Plasma deposition of a-C/B:H layers in textor

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Abstract - A new conditioning technique for fusion devices has been developed at the tokamak
TEXTOR in KFA Jülich. It is based on the plasma assisted vapor deposition of amorphous
hydrogen containing carbon (carbonization) and carbon-boron films (boronization) on the
plasma facing inner wall surfaces. Significantly increased plasma performance has been achieved
by this method, mainly based on the suppression of metal liberation from the inner walls, on the
reduction of hydrocarbon formation on carbonaceous surfaces and on the control of oxygen plasma
impurities via gettering processes.

INTRODUCTION

At present the most promising reaction on the way to a peaceful utilization of fusion power is the fusion of
deuterium and tritium atoms into 3.5 MeV alpha-particles and 14 MeV neutrons.

$$D + T = {}^4He\ (\ 3{,}25\ MeV\ ) + n\ (\ 14.01\ MeV\ )$$

80% of the total energy, carried by the neutrons, can be transformed into usable energy while 20% carried by
the alpha-particles have to sustain the burning plasma and compensate power losses. The main power losses
are: Bremsstrahlung, which is proportional to the second power of the atomic number Z and radiation from
not fully ionized atoms namely recombination and line radiation, which are proportional to $Z^{(3-4)}$.
This dependence shows the large weight of high Z impurities on the power losses. Moreover, impurities
dilute the D-T fuel of the tokamak plasma with nonreactive atoms. Figure 1 shows the dependence of the
maximum tolerable impurity concentration in a DT-plasma as defined by radiation losses as function of the
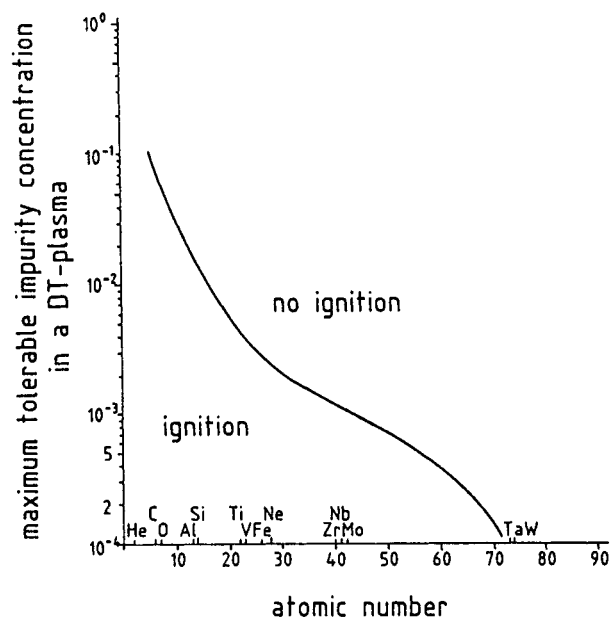atomic number Z of the impurities.

Fig. 1. Maximum impurity concentration of a plasma for ignition
depending on the atomic number Z of the impurity

High Z impurities, like tungsten,
can only be tolerated to
concentrations of roughly 10^{-4}
while the low Z impurities like
carbon should not exceed the
percent range.

The major aim of the new film
coating techniques is to keep the
impurity concentrations below
those limits. Usually the tokamak
plasma contains a mixture of
different impurities. A global
measure of the overall impurity
concentration is given by the
effective atomic number Z_{eff}
defined by:

$$Z_{eff} = \sum_i Z_i^2 \frac{n_i}{N_e / V}$$

With the ion densities n_i, total
number of electrons N_e and the
plasma volume V.

An ideal, clean hydrogen plasma
is characterized by $Z_{eff} = 1$.

Since the fuel gas is pure, it is obvious that all the impurities in the plasma originate from inside the tokamak. A process which is called plasma wall interaction. Figure 2 shows a schematic diagram of the most important processes. The plasma is surrounded by the inner wall of the fusion devices. Due to the incomplete confinement a flux of energetic particles from plasma reaches and hits the first wall, which is usually made from metal or graphite. The particles are either released back or they stick in the wall material.

