AN EXPERIMENTAL INVESTIGATION OF CHEMICAL SPUTTERING OF CARBON IN A TOKAMAK DISCHARGE

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Abstract

An experiment has been undertaken to see if chemical sputtering of carbon can be directly observed under tokamak operating conditions. The test material consists of a well instrumented probe limiter which can be heated up to 900°C under steady state conditions and to > 1200°C with additional plasma heating during a discharge. The carbon released from the limiter is monitored spectroscopically using a visible spectrometer and a CCD camera with interference filters. Measurements have been made from 20°C to 700°C in steady state conditions and up to 1200°C with plasma heating. Within experimental error there is no change in the carbon production rate over the whole temperature range. Compared with beam experiments the inferred chemical sputtering yields are low. A semi-empirical model predicts such low yields at high current densities of incident ions.

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1. Introduction

It has been shown in beam experiments using hydrogen ion bombardment that enhanced erosion of carbon occurs at $\sim 500\,^{\circ}\text{C}$ due to the formation of methane. These experiments have recently been reviewed by Roth (1). Chemical sputtering has thus been frequently predicted to occur in tokamaks, but until now it has not been the subject of a definitive experiment. There are some sound reasons for believing that the chemical sputtering of carbon limiters in a tokamak may be considerably lower than in beam experiments. Firstly, the current density at a limiter is some orders of magnitude higher than in beam experiments, and the yield of methane per incident ion is known to decrease with increasing current density. The production of methane competes with recombination of hydrogen atoms to form H2 molecules and the recombination rate increases with surface concentration. Secondly, the presence of metals on the surface inhibits methane formation even when the impurity concentration is low (2). and significant metal impurities are found on the surface of carbon limiters in tokamaks (3). Thus, the sputtering yield will be reduced by two independent factors in the case of actual graphite limiters.

In order to make a direct test of the rate of chemical sputtering, we have designed an experiment where the carbon production rate could be directly measured in a plasma over a wide range of limiter surface temperatures in the DITE tokamak.

Experiment

The DITE tokamak has a major radius of 1.17 m and a minor radius of 0.26 m. The experiments were carried out in deuterium with a plasma current of 150 kA, a line average density $\overline{n}_e \sim 3 \times 10^{19}$ m⁻³, a pulse length of 0.5 s and a safety factor $q \simeq 4$. Initially only ohmically heated discharges were used but later neutral beam heating at 1.1 MW was added. The fixed limiter is a single complete poloidal carbon ring. Measurements were made using a cylindrical ATJ graphite probe limiter 72 mm in diameter which was inserted into the plasma from the top of the machine, 25° off the vertical and 70° away toroidally from the fixed limiter, fig. 1. The probe is introduced via a vacuum lock and can be moved between discharges from a

minor radius of 0.17 m out to the wall at about 0.30 m. The details of the probe limiter are shown in fig. 2. An internal heater can be used to heat the probe from room temperature up to 900°C. For a few seconds during the tokamak discharge the heater is turned off to prevent damage, the heat capacity of the probe limiter being sufficient to maintain its temperature.

The limiter is instrumented with two chromel-alumel thermocouples and four single Langmuir probes. The thermocouples monitor the bulk steady state temperature. The Langmuir probes are made of carbon rods which are flush with the surface of the probe limiter and insulated using alumina sleeves. Two probes face the ion drift direction spaced 12 mm apart radially and another two are correspondingly arranged on the electron drift facing side. The probes are biased with respect to the probe limiter body and scanned from - 100 V to + 10 V at a repetition rate of 5 ms/scan. The probe limiter body is electrically isolated from the torus. Thus, the incident plasma ion current, electron temperature and floating potential (V_f) are directly measured as a function of time throughout the tokamak discharge.

