O. Box 969, Livermore, CA, USA

^b Lawrence Livermore National Laboratory, Livermore, CA, USA

Abstract

The Sustained Spheromak Physics Experiment (SSPX) will examine the confinement properties of spheromak plasmas sustained by DC helicity injection. Understanding the plasma-surface interactions is an important component of the experimental program since the spheromak plasma is in close contact with a stabilizing wall (flux conserver) and is maintained by a high current discharge in the coaxial injector region. Peak electron temperatures in the range of 400 eV are expected, so the copper plasma facing surfaces in SSPX have been coated with tungsten to minimize sputtering and plasma contamination. Here, we report on the characterization and conditioning of these surfaces used for the initial studies of spheromak formation in SSPX. The high pressure plasma-sprayed tungsten facing the SSPX plasma was characterized in situ using β -backscattering and ex situ using laboratory measurements on similarly prepared samples. Measurements showed that water can be desorbed effectively through baking while the removal rates of volatile impurity gases during glow discharge and shot conditioning indicated a large source of carbon and oxygen in the porous coating. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Plasma-surface interaction; Tungsten; Conditioning

1. Introduction

Over the past few years, improvements in performance in many tokamak experiments have resulted from the heightened attention paid to understanding wall conditions. Techniques for wall conditioning have reduced plasma impurity levels and lowered recycling of hydrogenic fuels, leading to improved confinement modes such as the super shot and VH-mode. It is to be expected then, that similar techniques will be needed to improve the performance of other innovative confinement devices included in the widening scope of the US Fusion Science program. While similarities to the tokamak exist, differences in the edge plasma and mag-

netic configuration of these experiments and renewed interest in refractory materials (due to codeposition of hydrogen with carbon) present new challenges to optimize plasma-wall interactions in these devices. The present work describes the initial efforts to characterize and condition the plasma facing surfaces in one such device, the Sustained Spheromak Physics Experiment (SSPX) at Lawrence Livermore National Laboratories.

The motivation for SSPX can be found in recent articles [1–3]. Initial experiments aimed toward establishing the spheromak magnetic configuration began in April 1999 and were accompanied by significant periods of wall conditioning. During this initial period of operations (through August 1999) and prior to the second period (October 1999), the diagnostic capabilities of the machine were limited (magnetics; gun voltage, current, and flux) and residual gas analysis was used as the primary indication of the effectiveness of wall conditioning. More recent analysis of the plasma impurity levels and plasma density will be reported by Wood [4].

* Corresponding author. Tel.: +1-925 294 3570; fax: +1-925 294 3231.

E-mail address: dabuche@ca.sandia.gov (D.A. Buchenauer).

2. Experimental setup

The configuration of SSPX is similar to that of CTX [5] and is shown schematically in Fig. 1. To form the spheromak plasma, a voltage is applied across the gap between the injector inner (negative) and outer (ground) electrodes as gas (H_2 , D_2 , or He) is introduced from 4 to 8 high speed gas valves mounted on the 45° shelf near the region labeled 'Injector Discharge Region'. $\mathbf{J} \times \mathbf{B}$ forces push the plasma out of the injector toward the flux conserver where reconnection occurs to form the spheromak. Given the high currents supplied at the injector (up to 450 kA) and expected in the main plasma (up to 1.2 MA), the solid copper flux conserver has been carefully shaped to minimize dissipation and provide

stabilization of global MHD modes. A more detailed description of spheromak formation schemes can be found in the recent book by Bellan [6].

The plasma facing surfaces in SSPX include regions in the injector and the main spheromak volume. All of these surfaces are made from 1.27 cm thick copper (OHFC) and protected with a 100 μm thick, high pressure plasma-sprayed (HPPS) tungsten coating. The protection derives from the higher sputtering threshold and melting temperature for tungsten compared to copper. The surface area of this coating exposed to the formation region is comparable to that exposed to the main spheromak plasma (see Table 1).