The interchange of the fuel gas hydrogen between plasma and wall, the so called recycling, (H is plotted here instead of D and T) affects the density control of the plasma. In case of a DT mixture is used, the recycling processes also affect the isotope ratio and the tritium inventory of the wall. In the following this topic will not be addressed. Instead this paper deals with the discussion of the plasma impurities.
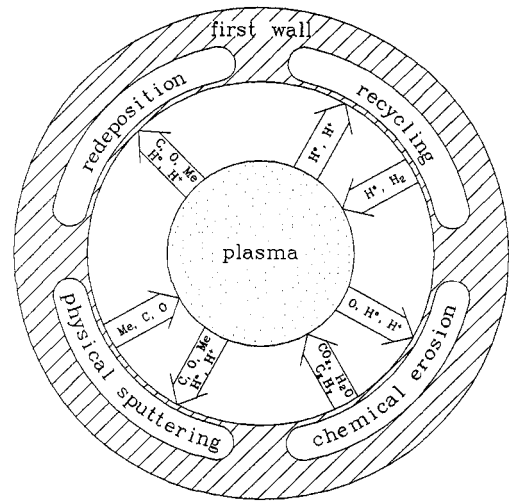

Fig. 2. Schematic diagram of the plasma wall interaction

Hydrogen in its atomic and ionic form can react at the wall surfaces to water or hydrocarbons, which are liberated from the surface and penetrate into the plasma. There they dissociate, become ionized and diffuse out of the plasma and start their own cycle in the chemical erosion with carbon walls. Oxygen as an example forms CO_x with a high yield (ref. 1). Due to their kinetic energy (above 100 eV) particles can cause physical sputtering of the wall components and liberate metal, carbon and oxygen. The gross erosion is partly balanced by the redeposition of material. The particles can stick at the wall and form layers. From this simple picture it becomes already clear that the metal release could be suppressed if the inner wall is made of light elements as for example graphite and the carbon release can be reduced if the concentration of oxygen is kept low.

DEPOSITION TECHNIQUE AND EXPERIMENTAL SET UP

At TEXTOR we have developed techniques called carbonization and boronization which aim at these two problems (ref. 2 - 5). TEXTOR is an acronym for Tokamak Experiment for Technological Oriented Research and is in operation since about one decade at Research Center Jülich. It is a machine with a toroidal stainless steel vacuum vessel with a major radius of 1.75 m and a minor radius of about half a meter. The vacuum vessel can be heated up to 300°C and has a total volume of about 17 m^3. A transformer with six yokes drives a plasma current of half a million ampere for approximately 4 sec. The plasma acts as the secondary coil of the transformer while the primary coil surrounds the central axis. 16 coils around the vacuum vessel generate a magnetic field of approximately 2.8 Tesla. This field confines the ionized plasma. The projected area of the inner surfaces of the vessel is of the order of 50 m^2. The true area is larger by 2 times 35m^2 because of an inbuilt metallic wall, the so called liner, which faces the plasma in a distance of about 10 cm. Since it is a special feature of TEXTOR to study the plasma wall interaction, the liner is designed to be exchangeable. It is heatable to 500 °C and made of 1mm thick Inconel 625. About 250 holes allow plasma diagnostic. 8 movable blades of a toroidal belt limiter mark the boundary of the plasma by direct contact with the plasma, respectively the last closed magnetic flux surface of the plasma. Plasma wall interaction is much more intensive on the limiter surfaces then on the liner surfaces. Nevertheless the contribution of the liner to the impurity production is not negligible because of its large inner area of 35m^2 compared to the 3.5m^2 of the limiters.

In the initial phase of operation it was difficult to obtain satisfactory plasma discharges. Plasma impurities were a major problem. During this time TEXTOR was conditioned only by baking and glow discharge cleaning in hydrogen. The wall consisted of the metallic liner and graphitic limiters. After several tokamak discharges the sputtered metal from the liner occurred in the plasma and was mainly redeposited onto the limiter surface. As a consequence the metal concentration in the plasma could not kept below 5 x 10^{-4}. The oxygen concentration in that situation approached roughly 3% in the plasma center and Z_{eff} was about 3. To get rid of the metal and oxygen the idea was to cover the whole inner surface with a thin protective layer of low Z material. Since TEXTOR was equipped with a radio frequency assisted glow discharge system from previous wall cleaning purposes the idea suggest itself to use this as a tool for plasma assisted chemical vapor deposition. This radiofrequency assisted DC - glow discharge is the basic tool for carbonization.