The surface temperature is measured using an AGA infrared camera with a spectral range 2-5 μm and a framing rate of 50 Hz. An in situ calibration of the camera is carried out using the internal probe limiter heater to raise the temperature to 900°C. All the infrared intensity data is digitized and computer analysed between discharges to obtain surface temperature as a function of spatial position $^{(4)}$. From an analysis of the time dependence of the surface temperature the deposited power density contours are derived.

The carbon production rate is monitored by direct viewing of the limiter with a SPEX spectrometer looking vertically upwards through a window, fig. 1. In the mode used in this study the SPEX scanned a 7 nm wavelength band with a repetition rate of 50 Hz. The band viewed can be selected from one discharge to the next. Typically three wavelength bands have been examined at each surface temperature covering the emission of CII, D $_{\alpha}$, D $_{\gamma}$, OII, OIII, FeI, CrI and a CD hydrocarbon band. Typical examples of the spectra observed in the three wavelength bands are shown in fig. 3. Table 1 lists the lines that have been identified. The bands, centred at 658, 432.5, 375 nm, were chosen because they contained most of

the impurity lines of interest and because they allowed direct comparisons between deuterium, carbon, oxygen and metal intensity ratios in a single discharge. In addition to the direct observation of the probe limiter a spatially scanning monospec spectrometer was used to survey the radial distribution of the main low Z impurity lines including CIII and CV. This spectrometer viewed the plasma from the top of the torus at a position displaced 180° toroidally from the probe limiter. Information on the spatial extent of the various lines measured with the SPEX is obtained with a TV camera based on a charge coupled detector (CCD) array and suitable interference filters (see fig. 1). Routine diagnostic measurements included, MHD signals, the line average electron density, and a bolometer that gave the total radiated power. The electron temperature profile was measured using an electron cyclotron emission Michelson interferometer.

Results

The time dependence of the main parameters of a typical ohmic tokamak discharge used is shown in fig. 4a. The plasma current flat top was 150 kA with an average density 3 x 10^{19} m⁻³ over the time from 200 to 450 ms. The central temperature $T_{\rm e}(0)$ was 650-750 eV. The plasma position is controlled both horizontally and vertically by active feedback controlled amplifiers and is maintained constant within \pm 5 mm from 100 to 450 ms.

Experiments were carried out with the probe limiter at two different radial positions, 0.255 and 0.230 m. The local plasma conditions at 0.255 m, as measured by the probe limiter as a function of time, are shown in fig. 5a and b. At this radius the electron temperature is in the range 15-20 eV with an ion saturation current density (j_s) from 5-12 mA/mm². The undisturbed "far plasma density" (n_e) on the field line that connects with the Langmuir probe is simply related to j_s provided there is no temperature gradient along the field lines, by the equation:

$$j_s = \frac{1}{2} en_e c_s$$

where c_s is the ion sound speed and we assume that $T_i = T_e$. Note the asymmetry in the signals between the ion and electron drift sides (probes 1 and 3, respectively); the ion saturation current density is roughly twice as large on the electron drift side. This asymmetry is also seen with the

CCD camera using a D filter. When in steady state the ion current flowing to the probe limiter will be equal to the flux of atoms leaving it, and therefore it is expected that j will be proportional to the local D $_{\alpha}$ emission. The SPEX spectrometer was focused so that the light collection area (* 200 mm²) was centred on the flat bottom of the probe limiter, roughly in the central region of the D $_{\alpha}$ emission observed with the CCD camera.

The integrated line intensities have been plotted in fig. 6 as a function of time. The hydrocarbon band due to the diatomic molecule CD has also been included but in this case the signal shown is proportional to the peak height near the CD bandhead ($\lambda \simeq 431$ nm) as noted in fig. 3. The ion saturation current densities as measured by the two probes LP1 and LP3 are shown for comparison. One can see that D follows j, as do all the lines except CrI and FeI, which decrease as the discharge progresses.

