Analysis of Material Removed From UCLA Tokamaks Microtor and Macrotor

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February 1979

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INTRODUCTION

The tokamak approach to magnetic confinement of thermonuclear plasmas is currently the lead program in the effort to achieve controlled nuclear fusion⁽¹⁾ and it is expected that scientific feasibility will be shown in the near future. However, in order to realize the enormous potential of the tokamak fusion reactor, many scientific, technical, and engineering problems must be solved.

In a tokamak, plasma particles are confined within an evacuated toroidal chamber by a magnetic field concentric to the chamber. Because the duration of the plasma discharge in present day tokamaks is much longer than the particle confinement time,⁽²⁾ virtually all of the material in the plasma has been in frequent contact with the wall (usually made of stainless steel). The reflux and emission of material by the wall is, therefore, a dominant factor controlling the properties of plasma in current tokamaks and must be understood and/or controlled before plasma discharges of the long duration and relative purity necessary for a power reactor can be achieved.

In the last few years, the extreme importance of the plasma-wall interaction has become generally accepted and experiments are being performed to examine various parts of the problem. These experiments can be grouped into three areas: the basic study of the interaction processes; simulation of the plasma-wall interaction in a controlled environment; and the study of wall properties of existing tokamaks. The necessary understanding will probably be obtained using results from all three of these methods.

The study of the surfaces of operating tokamaks began in recent years and the techniques developed have not been applied to all existing tokamaks. Since the various machines have important differences in parameters (size, power density, and impurity concentration), it is necessary to understand both their common characteristics and their differences.

Workers at the Massachusetts Institute of Technology (MIT) and the University of California at Los Angeles (UCLA) have been successful at producing high-purity (low- Z_{eff}) plasmas by using a low-energy ac discharge cleaning procedure on their tokamaks.⁽³⁾ However, little surface analysis

has been performed on these machines either to determine the effects of cleaning or to establish how the machines differ from other tokamaks with higher impurity concentrations.

This paper reports a first effort to examine the surface of the UCLA tokamaks, Microtor and Macrotor, by analyzing samples that have been exposed to plasma discharge and cleaning for long periods. The samples were sent to the Surface Science Section at the Pacific Northwest Laboratory (PNL). There, Auger electron spectrometry and sputter profile techniques were used to examine the samples, which had been handled in atmospheric conditions after being removed from the tokamak. Two objectives of the study were to learn

- whether the material collected varied with the type of machine operation and was correlated with changes in the plasma impurity content, and
- what types of information are available from samples handled in such a simple manner.

SAMPLES

The samples used in this study were either small sections of stock material placed in the machines for this experiment, or materials that were in the tokamaks for other purposes. The samples were therefore not specially treated prior to insertion in the tokamaks. They received only the normal degreasing and cleaning procedures used on all material placed in the machines.

Table 1 identifies the samples from the two UCLA tokamaks. The materials from Microtor were from various locations in the machine (see Figure 1) and had been exposed for periods ranging from 1 to 8 weeks between the spring of 1976 and the fall of 1977. Glass window no. 1, which covered a viewport, was in place during early operation of the machine and collected a film about 3500 Å thick while various cleaning procedures, wall treatments, and plasma pulses were being tested in the tokamak. The aluminum shutter protected a later version of the window and collected material at roughly the same port location. The stainless steel rod served as a shield for a microwave antenna and functioned to some degree as a limiter for the outside edge of the plasma. The second glass window and the wall flakes were removed from Microtor after a period of intensive wall processing using an argon glow-discharge. The flakes consisted of materials deposited on the wall during this procedure.

The first four Macrotor samples listed in Table 1 were exposed to an initial glow-discharge cleaning procedure during the winter of 1977. The first three metal samples, sections of stock material lying on the bottom of the port or chamber (see Figure 1), were in the machine at the same time. The glass material was a broken section of a support that fell into the plasma chamber. The final aluminum plate listed was in the center of the tokamak chamber and served as the anode during argon glow-discharge cleaning after a vacuum break.

