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SURFACE ANALYSIS OF TFTR VACUUM VESSEL
SAMPLES SUBJECTED TO THE POST-WELD
HEAT TREATMENT

BY

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Surface Analysis of TFTR Vacuum Vessel Samples
Subjected to the Post-weld Heat Treatment[†]

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ABSTRACT

To ensure the dimensional stability of the Tokamak Fusion Test Reactor (TFTR) vacuum vessel, it is necessary to perform a post-weld heat treatment (PWHT). This process consists of heating the vessel segments to $\sim 450^{\circ}$ C for 1.5h. The large size of the segments precludes a vacuum bake previous to installation. Effects of the PWHT on the vacuum vessel surface were studied using small samples of vessel material which were subjected to a variety of PWHT procedures, including inert gas purges and different oven designs. Changes in topography and near-surface chemistry were investigated with SEM and sputter-Auger electron spectroscopy. These samples were compared with the surface properties of non-baked UHV-quality stainless steel. The primary difference noted between the PWHT samples and the non-baked control was the thickness of the passivation oxide layer. The thickness of this mixed oxide ($\text{FeO}/\text{Cr}_2\text{O}_3/\text{NiO}$) on the control sample was $\leq 100\text{\AA}$. The thickness of the oxide layer on the heat-treated samples ranged between 230-350 \AA , depending on the method of the PWHT. The effect of hydrogen glow discharge cleaning on these thicker oxide layers, and the consequences of such heat treatment procedure relative to oxygen impurity production in fusion devices are discussed.

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Introduction

Among the many challenges in the construction of the Tokamak Fusion Test Reactor (TFTR), the vacuum vessel has proved to be a particularly formidable fabrication problem because of its large size, numerous penetrations (for diagnostics, pumps, neutral beams, etc.), and various requirements such as a 250°C bake-out capability. The vessel, which is made of 304 LN stainless steel and Inconel 625, is comprised of ten segments designed to be assembled at the TFTR site.¹ To ameliorate possible hysteresis in thermal expansion during bake-out cycles and also to relieve some of the residual stresses introduced during fabrication, each of the ten segments is subjected to a post-weld heat treatment (PWHT). In the PWHT, a segment is abrasively polished, chemically cleaned, heated in a gas-purged oven to ~450°C for 1.5h, and then allowed to cool. Since this process will affect the vacuum properties of the vessel, it is of interest to ascertain the changes in the surface characteristics which accompany the PWHT. In this paper we present analyses on small samples of 304 LN stainless steel which were subjected to two variations of the PWHT. Changes in surface topography and in near surface composition were investigated using SEM and sputter-Auger electron spectroscopy. Control measurements were also made on UHV quality stainless steel. Finally, samples were analyzed by AES and SEM following exposure to hydrogen glow discharge² cleaning, typical of that planned for use in initial TFTR operation.

Experimental Procedure

304 LN stainless steel samples were given four different PWHT's by the fabricator.³ (See Table I.) We will present data on the two samples, B and

E, which received the most different treatments. Sample B had been lightly sanded by hand, heated in air to $\sim 450^{\circ}\text{C}$ for 1.5 hours, and cooled in air to 100°C . Sample E was hand-sanded, then cleaned with acetone, alcohol, and wiped dry. The furnace was purged with purified Argon gas boiled off from liquid argon. The O_2 level in the oven was $< 0.1\%$ before heating. A purge of $1.7 \text{ m}^3/\text{h}$ was maintained throughout the entire 1.5 hour heat and hold period (450°C), and the cool-down to 35°C .

Control samples made of 304L stainless steel were only cleaned with alcohol. Samples B, E, and the control samples were mounted on the manipulator stage of our UHV analysis system and the system was pumped down to 5×10^{-10} Torr. The major constituents of the residual gas were H_2 , CO , and H_2O .

A PHI M/N 590 Scanning Auger Microprobe was used to analyze the samples. Secondary electron imaging was used to characterize the topography of the samples, and sputter Auger profiles were used to characterize the near surface elemental composition. Atomic concentrations were determined in the usual fashion.⁴ A 5 keV Ar^+ beam with a current density of $4 \times 10^{-4} \text{ A cm}^{-2}$ was used for sputtering. The sputter etch beam was usually rastered over a 10^{-2} cm^2 area to minimize crater-edge effects.

