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SURFACE IMPURITY STUDIES IN THE ISX-A TOKAMAK*

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ABSTRACT

The Impurity Study Experiment (ISX-A) was a tokamak designed to study impurity sources, plasma-wall interactions, impurity behavior within the plasma, and related phenomena. An Auger electron spectrometer, attached to the tokamak by means of a UHV sample transfer system, has been the primary tool used for surface studies of the first wall. Stainless steel samples positioned at the wall edge were used to follow the progressive "cleanup" of the torus wall. Cleaning consisted of low energy hydrogen discharge plasmas and later titanium gettering. The principal contaminants initially were carbon, oxygen, and sulfur. As cleaning proceeded, the surface oxide layers were reduced to sub-stoichiometric levels, and the carbon deposits appeared to be converted to metallic carbides. The sulfur was largely removed. A decrease in the effective nuclear charge of the plasma could be correlated with these surface changes. Discharge cleaning reduced Z_{eff} from ~ 4 to 1.6 over a period of several months while titanium gettering produced values approaching 1.0.

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INTRODUCTION

The impurity Study Experiment (ISX-A) at the Oak Ridge National Laboratory was a moderate size tokamak (major radius - 92 cm, minor radius -26 cm) designed for the study of the effects of impurities on tokamak plasmas.¹ Some specific items to be studied included sources of impurities, impurity behavior in the plasma, and the various interactions between the plasma and the first wall. Throughout the operational lifetime of ISX-A the changes in surface composition of metallic samples exposed to the machine conditions were followed by means of *in-situ* Auger electron spectroscopy (AES). Similar studies carried out at other tokamak installations have all made use of this or similar techniques.²⁻⁵ Carbon* and oxygen have been found in those studies to be the major impurities and the AES techniques are well-suited for their analysis.

EXPERIMENTAL APPARATUS

A description of the surface analysis station (SAS) is presented elsewhere^{6,7} and will only briefly be given here. A schematic diagram of the SAS is shown in Fig. 1. The analysis chamber, containing a cylindrical mirror electron spectrometer with a 5 μ diameter scanning electron beam for excitation of Auger electron communicates with ISX-A by means of a one meter long tube containing the sample transfer system. ISX-A is an iron core device in which the magnetic field decreases sharply with radial distance, thus permitting the close proximity of the spectrometer. The analysis chamber and the transfer tube are separately ion pumped.

Standard UHV techniques were used in the construction of the SAS. All major components are type 304-L stainless steel, and all metal sealed valves are used throughout. Following a mild bakeout, typical pressures are $1-2 \times 10^{-8}$ torr in the transfer tube and at least one order of magnitude lower in the analysis chamber. The end of the transfer tube away from the machine serves as a vacuum lock for the introduction and removal of samples. The various components of

*Recent studies on the T-12 tokamak at the Kurchatov Institute in Moscow as well as current experiments on Doublet III at the General Atomic Co. in La Jolla, CA, using AES techniques and a portable surface analysis station have also shown carbon to be a

the SAS are isolated by valves which are open only when the sample is being transferred. The vacuum lock and sample transfer system are described separately.⁷ In addition to the electron spectrometer, the analysis chamber contains a quadrupole mass spectrometer for gas analysis in thermal desorption studies, an ion gun and inert gas supply for sample preparation as well as for AES depth profiling, and equipment for the precise adjustment of sample position.

EXPERIMENTAL METHOD

ISX-A was constructed of type 304-L stainless steel, and it is this material that has been most extensively studied in the SAS. Stainless steels in the "as received" condition are covered by an oxide layer that is, typically, several tens of angstroms thick. In addition, a carbonaceous layer is present as well as smaller amounts of other impurities depending upon the previous treatment and environmental history of the metal. Upon exposure to the plasma within a fusion device, the stainless steel wall serves, at least initially, as an impurity source by virtue of various plasma wall interaction processes. Impurities from other sources within the device via the plasma or from other surfaces exposed to the plasma may be deposited on the wall. In any event, the wall composition may be expected to change with time, most rapidly at first and then more slowly as some equilibrium or steady state condition is approached.

Because of the obvious difficulty in monitoring both impurity ingress and egress using a single sample, a composite sample was fabricated with an aluminum as well as a stainless steel section. Each section consisted of a thin strip of the metal 0.3 cm wide and 1.3 cm long. A movable shield of magnesium, 0.07 cm thick, was used to cover one-half of each sample during inert gas sputter cleaning so as to maintain an "as-received" section on both metals. During exposure in

ISX-A, the shield was repositioned so that it covered one-half of the cleaned areas and one-half of the uncleared areas. Changes in the composition of the uncleared sides yields information on the clean-up rates while changes on the cleaned sides provide estimates of the recontamination rates. Comparison of the results from both parts of the sample then yield information on the equilibrium or steady state conditions expected for a given set of system parameters.

During the first four to six weeks of operation of the SAS few plasma diagnostics were operational, and the only tokamak operation consisted of nocturnal discharge cleaning. The progress of these operations was followed initially by means of a single stainless steel sample, analyzed before and after exposure to the discharge and, later, by use of the composite aluminum and stainless steel sample.