TABLE 1. Description of Tokamak Samples

<u>Microtor Samples</u>	<u>Location</u>	<u>Time</u>	<u>Machine Operation</u>
Glass Window no. 1	Port area	Spring 1976	Cleaning experiments Plasma discharge
Aluminum Shutter	Port area	Summer/Fall 1976	ac cleaning Plasma discharge
Stainless Steel Rod	Outside plasma edge	Summer/Fall 1976	ac cleaning Plasma discharge
Glass Window no. 2	Port area	Fall 1977	Argon glow-discharge cleaning
Wall Flakes	From chamber wall	Fall 1977	Argon glow-discharge cleaning
<u>Macrotor Samples</u>			
Stainless Steel Plate	Port area	Winter 1977	Exposed to oxygen, argon, and hydrogen glow-dis- charge during initial machine processing
Stainless Steel Plate	Bottom of plasma chamber	Winter 1977	
Aluminum Plate		Winter 1977	
Glass		Winter 1977	
Aluminum Plate	Center of plasma chamber	Winter 1978	Argon glow-discharge

FIGURE 1. Sample Locations in Microtor and Macrotor

ANALYSIS PROCEDURE

After exposure to the plasma and cleaning procedures, all samples were placed in plastic bags and sent to PNL where they were prepared for analysis. Each metallic plate was cut with shears while still in its plastic bag. The stainless steel rod and the glass samples required more elaborate cutting procedures that involved exposure of the surfaces to extraneous materials such as water or epoxy.

After being cut to an appropriate size, the samples were degreased in acetone, methyl alcohol, and ethyl alcohol. They were then examined by Auger electron spectroscopy (AES) and sputter profile techniques. The semi-quantitative use of these methods required us to make several assumptions.⁽⁴⁾ For example, we assumed that sputtering rates for all elements were equivalent to that of TaO, for which our ion gun is calibrated. We further assumed that the tables of sensitivity and the AES spectra reported in the Physical Electronics Industries Handbook of Auger Electron Spectroscopy⁽⁵⁾ were accurate and applied to our data. Unless otherwise noted, chemical effects on peak shapes and other similar problems were ignored.

The interpretation of data gathered from the samples was also based on several assumptions that appear reasonable but may be invalid in particular circumstances. The assumptions were that: 1) exposure to atmosphere and to any necessary cleaning procedures affects only outer surface layers of the samples; 2) material collected by the samples in the port areas is representative of the material incident on and removed from the wall; and 3) there is no significant thermal diffusion of the material between layers of different elemental concentrations as deposited on our samples.

RESULTS AND DISCUSSION

MICROTOR SAMPLES

The peak-to-peak amplitude of the Auger signal as a function of sputter time is shown in Figure 2 for Microtor's glass window no. 1. The data indicate that this viewport window primarily collected material removed from the tokamak's stainless steel wall. The sample had a film deposit approximately 3500 Å thick, and the relative amplitude of the signals for each element deposited provides some indication of how the elemental concentration of the deposited layers changed with varying machine operation.

Although the differences in operational mode were not documented for this sample, this observation suggests the answer to one of our first questions: the material deposited in the port areas does vary as the overall operation of the tokamak changes. The approximate relative composition of the deposit is given in Table 2 for sputter depths indicated by the arrows on Figure 2. For this early time in the lifetime of the machine, the major deposits on the glass were oxygen and iron. The amount of nickel on the glass, relative to the amount of iron, was not representative of uniform erosion of the 304L stainless steel wall; this was presumably due to several attempts to coat the tokamak wall with nickel.

Figure 3 shows the Auger depth profiles of the aluminum shutter. Through analysis of both the unexposed and exposed sides of the sample, the effects of the plasma on the uncharacterized sample can be determined and distinguished from any influence of handling or cleaning procedures. The side exposed to the plasma was visibly coated, although the deposit thickness was estimated to be only about 80 Å. Both sides of the sample had a peak in the oxygen signal followed by a nearly pure aluminum signal. A more detailed analysis of the Auger signal in this region showed a transition from an aluminum oxide signal to the standard aluminum lineshape. The presence of this Al_2O_3 -Al transition on both sides of the sample indicates that material was simply collected on the shutter and did not strongly interact with the surface material originally there. Although the surface of the sample was primarily oxygen, most of the

