The Design and Use of Tungsten Coated TZM Molybdenum Tile Inserts in the DIII-D Tokamak Divertor

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Abstract

Future tokamak devices are envisioned to utilize a high-Z metal divertor with tungsten as the leading candidate. However, tokamak experiments with tungsten divertors have seen significant detrimental effects on plasma performance. The DIII-D tokamak presently has carbon as the plasma facing surface but to study the effect of tungsten on the plasma and its migration around the vessel, two toroidal rows of carbon tiles in the divertor region were modified with high-Z metal inserts, composed of a molybdenum alloy (TZM) coated with tungsten. A dedicated two week experimental campaign was run with the high-Z metal inserts. One row was coated with tungsten containing naturally occurring levels of isotopes. The second row was coated with tungsten where the isotope ^{182}W was enhanced from the natural level of 26% up to greater than 90%. The different isotopic concentrations enabled the experiment to differentiate between the two different sources of metal migration from the divertor. Various coating methods were explored for the deposition of the tungsten coating, including chemical vapor deposition, electroplating, vacuum plasma spray, and electron beam physical vapor deposition. The coatings were tested to see if they were robust enough to act as a divertor target for the experiment. Tests included cyclic thermal heating using a high power laser and high-fluence deuterium plasma bombardment. The issues associate with the design of the inserts (tile installation, thermal stress,

arcing, leading edges, surface preparation, etc.), are reviewed. The results of the tests used to select the coating method and preliminary experimental observations are presented.

1. Introduction

To study the effect of a high-Z divertor a dedicated two-week experiment was run on the DIII-D tokamak with tungsten coated inserts installed in two rows of graphite tiles (Fig. 1). One of the primary goals of the experiment was to study the migration of the high-Z metal around the DIII-D vessel and in particular determine if the metal can migrate from the divertor region to areas where contamination of the plasma could more easily occur. To differentiate between the two row sources the inserts were coated with different isotopic concentrations of tungsten. The floor tile inserts were coated with natural tungsten (^{180}W 0.12%. ^{182}W 26.50%, ^{183}W 14.31%, ^{184}W 30.64%, ^{186}W 28.43%). The nose tile inserts were coated with tungsten where the isotope ^{182}W was enhanced from the natural level of 26% up to greater than 90%.

The original plan was to directly coat the surface of the graphite tiles. However, design, scheduling, and budgets prevented this plan. To resolve these problems the novel solution developed was to fabricate thin metal inserts that could be coated with tungsten and then mounted in a groove in the tile. The details of the metal insert fabrication and mechanical and thermal analysis are presented. Various methods of coating the metal inserts were investigated and the results will be presented. A brief discussion of the graphite tile/insert assembly behavior in the DIII-D following the two week campaign is presented.

2. Design considerations

Previously tiles in DIII-D were manufactured from the Fine Grade Graphite (FGG) Union Carbide ATJ. Because this material was no longer available, the graphite tiles were machined

from the FGG Sigrafine R6710. This grade graphite had been successfully used in the ASDEX Upgrade tokamak[2]. The mechanical properties of Sigrafine R6710 exceed those of ATJ.

To achieve better control of the tungsten exposure it was decided to coat only a thin, 5 cm, strip with tungsten. This provided for sufficient area of high plasma flux exposure on the tungsten and the plasma strike points could be moved on and off the strip depending on the experiment. Plasma shape development and vessel clean-up could also be performed without eroding the tungsten. Based upon sputter rates a minimum tungsten coating thickness of 1 μm was chosen.

The first coating technique investigated was Combined Magnetron Sputtering with Ion Implantation (CMSII)[1]. This technique, developed by the Romanian National Institute for Laser, Plasma, and Radiation Physics, has been used by both ASDEX[2] and JET[3]. An ATJ graphite tile was sent to the Romanian lab for coating. Due to the porosity of ATJ graphite (which contains small 10-20 μm pores) a requested coating thickness of 10 μm was used to limit the potential for exposed graphite in the tungsten strip. Initial scratch tests and a scotch tape pull test indicated a good mechanical bond between the tungsten and the graphite.