The HPPS tungsten coating was fabricated at Hayden Corporation (West Springfield, MA) by introducing

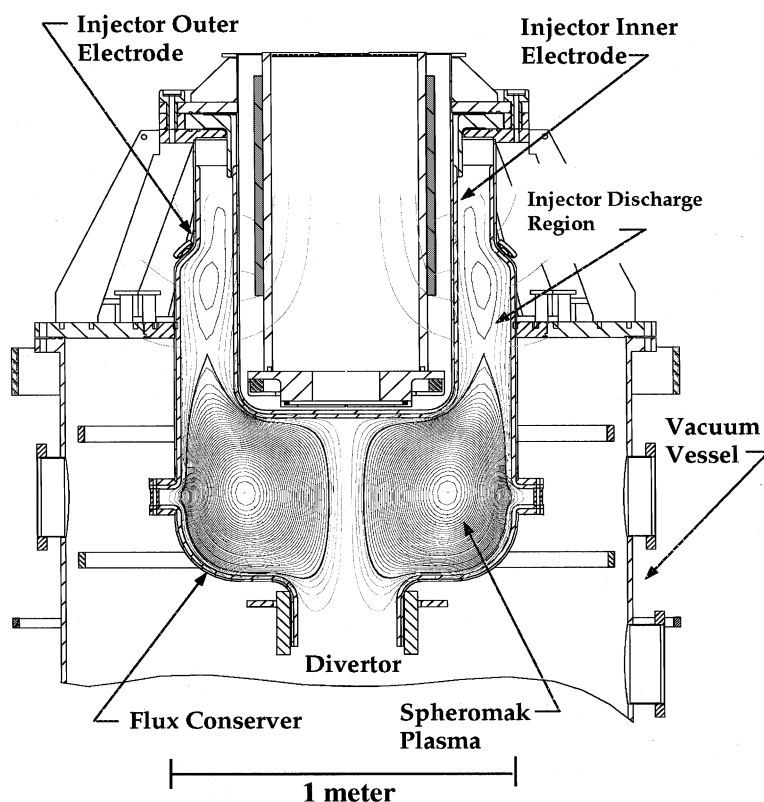


Fig. 1. Cross-sectional view of the toroidally symmetric SSPX device, showing the inner and outer electrodes, bias field coils, and flux conserver.

Table 1

	Inner electrode (m^2)	Outer electrode (m^2)	Flux conserver
Main spheromak plasma	0.36	0.71	4 m^2
Plasma formation region			
Upper ($d_{\text{gap}} = 10$ cm)	0.54	0.70	
Lower ($d_{\text{gap}} = 16$ cm)	1.1	1.6	

44 μm grain tungsten powder into an argon plasma torch (in air). While multiple passes of the torch can be used to build up a thick coating ($\approx 10 \mu\text{m}$ pass), the resultant porosity is known to be higher than that of low pressure techniques [7] and oxide formation between the layers can occur. The porosity is beneficial, however, in reducing thermal stresses that result from the thermal expansion mismatch between the tungsten and copper ($4\times$ at 293 K). Similar coatings were used on the CTX cathode and in HIT. The 100 μm thickness was based on the thickness-dependent, nitrogen desorption rates for the coating and SSPX vessel pump-out needs.

3. Characterization of HPPS tungsten

Characterization of the plasma facing surfaces in SSPX has been done by both in situ methods and laboratory techniques using similarly prepared coatings on 1.27 cm thick copper. Samples were also examined by thermal desorption spectroscopy for comparison with residual gas analysis during SSPX bake-out. Due to the high porosity of the coating, no solvent cleaning was attempted on the coated components installed in SSPX.

During initial inspection of the HPPS tungsten, variations in texture were observed on the flux conserver, suggesting that there were thickness variations. To determine the uniformity of the coating, β back-scattering measurements [8] were done on all components and calibrated using sample coatings of varying thickness. The hand-held device uses a gas proportional counter to measure the back-scattering of electrons from a $^{90}\text{Sr}/^{90}\text{Y}\beta$ source.

Toroidal measurements along the bottom of the flux conserver and at three heights on the inner electrode (bottom radius, 25 cm from the bottom, and 41 cm from the bottom) indicated that the uniformity of the coating was within the measurement accuracy (approximately 15%). In addition, spot measurements on all coated components showed that the coating thickness exceeded the 100 μm nominal value on all surfaces except on the inner electrode in the 'Injector Discharge Region' (25 and 41 cm from the bottom of the electrode), where values were about 15% below nominal. Subsequent measurements made at the end of the initial operations period did not show any erosion of these surfaces; however, the appearance of the inner electrode (used as the cathode) changed from the as-delivered dull gray to a shiny appearance. The role of shot conditioning in producing this change will be described below.