Analyses

Topography of both samples B and E was similar. Parallel grooves about 1μ deep with separation of about 4μ were observed on each sample (see Fig. 1). The grooves on sample E had more small scale structure ($.3\text{-}1\mu$), such as pit marks, than sample B. This difference was also discernible by the unaided eye. Samples A-E were gold in color. The control samples were silver colored.

Surface composition was determined with the AES electron beam rastered over a 300 μ area to average out surface inhomogeneity. The elemental composition of the surface for each sample is listed in Table 2. The control sample showed the usual carbon surface contamination common to any unprepared surface. Minor impurities that were detected on samples B and E included Na, Al, Si, and Mg. The Al, Si, and Mg are assumed to be residues from the abrasive treatment.

Figure 2 shows the atomic concentrations as a function of time during Ar^+ sputtering. The gold color disappeared after .1-.2 coulombs of Ar^+ sputtering. After this amount of sputtering the oxygen signal had fallen by a factor of six from its peak value. Exposure of the samples to .1 coulomb of H^+/cm^2 from H_2 G.D.C. also removed the gold color and the oxide film, (Figs. 3a and b).

The oxide thicknesses, defined as the 1/e point in the O fall-off in Fig. 2, on the various samples were 350 \AA (sample E), 230 \AA (sample B), and 70 \AA (control sample). The absolute depth was determined by multiplying the measured sputtering rate for Ta_2O_5 by the factor 1.4, which corrects for the difference between Ta_2O_5 and iron sputtering rates.⁵ This procedure underestimates the oxide thickness by 10-50% because of the smaller sputtering yield of iron and chromium oxides.

After sputtering for ~1 hour (5000 \AA), the atomic composition was calculated for all elements whose lines were discernable in the 10-2000 eV dN/dE spectrum. The results are shown in Table III.

Discussion

The oxide layer on sample E was thicker than that on sample B, which in

turn was thicker than that on the control sample. The oxygen content in the bulk (sputter time > 60 minutes) of sample E and D was greater by a factor of 1.5-2 than that of the control sample of steel, thus indicating diffusion of O into the bulk. These results show that the argon purge was an ineffective method of preventing oxidation during heat treatment.

Assuming that the observed surface conditions are representative of those of the vessel itself, we make the following inferences for TFTR operation. The increased oxidation of PWHT samples present an additional source of oxygen impurities accessible to high power discharges. H₂ glow discharge cleaning reduces the oxide layer to a large degree, but the samples provided still had greater concentration of oxygen at a depth of 5000Å which is inaccessible to the discharge cleaning flux. More importantly if the PWHT oxidation layer were left intact, a 500 Å thick oxide layer would likely trap 1-2 kC of tritium and would complicate the desorption at the 250°C vessel bake-out temperature.⁶

Light sputtering removed all of the Na, Si, Al, and Mg suggesting a loosely bound residue from abrasive treatment (sanding). Although H₂ G.D.C. cleaning of the assembled TFTR vessel can remove the surface oxygen as volatile compounds, it may not be able to remove the Na, Al, Si, and Mg. However, these elements have lower Z and lower concentrations than the constituents of stainless steel and should have comparable sputtering coefficients. Hence we believe these sanding residues will not be a problem to high power TFTR operation.

References

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Table 1

Types of Post-Weld Heat Treatments

<u>Sample</u>	<u>Pretreatment</u>		<u>Heat Treatment</u>		
	<u>Hand Sanding</u>	<u>Solvent Degreasing</u>	<u>Argon Purge</u>	<u>Temp. °C</u>	<u>Time h</u>
A	yes	no	yes .85 m ³ /h	450	1.5
B	yes	no	no	450	1.5
D	yes	yes	yes .85 m ³ /h	450	1.5
E	yes	yes	yes 1.7 m ³ /h	450	1.5

Table II

Elemental Surface Composition of the Stainless Steel Samples

Surface Atomic Percentages

<u>Sample</u>	<u>B</u>	<u>E</u>	<u>Control</u>
Element (Ar-Mo)	0.30	0.5	0.0
C	17.0	28.8	86.0
Ca	0.0	2.1	0.0
Cl	0.7	0.3	0.6
Cr	20.8	5.0	0.0
Fe	21.0	22.0	2.9
Ni	1.8	0.0	0.0
O	36.8	42.9	9.0
S	0.6	0.8	0.3

Table III

Element Surface Composition of the Stainless Steel Samples After
Sputtering Approximately 5000Å into the Bulk.