However, there arose an issue with using this method for coating the ^{182}W. Because evaporation and sputtering methods deposit materials everywhere within the vacuum chamber rather than being directed only at the surface, these methods generally have large inefficiencies. The ^{182}W is expensive and it was not economically feasible to try to coat the tiles with this method.

The ^{182}W was supplied by Oak Ridge National Laboratory's (ORNL) Nuclear Security and Isotope Technology Division. ORNL also has the ability to deposit tungsten using electron beam physical vapor deposition. As described above, this sputter method also requires a significant

amount of material. It was estimated that more than 200,000 mg would be needed to deposit 9,700 mg onto the tiles to achieve a 1 μm coating thickness. However, ORNL developed a method to capture and reprocess 80-95% of the non-directed material, which made it the only economically viable option.

Meeting the experimental schedule was a constraint. The large size of the tiles limited the number of tiles that could be coated during a run. This coupled with the need to mask each tile to obtain the 5 cm strip made the coating process very slow. There was not enough time to fabricate the tiles, bake them, and then send them out for coating.

3.1 Metal Tile Inserts: Design

An alternative to the graphite coating technique was to design inserts that could be mounted in the graphite tiles. The fabrication of the R6710 graphite tiles and the coating of the inserts could then be done in parallel. Due to their small size many inserts could be coated simultaneously reducing coating time and increasing the coating process efficiency, which reduced the amount of ^{182}W required.

The inserts where composed of the molybdenum alloy TZM (molybdenum alloyed with 0.5% titanium and 0.08% zirconium). TZM is readily available and has a higher strength at elevated temperatures and a higher recrystallization temperature than pure molybdenum. The inserts were 0.95 cm thick, 5 cm wide and either 17.2 cm long for the lower floor or 11.9 long for the nose tile. A groove was machined into the graphite tile and the insert was secured to the graphite with two #10-32 316 stainless steel screws (Fig. 2). The screw length was 2.8 cm for the floor tiles and 4.1 cm for the divertor nose tiles. The top of the insert was aligned to the graphite tile. If necessary thin Grafoil® sheets were used to shim the material to the proper height.

As many tiles in the two rows as possible were modified with inserts. However, there were some gaps in the toroidal row of tiles where a diagnostic prevented the tile being modified. For the floor row 46 of the 48 tiles had inserts. For the nose row 64 out of 71 had inserts.

3.2 Metal Tile Inserts: Mechanical Forces

A plasma disruption could result in a pulling force of up to 423 N on a metal insert. To react this force Belleville washers were preloaded to 1.13 N-m. The Belleville washers were made of Inconel X-750 with a working range of -100^{o} to 620^{o} c. This provided approximately 1335 N of force holding the insert to the tile. To reduce stresses caused by differential expansion of the metal insert relative to the tiles there was a radial gap of 0.13-.25 mm between the inserts and the tiles. However, even if the insert did start off touching the tile it was calculated that the stresses would not exceed the 66 MPa tensile strength of the Sigrafine R6710. The effect of the toroidal eddy current was ignored since there were tiles without inserts which would make the tile-to-tile conduction path fairly resistive.

3.3 Metal Tile Inserts: Thermal Modeling

Thermal modeling was used to determine the effect of the heat flux on the leading edge of the inserts. For DIII-D based upon an incident field line angle of 2.5^{o} a parallel heat flux of 149.6 MW/m^2 and a perpendicular heat flux of 6.5 MW/m^2 was assumed. From the geometry of the tiles it is assumed that the parallel heat flux is applied to a 0.2 mm height on the insert edge [Fig. 3]. This height is the estimated maximum vertical misalignment between tiles based upon past installations. Following installation the actual maximum vertical mismatch was measured at 0.30 mm. The variation in vertical mismatch was approximately the same for the floor and nose tile rows. The maximum horizontal spacing between the tiles was estimated to be 0.6 mm. The heat transfer from the insert to the tile was assumed to be through the bottom of the insert only with

approximately a 25% surface contact. To reduce the effect of the parallel heat flux the edge of the insert was rounded with a radius of 1.1 mm. This increases the area for the parallel heat flux from 10 mm^2 to 34 mm^2 decreasing the parallel heat flux from 149.6 MW/m^2 to 43 MW/m^2. For a discharge of 3.5 seconds this heat flux raises the average temperature of the insert to 700^o C with a maximum temperature on the leading edge of 1200^o C. The inserts return back to room temperature within 9 minutes. This temperature rise should not be a concern for the TZM. Given the small amount of material that rises to 1200^o C and the very short dwell time at temperature there did not appear to be any concern with embrittlement due to recrystallization[4] for the two week campaign. The screws and washers were assumed to follow the bulk temperature of the tiles. They had minimal affect on the heat transfer.