The technique is shown in figure 3 schematically in a poloidal cross section of TEXTOR. One can see the gas inlet, the vacuum vessel, the liner which acts as a cathode and the movable antenna as anode. It is surrounded by the light of the positive column of the glow. Both limiters, the inner belt limiter and the blades of the
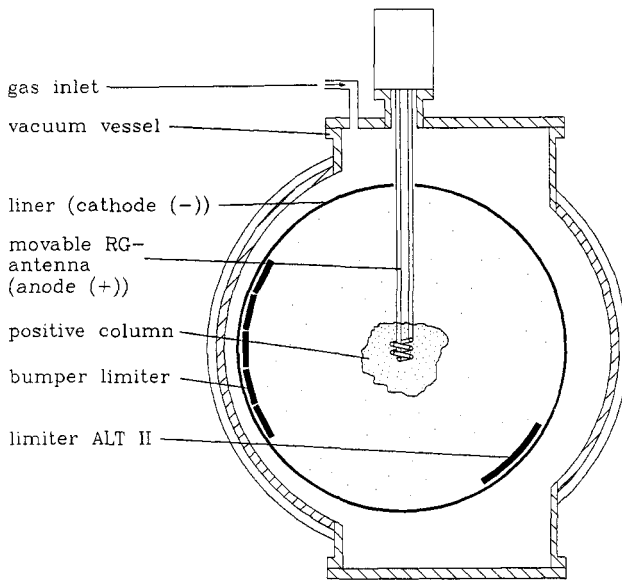
Fig. 3. Poloidal cross section of TEXTOR

toroidal limiter, are made of graphite. When a gas mixture of hydrogen and methane respectively CD_4 and D_2 enters the region between the anode and the cathode it becomes partly ionized. A DC-voltage of typically 500 Volts V between the electrodes generates a sheath potential at the cathode of the order of 300 Volts. The ions become accelerated to 300 eV and hit the wall which is typically heated up to a temperature between 150 - 200 °C. They decompose and form a layer with a growth rate of 1 monolayer per minute. Its composition is determined by the gas mixture and the other plasma parameters of the glow. The glow discharge is operated at typical total pressures of about 1×10^{-3} mbar in a total throughflow of 350 ccm/min. The system is equipped with two movable antennae with a total current of about 4 amps. This gives a current density of 1o microampere per cm^2 at the wall.

THE a-C:H LAYERS (CARBONIZATION)

In the first conditioning step (carbonization) a gas mixture of CD_4 and D_2 was used. The deposited layer covers the whole inner metallic liner. It is transparent and shows interference colors which allow thickness estimation. The thickness ranges between 50 and 150 nm depending on growth rate and deposition time. The layers are inert, hard and smooth and show a good adherence. The atomic ratio H to C is 0.4 + - .1 . And the density is about 1.4 g/cm^3.

The refractive index is about 2.4. The structure of the layer has been investigated by transmission electron microscopy at electron energies of 200 keV. The diffraction pattern of the film showed apart from the absorber for the primary beam concentric diffuse halos. The amorphous structure of the layer can be concluded from the lack of defined sharp Debey Sherrer rings. There is no long - range periodic order as in a crystal lattice (ref. 6).

All our observations showed that there was a strong resemblance to amorphous hydrogenated carbon layers as described in the literature. How those layers influenced the plasma wall interaction and consequently the plasma impurities will be described in the following.

The limiters were graphitic the liner still metallic but covered with a thin a-C:H layer. The major result of the carbonization was the suppression of metal to concentrations of about 2×10^{-5}. There was no further accumulation of metals on the limiters. Also the oxygen concentration decreased by a factor of roughly 5 to concentrations of $6-8 \times 10^{-3}$. Surprisingly the carbon concentration did not increase seriously and we found the same reduction as for oxygen. The measured Z_{eff} decreased from 3 to 1.6. The above data correspond to a fresh carbonizations, but did deteriorate with operation time because of oxygen accumulation in the machine. This necessitates weekly conditioning with baking and RG cleaning in hydrogen and monthly carbonization. But nevertheless these values led to unacceptable dilution of the plasma.

THE a-C/B:H LAYERS (BORONIZATION)

To overcome this problem we followed a proposal of our swiss colleagues to enrich the layers with boron. This new method is called boronization. The boronization is based exactly on the previous carbonization procedure except that the gas mixture is different. To add the boron atom into the deposition process we used the gas diborane (B_2H_6). It is toxic and explosive and measures had to be taken for its safe handling (ref. 7). An upper limit of 10% diborane in He in the gas bottle was also caused by the german safety rules in transportation. This mixture was added to the methane in the deposition process. All other parameter were the same as for the carbonization. Also the layers obtained by the boronization called a-C/B:H were very similar in appearance and structure. The density was somewhat lower, 1.2 - 1.3 g/cm^3 and, of course, the composition was different (ref. 8).

