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Surface and Impurity Studies in ORMAK and ISX

R. J. Colchin
R. E. Clausing
L. C. Emerson
L. Heatherly
R. C. Isler

MASTER

OAK RIDGE NATIONAL LABORATORY

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SURFACE AND IMPURITY STUDIES IN ORMAK AND ISX *

R. J. Colchin, R. E. Clausing, L. C. Emerson,
L. Heatherly, and R. C. Isler

Oak Ridge National Laboratory
Oak Ridge, Tennessee 37830, USA

ABSTRACT

The ORMAK vacuum liner is constructed of stainless steel overcoated with a thin platinum diffusion barrier and a final layer of gold. Gold was selected as the vacuum surface because it is chemically inert to the adsorption of common gases. However, gold surfaces do adsorb hydrocarbons, and carbon (along with oxygen) was the principal plasma contaminant during the first two years of ORMAK operation. Upon switching discharge-cleaning gases from hydrogen to oxygen, carbon levels dropped until carbon is no longer a significant contaminant; residual hydrocarbons can now be controlled either by hydrogen or by oxygen discharge cleaning. The principal measured plasma contaminant in ORMAK is now oxygen. Samples taken from the ORMAK liner and analyzed by Auger electron spectroscopy reveal the presence of iron and oxygen. There is evidence from a SXAPS (Soft X-ray Appearance Potential Spectroscopy) probe of iron and chromium diffusion from the stainless steel through the gold surface in spite of the platinum diffusion barrier. The iron and chromium provide surface oxidation sites, and SXAPS analysis shows that these metals exist as oxides.

In order to investigate tokamak impurity problems further, the ISX (Impurity Study Experiment) tokamak is presently under construction. It will provide a cleaner and more flexible vacuum system in which to conduct studies of surfaces and plasma impurities. The operating characteristics will be much the same as those of ORMAK (with ohmic heating) in terms of size, plasma current, and plasma temperature.

1. INTRODUCTION

Throughout the life of the ORMAK tokamak, Z_{eff} has typically been ≥ 4 . Various modes of discharge cleaning have been employed in an attempt

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to reduce impurities present through modification of wall conditions. As with other tokamaks, ORMAK discharge characteristics are quite sensitive to wall preparation. A summary of discharge cleaning and impurity experiments is given in the next section. A SXAPS (Soft X-ray Appearance Potential Spectroscopy) probe has been used to determine how surface conditions respond to discharge cleaning as well as to high-current tokamak discharges. A description of the SXAPS device is given in the third section, and results of ORMAK experiments are detailed in Sect. 4.

In order to expand and extend these impurity experiments, a new tokamak called ISX (Impurity Study Experiment) is being constructed. This device is aimed at providing a relatively clean and flexible vacuum system in which to perform studies of plasma-wall interactions and impurities. A general description of the ISX and anticipated experiments is given in Sect. 5.

2. DISCHARGE CLEANING AND PLASMA IMPURITIES IN ORMAK

The ORMAK vacuum system consists of inner and outer vacuum chambers. Plasma discharges are formed in the inner chamber (liner), which is constructed of 304L stainless steel overcoated with a thin ($\approx 500 \text{ \AA}$) layer of platinum and an outer ($\approx 1 \text{ }\mu\text{m}$) layer of gold. It was intended that the platinum layer act as a barrier to diffusion of the stainless components through the gold film. Ion microprobe tests¹ of similar material after it was baked for an hour at 300°C showed a marked decrease in surface impurities as compared with similar samples with no platinum barrier.

Discharge cleaning was used during the last four years to condition the surface of the liner. Initially, cleaning discharges were run in hydrogen at the rate of 120 Hz with each pulse lasting about 500 μ sec. Current-induced heating in the liner simultaneously caused a mild baking of the walls, at temperatures in the range 150-200°C. During this type of cleaning only hydrogen spectral lines were observed, but carbon and oxygen were present in the normal tokamak discharges. In the last two years, similar-length cleaning discharges have been run using oxygen in addition to hydrogen.^{2,3} The carbon concentration has fallen, and carbon is no longer a significant plasma contaminant. However, it is observed that the oxygen impurity level increases by nearly a factor of 2 after oxygen cleaning.

