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The removal of ion implanted deuterium from tungsten and stainless steel by transferred-arc cleaning

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Abstract

Tungsten and stainless steel samples have been ion implanted with deuterium in an accelerator to simulate hydrogen isotope ion implantation conditions in magnetic confinement fusion devices. The samples were characterized by ion beam analysis both before and after cleaning to determine deuterium concentrations present. The extent of transferred-arc (TA) cleaning was varied to determine the deuterium removal efficiency, surface roughening and sample erosion rate. The deuterium content was greatly reduced by the cleaning thus demonstrating the possibility of using the TA cleaning technique for removing deuterium from components exposed to D-T fuels. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

The unmitigated build up of tritium in the components of fusion reactors utilizing deuterium–tritium fuels is a serious concern for present and future experiments. Machine maintenance, removal and handling of tritiated components and accidental environmental release of the deposited tritium due to loss of vacuum pose a real threat to workers and the general population [1]. The tritium can be found implanted below the surface of the component or in a surface film often consisting of a codeposited material such as carbon [2,3]. Techniques such as laser thermal desorption [4], plasma discharges [2], oxygen exposure at elevated temperature [3], as well as the more traditional heating, purging, washing and vacuum desorption [1] are being considered for use on various components of fusion reactors.

Another possible cleaning technique for use in fusion reactors is cathodic-arc cleaning which is also known as transferred-arc (TA) cleaning. TA cleaning utilizes a dc plasma torch and a secondary power supply attached between the torch and the part to be cleaned to establish

arcing between the torch face (anode) and the part (cathode) as shown in Fig. 1. The torch produces a plasma which becomes the electrical conduction path for the cleaning arc. The arc attachment to the cathode is influenced by the surface shape, the surface roughness and surface contaminant layers (oxide, organic and/or metal film) which may be present [5]. The cathode contaminant layers are preferential sites for arc attachment. This is caused by ion charge buildup on the contaminants producing an enhanced electric field that increases in strength until break down of the film occurs resulting in arcing to the cathode [6]. Joule heating of the cathode causes erosion of the contaminant layer and the bulk cathode material beneath. As the contaminant is removed, electron emission ceases and the arc is displaced to another area with remaining contaminant. This process continues until the contaminants are removed. At this point, the arc may either extinguish or if sufficient conditions exist, the arc may transition to a different type that is characteristic of clean cathodes. This preference for attachment to the contaminant film produces a cleaning mechanism over cathode surfaces which is used for electrode conditioning for such varying applications as accelerator tubes to arc welding of highly reactive materials such as aluminum and magnesium. TA cleaning is also used in low pressure plasma spraying (LPPS) as a substrate pretreatment. The arcing first

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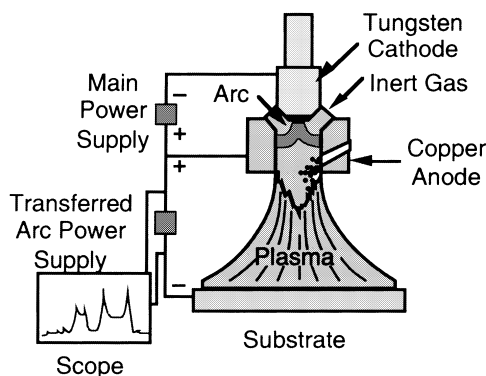


Fig. 1. Plasma torch and TA power supply configuration used for cleaning experiments.

cleans surface contaminants from the substrate then roughens the surface to provide enhanced mechanical bonding to the coating deposited subsequently [7].

Advantages of the TA cleaning technique for the removal of tritium and redeposited plasma facing component materials include:

- Localized technique with the ability to clean selectively in areas with varying buildup of films and/or implanted hydrogen so that only areas with contaminants are cleaned. This prevents unwanted erosion of components while providing sufficient material removal in areas containing contaminants.
- Effective removal of contaminant films and implanted hydrogen.
- Non-line-of-sight technique allowing the removal of material from hard-to-reach areas such as shadowed regions, gaps and recessed areas.
- Requires no bulk wall heating.
- If plasma spraying is used for in situ repair of damaged components, only an additional power supply and an electrical connection to the surface to be cleaned is necessary for TA cleaning.
- Does not rely on oxygen and thus does not produce the undesirable retained oxygen in the machine components requiring wall conditioning.
- Capture of gaseous residue at point of generation is possible for tritium reclamation.
- Minimal solid waste generation for cleaning operation.
- The cathode-arc cleaning effect has been extensively studied and its effects are well understood.

However, controlling the arcing process to yield the desired cleaning results for a particular set of arcing conditions must be investigated on an individual basis due to effects of surface morphology and contaminant effects on arc cathode spot behavior. This paper describes a preliminary investigation of TA cleaning for the removal of accelerator implanted deuterium ions from stainless steel and tungsten samples.

