

Title:

The Removal of Carbon/Deuterium from Stainless Steel and Tungsten by Transferred-Arc Cleaning

Author(s):

Kendall J. Hollis, Richard G. Castro, Carl J. Maggiore, Russell P. Doerner

Submitted to:

<http://lib-www.lanl.gov/la-pubs/00796050.pdf>

The Removal of Carbon/Deuterium from Stainless Steel and Tungsten by Transferred-Arc Cleaning

Kendall J. Hollis, Richard G. Castro, Carl J. Maggiore, Russell P. Doerner*

Los Alamos National Laboratory

Los Alamos, NM 87545 USA

*University of California, San Diego

La Jolla, CA 92093 USA

ABSTRACT

Tungsten and stainless steel samples have been contaminated with deuterium and carbon to simulate deposited layers in magnetic-confinement fusion devices. Deuterium and carbon were co-deposited onto the sample surfaces using a deuterium plasma seeded with varying amounts of deuterated methane. Deuterium was also implanted into the samples in an accelerator to simulate hydrogen isotope ion implantation conditions in magnetic confinement fusion devices. Cathodic arc, or transferred-arc (TA) cleaning was employed to remove the deposits from the samples. The samples were characterized by ion beam analysis both before and after cleaning to determine deuterium and carbon concentrations present. The deuterium content was greatly reduced by the cleaning thus demonstrating the possibility of using the TA cleaning technique for removing deuterium and/or tritium from components exposed to D-T fuels. Removal of surface layers and significant reduction of subsurface carbon concentrations was also observed.

KEYWORDS: cathode, arc, clean, deuterium, carbon

1. INTRODUCTION

The unmitigated build up of co-deposited carbon-tritium films in the components of fusion reactors utilizing deuterium-tritium fuels and carbon tiles 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 co-deposited 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 magnetic-confinement fusion reactors is cathodic-arc cleaning also known as transferred-arc (TA) cleaning. TA cleaning utilizes a direct current (DC) plasma torch and a secondary DC 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 (part) 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 surface contaminant. This process continues until all the surface contaminants are removed. At this point, the arc may either extinguish, or, if sufficient conditions exist, the arc may transition to a different cathode arc type that is characteristic of clean cathodes [7].

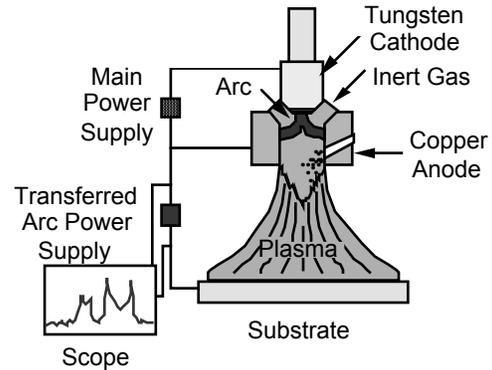


Fig. 1. Plasma torch and TA power supply schematic.

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

- Localized technique with the ability to clean selectively, preventing unwanted erosion
- Effective removal of implanted and surface contaminants
- Non-line-of-sight technique for cleaning hard-to-reach areas
- Requires no bulk wall heating
- Does not rely on oxygen and thus does not produce the undesirable retained oxygen in the machine components requiring wall conditioning before returning to normal operation
- Capture of gaseous residue at point of generation is possible for tritium reclamation
- Minimal solid waste generation from cleaning operation
- Cathode arc cleaning 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 described the TA cleaning behavior of stainless steel and tungsten samples with co-deposited deuterium-carbon films and ion implanted deuterium.

2. EXPERIMENTAL

2.1 Sample coating procedure

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. The samples were exposed to a deuterium plasma seeded with varying amounts of deuterated methane at the Univ. of California-San Diego PISCES-A [8] facility in order to deposit a film of carbon and deuterium. Several identical samples were exposed to deuterium plasmas containing varying amounts of

impurity fractions (from 0.1% carbon, up to 10% carbon). All samples were exposed for 1800 seconds.