The bulk temperature of the probe limiter was raised progressively with the heater between successive discharges from 25°C to 700°C. The power flux at 0.255 m ($\sim 100 \text{ W/cm}^2$) was small enough to limit the surface temperature rise during one shot to 50°C, a fact confirmed by the AGA infrared camera. Thus, during one discharge, the surface temperature was approximately constant at the pre-discharge value. The Langmuir probe data showed no dependence on temperature. The results of the CII, OII, OIII, D, FeI, CrI and CD band, during the steady state condition of the discharge (210 ms), are plotted as a function of the initial surface temperature in fig. 7. It is seen that there is no change in the principal impurity fluxes CII and OII within the experimental error over the whole temperature range. However, there is a decrease in the metallic fluxes with increasing temperature. The other noticeable change with temperature is the increase in the hydrocarbon CD band, although the intensity distribution within the band remains constant. This might be related to methane production at the probe limiter surface. However, the increase over the whole temperature range is only a factor of 1.7.

A second series of experiments was carried out with the probe limiter moved in to a radius of 0.230 m where there was a significant power flux due to the plasma and the temperature of the probe increased during the discharge. The local plasma conditions were found to be the same for both

the ion and electron drift sides of the probe limiter at this radius. The time behaviour of Langmuir probe 1 is shown in fig. 8. The electron temperature is in the range of 30-40 eV while the current density is about 35 mA/mm 2 . The time behaviour of the integrated line intensities have been plotted in fig. 9. The initial temperature had been raised to 600°C before the discharge using the heater. The incident power flux is sufficient to significantly heat the carbon surface during the discharge. The resulting temperature rise is greatest near the tip of the probe limiter and decreases with increasing minor radius along the probe limiter body. The incident power flux on the leading edge is of the order 12 MWm⁻² which leads to a surface temperature rise during the discharge of typically 500°C . As with the 0.255 m case most of the spectral intensities show a time dependence which is similar to that of the current density. The one exception is the FeI line which rises steeply near the end of the discharge. This is probably due to thermal sublimation of Fe caused by the high temperatures (> 1000°C) reached at this time. One may note that the OII line at 3749.49 A° also rises steeply at the end of the discharge but this may be due to the existence of a coincident FeI line. The initial temperature was varied in steps so as to vary the maximum temperature reached during a series of discharges. Again, apart from the FeI line, no relative change in the time dependence of the signals was observed as the initial temperature was varied between 25°C and 600°C. The CrI line was not monitored at this radial position. One should note that for the 600°C initial temperature a significant area of the limiter exceeded 1100°C, and at this temperature one might expect significant radiation-enhanced sublimation of carbon (1). However no change in the CII line was detected. The FeI signals, on the other hand, did vary considerably depending on the initial temperature of the limiter. These have been plotted in fig. 10 as a function of time for a number of initial temperatures. As one would expect the intensity near the end of the shot increased as the initial temperature was raised.

A third series of experiments was carried out using 1.1 MW neutral beam injection as additional heating. Three beam lines were used each pulse lasting 100 ms but with starting times staggered by 20 ms. The probe was kept at a radius of 0.255 m, and the initial temperature varied between discharges. Measurements were made with the initial temperature at 20°C, 500°C and 700°C. At each temperature different impurities were monitored

in successive discharges by looking at the same three wavelength bands in turn with the SPEX spectrometer.

The plasma conditions were as shown in fig. 4b. The plasma current is the same as the earlier discharges but the line average density increases by about a factor 2 when the neutral beam heating is turned on. This is mainly due to the particles in the beams themselves.

In these discharges the plasma conditions near the probe limiter do not change significantly when NBI is switched on. Although the total input power increases by a factor of nearly 4 the total radiation also increases by a large factor, to typically 1100 kW. When the reduction in the ohmic heating to ~ 280 kW during NBI is taken into account, the additional heat transported to the boundary is too small to be detected. This conclusion, based on the global energy balance, is confirmed by the observation that there is no change in power to the limiter as measured by the Langmuir probes and the AGA infra-red camera. The results for the neutral beam discharges are similar to the previous runs at 0.255 m in that no significant variation in the carbon impurity flux is detected either during the discharges or as the bulk temperature is raised to 500°C and 700°C.