Recently, more powerful though less frequent cleaning discharges in hydrogen have been tried. Discharges with a peak current of 16 kA and a safety factor $q = 1$ are initiated at the rate of two/sec. Figure 1 gives a comparison of spectra in the vacuum ultraviolet region resulting from the new and old discharge-cleaning techniques. Oxygen, carbon, and nitrogen impurities are evident in the more powerful discharges. In the initial tests, residual carbon and nitrogen underwent marked decreases during overnight cleaning, while the oxygen level decreased only slightly. The oxygen concentration during the normal tokamak discharges remained at the level observed prior to initiating the high-power, pulsed cleaning.

Spectroscopic measurements show that regardless of the type of discharge cleaning, oxygen is now the dominant light impurity observed in the plasma; this result is consistent with the observation that water vapor is the principal contaminant detected by the residual gas analyzer.

Fig. 1. (a) Partial spectrum of plasma observed during low-power 120-Hz discharges. No impurity lines are observed. The low-intensity features are lines of molecular hydrogen. Signal strength is in arbitrary units. (b) Partial spectrum of plasma observed during high-power, pulsed discharge cleaning. The intensities of the impurity lines and of the hydrogen lines are of the same magnitude. Signal strength scales differ between (a) and (b).

If carbon or nitrogen is observed at all in the plasma, their concentrations are less than 20% of the concentration of oxygen.

Iron is the most abundant heavy impurity which has been identified by spectral analysis, and its concentration is estimated to be no more than one-tenth the concentration of oxygen. The abundance of gold and tungsten (limiter material) remains uncertain; the wavelengths of lines from high ionization stages of these elements are not known, so that even their presence in the plasma cannot be verified. However, gold and tungsten could well be present as there are numerous unidentified lines. A typical vacuum ultraviolet spectrum is shown in Fig. 2.

Spectral lines with wavelengths longer than $50 \text{ \AA}$ are superimposed on a band of "continuum" radiation. The origin of this radiation is unknown, but it may be attributed either to closely spaced lines from a heavy element (such as tungsten or gold) or to recombination radiation near the wall. In any case, this "continuum" radiation accounts for a significant fraction of the radiated power and of the electron energy loss.

Besides spectroscopy, three other techniques of determining Z_{eff} have been used:² soft x-rays, plasma conductivity, and fast ion scattering. All impurity diagnostics have practical difficulties which lead to relatively large experimental errors; however, all methods give $Z_{\text{eff}} \geq 4$. Spatially resolved soft x-ray measurements have been made using a PIN diode array. These measurements, in which coronal equilibrium is assumed, indicate that light impurities dominate at low currents and that heavy impurities become important in high-current discharges. Although we observe a gradual increase of impurities with time, there is no preferential accumulation at the center of the plasma.

Fig. 2. Densitometer trace from 20 Å to 170 Å of spectra photographed during normal tokamak discharges. Most of the strong lines can be associated with ions of oxygen and iron, but several relatively intense features around 50 Å are unidentified.

3. SXAPS DESCRIPTION

A SXAPS probe has been used during the last four months to evaluate ORMAK surface conditions. Since this method differs from the Auger analysis systems used on the Pulsator (QWAASS)⁴ and PLT (SAS)⁵ tokamaks, a brief description is given below.

A review of soft x-ray appearance potential spectrometry has been given by Park and Houston.⁶ The ORMAK SXAPS⁷ probe is shown schematically

in Fig. 3. The surfaces which are analyzed lie on the faces of an eight-sided wheel. One face is exposed to normal plasma or to cleaning discharges and then rotated 180° to face the filament. A variable high voltage is applied to the wheel, and electrons accelerated from the filament release soft x rays from surface atoms. These soft x rays correspond to core-level excitations along with a bremsstrahlung background. Soft x rays falling on the gold-coated photocathode create

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SXAPS Probe for ORMAK

Fig. 3. Schematic of SXAPS probe as configured for use in ORMAK. The sample wheel is turned by a mechanical linkage. Data described in this paper was taken with the ion suppressor and photocathode held at ground potential, and the signal was taken from the collector.