When tokamak operation commenced, additional studies were carried out on material transport in the machine by means of the composite sample as well as individual samples of other metals. During this phase of ISX-A operation samples were exposed to tokamak discharges at various positions between the vacuum wall and the limiter inner radius in an effort to obtain information on material transport as a function of radial distance.

The final surface physics experiment carried out just before ISX-A ceased operation consisted of rotating an aluminum cylinder behind a 0.1 cm slit within the limiter shadow during tokamak discharges. AES analysis of the periphery of the cylinder yielded a time resolved analysis of impurity deposition during the course of the tokamak discharges.

RESULTS AND DISCUSSION

With proper calibration AES techniques are capable of yielding quantitative analyses for most elements. The results reported here are given in atomic percent obtained using published techniques.⁷ For those cooperative studies relative values are satisfactory and calibrations for the various elements were not necessary.

The rate at which impurities are removed from the

tokamak discharges or by plasma discharge cleaning as well as the rates at which "clean" surfaces become contaminated are dependent on the plasma conditions, the machine history, the materials involved and other parameters. The interaction of these parameters is complex and as a result, "clean up" phenomena are not well understood. The results reported here were observations made during the course of plasma and confinement experiments that required a wide range of machine conditions. As a result, it was not always possible to correlate one set of data with another. However, some significant trends in impurity removal rates and deposition rates were observed. Some of the more significant results were reported elsewhere.⁶ We report here some of those results, but more importantly, we report the general behavior of certain specific impurities throughout the lifetime of ISX-A. Much has been learned that will be of value in the design of future experiments and in the operation and "clean up" of tokamaks in general.

Because of the differences in the behavior of the various materials in the tokamak environment each of the major impurities will be discussed separately. The minor impurities on those with a similar behavior pattern will be discussed collectively.

Sulfur

This material is a minor, although common, impurity present both on the surface and in the bulk of stainless steels. Samples of the material typically exhibit atomic concentrations in the range 0-2 percent. As previously reported,¹⁰ a large build-up of sulfur was found on the stainless steel sample exposed at the wall position during the initial discharge cleaning in ISX-A. This discharge cleaning, using hydrogen as the working gas, lasted only 20 min, during which the concentration of sulfur on the surface increased from 0.7 atomic percent to 11.5 atomic percent. Later studies showed a smaller, but still significant, build-up of sulfur on sample surfaces even under high vacuum (2×10^{-7} torr) static conditions. The impurity agent is apparently activated by the discharges and evidently exists in the machine in the form of a gaseous compound even after the discharge ends. Control samples showed that the observed increase was not due to the sulfur within the bulk of the sample since our sample temperature never exceeded 80°C, and, hence, the source must be within ISX-A itself.

Over a period of one month of periodic discharge cleaning using both hydrogen and helium, the surface sulfur concentration gradually fell to <2 atomic percent. These values remained low until the machine was vented to air on January 25-26, 1978. Following this, sulfur levels as high as 12.3 atomic percent were observed. As before, these levels were gradually reduced by routine discharge cleaning. A second vent occurred on February 21-22, 1978 that was not followed by abnormal sulfur levels. These observations suggest that the sulfur may have been inadvertently introduced into the machine during the up-to-air periods. One possible source of sulfur is the commercial antigalling compound extensively used to coat the flange bolts on ISX-A. This material contains molybdenum disulfide which may, if introduced into ISX-A, decompose upon exposure to ion bombardment. Thus, the presence of sulfur in ISX-A could be explained via a misplaced set of fingerprints, contaminated gaskets, a dropped nut or bolt, or by one of any number of incidents.

While sulfur has been observed on surfaces of at least one other tokamak³ the amounts involved were very small. Other surface studies^{2,4} have not reported the presence of sulfur.*

Carbon

Carbon is an impurity seemingly common to all tokamaks. It is the dominant surface impurity on the stainless steels and is omnipresent in the form of hydrocarbons. Typical surface levels on "as-received" samples of stainless steel may range as high as 60-70 atomic percent.

Discharge cleaning in hydrogen in ISX-A rapidly reduced surface carbon levels to 15-20 atomic percent. Concurrent with this partial removal of carbon was the apparent conversion of the remaining carbon to metallic carbides as evidenced by the shape of the Auger peaks.¹² These materials are not further removed even by extensive discharge cleaning. No appreciable changes in carbon removal rates were observed during discharge cleaning when using either deuterium or

*Sulfur has also been observed on samples exposed at the wall position during recent experiments on discharge cleaning at both the T-12 and Doublet III tokamaks.

helium as the working gas. Discharge cleaning in oxygen is known to be very effective in removing carbon deposits,¹³ but oxygen was not used in ISX-A because of its high atomic number.

Following the initiation of titanium gettering, in connection with the impurity flow reversal experiment,¹⁴ in mid-January 1978, the carbon levels were reduced to <10 atomic percent. During this time the vacuum conditions in the machine also improved by an order of magnitude with the base pressure approaching 1×10^{-8} torr. Even following the January venting, the carbon levels quickly fell to the previous <10 atomic percent level. These levels remained low during the time extending to the machine vent in February. It was also during this time that low values of Z_{eff} were observed,¹⁴ generally, between 1 and 2.