In parallel with the experiments described routine measurements of the spatial distribution of CV were taken with the monospec spectrometer (5). No significant change was observed either in the absolute value or in the shape of this profile as the probe limiter temperature is increased.

Because it is known that metallic and other impurities on the surface reduce the chemical sputtering yield, the probe limiter was removed immediately after the end of the series of experiments. Measurements were made of the surface impurity concentration using Rutherford Backscattering Spectroscopy (RBS), and Particle Induced X-ray analysis, (PIXE), using an incident ion beam of 2.0 MeV He⁺ ions. The major impurity was iron with a typical surface concentration of 10^{21} atoms m⁻². Other impurities observed were nickel, chromium and titanium in approximately the correct ratios for stainless steel, which clearly come from the vacuum vessel wall. In addition small amounts of calcium, chlorine, sulphur and silicon were observed. The RBS spectrum shows a broad peak in which the Fe, Cr, and Ni cannot be resolved.

4. Discussion

The main result of this experiment is that there is no evidence for chemical sputtering of carbon under deuteron bombardment. This is most clearly seen in the curve of the CII emission as a function of surface temperature (fig. 7). Each methane molecule leaving the probe limiter surface will result in the production of a CII ion (6). This molecular dissociation can follow a number of routes, some of which result in the production of a CD radical as an intermediary. The results with the probe at 0.255 m where the surface temperature is essentially constant throughout the discharge, show no change in CII emission, within experimental error, when the surface temperature is changed from 20°C to 700°C in different discharges. This is to be contrasted with the sputtering yields obtained from beam experiments at low current density. The curve shown in fig. 7 is the CII emission one would expect based on the total carbon sputtering yield H+C (at 100 eV) including the enhancement due to CH, formation found in a beam experiment at a current density of $\sim 10^{-6} \text{ A.mm}^{-\frac{7}{2}(7)}$. The set of results at a radius of 0.23 m, where the probe limiter had different initial temperatures, but was also heated during the discharge by the plasma energy, support the results at 0.255 m.

Up to now we have discussed only chemical sputtering i.e. the production of methane at the graphite surface by deuterons. Typically this has a maximum yield at 500°C in beam experiments. At higher temperatures a non-chemical erosion occurs due to radiation enhanced sublimation (1). beam experiments with energies of 1 keV this starts to be significant at $\sim 1000^{\circ}$ C, and by 1200°C the yield, due to both deuterium and helium ions, has increased by a factor 4 over their physical sputtering yields. In the case of the probe limiter results at 0.23 m radius the surface temperature reached close to 1200°C and some radiation enhanced sublimation might have been expected. None was observed. This is consistent with data from the T10 tokamak where although an enhanced emission occurs at higher temperatures no effect is observed until the surface temperature exceeds 1300°C⁽⁸⁾. A theoretical model of radiation enhanced sublimation predicts that the enhancement will be less at high fluxes (9). In addition a recent ion beam experiment $^{(10)}$ has shown that metal contaminants also reduce the radiation enhanced yield.

The only carbon spectral emission which shows a dependence on surface temperature is the CD band at 4296 to 4315 Å. This increases by a factor of 1.7 when the temperature rises from 20° to 600° C. The origin of the CD radical is uncertain but it possibly comes from carbon on the torus wall in the vicinity of the probe limiter that is released as methane by D atom bombardment. This would explain a CD signal at 20° C - a temperature at which chemical sputtering is negligible even in ion beam experiments. However, it would not explain the observed temperature dependence. The fact that the CII signal does not change with temperature, to within $\sim 10\%$, implies that the chemical sputtering contribution to the total carbon emission observed is negligible.