4.1 Coating of the metal inserts: Introduction

The first row of inserts to be coated by ORNL with ^{182}W could be completed just in time for the experiment. The second set of inserts to be coated with natural tungsten would need to be coated by a different supplier. The Romanian lab was unable to meet the schedule requirements so we were unable to utilize the CMSII technique. Three different commercial vendors, each utilizing a different coating technique, were identified and samples from each were obtained for analysis and testing.

4.2 Coating method for the ^{182}W

The coating of the ^{182}W onto the TZM inserts was to be done at ORNL using a custom built electron beam physical vapor deposition (PVD) system with a Model 264 Telemark e-beam tool. The base pressure for the tool is $8.3x10^{-4}$ Pa. The tool was further modified with extra cooling to allow evaporation of W metal. To achieve good adhesion the surface was abraded with 320 grit loose silicon carbide abrasive using a Comco Microblaster. This provided a uniform texture and

removed oxidation from the surface. Surfaces were ultrasonically cleaned with deionized water before coating with the ^{182}W isotope. The target coating thickness for the ^{182}W was 1-2 μm. The test sample from ORNL was slightly thinner at approximately 0.85 μm. A Secondary Electron Microscope (SEM) image[5] of the ^{182}W coating shows a smooth surface with small irregularities on the order of 1 μm (Fig. 4).

4.3 Coating methods for the natural tungsten

For the second row to be coated with natural tungsten three commercial vendors were identified who were able to quickly coat samples for analysis. The first vendor used Chemical Vapor Deposition (CVD) with WF_6 gas with the inserts heated to 600^o C. The oxides were removed by an initial flow of hydrogen to reduce the oxygen on the surface. The surface image from the SEM shows slightly jagged features on the order of 2 μm (Fig. 5). There was concern over the fluorine from the WF_6 feeder gas, but X-Ray Dispersive Spectroscopy (EDX) of the surface shows no detectable fluorine levels in the coating. The coating thickness for the test sample was approximately 12 μm, which is above the target value of 1-2 μm. However, given the roughness of the coating this thickness was considered acceptable to achieve good coverage.

The second vendor used electroplating where the entire insert is dipped into a heated tungsten oxide bath. This technique is known to provide a very controlled deposition. SEM analysis showed a smooth surface with irregularities less than 1 μm. While this approach looked promising, the vendor could not meet the schedule.

The third vendor utilized a plasma spray technique. This method produced a visibly rough surface coating that was not acceptable for the experiment.

4.4 Testing of Coatings

The first test of all the samples was a simple scotch tape pull test and a scratching of the surface using the dulled edge of a hardened metal stylus. All the samples survived this initial test.

The coatings were then subject to two types of stress tests. Both tests were performed at the PISCES facility located at the University of California San Diego. For the first test a high power laser was used to the heat the samples. The laser provided a 1 second on, 4 second off pulse for 1,000 cycles onto a 2 mm spot in the middle of the sample with an estimated absorbed power density of approximately 100 MW/m^2. This repeatedly raised the surface temperature of the spot to 1200^o C. The laser had no visible effect on the coating and no material came off during a subsequent scotch tape pull test for either the CVD or electroplated coating. The CVD coating was hand-scratched with a hardened metal stylus and then sectioned and looked at under SEM. No effect could be seen from the laser test or from the scratch. Scheduling did not permit laser testing of the ORNL sample.