2.2 Sample ion implantation

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.

2.3 TA cleaning conditions

The TA experiments were carried out in an argon environment at 2.7 kPa. The plasma torch used was a Praxair SG-100. The cleaning conditions for all samples were torch gas of 40 slm argon and torch current of 200 A. The distance between the torch and the sample to be cleaned (TA electrode separation) was 12 cm. The TA power supply used was a 250 A constant current DC supply built by Miller Electric, Inc.

2.3 Sample measurements

The samples were weighed before and after cleaning using a Mettler-Toledo AB104 balance. The TA power supply current was measured with a Tektronix TDS 420A digital oscilloscope using a Tektronix 503S current probe system with a high DC current probe.

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 [9]. Areal density measurements give the number of atoms detected per unit of sample surface area. The sampling volume for each measurement is determined by the spot size and the sampling depth. An incident beam of 5.6 MeV alpha particles was focussed to a 4 mm^2 spot on the sample that was tilted at 75 degrees from beam normal. The recoiling deuterium was detected at a forward scattering angle of 30 degrees. 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 Si solid-state detector. Carbon was detected using Rutherford Backscatter Spectroscopy (RBS) analysis of incident alpha particles. The incident energy of 5.6 MeV was chosen to enhance carbon sensitivity using the resonant backscattering technique. The RBS technique also has a sampling depth of $\leq 5 \mu\text{m}$. These ion beam analysis techniques have a resolution limit of $5 \times 10^{14} \text{ atoms/cm}^2$ so values less than this are not reliable.

3. RESULTS AND DISCUSSION

3.1 Deuterium implanted samples

The deuterium areal densities implanted prior to cleaning are shown in Fig. 2. An areal density of $1 \times 10^{15} / \text{cm}^2$ corresponds approximately to one monolayer. As can be seen in the figure, the deuterium was greatly reduced by the TA cleaning with all but one sample below the resolution threshold of $5 \times 10^{14} / \text{cm}^2$. This represents minimum cleaning efficiencies of 95% to 99.9%. 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/cm^2 .

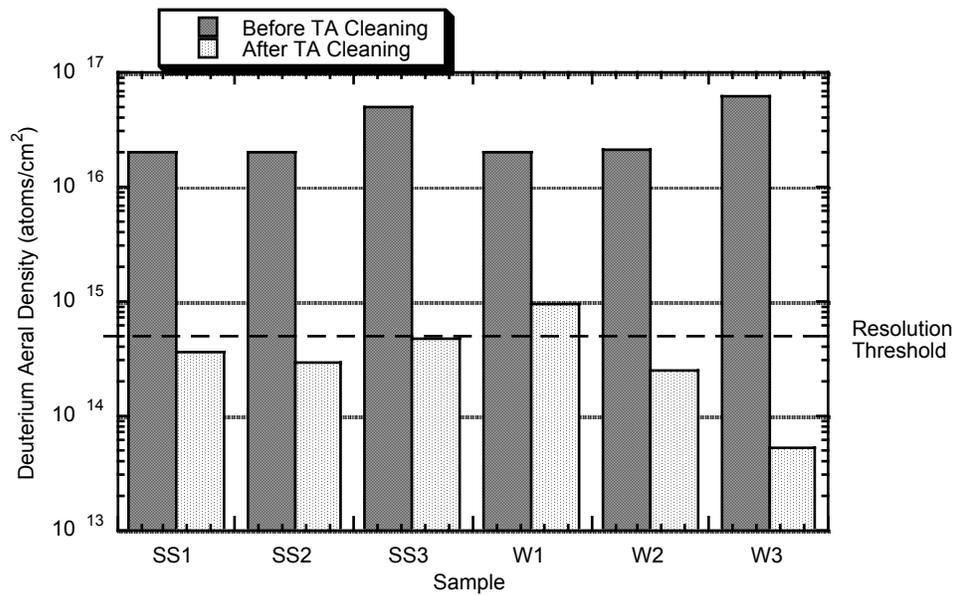


Fig. 2. Ion implanted deuterium areal densities before and after TA cleaning on stainless steel (SS) and tungsten (W) samples.