2. Experimental

2.1. TA cleaning conditions

The TA experiments were carried out in an inert environment LPPS chamber. In order to purge the air from the system, the chamber was pumped down to 13 Pa and twice back filled with argon before conducting experiments. Chamber pressure was maintained at 2.7 kPa during cleaning. The plasma torch used was a Miller SG-100 using a 40 kW Plasmadyne dc power supply. The cleaning conditions for all samples were as follows: torch gas of 40 slm argon; torch current of 200 A; TA electrode separation of 12 cm. The TA power supply used was a 250 A maximum constant current dc supply built by Miller Electric. For all experiments, the plasma torch-arc was started with the torch away from the sample to be cleaned. The torch was then moved close to the sample and the TA power supply engaged manually. The torch was passed over the sample, reversed direction, then passed back over the sample to the starting position. Voltage signals from the TA power supply were recorded with a Tektronix TDS 420A digital oscilloscope and P5200 high voltage differential probe. The TA power supply current was measured with the same oscilloscope using a Tektronix 503S current probe system with a high dc current probe.

2.2. Sample measurements

The samples used for this experiment were 2.5 cm diameter discs with a thickness of 1.3 mm. The sample materials were 304 stainless steel and tungsten. Prior to ion implanting, two samples of each material were mechanically polished and one sample was left in the as-received condition. The surface roughness was measured before and after cleaning to determine the roughness induced by TA cleaning. Sample surface roughness measurements were performed using a Sheffield Measurements Handysurf E-21A surface texture analyzer. Thermocouple holes 1.5 mm in diameter were drilled half way through the sample thickness for temperature measurements during cleaning. The samples were weighed before and after cleaning using a Mettler-Toledo AB104 balance to determine the mass lost during cleaning.

The samples were implanted with deuterium at an incident energy of 33 keV. The calculated depth of implantation in stainless steel is 240 nm and the depth in tungsten is 150 nm. The measured doses and depths of the implanted samples agreed with the calculations and the integrated dose measured at the time of implantation.

The deuterium areal density of the near surfaces ($\leq 5 \mu\text{m}$ sampling depth) of the tungsten and stainless steel samples before and after cleaning were measured

using elastic recoil detection of the deuterium [8]. An incident beam of 5.6 MeV alpha particles was focussed to a 4 mm² spot on the sample that was tilted at 75° from beam normal. The recoiling deuterium was detected at a forward scattering angle of 30°. A combination of Al and mylar absorber foils were used to stop the forward scattered alpha particles but allow transmission of the recoiling hydrogen and deuterium to the detector Si solid state detector.

3. Results and discussion

3.1. Results of sample measurements

The TA voltage was monitored during sample cleaning. The values varied only slightly with cleaning conditions giving an average voltage for stainless steel of 37 V and for tungsten of 48 V. The difference in voltage represents differences in material properties such as first ionization potential, boiling point, heat capacity and thermal conductivity. The 11 V difference agrees well with other reported results [9].

The deuterium areal densities implanted prior to cleaning are listed in Table 1. An areal density of 1×10^{15} cm⁻² corresponds approximately to one monolayer. There was no measurable peak detected in the data for deuterium after cleaning so levels were near or below the detection limit of 1×10^{14} cm⁻². This represents minimum cleaning efficiencies of 99.5–99.8%. Since relatively aggressive cleaning was used in all cases and no residual deuterium was detected, the minimum arcing needed for deuterium removal is not known. An indication of the amount of TA cleaning is represented by the charge in coulombs (current integrated over the cleaning time) divided by the area cleaned. The values of cleaning charge/area for these samples were between 6 and 37 C/cm². These values represent a maximum required charge/area as the deuterium was likely removed some time before the arcing was terminated.

During TA cleaning, the sample surface was eroded at the location of the moving cathode spot. The mass lost during cleaning was measured and divided by the total charge flow to give an erosion rate per coulomb of

charge. The erosion rates for tungsten and stainless steel were found to vary for differing cleaning conditions but fell in the range of 20–60 µg/C. These values are in good agreement with other measurements of cathode erosion rates [10].

Since the erosion takes place locally at the cathode-arc spot, the surface is roughened by differential material removal. The before and after cleaning average roughness values are given in Table 1. After cleaning, the surface roughness varied with the TA cleaning charge per pass which is also shown in Table 1. Higher TA cleaning charge per pass produces higher surface temperatures and larger craters [11] resulting in a rougher surface. The surface of sample W3 after cleaning is shown in Fig. 2. This degree of roughening is not necessarily required for the deuterium cleaning effect. Refinement of the cleaning process will allow for cleaning with less roughening if that is the desired outcome.