For the second test, all the samples were exposed to 50-60 eV D_2 ions with a flux of $1\text{-}2x10^{18}$ $cm^{-1}s^{-1}$ for approximately one hour. This resulted in a surface temperature rise of $250\text{-}300^o$ C. There was no visible damage to the CVD or electroplated samples and they were unaffected by the scotch tape pull test and the mechanical scratching. There was discoloration on the ORNL e-beam PVD coating following the D_2 plasma exposure but the coating still had mechanical integrity based upon a scotch tape pull test and the mechanical scratch test. An EDX analysis showed levels of Mo through the W suggesting either that there was some erosion of the W or that the film was too thin to provide adequate coverage. However, since the production thickness of 1.5 μm was double the test sample thickness it was considered an acceptable risk for the two week experiment.

Based upon experimental requirements it was decided to use the ORNL enhanced ^{182}W coating on the nose tile insert since it had a slightly smoother surface finish.

4.5 Measurement of coating thickness

For every tile insert, the thickness of the W coating was measured in order to verify adequate W coverage and uniformity. Measurements were made using a non-destructive X-ray fluorescence method with an Olympus Innov-X DP-6000 instrument. The W coating in each case was thin enough to transmit both the excitation beam energy (45keV) and the Mo fluorescence lines (Mo kA 17.5keV, Mo kB 19.6keV) originating from the underlying substrate. Due to the attenuation of the Mo fluorescence lines through the W layer, the ratio of the relative W and Mo fluorescence lines can be translated to an effective W thickness based on known calibration standards. Using this technique the average thickness of the film at the midpoint for each insert was measured. The CVD films had a W coating thickness of 10.1 +/- 3 μm, and the ORNL films a thickness of 1.5 +/- 0.3 μm. For comparison, vendor supplied thickness levels based upon micrometer measurements at the midpoint for the CVD films were 18 μm +/- 7 μm. The results are in reasonable agreement. The difference between two methods could also be partially explained by the expectation that the micrometer would measure surface peaks while the X-Ray fluorescence would average out the surface roughness. The agreement was sufficient to validate that the ORNL film thickness was greater than the required value of 1.0 μm. For both the ORNL and CVD films a single insert was selected and a ten point scan was taken down the length of the insert. For both inserts the film thickness varied linearly with the thickness on one end approximately a factor of two greater than the other end.

5. Preliminary results

Following installation of the tiles in the DIII-D vessel a successful two-week experimental campaign was run utilizing the tungsten metal rings. Maximum perpendicular peak heat fluxes were 4.8 MM/m^2 for the floor tiles and 3.0 MW/m^2 for the nose tiles. Maximum surface temperatures were 820^o C for the floor tiles and 320^o C for the nose tiles. Following operations, there was an in-vessel inspection of the tile/insert assembly. Considerable differences were observed between the nose and floor tiles. For both rows, a visible inspection did not show signs of arcing in the ^{182}W coating. An initial measurement of thickness using the XRF did not show measurable erosion of the coating. For the nose tiles both the graphite tile and the insert were in good condition showing little damage or erosion.

However, for the floor tiles both the graphite tiles and the inserts showed damage. Small pieces of graphite at the edges of the tile had chipped off. The initial thermal modeling had assumed that the bulk of the power would be deposited on the metal inserts. However, during the experiment, a considerable amount of power was also deposited on the graphite tiles. Further modeling indicated that this could lead to significant stresses in the tile. On the floor there were two tiles without inserts. The first tile made of Sigrafine had chipping damage. The second tile made of ATJ did not show damage. This chipping damage has also not been seen previously on ATJ tiles. The current speculation is that the damage to the Sigrafine R6710 graphite may be caused by constraints in the floor tile movement coupled with R6710's higher thermal expansion coefficient (4.2e10^{-6} K^{-1}) as compared to ATJ's (2.3x10^{-6} with the grain and 3.4x10^{-6} against the grain).

Approximately 40% of the floor tile inserts showed signs of bowing and melting on the edges. This ranged from very slight up to pockets of melt several mm deep. The areas of highest melting generally correlated with those inserts with the highest misalignments. While the floor

tiles were exposed to higher power shots, it is not certain that this is the principal reason for the damage. The tiles for the two rows are designed differently, and are secured in different fashions to the vessel. The floor tiles are secured to the vessel perpendicular to the inserts while the nose tiles are secured to the vessel parallel to the inserts. During a shot this perpendicular attachment may have allowed for the hotter edges to expand relative to the middle increasing the misalignment. The expansion and relaxation may have resulted in permanent deformation of the inserts. However, further modeling and investigation is needed to confirm this speculation.

