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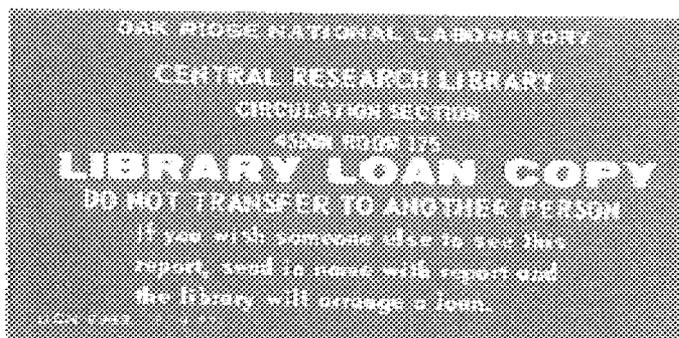


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ORNL/TM-10310

Hydrogen Recycle and Isotope Exchange from Dense Carbon Films

R. E. Clausing
L. Heatherly



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Printed in the United States of America. Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road, Springfield, Virginia 22161
NTIS price codes--Printed Copy: A03; Microfiche A01

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UC-20, -20c

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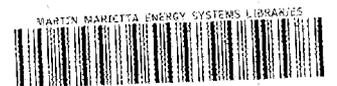
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Date Published: March 1987

Prepared for
Office of Fusion Energy

Prepared by the
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Oak Ridge, Tennessee 37831
operated by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
U.S. DEPARTMENT OF ENERGY
under Contract DE-AC05-84OR21400



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CONTENTS

ABSTRACT	1
INTRODUCTION	1
EXPERIMENTAL PROCEDURE	2
RESULTS OF RECYCLE MEASUREMENTS	3
GENERAL OBSERVATIONS	3
EFFECT OF TIME BETWEEN PULSES	5
EFFECTS OF HYDROGEN CONTENT IN THE FILM	5
RESULTS OF ISOTOPE CHANGEOVER MEASUREMENTS	8
DISCUSSION OF RESULTS	9
THE LARGE INITIAL RECYCLE COEFFICIENT	9
RELATION TO EXPERIMENTAL FUSION DEVICE	10
TRITIUM MANAGEMENT	11
SUMMARY AND CONCLUSIONS	11
REFERENCES	12

HYDROGEN RECYCLE AND ISOTOPE EXCHANGE FROM DENSE CARBON FILMS*

R. E. Clausing and L. Heatherly

ABSTRACT

Dense carbon films were prepared by deposition from hydrogen plasmas to which methane was added. The initial hydrogen recycle coefficient from the films ranges from more than two to less than one. The films contain large amounts of hydrogen (up to 50 at. %). They adjust themselves to provide recycling coefficients near unity. Isotope changeover times tend to be long. The reservoir of hydrogen instantly available to the plasma to maintain or stabilize the recycle coefficient and isotopic composition of the plasma is 10^{15} cm⁻² or greater depending on film preparation, temperature, and prior plasma exposure conditions. Simulator observations tend to support and improve the understanding of the observations in TEXTOR and JET; however, they also point out the need for control of film deposition and operating parameters to provide desirable and reproducible properties. The films and the hydrogen isotopes they contain can be removed easily by plasma processes. Since the hydrogen in these films is relatively immobile except in the zone reached by energetic particles, or at temperatures above 400°C, dense carbon films may be useful in managing the tritium recovery from near-term fusion experiments.

INTRODUCTION

Carbon films have useful properties as wall materials for fusion devices.¹ They are low-Z materials and can be easily deposited and removed. These films can be used to cover metallic substrates, thereby reducing plasma contamination with higher-Z materials. The processes occurring during application and removal of films also remove other impurities such as oxygen, chlorine, nitrogen, and sulfur.^{2,3} If graphite is used for high-heat-flux components, carbon-covered walls complement these components and protect them from contamination.

*Research sponsored by the Office of Fusion Energy, U.S. Department of Energy, under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc. This report was adapted from a paper presented at the Seventh International Conference on Plasma-Surface Interactions in Controlled Fusion Devices, May 5-9, 1986, Princeton, N.J.