Figure 4 shows a sputter Auger depth profile of an a-C/B:H film on a silicon substrate. The sample was exposed during a boronization in TEXTOR by a transfer system at wall position and taken out immediately after the termination of the process. The sputter Auger electron spectroscopy gives the normalized concentration of the atoms in percent as a function of depth. The layer is homogeneously deposited onto the silicon substrate. Over the whole depth we have more or less a constant B/C ratio of 1. The depth scale was obtained from the crater depth sputtered by 3 keV Xe ions. AES is not sensitive to hydrogen. Thermal desorption measurement showed that the ratio of H/H+B is about 0.4 as obtained in a-C:H layers. No other species was found except some oxygen at the interface, which is probably due to a contamination of the silicon before boronization.

The effect of the boronization on the plasma properties is clearly revealed by spectroscopical observation.

Figure 5 shows impurity fluxes from the limiter normalized to the deuterium flux from the limiter as a function of the electron density in the plasma. The upper traces show that the oxygen fluxes after boronization are reduced to roughly one third compared to carbonization and the lower curves show the almost identical behavior for the carbon fluxes. This indicates the liberation of oxygen and carbon as CO via the chemical erosion.

Figure 6 shows the power loss of the plasma by radiation normalized to the ohmic input power as a function of the electron density of the plasma. A clear drop in the absolute amount of the radiated power can be seen when going from carbonization to boronization. In addition, the density dependence of P_{rad} in the boronized case is much less pronounced than with carbonization. The comparison of impurity concentrations in the plasma shows that the metal concentration after boronization is below the detection limit of the soft X ray analysis, which is 1×10^{-5}. The oxygen concentration is decreased to 2×10^{-3} and the carbon concentration is 3×10^{-3}, less than half of the carbonization values. The measured Zeff droped to 1.1. This indicates the net cleaning effect of boronization despite some boron is observed in the outer region of the plasma.

This reduction in the oxygen impurity level is probably caused by the formation of a chemical bond between oxygen and boron which leads to a stable boron- oxygen compound. The formation of the boron oxygen bond occurs during the plasma discharges. This is indicated by the observation of oxygen in redeposited layers with a ratio of O/B ~ .7, while only trace amounts of oxygen (less than 5%) were
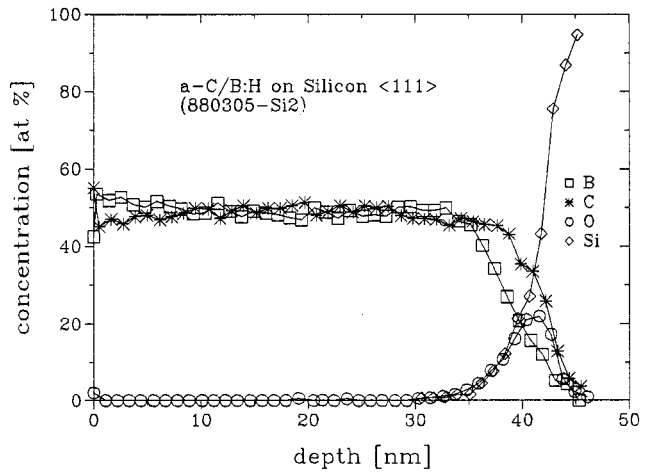
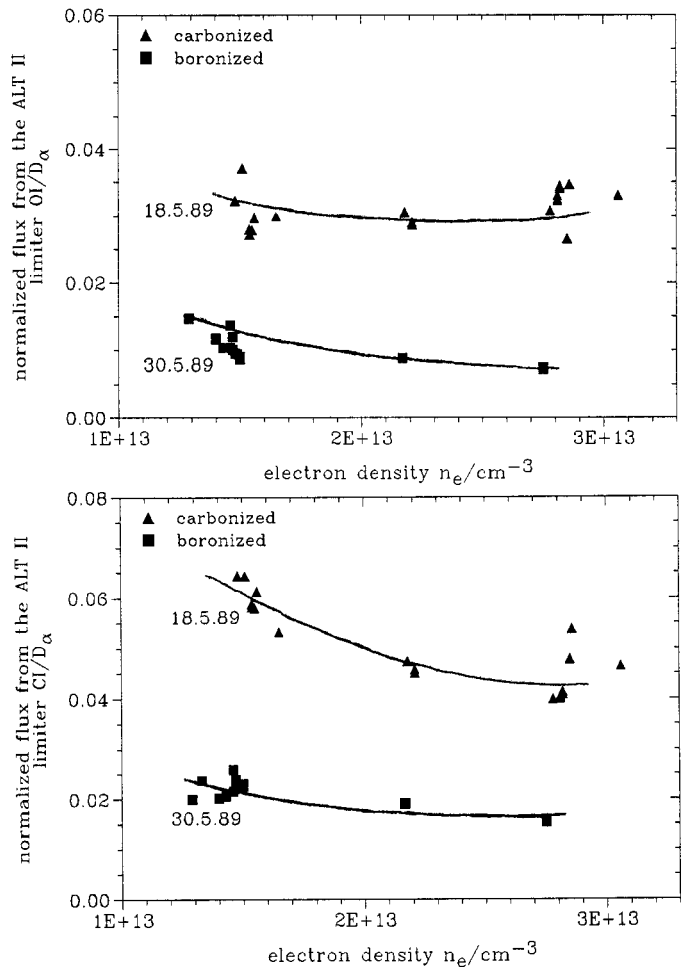Fig. 4. Sputter Auger depth profile of an a-C/B:H film on a silicon substrate