6. Conclusion

To study the effect on plasma performance and the migration of tungsten in the DIII-D tokamak a dedicated two week experimental campaign was run with two rows of tiles in the divertor modified to contain tungsten coated metal inserts. The use of inserts arose as a solution to challenges to meet both the schedule and to coat the inserts with both natural W and isotopically enhanced ^{182}W. The two-week experimental program was successfully completed. Initial XRF measurements of the tungsten coatings for both the electron beam physical vapor deposition and the CVD did not show measureable erosion, nor was there visible evidence of arcing. The graphite tile/insert assembly showed considerable differences between the nose and floor tiles. For the nose row both the graphite tiles and inserts showed little damage or wear. For the floor, there was chipping of the graphite tiles and melting of the insert's leading edge. The melting appears correlated with vertical misalignment between the inserts which may have increased due to deformations of the inserts caused by differential expansion of the graphite tiles during plasma discharges. The damage to the graphite tiles may be due to the amount of heat flux on the graphite and Sigrafine R6710's higher thermal expansion coefficient (as compared to ATJ). Further analysis is needed to understand these observations.

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References

[1] Tungsten coatings deposited on CFC tiles by the combined magnetron sputtering and ion implantation technique, C. Ruset, et al, Physica Scr. T128 (2007) 171-174

[2] Ten Years of W programme in ASDEX Upgrade – challenges and conclusions, R. Neu, et al., Phys. Scr. TI38 (2009) 014038 (6pp)

[3] Qualification of tungsten coatings on plasma-facing components for JET, H. Maier, et al, Physc. Scr. T138 (2009) 014031 (5pp)

[4] High head load properties of high purity CVD tungsten, S. Tamura et al, Journal of Nuclear Materials, 307-311 (2002) 735-738

[5] Secondary Electron Microscope Images are provided by B. Stahl, General Atomics (2016).

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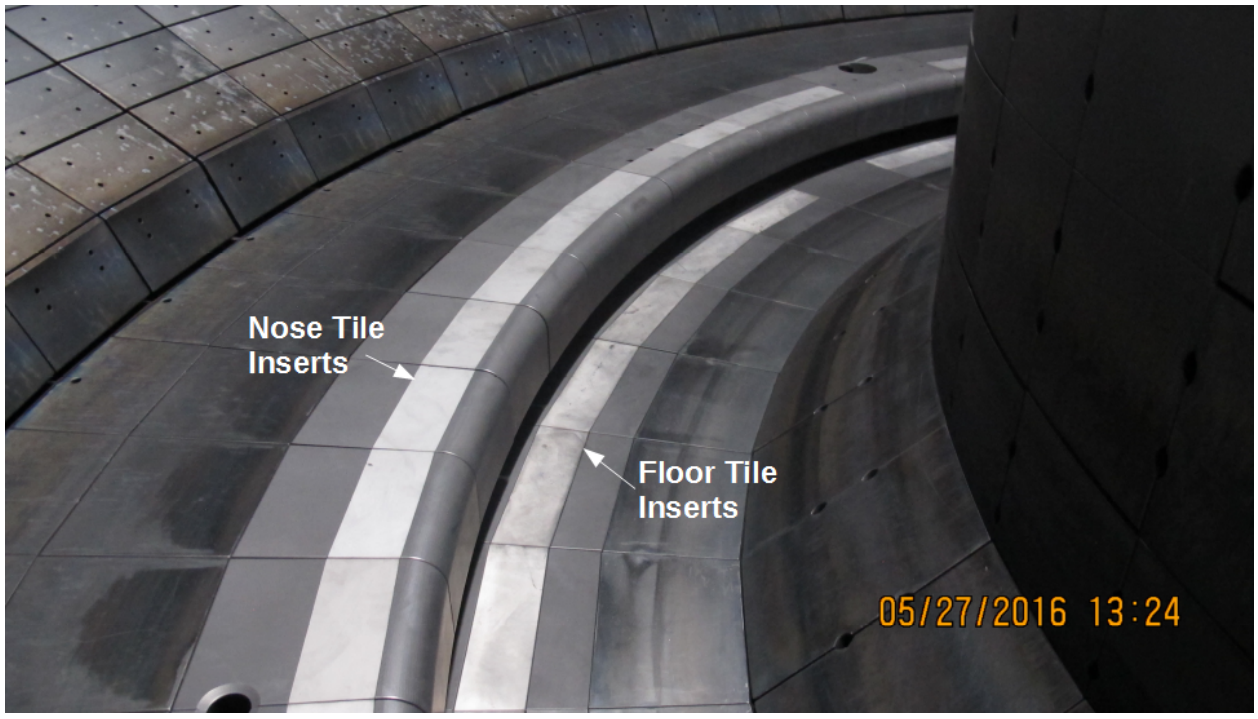