Carbon films have been tested in DIVA, JET, TEXTOR, ASDEX, JIPPT II-U, and other devices recently.⁴⁻¹⁰ Many of the results (especially the reduction of oxygen and metal plasma impurities) are favorable; however, much remains to be learned about sputtering, chemical erosion, and especially the hydrogen recycle from such films. We are using a plasma materials interaction test facility (Recycle, Impurities, and Conditioning Simulator, known as RICS) to produce and study carbon films formed in situ by the action of hydrogen plasmas to which methane has been added. We report here some observations concerning the hydrogen recycle characteristics of these films.

EXPERIMENTAL PROCEDURE

The principles of the RICS apparatus and the measurement techniques have been described previously.^{11,12} The method basically depends on measuring the composition and pressure changes in a plasma chamber during and after plasma discharges. If the flux and species of energetic hydrogen going into the walls are known, recycle coefficients can be calculated and much can be learned about hydrogen recycling.¹² The plasma chamber is 14.5 cm in diameter and 60 cm long. Its temperature can be controlled from room temperature to 400°C at present. Data acquisition and certain system operations are under computer control.

Dense carbon films are formed on the inside walls of the chamber by deposition from a hydrogen glow discharge plasma to which methane has been added. During film deposition the pumping speed is normally set to 3 L/s, a manually controlled methane leak valve is adjusted to raise the pressure to 0.8 Pa, and then a servo valve is set to maintain the total pressure at 2.0 Pa by adding hydrogen. The chamber wall is the cathode of a 1-A, 600-V discharge. As the film forms, the voltage usually drops from 600 V to about 260 V in hydrogen or 350 V in deuterium. At 250°C, a 150-Å film is deposited in about 30 min. This film and these deposition conditions have served as standard reference conditions for this study. The films are removed after each experiment by a hydrogen glow discharge plasma. This leaves a reproducible, clean, carbide-rich surface on the stainless steel as a starting point for the next film deposition.

Recycle measurements are made by observing pressure changes, optical spectra, and gas composition changes accompanying plasma pulses. Ion fluxes, ion energy, pulse duration, pulse intervals, wall temperature, and hydrogen or deuterium feed gas can be chosen as desired. The standard pulse for these studies was 1 s long, 2×10^{15} of H_2^+ ions $cm^{-2}s^{-1}$ at 100 eV per ion with 5 min between pulses, beginning 15 min after film deposition. The pumping speed for hydrogen is set at 3 L/s, and the usual filling pressure is 2.0 Pa. The calculated recycle coefficients are corrected for vacuum system pumping and gas input during the plasma pulse.

The recycle coefficient, R , is defined as the hydrogen flux out of the wall divided by the hydrogen flux into the wall, where the flux out of the wall is obtained from a hydrogen mass balance and the flux into the wall from the measured molecular ion abundances and current to the wall.

Both optical spectroscopy and mass spectroscopy were used to monitor the changing isotope ratio in the plasma during 5-s plasma pulses. The standard procedure is to make a film using one isotope and then conduct a series of standard recycle plasma pulses as described above, except that the plasma pulses are longer. A few pulses are run with the hydrogen isotope used during film formation, and then for the changeover measurement the other hydrogen isotope is used for the fill gas. Fill gas is continuously admitted as during the standard recycle measurements. Optical spectroscopy using $H\gamma$ and $D\gamma$ lines gives excellent time resolution, while mass spectroscopy gives better isotopic ratios and continues to monitor the isotope ratio after the plasma pulse.

RESULTS OF RECYCLE MEASUREMENTS

GENERAL OBSERVATIONS

The recycle measurements reported here were made on films deposited and tested at 250 and 330°C. Figure 1 shows a comparison between the pressure changes accompanying a standard 1-s recycle plasma pulse on (a) a "clean" stainless steel surface at 250°C and (b) a carbon film deposited and tested at 250°C. Calculated recycle coefficients are displayed in Fig. 1(c). The contrast is large and important. The carbon film has an