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 $\mu\text{g}/\text{C}$. These values are in good agreement with other measurements of cathode erosion rates [10].

3.2 Deuterium coated samples

Four of the stainless steel samples (SS4, SS5, SS6, and SS7) and one of the tungsten samples (W4) showed measurable deuterium deposited on the surface from plasma deposition in PISCES-A. All of the deuterium measurements after cleaning were at or below the resolution threshold of 5×10^{14} atoms/cm². Therefore, the values shown in Fig. 3 for after cleaning represent maximum possible values rather than accurate measurements.

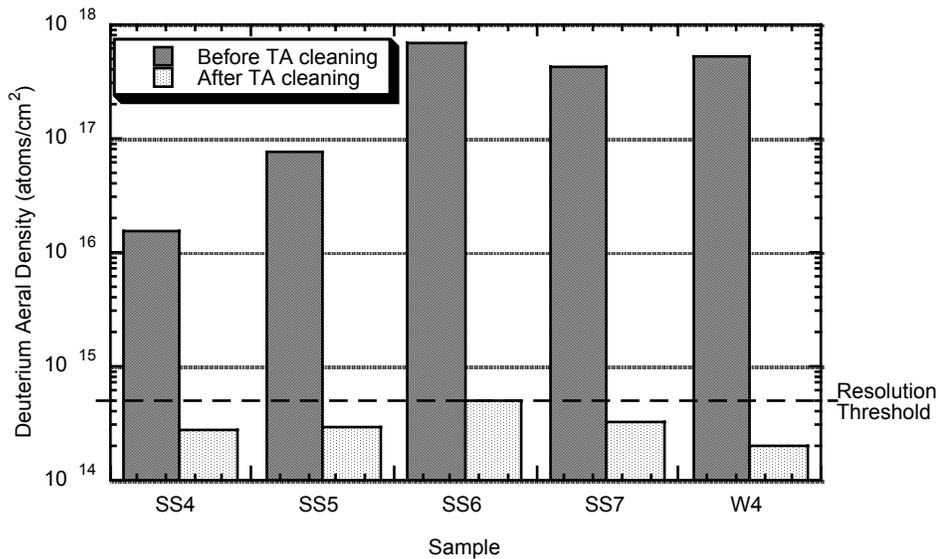


Fig. 3. Surface deuterium areal densities before and after TA cleaning on stainless steel (SS) and tungsten (W) samples with plasma deposited films from PISCES-A.

3.4 Carbon coated samples

The samples had carbon deposits of varying areal density after plasma deposition. Samples were given varying amounts of cleaning charge in order to determine the effect of cleaning charge on contaminant removal efficiency. Fig. 4 shows the areal densities of carbon on the surface before and after cleaning demonstrating large reductions in all cases.

3.5 Erosion rate measurements

In order to determine the cleaning speed for a given object, the rate of material removal as a function of cleaning charge must be measured. With the surface area of the object and the desired amount of material to be removed, the electric current and time needed to clean the object can be calculated. The values of eroded mass plotted against cleaning charge are shown in Fig. 5 for the stainless steel samples. The lower total charge flow samples (<100 C) showed a mass loss rate of approximately 20 $\mu\text{g}/\text{C}$ while the higher total charge flow samples (>150 C) gave a mass loss rate of approximately 77 $\mu\text{g}/\text{C}$. These two different mass loss rates are indicative of the fact that there are actually two types of arcing occurring for each of the cleaning experiments. The initial arcing on the samples results in the cleaning of the surface films. Once the surface has been cleaned, a new mode of arcing occurs which attaches to the