Figure 1. The metal inserts were installed at two locations in the lower divertor. The outer divertor strike point can be placed on either of the two locations.

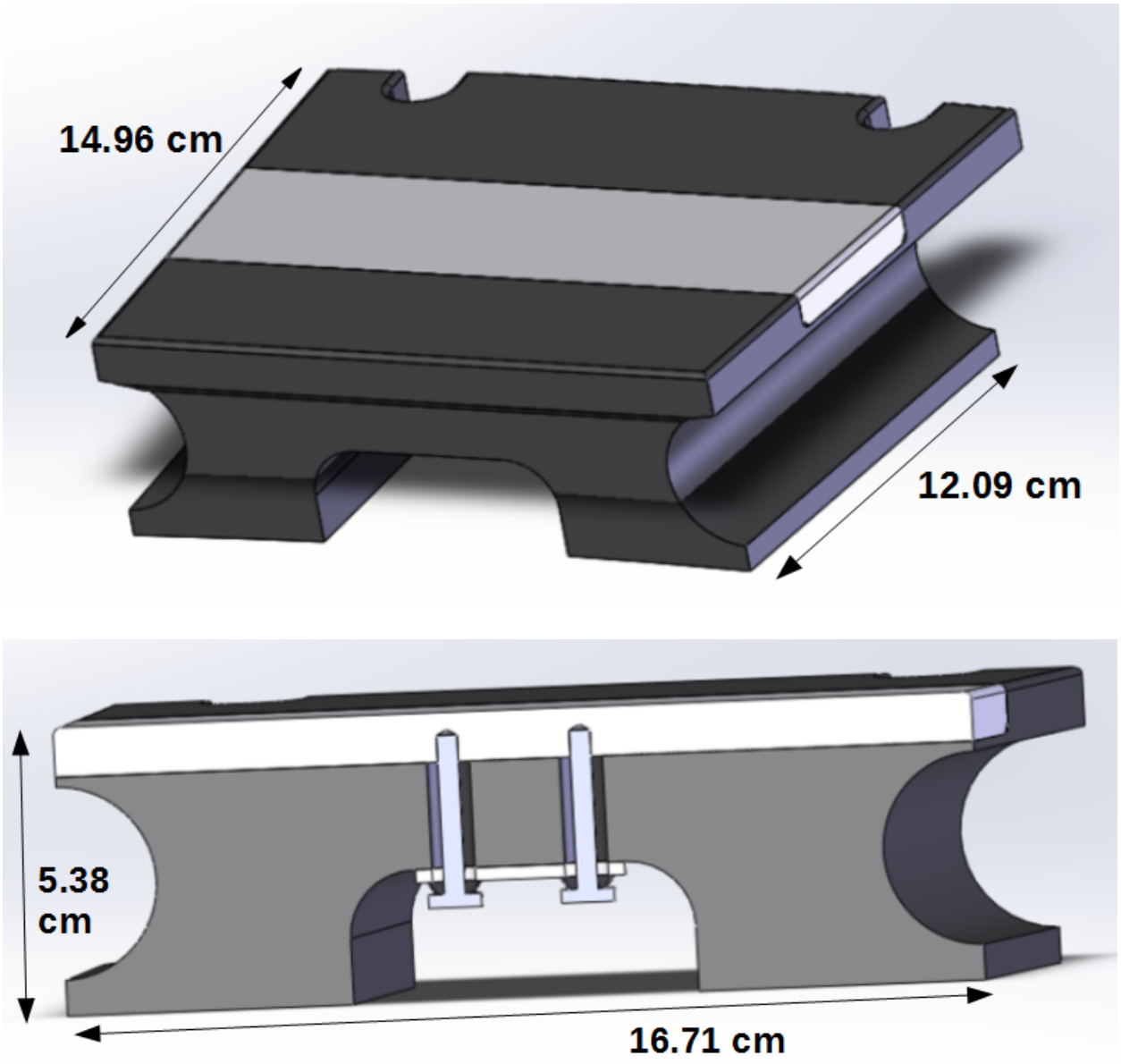

Figure 2. The metal insert is installed into a slot in the graphite floor tile and secured to the tile with two #10-32 316 stainless steel screws. Belleville