The conclusion that chemical sputtering contributes negligibly to the total D+ yield is based on the assumption that sputtering by deuterons is not dominated by some other C erosion mechanism; impurity sputtering by C or O ions, for instance. Examination of the spectral fluxes indicates that oxygen (which has a high sputtering yield ~ 1 atom/ion on carbon (11)) is the only likely candidate. The possibility of oxygen sputtering being dominant has therefore been examined in some detail. From the spectral intensities we can get the ratio of the photon fluxes due to deuterons, carbon and oxygen atoms leaving the probe limiter surface. From these the ratio of particle fluxes can be inferred knowing the local electron temperature from the Langmuir probe data and using theoretical calculations of photon efficiencies (photons/ionisation event) (12). In this way we have calculated the ratio of fluxes D:C:O to be 1:0.03:0.02 when the probe limiter is at 0.255 m. These values are probably accurate to within a factor of 2. If we assume that the incident D+ flux equals that leaving the surface then the contribution to the D:C ratio due solely to D+ physical sputtering is 0.02, based on an ion impact energy of 5 kT (100 eV) and a known sputtering yield (13). Oxygen leaves the surface mainly in the form of ${\rm CO}^{(14)}$; therefore, the contribution by 0 to the D:C ratio is 0.02. Considering the errors involved we can only conclude that D and O sputtering are roughly comparable. However, when the probe is at 0.23 m the O flux as indicated by the OII line at 371.2 nm increases by a factor of 1.6 during the steady state portion of the discharge (fig. 9) with no corresponding increase in the C flux (to within 10%). Thus the contribution of the O sputtering to the total carbon yield must be small. In any case, a significant change in the sputtering yield of deuterons due

to chemical effects should be readily observed. If the yield were of the same order as in beam experiments ~ 0.1 atoms/ion the contribution due to oxygen or other ion sputtering would have to be unrealistically large to mask the chemical sputtering.

As outlined earlier the negligible effect of chemical sputtering could be due to one or both of two separate processes. The first is the increased competition of recombination of hydrogen atoms to form molecules. A simple model of methane production (15) can be written in the form

$$R = \frac{J_o A \exp (-E_R/RT)}{J_o \sigma + K \exp (-E_A/RT)}$$

where J_{0} is the incident ion current density, E_{R} is the activation energy for methane production, E_{A} is the activation energy for thermal desorption of methane, σ is the cross-section for desorption of surface D atoms by incident ions, K is a constant associated with recombination and A is a normalizing constant. Using the data derived in ref. (15) we can extrapolate to high incident fluxes. The results are shown in fig. 11. It is seen that on increasing the incident flux from 10^{15} ions cm⁻² s⁻¹ to 10^{19} ions cm⁻² s⁻¹ the methane yield is suppressed by a factor of 100 to about 10^{-3} atoms/ion i.e. a factor of ~ 15 less than the yield for physical sputtering.

The second mechanism for suppressing the chemical sputtering is the presence of metal contamination on the surface. It has been shown experimentally by Roth et al. $^{(10)}$ that a surface concentration of $\sim 10^{15}$ metal atoms cm⁻² depresses the chemical sputtering yield for 500 eV H⁺ by a factor of 2. The mechanism by which the presence of metal can suppress chemical sputtering has not been positively identified but it could also be related to the competition of hydrogen atom recombination. The rate of recombination on metal surfaces is many orders of magnitude higher than for non metals $^{(16)}$. An increase in the recombination coefficient K by an order of magnitude would decrease the methane production by a factor of \sim 4, see fig. 11.

The other principal change of impurity flux with surface temperature is that of FeI. The results with the probe at $0.255~\mathrm{m}$ show both the FeI

and CrI fluxes decreasing by about a factor of 3 with increasing surface temperature, fig. 7. This is probably due to diffusion into the bulk carbon between shots when the temperature is being raised with the heater. The metal fluxes increase again once the probe limiter is cooled, suggesting that the surface is recontaminated with metals in a few discharges. The metal fluxes with the probe limiter at 0.255 m also decrease as time progresses through a discharge (see fig. 6). This is probably not a thermal effect since the surface temperature rise is small in magnitude and short in duration. The decrease is possibly due to slight shifts in the quasi-equilibrium between deposition and erosion. The metal content in tokamak plasmas is known to decrease as \overline{n} increases. Since \overline{n} increases with time the rate of metal deposition decreases. With a constant erosion rate (that is, with constant local values of \overline{n} and \overline{n} the surface concentration of metals should decrease.