Samples of 125 μm thick HPPS tungsten were studied using optical microscopy and scanning electron microscopy (SEM) to evaluate the coverage and structure of the coating. The surface topology revealed lamella structures characteristic of plasma-sprayed materials (see Fig. 2). The apparent porosity is connected to the

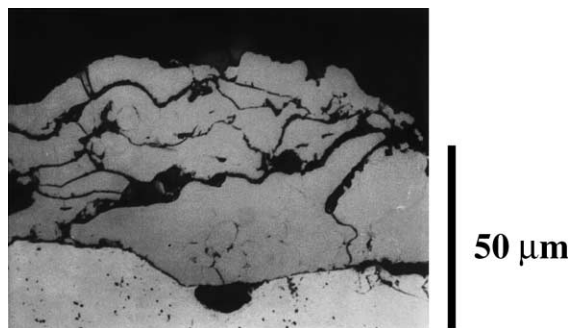


Fig. 2. Optical micrograph of a 125 μm (nominal) thick HPPS coating on copper.

surface and results in a larger effective surface area (up to six times the planer area using features above 1 μm). Lower porosity regions were also observed that contain larger spherical structures, indicative of inadequate melting of the tungsten powder in the plasma torch. The high porosity and lack of melting in some areas can result in low thermal conductance in the coating and between the coating and copper substrate.

Surface composition measured by Auger Electron Spectroscopy (AES) on an outer layer lamella showed concentrations of oxygen and carbon on the outer surfaces (45% and 30%, atomic) typical of metal surfaces. Depth profiling indicated that the carbon was only on the surface and that the oxide layer was 15 nm thick. Since the cooling of the tungsten layers was done in atmosphere, growth of an oxide layer during each pass of the plasma torch could lead to significant quantities of oxygen contamination within the coating. To examine the internal layer surfaces, a sample was fractured and examined by a JEOL-6400 SEM. Measurements of the atomic percentage of oxygen by Energy Dispersive X-Ray Spectroscopy (EDS) were made at several locations on both the top surface of lamella and on the fractured surface. The concentrations on the outer lamella surfaces were comparable to those on internal surfaces and are in rough agreement with the 15 nm depth determined by AES (levels on the fractured surfaces were three times lower). The observations are consistent with a HPPS growth sequence in which each newly deposited layer of tungsten forms an oxide surface, with incomplete bonding of the lamella to the next deposited layer. Since the vaporization of tungsten oxide occurs well below the melting point of tungsten, the vapor could provide a mechanism preventing the bond formation.

4. Conditioning methodology

Since the porous HPPS tungsten was not cleaned with solvents, it was expected that the carbon and water

contamination on the plasma facing components in SSPX could be considerably higher than that found on smooth metal surfaces found in early tokamaks. For comparison, the use of glow discharge conditioning in PDX resulted in the removal of 100 monolayers of equivalent carbon [9]. For SSPX, the mass flow of volatile impurity species was used as a metric for evaluating the effectiveness of the initial conditioning.

The initial conditioning of the HPPS tungsten consisted of baking for water removal, glow discharge conditioning (GDC) for surface carbon and oxide reduction, and shot conditioning to remove residual surface contamination and more deeply trapped impurities (by surface heating). During this time period (May–August and October 1999), conditioning periods were inter-dispersed between machine operations (one partial loss of vacuum) and diagnostic installations. Following this period, titanium gettering of the HPPS tungsten has been used to further improve vacuum conditions and reduce plasma impurity levels.