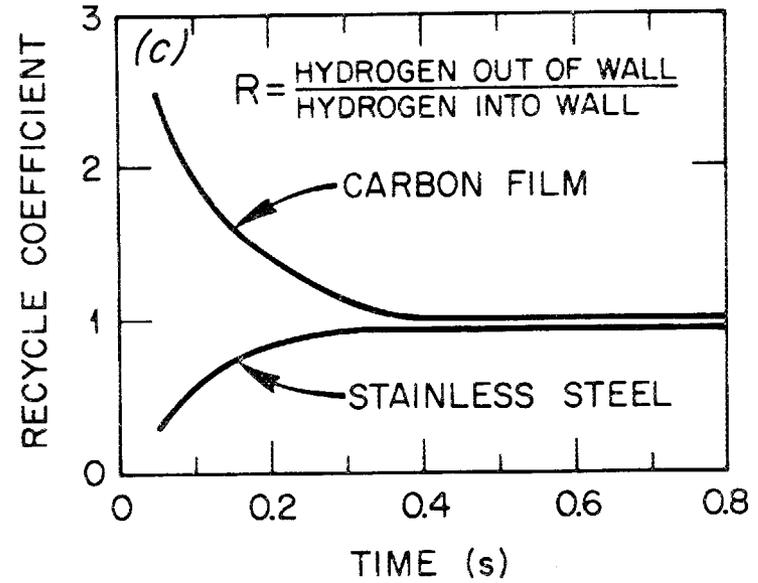
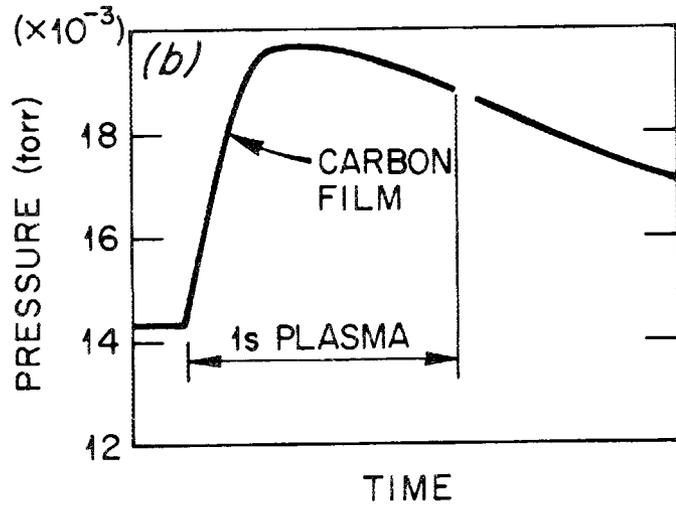
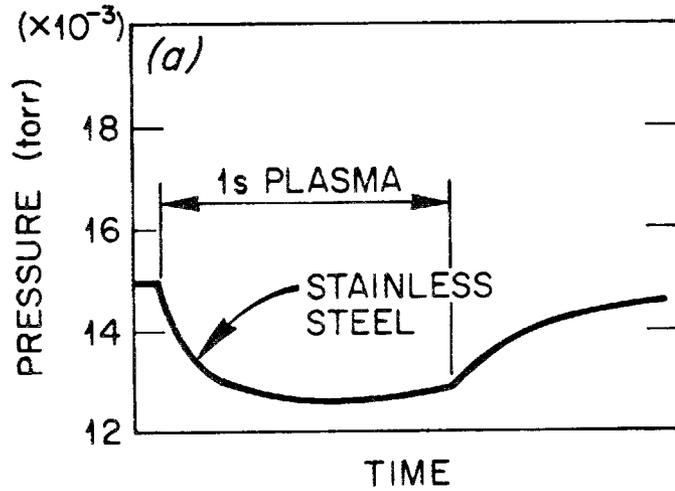


Fig. 1. A comparison between the pressure changes resulting from a standard plasma pulse on (a) a clean stainless steel surface at 250°C and (b) a carbon film at 250°C. (c) Calculated recycle coefficients for the data of (a) and (b).

initial recycle coefficient >2 , and it causes a large increase in the hydrogen gas pressure when the pulse begins. On the other hand, the metallic surface absorbs hydrogen and causes the pressure to decrease. Similar changes will occur to the plasma density in fusion devices and could make density control difficult. A decrease in density can be compensated by gas puffing, but an increase might have to be removed by a pump limiter or divertor. Both surfaces soon adjust their recycle coefficients to near unity. R for the metallic surface remains slightly <1 due to the diffusion of hydrogen into the bulk. The diffusion of hydrogen into or out of the carbon film is slow at this temperature, so its recycle coefficient is quickly forced to unity.