Si	1.6	0.7	0.8
Cl	0.6	0.4	0.2
C	4.2	4.0	10.8
Cr	18.9	16.6	16.7
O	3.6	4.5	2.3
Fe	63.0	65.0	62.0
Ni	7.9	8.4	7.1

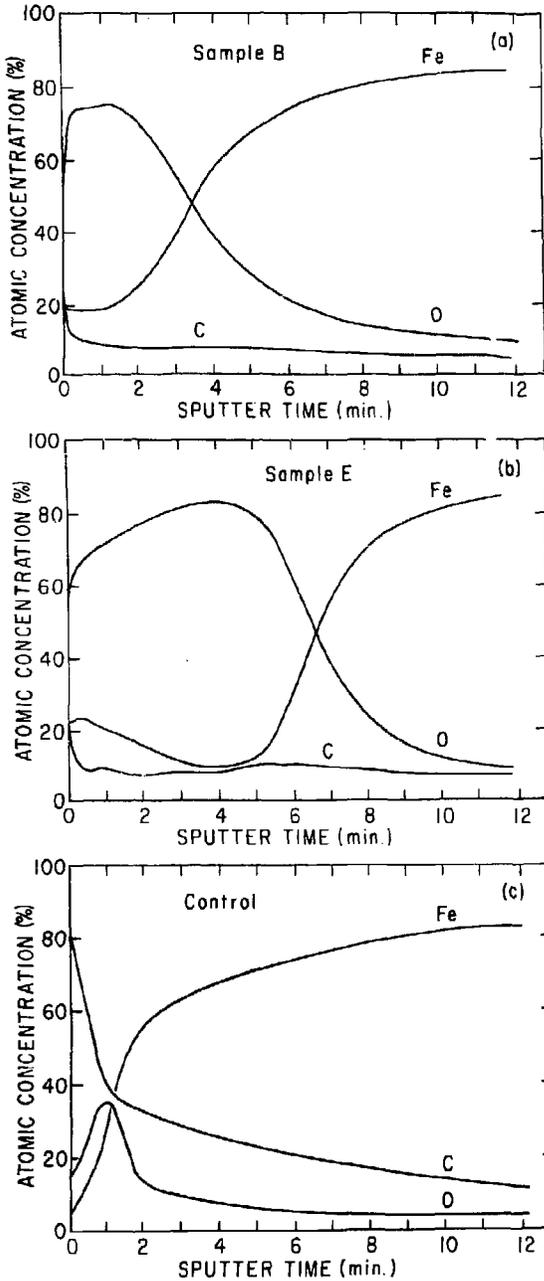


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Fig. 1(a). SEM at 250x of 304 LN stainless steel sample B. This sample was hand-finished with abrasive paper and then heated to 450°C for 1.5 hours in atmosphere.



(PPPL-806220)

Fig. 1(b). SEM at 250x of 304 LN sample E. This sample was also hand-finished, then degreased with acetone and methanol prior to a heat treatment of 450°C for 1.5 hours in an argon-purged atmosphere.



(PPPL-806490)

Fig. 2. Sputter-Auger profiles of the heat-treated 304 LN sample B (a) and E (b). For comparison the profile for typical UHV quality 304 L stainless steel is shown (c). The samples were profiled with a 5 keV A^+ beam with a current density of 4×10^{-4} A cm^{-2} . The sputter rate at the density is equal to 1 \AA s^{-1} for bulk stainless steel.