□

secondary electrons which are attracted to the collector wire. In order to separate sharp lines produced by core-level excitations from the bremsstrahlung, the collector current signal is twice differentiated.

Soft x-ray appearance potential spectroscopy has not undergone as extensive a development as has AES (Auger Electron Spectroscopy) and is not a quantitative measurement technique. As pointed out by Musket,⁸ the amplitude and detailed shape of the SXAPS spectrum is sensitive to chemical binding and hence gives chemical information not obtainable from AES. SXAPS can detect most of the tokamak surface contaminants such as stainless steel (Fe, O, Ni, and Cr) and carbon, but is insensitive to gold. It has been our experience that even though SXAPS does not give quantitative estimates of surface coverage, it provides a very convenient monitor of liner surface conditions.

Figure 4 shows the SXAPS spectra of a stainless steel sample taken with a test probe at the ORMAK site. SXAPS is quite sensitive to iron, chromium, and nickel and somewhat less sensitive to carbon and oxygen, although carbon contamination is easily detected (as may be seen in Fig. 4). We find that contaminated specimens exhibit noisier spectra than do clean specimens.

Fig. 4. SXAPS test spectrum of a stainless steel sample. This data was taken with a test probe at the ORMAK site.

4. SXAPS RESULTS

Data have been taken using two different eight-sided wheel, both of which were made of stainless steel and coated with gold and platinum as described in Sect. 2. The second wheel contained a much thicker platinum layer than the first. One of the eight sides of each wheel was bare stainless steel, and a two-mil-thick gold foil was welded to a face of the second wheel.

Before discharge cleaning the first wheel, only carbon was observed on the gold faces. After discharge cleaning in hydrogen over a weekend,

the components of stainless steel appeared on all faces. Similar contamination has been observed on liner samples taken from ORMAK and analyzed by AES.⁷ A typical spectrum from the stainless steel face is shown in Fig. 5. The two peaks following the initial oxygen K peak are probably due to surface plasmon oscillations.⁹ According to Houston and Park, the peak following the chromium L_2 peak appears as the chromium becomes oxidized and thus gives chemical information. The relative height of this peak did not change either with discharge cleaning or with normal plasma discharges, indicating that the amount of Cr_2O_3 remained

Fig. 5. SXAPS spectrum of the stainless steel face of the ORMAK probe. This face had been exposed to several days of both discharge cleaning and normal tokamak discharges. The peak at 364 eV is due to calcium.

constant. Likewise, the shape and height of the iron L_2 peak is sensitive to oxidation, the peak becoming broader and shorter as Fe_3O_4 and Fe_2O_3 are formed.¹⁰ The SXAPS spectra observed in ORMAK were similar to those of Ref. 10 for oxidized iron. The small, unlabeled peak at 364 eV is due to calcium L_3 and L_2 transitions. The spectrum of Fig. 5 was taken after overnight discharge cleaning with hydrogen gas. Similar spectra were observed after oxygen discharge cleaning, except the oxygen peaks were larger and not as clearly separated.

For all SXAPS experiments the current to the sample wheel was fixed at 5 mA (at voltages above 200 V). This current was sufficient to polymerize residual hydrocarbons and cause a buildup of carbon on the wheel surface which we bombarded. This buildup of carbon is illustrated for a series of spectra in Fig. 6. The first spectrum, labeled (a), was taken after the stainless steel face had been discharge cleaned overnight in hydrogen. Spectrum (b) was taken 25 min after the first spectrum and shows signals from carbon starting to appear above the noise; the chromium and iron peaks simultaneously became smaller as the surface was covered by carbon. In spectrum (c), taken 25 min after (b), carbon is quite evident, while the other peaks have shrunk still further. After discharge cleaning overnight the carbon is no longer evident and the chromium and iron peak heights have been restored, as may be seen in spectrum (d).