EFFECT OF TIME BETWEEN PULSES

While the data of Fig. 1 is characteristic of dense carbon films deposited and characterized at 250°C , the exact behavior can be strongly influenced by the time interval between pulses. Figure 2 shows this effect. A series of 1-s pulses were run with increasing time intervals between pulses. The initial pressure increase gets progressively larger as the interval becomes longer, up to 16 min. Figure 3 shows that for times longer than this the pressure increase becomes less. Apparently an easily desorbed phase, we will call it "D" phase, is formed fairly rapidly and is subsequently depleted by another process.

EFFECTS OF HYDROGEN CONTENT IN THE FILM

Films formed and measured at 330°C show similar behavior but also additional effects, which may be due to depletion or an excess of hydrogen in the bulk of the film (relative to a steady-state amount). Figure 4 shows the effect of a series of pulses made on a film loaded with hydrogen as the result of a 15-min plasma exposure. Figure 5 shows a similar series made after about 5 h of exposure to vacuum at 330°C . The vacuum exposure apparently has slightly depleted the bulk of hydrogen so that the recycle coefficient goes to less than one as the bulk is replenished, but this process is more or less separate from the formation and depletion of the easily desorbed "D" phase. Figure 6 shows the recycle coefficients

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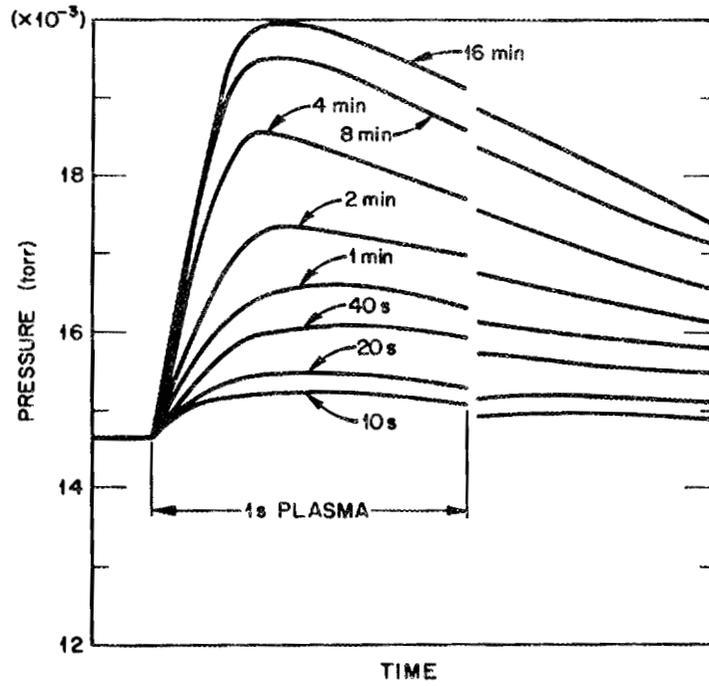


Fig. 2. Increasing the interval between plasma pulses up to 16 min (at 250°C) increases the pressure excursion.

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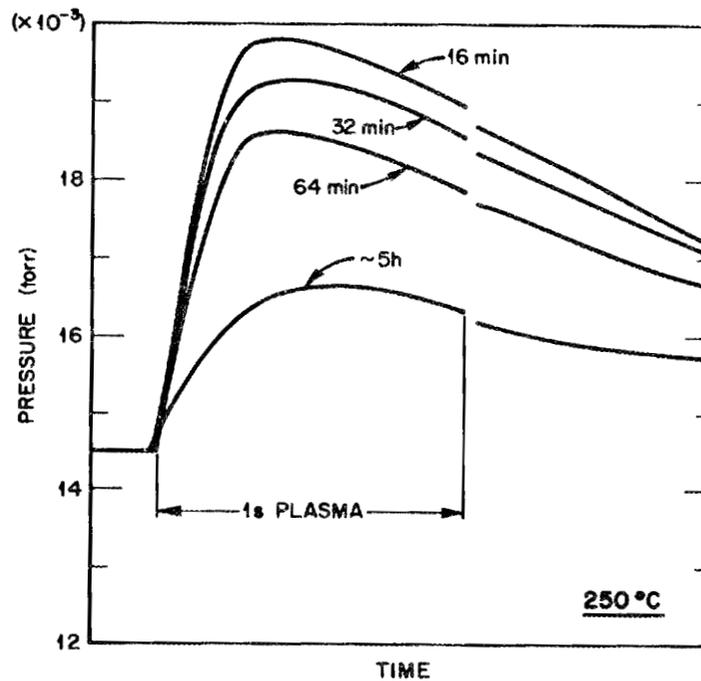


Fig. 3. Longer pulse intervals result in smaller pressure increases by decreasing the amount of desorbable gas at the carbon film surface.