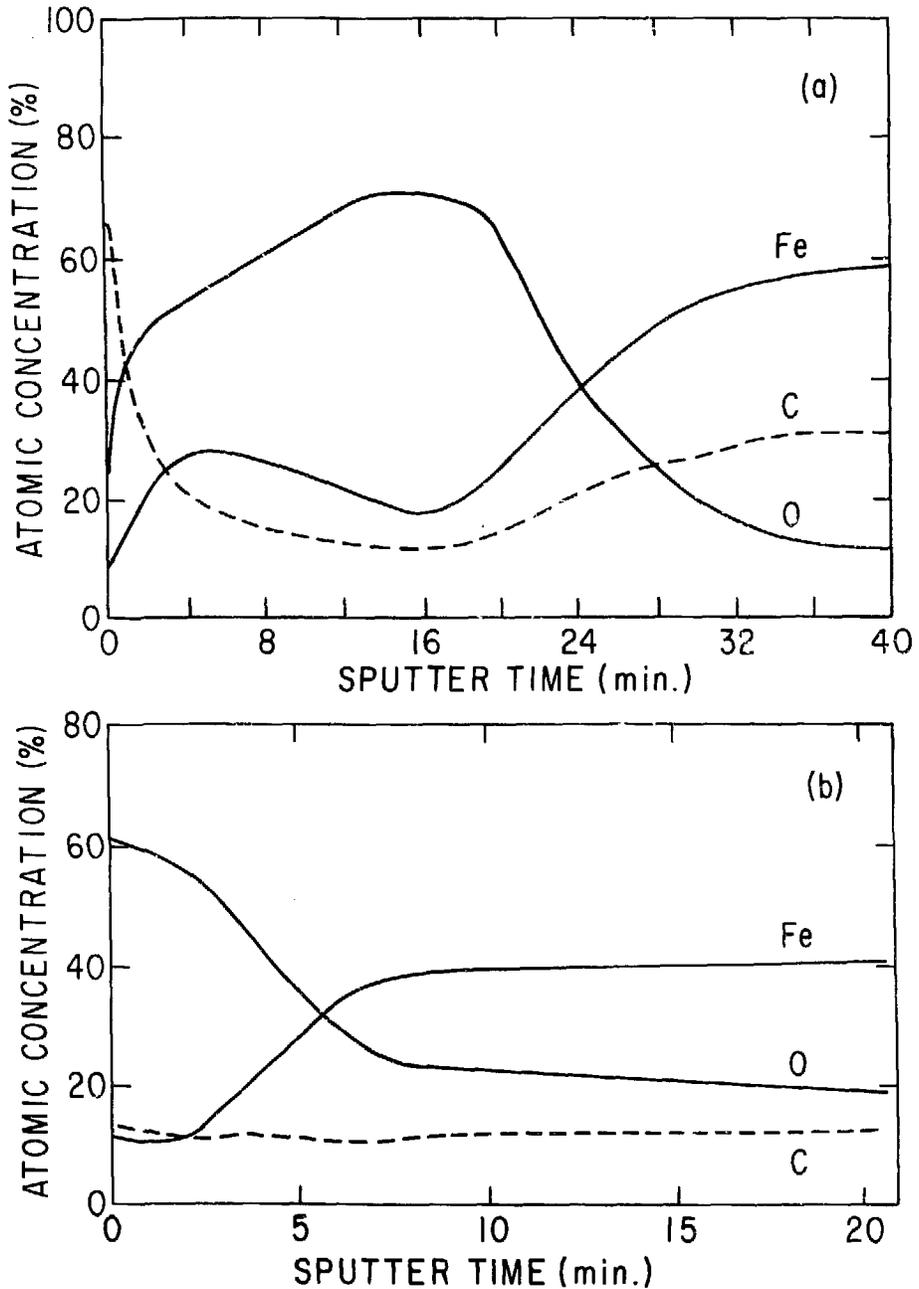


Fig. 3. Sputter-Auger profiles of 304 LN sample E before (a) and after (b) exposure to hydrogen glow discharge cleaning (GDC). The H^+ dose during GDC was $\sim 10^{18} H^+ cm^{-2}$. Sputter rates are the same as given in Fig. 2.