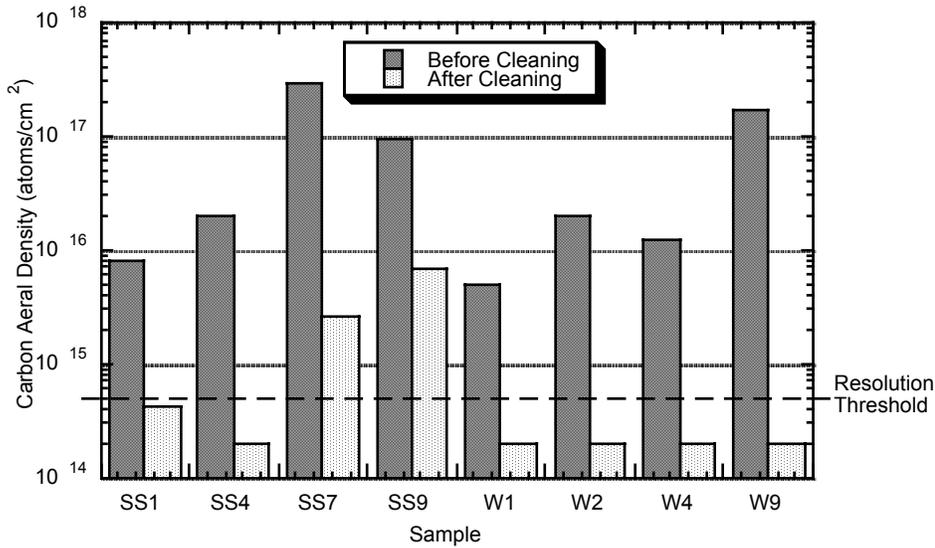


Fig. 4. Surface carbon areal densities before and after TA cleaning on stainless steel (SS) and tungsten (W) samples.

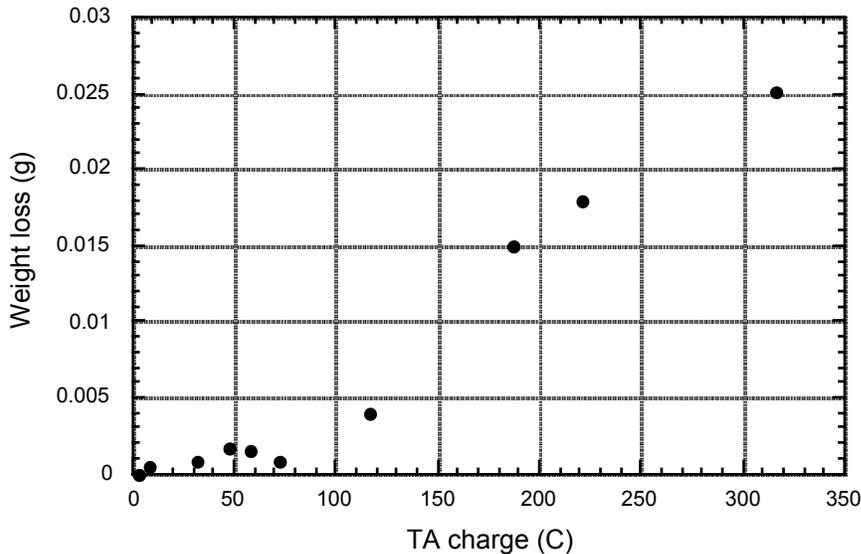


Fig. 5. Weight loss from stainless steel samples as a function of TA cleaning charge. bulk cathode material and causes it to erode. In general, the cleaning arc produces erosion rates of $<10 \mu\text{g/C}$ while the bulk cathode arcing produces erosion rates of $10\text{-}100 \mu\text{g/C}$ [10]. The value of $20 \mu\text{g/C}$ results from the dominance of cleaning arc erosion which occurred on the lower total charge flow samples. The value of $77 \mu\text{g/C}$ results from the dominance of bulk cathode erosion which occurred on the higher total charge flow samples. The dominance of cleaning arcing for samples with $<100 \text{ C}$ total charge and bulk cathode arcing for samples with $>150 \text{ C}$ was confirmed by video observation of arc emission intensity during cleaning. For the tungsten samples, the erosion rates did not depend strongly on the total TA current flow. The mass loss rate was approximately $20 \mu\text{g/C}$ for all samples. This is due to the fact that metal

arcing erosion was largely absent from the tungsten samples owing to the much higher voltage required for tungsten arc attachment.