The SSPX vessel (volume 7900 l) is pumped by a 1000 l/s (H_2) turbomolecular pump, backed by an oil free scroll pump. Up to pressures of 10^{-4} Torr, the vessel pumping speed is conductance limited by the pumping duct (550 l/s). GDC is operated at pressures above 10^{-4} Torr, where the pumping speed for H_2 and other volatile impurity species is significantly lower. The GDC working gas pressure above 10^{-3} Torr is measured by a 10 Torr (now 1 Torr) capacitance manometer. Residual gas pressures in the vessel are monitored by a differentially pumped mass spectrometer (1–100 amu). Sampling of the vessel gasses during GDC is done through a restricted conductance valve to keep the total pressure at the mass spectrometer below 10^{-4} Torr. To relate the partial pressure of volatile impurity species in the vessel to those at the mass spectrometer during GDC, a calibration was performed by flowing a measured amount of the dominant volatile impurity gases (CH_4 , CO , and CO_2) into SSPX at several H_2 pressures corresponding to GDC conditions. The mass spectrometer to vessel pressure ratio is then given by dividing the calculated vessel pressure ($p_x = Q_x/S_x$) by the mass spectrometer pressure. Here p_x is the vessel partial pressure of volatile species x , Q_x is the mass flow of species x out of the vessel (and in this case, into the vessel), and S_x is the vessel pumping speed of species x .

5. Results

Fig. 3 shows a thermal desorption spectrum for a 125 μ m thick HPPS tungsten coating as measured in the Armor Conditioning Experiment (ACX) at Sandia. 18% of the water was removed during the first 3000 s by ramping the temperature to 210°C (SSPX vessel temperature limit), with another 80% being removed by

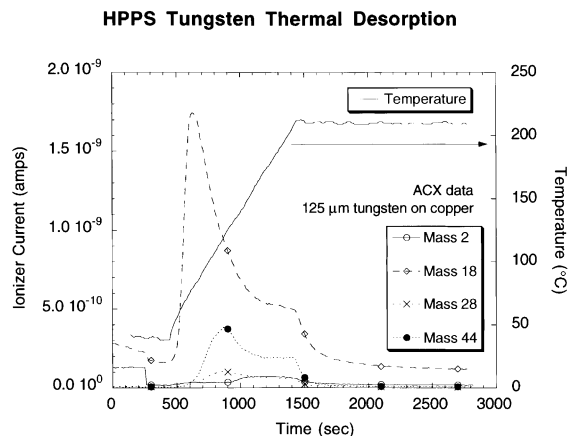


Fig. 3. Ionizer current from the mass spectrometer versus time from a 125 μ m (nominal) thick HPPS coating during thermal desorption.

maintaining this temperature for about 70 h (the remaining 2% was desorbed through a thermal ramp to 600°C). From the shape of the water desorption curve, it is clear that temperatures above 150°C are adequate to remove water. While this system was not absolutely calibrated for water removal rates, the quantity of water liberated greatly exceeds other non-porous metals examined and is larger than any of the other volatile impurities liberated from this sample.

Similar trends were observed in the residual gas analysis made during bake-out cycles in SSPX, where H_2O dominates the initial vessel pressure by at least a factor of 10. The partial pressure of mass 16, 18, 28, 32 and 44 were all reduced by an order of magnitude by baking the vessel at 170–180°C for 400 h. Baking is typically operated until the H_2O peak falls significantly below the H_2 peak (ultimate pressure of 10^{-8} Torr).

During H_2 plasma operations, it was observed that H_2O levels could rise to pre-bake levels, dominating the residual gas spectrum. Since it is well-known that reduction of tungsten oxide by hydrogen occurs if the temperature approaches 600°C, it is possible that either (1) small regions of the HPPS tungsten coating are heated to high temperatures during discharges, or (2) hydrogen ion induced reduction can occur at lower temperatures. Tungsten oxide does not volatilize below 800°C, therefore, reduction should occur first. Observations of the H_2O and D_2O partial pressures during the first deuterium plasma operations also indicated the formation of water in the HPPS tungsten coating. Following a large set of He fueled discharges (in which the H_2O levels fell), water levels were observed to rise several orders of magnitude. First mass 18 levels increased two orders of magnitude during a set of 13 H_2 fueled shots, and then mass 20 levels increased to almost the same level during a set of 14 D_2 fueled shots (mass 18

levels remained constant). This level of D₂O is too large to result from the isotopic abundance of ²H (1.5×10^{-4}) or ¹⁸O (2×10^{-3}) while isotopic exchange is slow and would result in a reduction of the mass 18 peak.