washers pre-load the tile against pulling forces from disruptions.

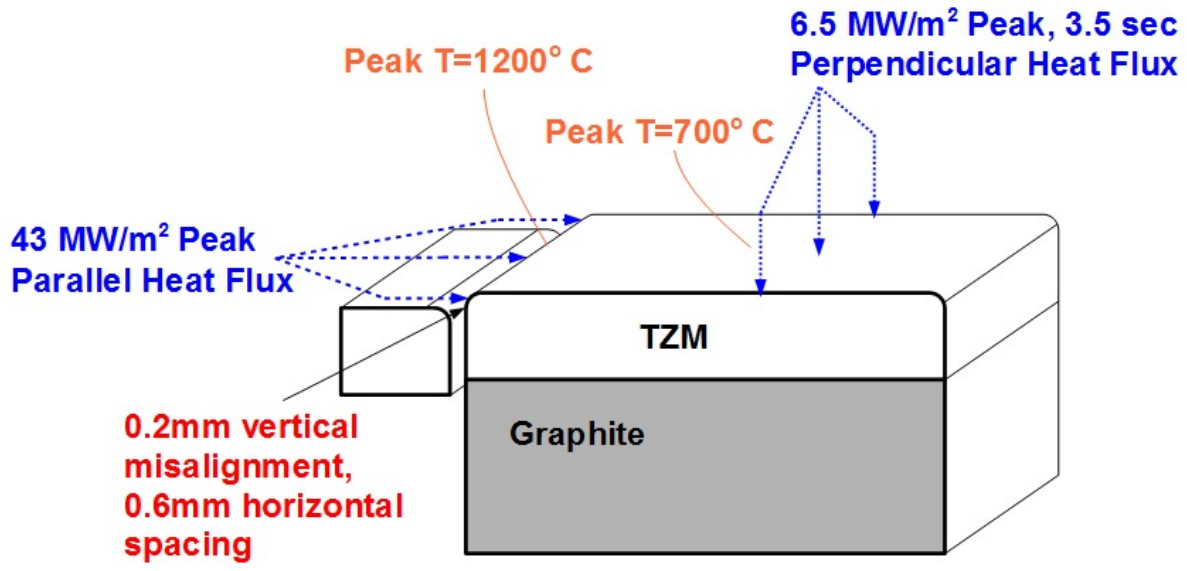

Figure 3. For a 3.5 second discharge the peak heat flux (assuming a 0.2 mm vertical misalignment and a 0.6 mm horizontal spacing between tiles) results in a peak temperature of 1200^{o} C on the leading edge and a bulk temperature rise of 700^{o} C.