When the surface is heated to high temperature $\sim 1200\,^{\circ}\text{C}$, during the discharge with the probe limiter at a radius of 0.23 m, the FeI flux increases rapidly, fig. 9. The rapid increase occurs at a temperature corresponding to that at which the iron vapour pressure is $\sim 10^{-4}$ Torr (or $\sim 10^{16}$ atoms cm⁻² s⁻¹, assuming a pure Fe surface), and is therefore consistent with sublimation. Between 300 and 400 ms the surface temperature rises slowly at a constant rate $\sim 0.5\,^{\circ}\text{C/ms}$. The Fe emission increases exponentially with time during this period as expected. The calculated heat of sublimation is $\sim 130\,$ kcal/mol which is close to the value for Fe (99 kcal/mol). After 400 ms the FeI signal flattens out and then drops despite the fact that the temperature is continuing to rise. This is to be expected as the surface concentration is depleted.

5. Conclusions

Experimental measurements have been made of the sputtering of carbon at an instrumented probe limiter in a tokamak edge plasma under a range of operating conditions. The steady state surface temperature has been varied from 20°C to 700°C and the transient surface temperature has reached 1200°C due to plasma ion bombardment. There has been no change in the rate of carbon release within an error of \pm 10% during any of these measurements. The carbon to deuterium flux ratio has remained constant throughout the

experiments. Local plasma conditions have been monitored using Langmuir probes inset in the limiter. Plasma conditions have been varied from $2 \times 10^{18} \, \mathrm{m}^{-3}$ and 15 eV to $8 \times 10^{18} \, \mathrm{m}^{-3}$ and 40 eV. The low chemical sputtering yield compared with that observed in ion beam experiments is explained by competition with hydrogen atom recombination at high incident fluxes and possibly also by the presence of metallic impurities on the surface of the carbon after operation in the tokamak.

Although we have demonstrated that there is no temperature dependence of the carbon emission at a graphite limiter i.e. no enhancement above the room temperature value, this conclusion does not necessarily apply to other graphite surfaces where there is a lower current density. It is thus important to investigate further whether the suppression is due to high current density or to the presence of metal contamination.

The results obtained from a tokamak experiment emphasizes the difficulties involved in extrapolating from beam experiments. This is primarily because of the difficulty of reproducing the high ion fluxes at low energies. The situation is further complicated by the complex surface composition which develops on all surfaces exposed to the plasma.

Acknowledgements

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TABLE 1 - Spectral lines identified in this study. Asterisks indicate lines that have been plotted in Figures 6, 7, 9, 10.

Species	Wave length nm	
,	656.28	*
H _α	434.05	
Η _γ	656.10	*
D α	433.93	
D Y CII	426.70	
GII	426.73	
	657.81	*
	658.29	223
OII	371.28	*
OII	372.73	
	374.95	*
	431.71	
	431.96	
	435.13	
	436.69	
OIII	375.47	
0111	375.99	*
FeI	371.99	*
	373.49	
	373.71	
	374.56	
	427.18	
CrI	427.48	*
	428.97	
CD, CH Molecular Ban	d 429.6-431.5	*

(v',v'') = (0,0)