TABLE 2. Composition of Microtor Samples

Sample	Sputter Depth	Percent Composition of Sample				
		O	C	Fe	Ni	Cr ^(a)
Base Wall Material (304L Stainless Steel)				70	9	21
Glass Window no. 1	I	36	10	35	13	6
	II	31	13	41	15	(a)
	III	42	10	33	15	(a)
Aluminum Shutter		8	22	38	16	15
Stainless Steel Rod		10	8	45	19	19
Wall Flakes	I ($\sim 160 \text{ \AA}$)	3.5	20	57	6	13
	II ($\sim 350 \text{ \AA}$)	2	18	57	6	16
	III ($\sim 1,120 \text{ \AA}$)	0.5	8.5	62	6	23
	IV ($\sim 4,600 \text{ \AA}$)	-	2	65	6	27
Glass Window no. 2	I ($\sim 500 \text{ \AA}$)	6.5	9	58	8.5	18
	II ($\sim 2,400 \text{ \AA}$)	8	8	58	8.5	18
	III ($\sim 10,000 \text{ \AA}$)	7.5	5	60	8.5	20

(a) The Cr signal cannot be precisely measured in the presence of a large oxygen signal.

FIGURE 2. Depth Profile of Glass Window Sample No. 1 (Microtor)

FIGURE 3. Depth Profiles of Aluminum Shield Sample (Microtor)

material deposited had a much smaller oxygen content. We assume that the material coming out of the plasma is best represented by the elements collected at the sputter depth marked by the arrow in Figure 3 and roughly corresponding to the peaks in the metallic deposition. The approximate composition at this region is given in Table 2. Note that this region did not have an Fe/Cr/Ni ratio representative of the 304L stainless steel wall, but had a larger concentration of nickel, as did the glass sample. The decreased amount of oxygen deposited on the aluminum (as compared to the amount on the glass sample) is consistent with a concomitant decrease in the oxygen concentration of the plasma discharges. This observation indicates that the amount of oxygen collected in the port area was directly correlated to observable changes in plasma parameters.

Figure 4 shows the Auger sputter profiles of the stainless steel rod that functioned to some extent as a limiter at the outside edge of the plasma. One side of the rod had feather-like lines believed to be due to run-away electrons. This is labeled the "electron side" in Figure 4. The side opposite these marks had been exposed to plasma ions and is called the "plasma side." A comparison of these profiles with those of stainless steel samples not exposed to the plasma suggests that both sides were altered by exposure to the tokamak discharges, although the profile of the electron side is more nearly like that of unexposed stainless steel. The plasma side of the rod was significantly altered by interactions with the plasma. The concentration profiles for chromium, iron, and nickel changed significantly in the near-surface region. It seems likely that this altered region represents an equilibrium between deposition of material and plasma-induced erosion, such as occurs at the wall. Note that the composition of the rod in the region where the largest change occurred (marked by the arrow) was similar to that of material collected on the aluminum shutter. As can be seen from Table 2, the ratios of O/Fe/Cr/Ni on the two samples were nearly identical. The reason for a major difference in carbon content is not clear.

The observation of different depth profiles on the electron and plasma sides of the rod may provide some indication of the mechanism that transports material around the tokamak. Any process that would produce a uniform distribution of material would seem to be ruled out. For example, it is difficult to explain the distribution of material on the stainless steel rod solely in terms of sputtering of wall material by charge exchange neutrals, as such sputtering would be expected to produce a more isotropic distribution of material. At the moment, a more definitive statement about the transportation mechanism cannot be made and further experiments are in progress.

In September 1977, Microtor was shut down for repairs and improvements. The machine was disassembled and the main vacuum components were cleaned initially by argon sputtering. During this process, material was deposited on the windows in the port areas and on the inside walls of the main chamber. Material collected on the windows and flakes accumulated on the chamber wall were sent to PNL for analysis.

FIGURE 4. Depth Profiles of Stainless Steel Rod (Microtor)

Roughly 10,000 Å of material collected on the glass window and the depth profile showed a relatively uniform composition as indicated by data in Table 2. The buildup had the same composition as the stainless steel wall (iron, chromium, and nickel), with the addition of oxygen and carbon. The material on the window probably represents the flow of sputtered atoms from the main chamber to the port area. There they collected on the relatively cool window, out of the range of primary sputtering activity. Note that the Fe/Cr/Ni ratio (7/2.3/1) is slightly different from that of bulk stainless steel (10/2.3/1); this indicates that more iron was reaching the window than would be expected from the base wall composition.