Fig. 5. Spectroscopical observations of the normalized CI- and OI-line (carbon and oxygen) as a function of electron density of the plasma

found in the boronization layers itself. This means that the boronization layer acts as a sources for a boron consuming codeposition process by which the oxygen is gettered simultaneously. This and the erosion-redeposition effect of the carbon limiters explains also the long term degradation of the positive boronization effect.
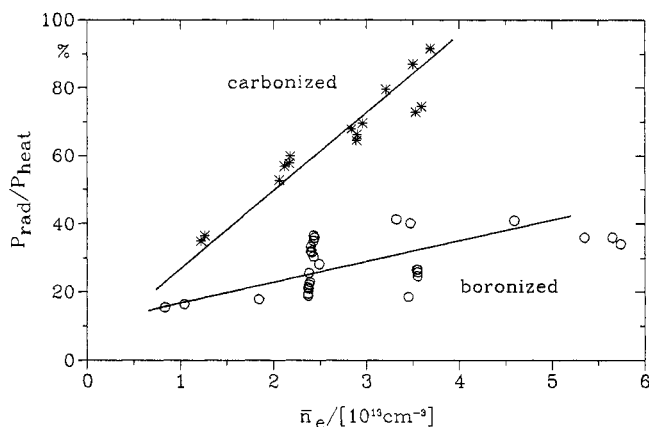

Fig. 6. Power loss of the plasma as function of electron density

CONCLUSION

The new conditioning techniques carbonization and boronization has led to significant improvements in plasma performance.

With carbonization, the metal impurities in the plasma could be suppressed. This led, e.g., to good working conditions for wave heating experiments (ref. 8). The boronization reduced in addition the oxygen impurity concentration due to gettering and the chemical erosion of carbon. This yields plasmas with very low concentrations of the impurities C and O ($Z_{eff} \sim 1$). New plasma regimes with favorable properties could be achieved. The techniques have been applied to other fusion devices worldwide ASDEX, JET, TORE SUPRA, JIPP-TIIU, HELIOTRON, TFTR, TdeV, DIIID. A shortcoming of the techniques is the limited lifetime of the films on high heatflux components. In the case of carbonization graphite plates and divertor tiles are used successfully.

At present, boron containing bulk materials on carbon basis are under investigation (ref. 10) which may be used as high heatflux components.

Acknowledgement

REFERENCES

1. V. Philipps, E. Vietzke, M. Erdweg, et al., Plasma Phys. Contr. Fusion, 31, 1685 (1989)
2. J. Winter, J. Nucl. Mater., 145-147, 131 (1987)
3. J. Winter, J. Nucl. Mater., 161, 265 (1989)
4. J. Winter, H.G. Esser, L Könen et al. J. Nucl. Mater., 162-164, 713 (1989)
5. J. Winter, J. Nucl. Mater., 176-177, 14 (1990)
6. J.Winter, H.G. Esser, P. Wienhold et al., J. Nucl. Instr. Meth. in Phys. Res., B23 538 (1987)
7. H.G. Esser, H.B. Reimer, J. Winter, D. Ringer Fusion Technology, 1, 791 (1988)
8. J. v Seggern, P. Wienhold, H.G. Esser, J. Nucl. Mater., 176+177, 357 (1990)
9. G.H. Wolf, H.L. Bay, G. Bertschinger et al., Plasma Phys. Contr. Fusion, 28, 1413 (1986)
10. J. Linke, H. Bolt, R. Doerner et al. J. Nucl. Mater., 176-177, 856 (1990)