Results using the second wheel were similar to those using the first. Before discharge cleaning all faces were badly contaminated by carbon. The carbon disappeared upon exposure to discharge cleaning in hydrogen overnight, but it remained on unexposed faces. After four days of discharge cleaning, chromium appeared on the wheel faces that were

Fig. 6. Comparison of SXAPS spectra showing electron-induced adsorption of carbon on the stainless steel sample wheel face as a function of bombardment time. Spectrum (a) was taken after overnight discharge cleaning using H_2 gas, (b) was taken 25 min after (a), and spectrum (c) was taken 25 min after (b). Spectrum (d) was taken after the face was again exposed to overnight discharge cleaning in H_2 .

vacuum-coated with gold but not on the face covered with gold foil. No clear evidence of any contamination (other than hydrocarbon) was observed on the foil-covered face in over two weeks of operation. This implies that gold does provide a clean vacuum surface if it is applied in sufficiently thick layers.

5. ISX (IMPURITY STUDY EXPERIMENT) TOKAMAK

The ISX tokamak¹¹ is being built as a joint project between the Oak Ridge National Laboratory and General Atomic Company. This experiment will be dedicated to continuing and broadening the scope of the surface and plasma impurities studies presently initiated in ORMAK. Construction is under way, and experiments are scheduled to begin in the spring of 1977.

ISX is an iron-core tokamak of simple basic design; it is slightly larger than ORMAK. Toroidal field coils are rectangular and are joined so that the top of each coil can be removed. Ohmic heating and vertical field coils are wound on fiberglass cylinders encircling the central iron core. When the coil tops and the upper leg of the iron core are removed, the vacuum chamber is free to be removed vertically from the tokamak. This makes possible the easy interchange of plasma chambers. The estimated turnaround time is two weeks, working two shifts a day. Another consequence of simple design is large diagnostic access.

Basic machine parameters are listed in Table I. Plasma parameters are based on values routinely obtained in ORMAK. The vacuum chamber, shown in Fig. 7, is of all-metal 304L stainless steel construction. The pumping system is a combination of cryosorption and turbomolecular pumps;

Table 1. ISX parameters

Toroidal field	18 kG
Transformer flux swing	0.9 V-sec
Ultimate vacuum pressure	2×10^{-9} torr
Major radius	93 cm
Minor radius	26 cm
Plasma current ($q = 5$)	150 kA
Central electron temperature	800-1800 eV
Central ion temperature	300-500 eV
Shot repetition rate	1 per minute
Flat (equilibrium) current duration	250 msec

Fig. 7. ISX vacuum chamber and pumping system as viewed from above. Sorption pumps will be used for rough pumping. High vacuum will be maintained by a combination of turbomolecular pumps and cryosorption pumps.

no pumps using oil will be used. Assuming an outgassing rate of 10^{-11} torr-l/cm²-sec, the ultimate pressure should lie in the low 10^{-9} torr range. Discharge cleaning, titanium evaporation, and baking to 400°C will be used to achieve and maintain a low outgassing rate.

The ISX experimental program will be centered around surface-impurity studies and the transport and control of plasma impurities. Surface experiments will include detailed tests of various cleanup techniques, initially using the stainless steel vacuum chamber shown in Fig. 7. These studies will be later extended to other materials, such as low-Z walls and walls made from refractory materials. Initial plasma studies will involve a test of the impurity flow reversal scheme proposed by Ohkawa.¹² Later studies will focus on plasma impurity transport and impurity accumulation.

6. SUMMARY

The ORMAK vacuum liner surface has undergone a continual evolution. The gold surface was initially contaminated by hydrocarbons. After oxygen discharge cleaning, carbon no longer was a significant contaminant. Residual hydrocarbons can now be controlled either by hydrogen or oxygen discharge cleaning. Iron oxides now appear on the liner surface, and SXAPS results indicate the iron may have originated by diffusion through the ~ 1 μ m-thick gold surface coating. Oxygen is the main light ion contaminant of the plasma, and iron is the major identifiable heavy contaminant.

A new tokamak, ISX, is being constructed to study wall and impurity problems in more detail. ISX is slightly larger than ORMAK and features

an ultrahigh vacuum system for surface studies and ample diagnostic access for plasma impurity experiments. Operation is scheduled to begin in the spring of 1977.

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