During initial operation of SSPX, hydrogen GDC was used routinely to reduce surface carbon and oxide levels. The hydrogen pressure varied from a lower limit of 40 mTorr needed to sustain the sheath to 120 mTorr required to access the gap between the inner and outer electrodes. The reduced pumping speeds obtained at these pressures are shown in Table 2. The pumping speeds of volatile impurity gases produced by the GDC are measured by fitting their pump-out curves in the working gas flow (see Fig. 4).

The glow discharge current is supplied by a current limited, 600 V, 11.25 A DC power supply, using a 1 Ω resistor in series to measure the current. Typical working voltages vary between 250 and 400 V, with currents ranging from 100 to 1400 mA. The voltage required for a given current has decreased as the wall conditions have improved. During the initial period of operations, the inner electrode was alternatively biased as cathode and anode. Simultaneous conditioning of all tungsten surfaces is now possible using a movable anode (3.8 cm × 12.7 cm).

Table 2

Pressure (mTorr)	Pumping speed (l/s)	Gas flow (Torr l/s)	Residence time (s)
40	35	1.3	220
120	16	1.9	500

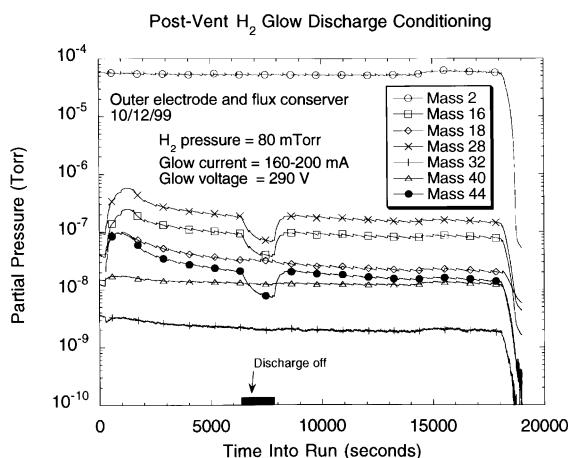


Fig. 4. Mass spectrometer partial pressures (sampled through the small conductance) observed during hydrogen GDC. Pumping speeds for the volatiles are calculated using the e-folding drop of pressure when the discharge is turned off. The glow current was not held constant during the first thousand seconds.

Fig. 4 shows the evolution of volatile impurity species during a typical shift of hydrogen GDC following a vessel bake-out. The dominant species (CO and CH₄) and CO₂ increase by 1.5–3 during the discharge while O₂ and H₂O do not show an increase. Oxygen is typically difficult to observe in vacuum systems while the H₂O production through GDC has been shown to be negligible [10]. Operation with helium as the working gas did not increase the volatile impurity species pressures. Dylla [11] has attributed this effect on stainless steel to the role of atomic hydrogen in the formation of CH₄, CO and CO₂ in the carbon/oxide layer on the metal surface.

The mass flow out of the vessel can be estimated from the partial pressures of volatile impurities and their pumping speeds in the working gas: $Q_x = S_x p_x$. Using the calculated vessel partial pressures for CH₄, CO and CO₂ from the mass spectrometer partial pressures in Fig. 4 and the measured pumping speeds from the pressure drops in Fig. 4 ($S_{16,28,44} \approx 28$ l/s), the mass removal rate for carbon-containing molecules is 7×10^{-3} Torr l/s, or 2.5×10^{17} carbon atoms/s. Given the GDC cathode area from Table 1 for these conditions, this represents approximately 0.004 monolayers of carbon equivalent/s being removed by the GDC. Mass flow rates during the initial conditioning of SSPX were about twice this rate.

During the initial period of SSPX operations, hydrogen GDC was performed for 32.5 h on the inner electrode (inner electrode as cathode) and for 15.5 h on the outer electrode and flux conserver (inner electrode as anode). For nearly identical hydrogen pressure and current, the GDC partial pressures of CO, CH₄, and CO₂ decreased by only a factor of 2 over this four-month period. Since the above rates would imply several 100 equivalent monolayers of carbon removal, the modest drop in removal rate of carbon is in marked contrast to experience on early metal wall tokamaks [9,12], where the observed drops in removal rates were about 2 orders of magnitude. The amount of carbon removed is also much higher than expected from the AES or EDS surface measurements.