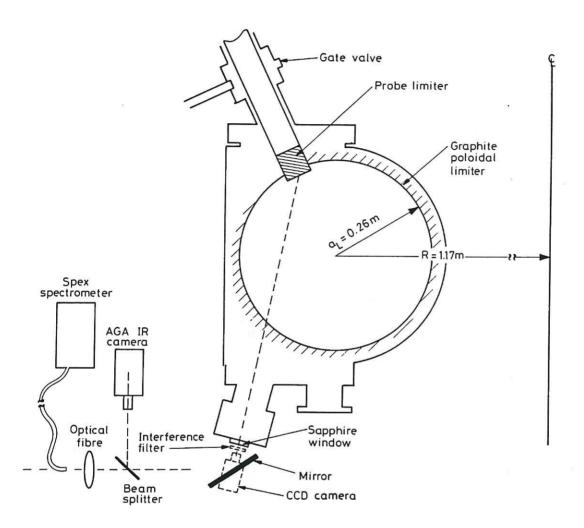


Fig. 1 Schematic of the DITE tokamak cross-section showing probe limiter and viewing arrangement for the spectrometer, the infra red and CCD cameras.

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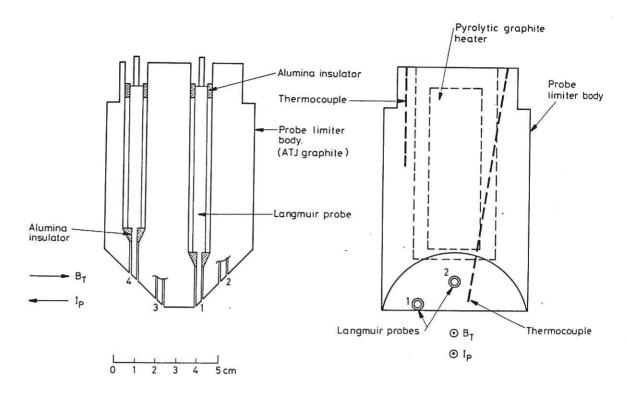


Fig. 2 Cross-sections of the carbon probe limiter showing arrangement of heater, thermo-couples and Langmuir probes.

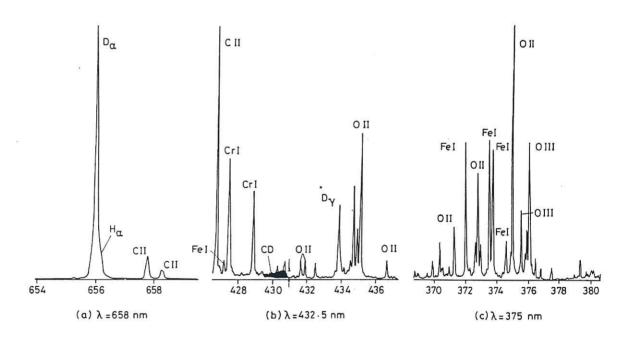


Fig. 3 Typical spectra from the SPEX spectrometer measured viewing the probe limiter at 0.255 m radius. Three spectral bands are shown centred at (a) 658 nm, (b) 432.5 nm and (c) 375 nm.

CLM-P775

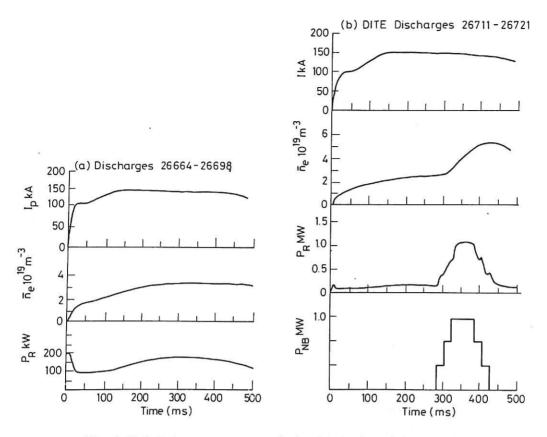


Fig. 4 Global plasma parameters during (a) ohmic and (b) neutral beam heated discharges; plasma current (I_p) , line average density (\bar{n}_e) , total radiated power (P_R) , and neutral beam power (P_{BN}) .