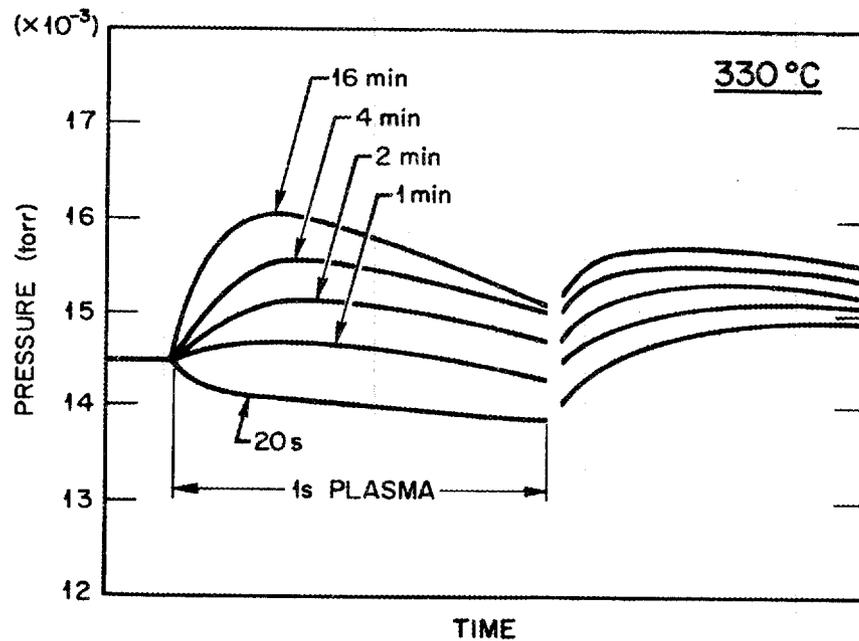


Fig. 4. The effect of changing the time interval between pulses (at 330°C) with a film that was recharged with hydrogen for 15 min before the series began.

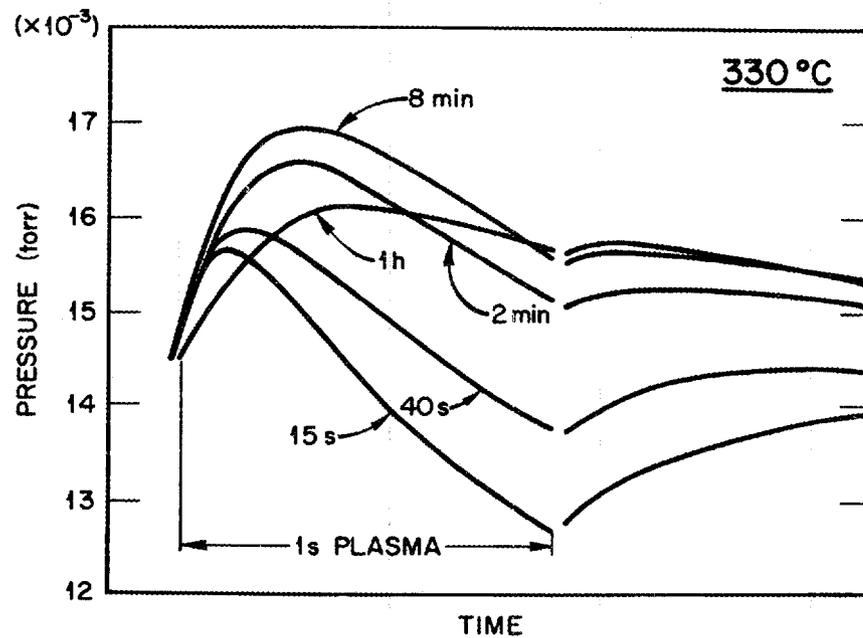


Fig. 5. The effect of changing the interval between pulses (at 330°C) with a film that was degassed for 5 h before this series began.