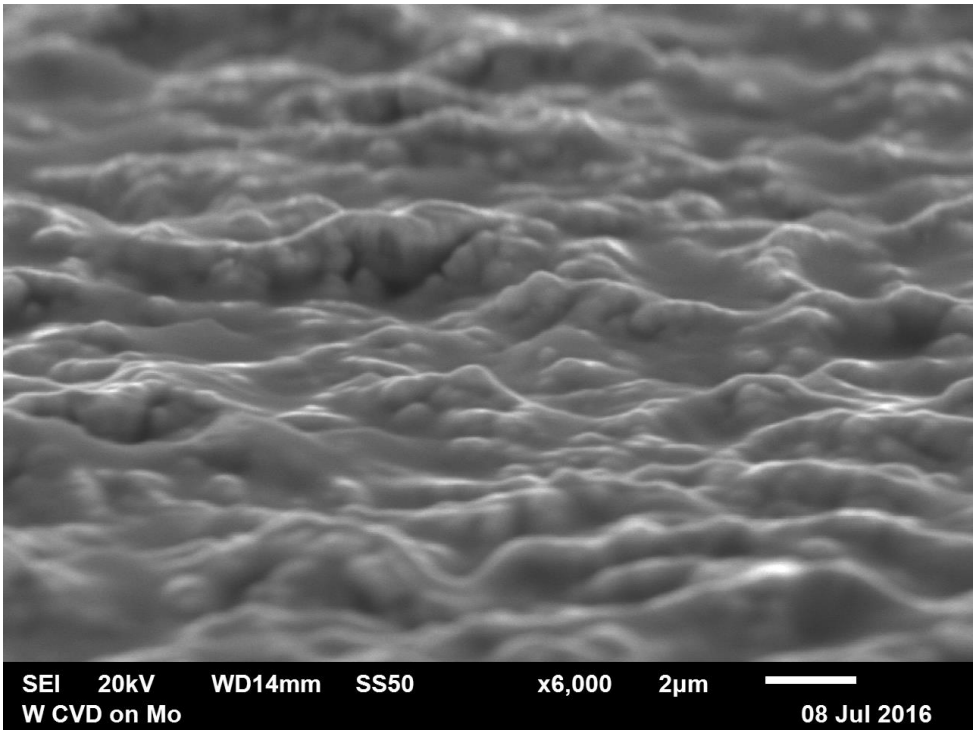

Figure 4. A SEM image of the ^{182}W coating from electron beam sputtering physical vapor deposition shows a smooth surface with small irregularities on the order of 1 µm.

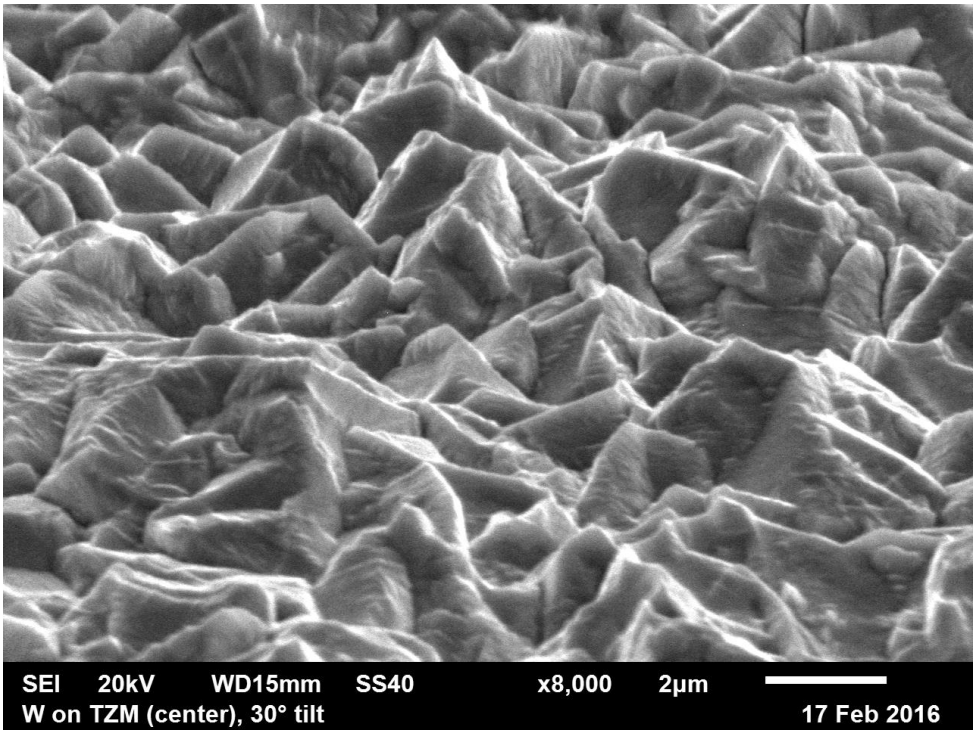

Figure 5. A SEM image of a natural W coating from WF_6 CVD deposition shows spiky surface irregularities on the order of 1-2 μm.