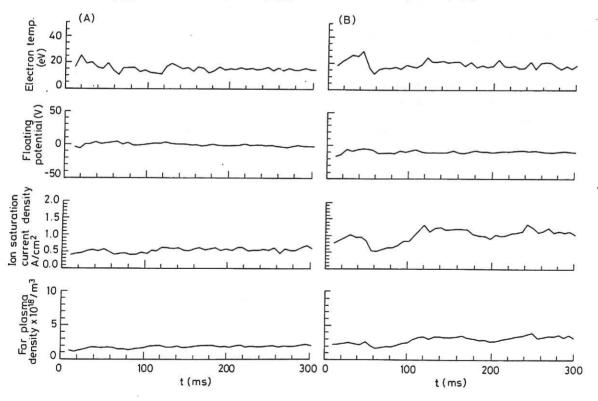


Fig. 5 Local plasma parameters T_e , V_f , j_s and n_e calculated from the probe limiter Langmuir probe data (a) on the ion drift side (LP1) and (b) on the electron drift side (LP3). Probe limiter at 0.255 m minor radius.

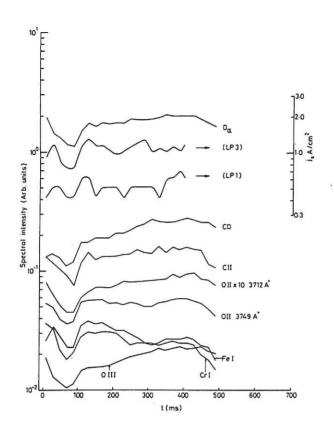
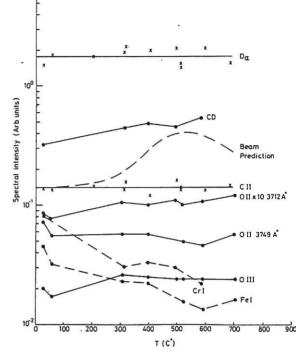


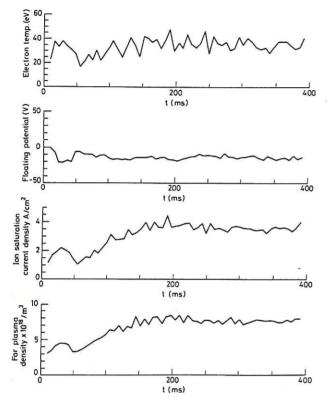
Fig. 7 Integrated spectral line intensities as a function of probe limiter temperature in successive similar deuterium discharges at t=210ms. Probe limiter at 0.255 m, where heating by the plasma is insignificant.



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Fig. 6 Integrated spectral line intensities for OII, OIII, CII, FeI, CrI and D_{cs} , the height of the CD molecular band and the ion saturation current density from the Langmuir probes LP1 and LP3 as a function of time during a discharge. Probe limiter at 0.255 m.

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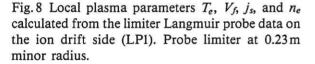


t(ms) Fig. 9 Integrated spectral line intensities, js, and tip surface temperature (Ti) as a function of time during an ohmically heated discharge. Limiter position 0.23 m minor radius, and initial temperature

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Spectral intensity (Arb. units)

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600°C.

The surface temperature T_l increases during the discharge as illustrated, due to plasma heating.

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C 11 011 3749 Å

OIII OII 3712 A

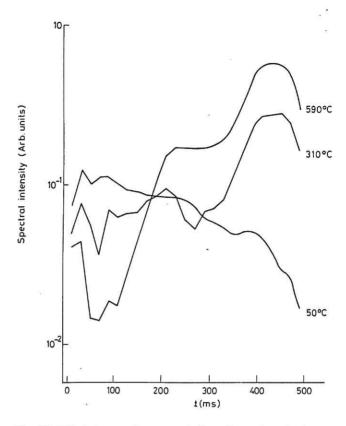


Fig. 10 FeI integrated spectral line intensity during a discharge for various limiter starting temperatures. Limiter position 0.23 m.

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