



## Erosion of a-C:H in the afterglow of ammonia plasma



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### ABSTRACT

Amorphous hydrogenated carbon (a-C:H) deposits were eroded in the afterglow of a NH<sub>3</sub> plasma, created with an inductively coupled RF generator in pure NH<sub>3</sub> at the gas pressure of 50 Pa. The plasma system was characterised by optical emission spectroscopy and mass spectrometry, and the erosion process was monitored in-situ with a laser interferometry system. Based on the mass spectrometry measurements, the degree of dissociation of the NH<sub>3</sub> molecules was estimated at 90% at the highest generator forward power in the discharge region, however the densities of N and H atoms were significantly smaller at the location of the sample holder. The erosion rates were found to increase with surface temperature and forward generator power. In the high dissociation regime, the composition of the afterglow and the reaction products highlight the role of N atoms in the erosion process.

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### 1. Introduction

In recent years, a lot of research efforts were invested in the problem of removal of amorphous hydrogenated carbon (a-C:H) layers [1–6]. The formation of these layers occurs when carbon-based plasma facing materials are eroded by H species from the plasma. The resulting C<sub>x</sub>H<sub>y</sub> complexes are re-attached to the surface, forming a-C:H deposits [7–10].

As ITER was planned to operate with carbon based divertor components, the formation of a-C:H layers would lead to in-vessel tritium retention, a serious concern from the legal, safety and operational point of view [7]. Ultimately, a decision has been reached to start ITER without carbon based components, owing to the high susceptibility of carbon to chemical erosion by hydrogen species and subsequent growth of a-C:H layers. However, owing to their favourable power handling capabilities, and the low Z of carbon, many fusion devices still operate, or will be starting to, with carbon (graphite or carbon fibre composite) plasma facing components. Moreover, while the carbon content in the plasma has been significantly diminished after switching to full metal inner wall configuration in AUG and JET, it has nonetheless not been removed completely [11,12]. As another campaign of D–T operation

is foreseen for JET, the problem of tritium retention in carbon codeposits could still persist albeit on a significantly smaller scale.

Even without D–T operation, coverage of surfaces with a-C:H could lead to several problems. As a-C:H deposits are easily eroded by the plasma, a change in the plasma configuration could erode deposits accumulated on previously non-wetted areas. This could result in rapid release both of impurities and hydrogen species into the plasma, causing problems with recycling and impurity control.

Coverage of inner vessel surfaces with a-C:H affects the low-energy end of their plasma-wall interaction properties, as it alters the probability of heterogeneous recombination of neutral atoms [13,14] which in turn affects the fuel recycling properties. The accumulation of a-C:H also impedes optical spectroscopic diagnostics when the a-C:H deposits cover the surfaces of the mirrors [15].

The research of chemical methods of a-C:H removal has been approached from two sides. The conventional approach was based on using chemically active species to remove the a-C:H deposits from reactor vessel walls. The chemically active species would either be formed in the vessel, as was demonstrated with glow discharge cleaning in oxygen [3,4], or produced externally (in a remote plasma system) and puffed into the reactor vessel [16]. The drawback of using oxidative species is the risk of damage to plasma-facing and structural components. The other approach was to use chemically active species to prevent the formation of a-C:H deposits by binding to the precursors, i.e. the scavenger effect [17].

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Suppression of a-C:H layer formation by injecting ammonia ( $\text{NH}_3$ ) has been achieved in PSI-2 [18] as well as in TEXTOR [19], however in the “real-life” setting of fusion devices and linear plasma devices, the observed effect is most likely due to combined action of the scavenger effect and removal of already formed a-C:H layers by active species, produced upon the  $\text{NH}_3$  injection. Complementary to laboratory experiments on the scavenger effect, the work presented in our paper focuses on the a-C:H removal effectiveness of radicals produced in an  $\text{NH}_3$  plasma. Our previous study demonstrated that the ionic species of a  $\text{NH}_3$  plasma can erode a-C:H deposits [20]. In this work, we focused on the neutral radicals that are formed in a  $\text{NH}_3$  discharge.

## 2. Experimental

The main vacuum vessel of the plasma reactor, shown in Fig. 1, was a borosilicate glass tube with an inner diameter of 36 mm, and two side arms which allowed for mounting of the sample holder and the optical view-port for the laser interferometry system. The distance between the side arms and the discharge coil was 35 cm. The vacuum system was pumped with a two stage rotary pump with a pumping speed of  $80 \text{ m}^3/\text{h}$ , which could be attenuated with a butterfly valve. Ammonia was leaked into the system through a mass-flow controller at a flow rate of 183 sccm, and the butterfly valve was set so that the pressure in the reactor chamber was 50 Pa. The effective pumping speed was thus  $16.6 \text{ m}^3/\text{h}$  ( $4.6 \cdot 10^{-3} \text{ m}^3/\text{s}$ ). The average drift velocity of gas through the glass tube was 4.6 m/s and the gas required about 0.08 s for drifting from the coil to the sample.