3.6 Contaminant removal mechanism discussion

The mechanism for removal of ion implanted 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 near the surface of the sample. 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. 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 nm 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.

The removal of the deuterium/carbon surface films was also due to the material removal mechanism of cleaning. As the arc attached to the surface of the material, intense surface heating caused the contaminant film as well as some of the underlying metal sample to be evaporated and/or ejected as small molten particles. The entire surface is thus cleaned due to the migrating action of the arc which moves from one area of contaminant film to another until the surface film is completely removed.

4. CONCLUSIONS

The following conclusions resulted from this study of the removal of ion implanted deuterium and plasma deposited deuterium/carbon films from stainless steel and tungsten samples by transferred-arc cleaning:

- Ion implanted and plasma deposited deuterium are effectively removed from stainless steel and tungsten by the TA cleaning process.
- Surface carbon was effectively removed from the surface of samples by TA cleaning.
- Two different erosion rates were observed for the stainless steel samples. These different rates are indicative of the two different types of arc phenomena.
- The carbon and deuterium were removed from the samples by a combination of thermally induced diffusion and evaporative erosion.
- Transferred-arc cleaning of magnetic-confinement fusion reactor components for removal of contaminant species has many promising characteristics. However, further work is needed to test the usefulness of the technique in actual reactor use.

Future research plans include the investigation of TA cleaning for use on actual magnetic-confinement fusion device components which have been contaminated by films containing deuterium, tritium and carbon. This future work will help in evaluating the suitability of TA cleaning for present and future requirements.

REFERENCES

- [1] K.Y. Wong, B. Hircq, R.A. Jalbert, W.T. Shmayda, *Fusion Eng. and Design* 16(1991) 159-172.
- [2] W. Jacob, B. Landkammer, C.H. Wu, *J. Nucl. Mater.* 266-269 (1999) 552-556.
- [3] A.A. Haasz, J.W. Davis, *J. Nucl. Mater.* 256 (1998) 65-68.
- [4] C.H. Skinner, H. Kugel, D. Mueller, B.L. Doyle, W.R. Wampler, *Proceedings of the 17th IEEE/NPSS Symposium on Fusion Engineering, San Diego, CA, Oct 6-10, 1997, v.1, p.321-324.*
- [5] A.E. Guile, *Proc. Inst. Elect. Eng, London, Sept 1971, v. 118, no. 9R, p. 1131-54.*
- [6] A.E. Guile, B. Juttner, *IEEE Trans. Plasma Sci., v. PS-8, no. 3, p. 259-69.*
- [7] B. Juttner, V. Puchkarev, E. Hantzsche, I. Beilis, "Cathode Spots", *Handbook of Vacuum Arc Science and Technology*, R. Boxman, P. Martin, D. Sanders, Eds., Noyes Publications, 1995, p 75.
- [8] D. M. Goebel, J. Bohdansky, R. W. Conn et al., *Nucl. Fusion* 28 (1988) 1041.
- [9] *Handbook of Modern Ion Beam Analysis*, Eds. Joseph R. Tesmer and Michael Nastasi, Materials Research Society, Pittsburgh, PA (1995).
- [10] B. Juttner, V. Puchkarev, E. Hantzsche, I. Beilis, "Cathode Spots", *Handbook of Vacuum Arc Science and Technology*, R. Boxman, P. Martin, D. Sanders, Eds., Noyes Publications, 1995, p 124,257.