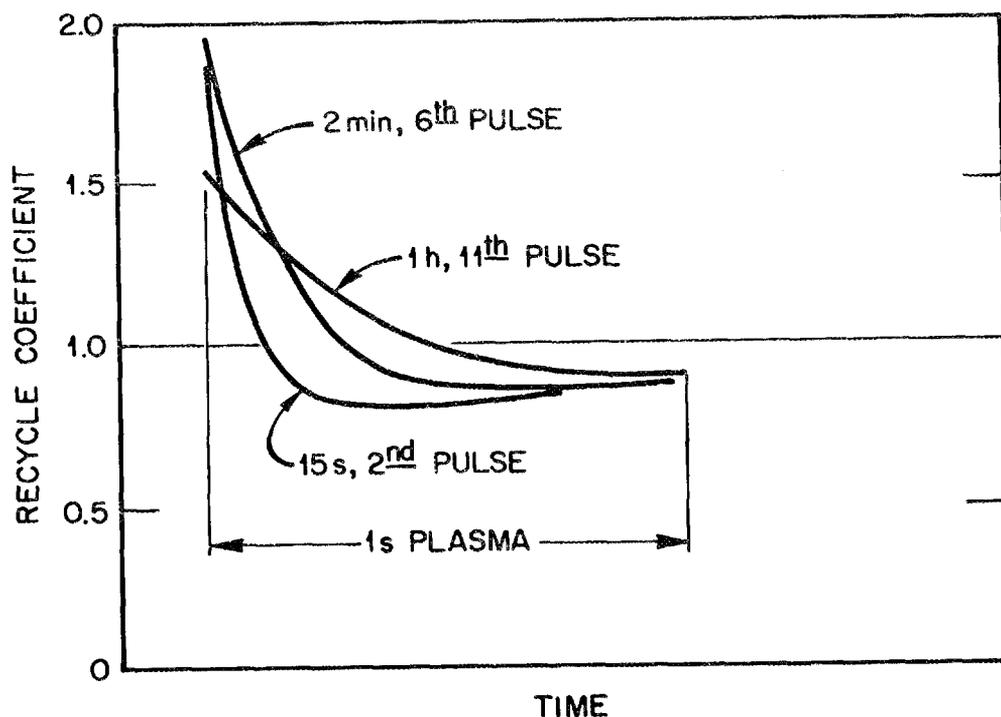


Fig. 6. Recycle coefficients for several of the pulses of Fig. 5.

for several of the pulses of Fig. 5. The recycle coefficients from this last series at 330°C range from >2 to about 0.8. All of the pulses from degassed films ended with $R < 1$.

Note that when the hydrogen content of the film is high, as it is in Fig. 4, a thermally activated evolution of gas occurs after the plasma pulse is finished. Some thermal evolution also is apparent in Fig. 5. The recycling of hydrogen back to the plasma from the film at 330°C apparently is via both thermal and ion-induced processes since the thermal process continues briefly after the ion pulse. The 250°C recycling is mostly the result of ion-induced processes.

RESULTS OF ISOTOPE CHANGEOVER MEASUREMENTS

One isotope changeover experiment will be briefly reported here to indicate the general behavior of our reference films. Experiments on this and other films will be reported in detail elsewhere.

A film prepared at 250°C with hydrogen was subjected to a series of 5-s plasma pulses in a deuterium atmosphere at 250°C. The first pulse (gas filled with 100% deuterium) was 60% hydrogen after about 2 s and 62% hydrogen at the end. The second and third deuterium plasma pulses ended at 44% and 30% hydrogen, respectively. After ten pulses the level was about 10% hydrogen and falling slowly.

The amount of hydrogen desorbed and still remaining in the plasma at the end of the first deuterium plasma pulse is equivalent to $2.4 \times 10^{15} \text{ cm}^{-2}$. This indicates the size of the reservoir of hydrogen instantly available to interact with the plasma. The results will be different if other film preparations, temperatures, pulse durations, intervals, or flux intensities with higher or lower ion energies are used, but those are subjects of a continuing investigation.

DISCUSSION OF RESULTS

THE LARGE INITIAL RECYCLE COEFFICIENT

Perhaps the most interesting result is the effect of the pulse interval on the initial recycling coefficient. At both 250 and 330°C an easily desorbable surface "D" phase grows in a matter of minutes to the point of being able to supply 10^{15} or more hydrogen atoms to the plasma per square centimeter of wall area in less than a second at initial recycle coefficients of two or more. This "D" phase is subsequently slowly desorbed, removed, or changed and made less available to the plasma. In an attempt to determine if the presence of hydrogen gas or atomic hydrogen from a hot tungsten filament in our system might assist in the growth of the "D" phase, we saw little if any change in the formation rate when the system was evacuated to about 10^{-4} Pa and the filament turned off. Either the phase grows from internal resources or the rate determining step is not influenced by changing the hydrogen pressure in the range indicated.