During the February vent retractable carbon limiters (ATJ-S graphite) were installed in one diagnostic section of ISX-A. Following the next H₂ discharge cleaning with the carbon limiters in the retracted position, carbon levels on the stainless steel samples were found to be 25-30 atomic percent. Concomitant with this increase other impurity levels were also elevated and Z_{eff} rose to values approaching six.¹⁴ Surface carbon levels gradually fell during the final two weeks of ISX-A operation but generally remained above 15 atomic percent. No appreciable difference was observed during operation with the carbon limiters extended. It appears that carbon in the form of carbides does not prevent the attainment of low Z_{eff} values.

Nitrogen

Nitrogen, although generally present on the surface of stainless steel in the range from 1-2 atomic percent, was found always to exhibit levels much below that of carbon. Initially, nitrogen levels were also below that of oxygen, but after routine tokamak operation, interspersed with discharge cleaning, surface concentrations of these materials became comparable. Discharge cleaning was ineffective in removing nitrogen from stainless steel; in fact, frequent increases in nitrogen levels to 3-5 atomic percent were observed during those times when the carbon levels were low.

Following the initiation of titanium gettering it was no longer possible to monitor the surface nitrogen level routinely because of interference between the titanium and nitrogen Auger peaks at 383 eV and 381 eV respectively.

When the sample surfaces were sputter cleaned to a point where the 418 eV titanium peak disappeared, nitrogen concentrations were usually in the range of 3-5 atomic percent before installation of the carbon limiters and 5-10 atomic percent after this.

Oxygen

Oxygen exists on typical stainless steel surfaces in the form of metallic oxides. Typical concentrations as determined by AES of "as-received" surfaces may range from 10 to 20 atomic percent. Hydrogen discharge cleaning has been particularly effective in reducing this oxide layer.

In ISX-A several weeks of discharge cleaning reduced these levels to <10 atomic percent on "as-received" samples and to <5 atomic percent on samples that had been lightly sputter cleaned. Deuterium discharge cleaning was more effective than hydrogen in removing oxygen from both stainless steel and aluminum surfaces. A single 36 hr exposure reduced the oxygen concentration on type 304-L stainless steel to 3.8 atomic percent and, on series 1100 aluminum, to ~0.5 atomic percent.

Unlike other impurities, oxygen builds up on "clean" surfaces even under good vacuum conditions. Surface concentrations were found to increase from 4.6 to 16.5 atomic percent during a one week exposure to the static ISX-A environment. However, this layer was quite thin and was easily removed by subsequent discharge cleaning.

Following titanium gettering, oxygen levels were found to be quite low throughout the topmost 10-15 atom layers of the deposit. The surface levels, following gettering, ranged from 10-15 atomic percent. Sputter cleaning for 30-40 sec (corresponding to approximately one atom layer) generally reduces the oxygen levels to <1 atomic percent.

Other Contaminants

At various intervals throughout the ISX-A operation a number of other impurities were observed. The concentrations were generally only a fraction of the carbon levels and were typically in the range of several atomic percent. The most predominant of these were phosphorus, chlorine, and calcium, but occasionally small amounts of sodium, silicon, and copper were observed. The values were always small, and their appearance did not seem to follow any pattern.

Following the installation of the carbon limiters, potassium was observed on both the stainless and aluminum surfaces. Peak concentrations near 10 atomic percent were observed on the aluminum samples.

CONCLUSIONS

Information on impurity behavior in the ISX-A tokamak was obtained under various operating conditions by surface analysis of stainless steel and other samples before and after exposure to tokamak discharges and low-power discharge cleaning in various gases.

Transfer of samples between ISX-A and the analysis chamber were made under clean ultra-high vacuum conditions. As expected, carbon, nitrogen, and oxygen were initially found on all samples exposed parallel to the tokamak wall. Significant amounts of sulfur were also observed following discharge cleaning in both hydrogen and helium. Over a period of 4-6 weeks, the oxygen levels were reduced by factors of 2-4 while sulfur was essentially eliminated. Discharge cleaning was found to be ineffective in removing nitrogen, but carbon levels were significantly reduced. The carbon remaining appeared to be in the form of metallic carbides that were not appreciably removed by discharge cleaning. Impurity removal rates were significantly increased during the final weeks of ISX-A operation when titanium getter pumping was introduced. Replacement of the normally used stainless steel limiters by carbon limiters near the end of ISX-A was accompanied by a 2-fold increase in the surface carbon levels. Significant amounts of potassium also appeared for the first time. This change also resulted in an abrupt increase in Z_{eff} from values near 1, to ~6; however, near the end of ISX-A operation values of Z_{eff} and the surface carbons levels were 50% lower.

The use of *in-situ* surface analyses in fusion devices has provided useful information on the efficiency of various cleanup techniques in ISX-A. The use of similar surface analytical techniques should provide additional diagnostic capabilities for present and future generation devices.

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FIGURE CAPTIONS

1. Schematic diagram of the surface analysis station on ISX-A.