The wall flakes were also composed primarily of chromium, nickel, iron, oxygen, and carbon. There is a continual process of deposition and removal of material during the sputter process; however, the flakes formed on the

inside wall indicate that in this area deposition was greater than sputter removal. The flakes had a somewhat less uniform composition than the deposit on the glass, as shown in Table 2. One difference was a significant amount of carbon that decreased as we sputtered into the material. In addition, at shallow sputter depth the Fe/Cr/Ni ratio of 9.5/2.25/1 was nearly that of bulk stainless steel, but in the interior of the flake, an increased amount of chromium was present with a resulting ratio of 10.8/4.5/1. It is tempting to try to relate this observation to the composition of the Microtor chamber wall after a year of plasma processing, but we will not try this because the complications of the sputter process make any models questionable.

MACROTOR SAMPLES

The depth profiles of the stainless steel sample located in the port of Macrotor are shown in Figure 5. A comparison of the profiles for the exposed and unexposed sides of the sample suggests that the glow-discharge cleaning procedure moved a lot of material to the port area. As shown in Table 3, the dark deposit layer that was visible on the sample consisted mostly of oxygen, with some elements of stainless steel. By contrast, the stainless steel sample in the chamber had no visible deposit of any kind and until analysis showed argon from the discharge cleaning process on the surface of the sample, there was no indication of which side had been exposed to the cleaning process. However, the depth profile for the chamber sample (Figure 6) disclosed differences which suggest that the cleaning procedure produced a restructured surface consisting of more nickel and less iron and chromium than were found in the unexposed side of the sample. The shoulder in the oxygen profile is similar to that observed after the discharge cleaning procedure on JFT-2⁽⁶⁾ and may indicate that oxygen was being implanted in the sample.

FIGURE 5. Depth Profiles of Stainless Steel Sample in Port (Microtor)

TABLE 3. Composition of Macrotror Samples

Sample	Sputter Depth	Percent Composition of Sample						
		O	C	Fe	Ni	Cr	Al	S
Base Wall Material (304L Stainless Steel)				70	9	21		
Stainless Steel (Port)	52		<3	33	5	7		
Stainless Steel (Chamber)	28		<3	49	8	13		
Aluminum (Chamber)	47		5	19	2	0.6	24	1
Glass	62		<1	24	2	12		
Aluminum (Inside Surface)	I	48	1	37	7	7		
	II	57	0.5	30	7	6		
	III	63	2.8	27	4	3		
Aluminum (Outside Surface)	I	43	1	41	6.5	8		
	II	48	2.5	35	8	7		
	III	42	17	31	2	9

FIGURE 6. Depth Profiles of Aluminum Shield Sample (Macrotror)

The depth profiles of the aluminum samples from Macrotror are shown in Figures 7 and 8. The comparison of the exposed and unexposed sides of the sample is particularly interesting when also compared to the aluminum sample from Microtror (Figure 3). The unexposed side of the Macrotror sample shows an oxygen peak similar to that of the Microtror sample. However, the exposed side has been altered; the $\text{Al}_2\text{O}_3\text{-Al}$ region no longer exists and has been replaced by quantities of oxygen, iron, chromium and nickel that decrease as one sputters into the metal. This observation, in conjunction with data from the other samples, supports the following picture: samples in the port areas primarily collect material; on the other hand, samples that interact directly with the plasma achieve a dynamic equilibrium between erosion and deposition processes. The composition of the near-surface region of this sample is given in Table 2.

FIGURE 7. Depth Profile of Aluminum Sample, Shielded Side (Macrotror)

FIGURE 8. Depth Profile of Aluminum Sample, Exposed Side (Macrotror)

Figure 9 shows the depth profile of a film deposited on a section of broken glass that fell into the plasma chamber. This sample seemed primarily to collect material and the deposit (Table 3) was roughly the same as that collected on the stainless steel sample in the port area and on the reworked aluminum surface.