Fig. 5 shows volatile partial pressures measured for conditioning shots during the initial period of SSPX operations. An average of the peak partial pressures from three discharges is used for each point on the plot. H₂O and CO₂ are observed to drop by over a factor of 10 during this period; however, the CO and CH₄ partial pressure drops are not as large. While a direct comparison of the volatile removal rates is difficult, the removal rate of equivalent carbon for shot conditioning appears to be smaller than that obtained during GDC.

Observations of the HPPS tungsten surfaces suggest that the majority of this conditioning is occurring on the inner electrode (cathode). A low density of arc tracks observed prior to this period grew to cover all of the surfaces in direct contact with the formation and

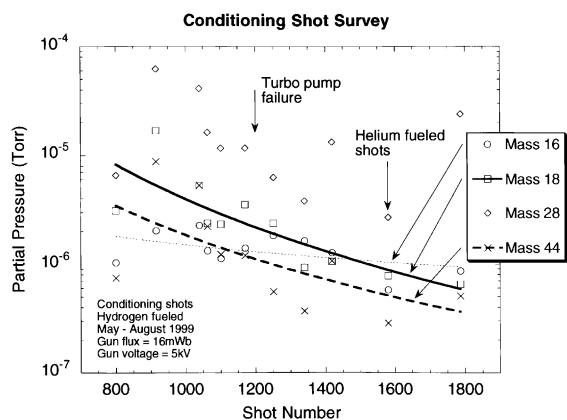


Fig. 5. Peak values of mass spectrometer partial pressures (sampled through the large conductance) generated by shot conditioning during the initial operations period.

spheromak plasma. The localized heating which occurs during arcing can remove more deeply trapped impurities than is accessible to the GDC.

Despite these conditioning efforts, the resistive decay times for discharges during this initial period of operations were short, indicating potentially low electron temperatures. Due to indications of significant hydrogenic wall pumping by the HPPS tungsten coating (and a lack of plasma density measurements), it was not clear if these low temperatures were the result of impurity radiation levels or high density plasmas (lack of density control). While the high porosity of the coating was beneficial in preventing cracking from thermal shock, impurities trapped during fabrication and the connected porosity were likely limiting the plasma performance. Low pressure plasma spraying can be used to reduce this porosity to a few percent, although at a much higher cost.

Recent operation in SSPX has benefited from titanium gettering of the HPPS tungsten surfaces. Residual gas analysis suggests that H_2O is well pumped, while CO and CO_2 exhibit some pumping. CH_4 and other hydrocarbons are not pumped and the H_2 pumping capacity of the film is approximately the gas fueling of one discharge. The affect of titanium gettering on plasma performance is described by Wood [4].

6. Summary

We have characterized the HPPS tungsten using both in situ studies and ex situ measurements of samples. In situ β back-scattering has been used to show that the coating exceeds its nominal value of 100 μm on all surfaces except in the plasma formation region of the inner electrode. Micrographs and thermal desorption of samples have shown a connected, porous structure ca-

pable of absorbing high levels of water. Baking, hydrogen GDC and shot conditioning have been used to reduce water and surface carbon levels. Laboratory measurements and results from SSPX bake-out cycles have indicated that the 230°C baking capability of SSPX is sufficient to remove large amounts of water. The effectiveness of GDC and shot conditioning has been evaluated using removal rates of carbon calculated from the volatile species liberated by the plasma. In situ mass spectrometry indicated initial carbon removal rates of 0.01 monolayer/s during hydrogen glow discharge conditioning at 100 mTorr (abnormal glow). While GDC is used to condition the inner electrode and outer electrode/flux conserver, shot conditioning is most effective in removing impurities from in the inner electrode, with CO_2 and H_2O removal rates dropping by a factor of 10 over ≈ 1000 shots. Titanium gettering on the tungsten surfaces has been used to increase the pumping for some of the volatile impurity species, but effective pumping of hydrogen has been limited.

Acknowledgements

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