We suppose that the "D" phase may be either near-surface hydrogen replenished from the bulk or a CH_x or CH_3 radical. Vietzke, Flaskamp, and Phillips showed that such radicals were formed on graphite surfaces bombarded with atomic hydrogen and/or hydrogen ions.¹³ As they suggest, the

CH_3 could form from CH_x radicals formed during the previous irradiation. The decrease in the amount of the "D" phase with time could be due to desorption of near-surface hydrogen but also appears to be consistent with their suggestion that the CH_3 can be desorbed as CH_4 by combining with a hydrogen atom or that a CH_x radical combining with hydrogen may have enough excess energy to allow it to desorb. It probably would pick up additional hydrogen from subsequent surface interactions to become CH_4 . Desorbed CH_4 would be difficult to detect during the plasma pulse. We do see thermally desorbed hydrogen and CH_4 after plasma pulses but haven't yet determined if "D" phase is CH_3 or simply hydrogen.

RELATION TO EXPERIMENTAL FUSION DEVICES

Both TEXTOR and JET have used carbon films extensively, TEXTOR at about 250°C and JET at 250 to 300°C . At both facilities, little if any gas puffing was needed to keep the plasma density up once the plasma discharge was begun. JET has had some difficulty attaining low density plasmas after running high-density plasmas. Investigators at both facilities have indicated that a long time was required to change isotopes unless glow discharge conditioning was used to remove the film or replace the unwanted isotope. All of these results are consistent with the observations in this study of recycle coefficients initially greater than one going toward one as the shot progresses and with the large hydrogen capacity of the films.

The JET staff has also reported that the carbon films tend to soak up excess hydrogen as the plasma density is ramped down at the end of a shot. This implies a recycle coefficient less than one. We show that films which are depleted in hydrogen, Fig. 5, can exhibit recycle coefficients less than one after the "D" phase is depleted. Thus, if a film were heated high enough or long enough to desorb some of its hydrogen, it could subsequently soak up hydrogen. This could happen during one shot or during the time between shots, especially if the plasma dumps the particles at the end of the shot on an area that has not been heavily bombarded during the main part of the shot. In any event, the surfaces inside any complex machine will not be uniform or uniformly treated, so areas near the limiters or in divertor regions will have behavior different from others. One

area may evolve hydrogen while others absorb it. The hydrogen capacity of these films is so large that large quantities of gas relative to that in the plasma can be involved.

TRITIUM MANAGEMENT

The use of carbon films in plasma experiments involving tritium may produce problems because of large hydrogen (tritium) inventories. However, at low temperatures, all hydrogen isotopes in dense carbon films are relatively immobile, and tritium probably will not penetrate much beyond its implantation depth (which is small for low-energy particles from the plasma edge). Perhaps a high recycling coefficient from a thin surface layer on a relatively impervious underlying layer could be engineered to advantage. In any event, the films and their hydrogen isotopes are easily and completely removed by in-situ plasma processes.

SUMMARY AND CONCLUSIONS

In summary, we find that the hydrogen recycling behavior of these films is complex and can be caused to vary greatly, but it is predictable and reproducible if adequate control of the many variables affecting recycling is exercised. Some important conclusions follow.

1. The recycling of hydrogen from dense carbon films is not describable by a recombination coefficient. Recycling at 250°C (and below) is activated almost entirely by ion bombardment, but some thermally activated desorption becomes apparent at 330°C for the conditions of these experiments. The film formation conditions, the thermal and ion bombardment history, and the temperature are strong influences.

2. The interval between plasma pulses can substantially alter the initial recycle behavior of the following pulse. It appears that an easily desorbed phase is replenished or formed and subsequently depleted over a period from seconds to hours.

3. Some desorption of hydrogen and methane occurs after a plasma pulse, but several hours are required at 330°C to affect the bulk hydrogen content significantly. The surface composition that influences recycle is changed in minutes, however.

4. The amount of hydrogen from the film that can interact and come into equilibrium with the plasma is several times 10^{15} cm⁻².

5. Recycle coefficients less than one can be obtained by depleting the hydrogen in the film.

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