The plasma was created with an inductively coupled radio-frequency (RF) generator. The generator was operating at the frequency of 13.56 MHz with the maximum available output power of 1000 W. The generator was coupled to the plasma system through a matching network and six turn, water-cooled coil, wound around the glass tube, which defined the discharge region in the reactor. The discharge region was actively cooled by compressed air.

Prior to measuring the erosion of a-C:H layers, the plasma system was characterised by optical emission spectroscopy and residual gas analysis. Optical emission spectroscopy (OES) was performed with an Avantes AvaSpec 3648 optical emission spectrometer. The spectrometer was linked to the system with an optical fibre which was mounted very near (less than 1 cm away) to the inductive winding, as indicated in Fig. 1. A differentially pumped Pfeifer PrismaPlus QMG 220 Residual Gas Analyser (RGA) was mounted downstream of the discharge. It was used to record continuous mass spectra of the ammonia discharge, in the 0–35 Atomic Mass Unit (AMU) range, with the electron energy set at 70 eV. Because the pressure in the reactor chamber was

considerably higher than the maximum allowed operating pressure of the RGA, it was connected to the reactor through a glass-tube flow restrictor which kept the pressure in the RGA chamber below  $5 \cdot 10^{-3} \text{ Pa}$ . This pressure drop of 5 orders of magnitude unfortunately made it impossible to calibrate the RGA against partial pressures in the reactor chamber, or to carry out any gas-balance analysis. The cracking patterns of molecules, detected with the spectrometer, are provided by the manufacturer.

Samples of approximately 250 nm thick a-C:H thin films with an approximately 40 at % hydrogen content were produced by a previously described procedure [14], and deposited on polished stainless steel substrates.

In the erosion experiments, the samples were mounted on a temperature controlled holder, positioned 35 cm away from the discharge region as shown in Fig. 1. Flash-dry silver paint (SPI Supplies) was used to ensure a good thermal contact between the samples and the sample holders. The erosion of the amorphous hydrogenated carbon deposits was monitored with a laser interferometry system. The system consisted of a laser module that produced laser light with a wavelength of 635 nm and an intensity of 3 mW. The light was passed through a quartz window to the sample. The intensity of the reflected light was measured with a Hawkeye<sup>®</sup> laser detector. The details of such in-situ erosion monitoring are described elsewhere [6].

## 3. Results and discussion

### 3.1. Characteristics of the plasma system

The characterisation of the  $\text{NH}_3$  discharge by RGA is shown in Fig. 2a and b. Mass spectra were acquired without plasma operation, and during plasma operation at generator forward powers ranging from 100 to 1000 W. Spectra recorded without plasma operation (0 W), at 200 W and at 700 W forward power are shown in Fig. 2a, and peak intensities at 2, 16, 17 and 28 AMU/ $e_0$  are shown in Fig. 2b versus generator forward power. The most prominent characteristic of the system is that it operates in two modes, the so called E-mode and H-mode [21]. The difference in the two modes is related to the coupling of the power supply to the plasma system and is reflected on the degree of dissociation of the source gas molecules. The power at which the transition between the two modes occurs is pressure dependent. In the presented system, operating in pure  $\text{NH}_3$ , it was not possible to reliably achieve the H-mode at pressures above 50 Pa. At this pressure, the system operates in the E-mode up to the forward generator power of 350 W, and in the H-mode at generator forward powers above 600 W. At powers between 400 and 650 W, the system is in a transitory mode in which prolonged operation is potentially damaging to the RF

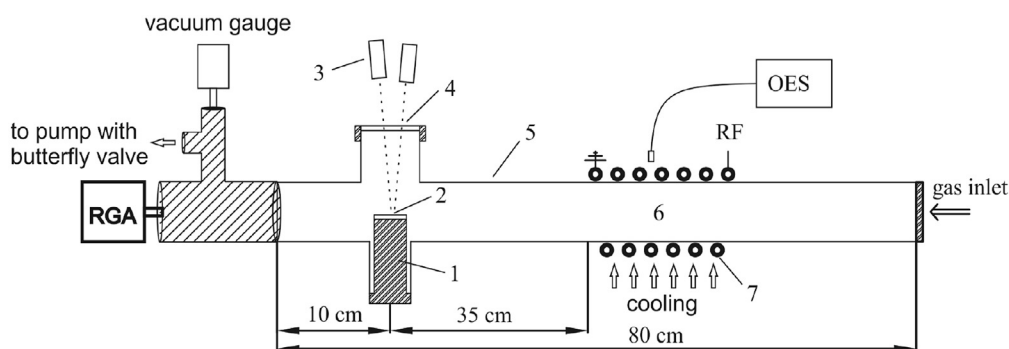


Fig. 1. Experimental set-up. 1 – temperature controlled sample holder, 2 – sample, 3 – laser interferometry system, 4 – optical viewport, 5 – glass tube, 6 – discharge region, 7 – inductive winding, coupled to the RF generator.

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