Temperatures during cleaning for tungsten samples W1 and W2 were measured by imbedded thermocouples. Temperatures for the remaining samples were not resolvable due to electrical interference in the thermocouple signal. The substrate temperatures varied with

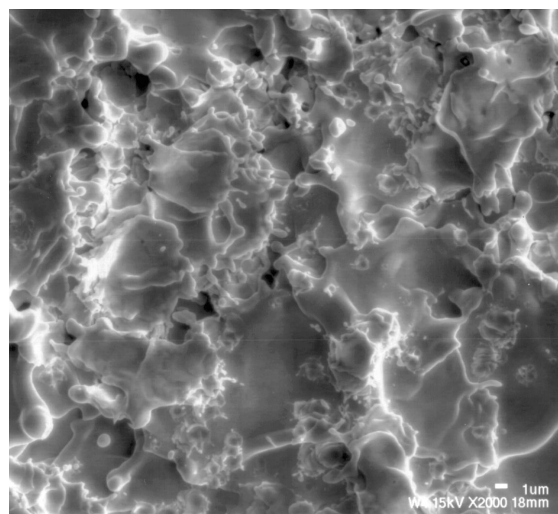


Fig. 2. Surface of tungsten sample W3 after TA cleaning.

Table 1
Deuterium implanted sample results

Sample	D before cleaning ($\times 10^{15}$ cm ⁻²)	TA charge (C)	TA charge/pass (C/pass)	R_a before cleaning (µm)	R_a after cleaning (µm)
W1	19.5	135	135	1.6	2.2
W2	20.7	88	44	0.08	1.4
W3	59.8	117	39	0.08	0.85
SS1	20.1	32	16	0.27	2.1
SS2	19.5	187	93.5	0.08	7.0
SS3	50.0	48	43	0.08	2.5

the total charge transferred per cleaning pass; more charge transferred gave higher temperatures. Sample W1 (135 C/pass) had a peak temperature of 650°C while sample W2 (44 C/pass) had peak temperatures of 192°C and 193°C for the two cleaning passes, respectively. The substrate temperature was 25°C before cleaning in all cases. The plasma torch contribution to heating was approximately 30°C; the remainder is due to TA cleaning. In cases where the TA charge per pass is kept low (<10 C), the TA contribution to heating is similar to the torch contribution and total temperature increase can be below 50°C. However, the local surface temperature at the location of the cathode-arc is in excess of the melting point of the material. This is evident from the cratering and splashing of material visible in Fig. 2.

3.2. Cleaning mechanism

The mechanism for removal of deuterium by TA cleaning is most likely a combination of thermal desorption and material removal. The current density of cathode spots is approximately 10^8 A/cm² [6]. For a typical cathode voltage drop of 15 V, this gives a power density of 1.5×10^9 W/cm². At such high power densities, large thermal gradients are formed on the surface of the sample even for materials with high thermal conductivity. This large thermal gradient acts as a driving force to displace the deuterium from under the sample surface to the sample surface where it can be transferred to the surrounding atmosphere.

Material removal from the sample surface is also partly responsible for deuterium losses. The mass eroded during cleaning can be used to estimate the thickness of material removed from the cathode. However, the material is preferentially removed from the edges of the sample due to more frequent arcing caused by geometric field concentration effects. As an estimation, half of the mass lost was assumed to be from the sample edge and half from the top surface. This division of mass loss is based on video observations of the frequency of cathode-arc spot attachment on the edge and the top surface of the samples. The eroded depths of the samples were then calculated. Sample SS2 had an eroded depth of 1800 nm due to the aggressive cleaning while the other samples ranged from 9 to 200 nm with an average of 126 nm. When compared to the implantation depth of 140 and 250 nm for tungsten and steel, respectively, it is evident that material erosion is at least partly responsible for deuterium removal in all samples.

4. Conclusions

The following conclusions resulted from this study of the removal of ion implanted deuterium in stainless steel and tungsten by TA cleaning:

- Implanted deuterium is effectively removed from stainless steel and tungsten by the TA cleaning process.
- As a result of TA cleaning, the sample surface is eroded by the action of the arc at the location of the cathode spot. The arc attachment leaves craters from displaced material that was melted and flowed during arcing.
- Values of surface erosion, surface roughness and bulk sample temperature vary with the aggressiveness of cleaning. Within limits, these values can be tailored to the demands of the particular application.

Research into the removal of codeposited carbon/deuterium films and the refinement of the TA cleaning process for the particular needs of the magnetic confinement fusion community is planned. Future work will help in evaluating the suitability of TA cleaning for present and future requirements.

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