During the spring of 1978, some aluminum plates were placed in the center of Macrotron during machine processing and argon sputtering. Although deposits built up on both sides of the material (see Figure 10), the deposit toward the outside of the torus was thicker, with a depth ratio for inside/outside of 0.6. The total accumulation of the material was about 10,000 Å on the outside surface. Again, the elemental composition consisted mostly of iron, chromium, nickel, oxygen, and carbon. If the material collected on the aluminum is representative of what was on the tokamak wall, the sputter profile must be read backwards from the Al_2O_3 -Al signal to learn about the wall condition as a function of time.

FIGURE 9. Depth Profile of Coated Glass Sample (Macrotron)

FIGURE 10. Depth Profiles of Aluminum Plate Sample (Macrotron)

The first region moving backwards (region III) showed the first buildup of material on the aluminum. Note that the outside surface had a buildup of carbon, while the inside surface did not. This is consistent with the observed plasma position during normal tokamak operations; since the plasma is usually close to the inside radius of the tokamak, this inside surface gets warmer and sees a greater flux of ions from the plasma. The result is a clean surface. The outside wall, however, has less contact with the plasma and will accumulate carbon, carbon monoxide, and carbon dioxide that happen to be in the machine.

Region II of both profiles showed the greatest amount of oxygen accumulated in each deposit. As further processing occurred (moving toward region I), less oxygen was visible. Although it seems likely that not all of the oxygen in the deposit was from the tokamak wall, the decrease in oxygen (going from region II to region I) is consistent with machine cleanup.

The Fe/Cr/Ni ratios for both deposits were similar for the respective regions (see Table 2), but they were not similar to the ratio for bulk stainless steel. Although the chromium signal was somewhat confused because of the presence of the large oxygen signal, it appears that the elements were not deposited in proportion to the bulk composition of the wall. It is not known whether this difference is characteristic of the sputter process in this setup in the machine or due to the composition of the wall as it was altered by the plasma discharges.

The difference between the thickness of the inside and outside surface deposits is primarily due to the larger surface area available to the outer side of the aluminum plate. It is possible to make a rough estimate of the ratios of material that should be collected on each side of the aluminum plate by considering the effects of both surface area and electric field line on sputtering. The rough estimate is consistent with the observed results; this agreement indicates that the sputter process was operating as expected on each side of the aluminum plate.

Although most of the Microtor and Macrotor samples were not produced under similar conditions and therefore cannot be directly related, it is interesting to compare the material that collected on the samples from the two machines (see Tables 2 and 3). As already noted, Microtor had been coated with nickel which tended to appear both on samples in the port area where material was deposited and on the stainless steel rod where material was deposited and removed. The Macrotor samples collected smaller quantities of nickel, but the amounts were often greater than the relative amount characteristic of the base wall material. This difference could be attributable to the slightly enriched nickel concentration near the surface of unexposed stainless steel (see Figures 5 and 6); the greater surface concentration makes it more likely that the nickel will be moved around. Given a net flow of material to the port areas, the excess nickel at the wall surface will eventually be depleted unless it is resupplied to the wall by diffusion or some other means. Thus, it is interesting to consider the long-term effects and potential benefits of nickel-coating the tokamak wall, as was done on Microtor.

CONCLUSIONS

This analysis of samples that were not specially designed for use in a tokamak and not continuously handled under high vacuum conditions provided more information than was initially expected. Although it is not possible to analyze the effects of a particular pulse or even a particular pulse sequence, we find that the material collected varies both with the mode of machine operation and with the impurity content of the tokamak. This finding indicates that offsite analysis of tokamak samples can provide useful information, particularly when onsite continuous-vacuum experiments are not possible.

Material that collected in a port area of Microtor was found to be similar to material in the near-surface region of a stainless steel rod that functioned as a limiter. The observation that the electron-exposed side of this rod was not markedly altered may help eliminate some of the several theories proposed to explain how material is transported in a Tokamak.

The analysis of samples from both Microtor and Macrotor supports the view that samples away from the plasma tend to collect material, while samples near the plasma achieve an equilibrium between erosion and deposition of material.

The Auger analysis of the samples described above provides an approximate indication of the elemental composition of the samples. The changes in elemental composition during the depth profiles are probably accompanied by similar changes in the chemical composition of the material. Except for special cases (such as the Al-Al₂O₃ transition), these changes are not observed by Auger analysis. The chemistry of the plasma-wall interaction and of the material deposited is an important area that needs study.

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