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PAPER

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Oxygen content dependent etch rate of single polymer microparticles confined in the sheath region of a low pressure radiofrequency argon/oxygen plasma

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Abstract

To study the etching of polymer microparticles confined in low pressure radiofrequency plasmas, the size and refractive index of single 2 μm particles are experimentally obtained as a function of both time and oxygen content (0%–50%) added to the argon background gas. The etch rate was found to depend heavily on the oxygen (O_2) content, especially for mixtures with low fractions of O_2 . As expected the etch rate was found to be close to zero in absence of O_2 and increases to a value of 2 nm min^{-1} for 0.5% O_2 and to roughly 3.5 nm min^{-1} for 5% O_2 . Above 5% O_2 the etch rate saturates. It is shown that these results are consistent with a steady state etch model taking the effects of both atomic oxygen and positive ions into account.

Keywords: microparticle etching, plasma sheath, argon/oxygen plasma

(Some figures may appear in colour only in the online journal)

1. Introduction

Increasingly frequently, plasmas are utilized to synthesize and modify small nano- to micrometer sized particles [1, 2]. Whereas these so-called ‘dusty plasmas’ are most often thought of in terms of growth [3, 4], i.e. plasma enhanced polymerization to trigger nanoparticle formation [5, 6], particle coagulation into larger particles [6, 7] and steady state particle growth due to deposition of mainly plasma-generated radicals on their surface [8, 9], etching of the particle material by the surrounding plasma may be significant as well, but has appeared hard to study.

It is especially the permanent negative charge, which plasma-immersed particles obtain due to the difference in mobility between (positive) ions and electrons, together with the plasma potential which is positive in relation with the

surroundings, that can make the particles being confined for considerable amounts of time in the plasma volume. Being confined in the space charge layer of plasma (for micrometer sized particles [10]), these negatively charged particles are subjected to influx of plasma-produced radicals and violent bombardment by ions. Both radicals and impinging ions may be responsible for plasma etching, often in a synergetic manner [11]. Well known etching species (etchants) are for example species containing fluor (e.g. CF_4) and species containing chloride (e.g. CL_2), for example to the end of etching silicon targets [12]. Oxygen is a well known etchant for treating organic polymers [13, 14].

In this paper, we present the experimentally determined oxygen content dependent etch rate of polymer microparticles confined in the sheath region of a low pressure radiofrequency (RF) argon/oxygen plasma. To this end, we utilize the previously developed *in situ* and angular-resolved Mie scattering technique in combination with a routine to fit these data to

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Figure 1. Picture of a microparticle confined in the plasma sheath near the powered electrode.

a Mie scattering model [15]. This method delivers the size, refractive index and etch rate under controlled plasma conditions where for instance oxygen content is changed.

The conducted experiments are relevant (I) to the field of complex and dusty plasma physics since polymeric (e.g. melamine formaldehyde (MF)) particles are often used in experiments [16–19] where they are often subject to plasma etching (e.g. due to non-sublime vacuum conditions), and (II) to all studies and experiments involving plasma etching of polymeric surfaces using oxygen (containing) plasmas [11, 20, 21].

This manuscript is organized as follows. Section 2 will elaborate briefly on the concept of plasma etching and the equilibrium confinement position of micrometer-sized particles in the plasma sheath. Section 3 discusses the used experimental setup and the Mie scattering diagnostics. Section 4 contains the experimental results with as main result the obtained etch rates of a single plasma confined polymeric microparticle as a function of O_2 content in a background argon (Ar) plasma. For analysis of the experimental results, an etch model is introduced in this section as well. Finally, discussion and conclusions drawn from this work are discussed in section 5.

2. Etching and confinement position of microparticles

Oxygen plasmas can be used to etch a variety of organic polymers [22]. Although the exact mechanism of polymer etching by oxygen is not fully understood, atomic oxygen is believed to play a dominant role. Etch rates of polymer surfaces have been found to be correlated with the density of O and etching has been observed downstream, but in the vicinity of, oxygen plasmas where atomic oxygen still persist [14].

Plasma etching can be divided in four types of mechanisms [13]:

- (I) Physical sputtering by high energetic ions (at least a few tens of eV). Surface material is ejected due to the impact of the ion. This mechanism occurs mainly at low pressure, where the mean free path is sufficiently long and ions can be accelerated to high energies (>100 eV in industrial

applications). As no chemistry is involved, possibilities of selecting the material to be etched are limited and the etch rates are low.

- (II) Chemical etching by plasma produced species. This is a purely chemical mechanism, where a neutral species reacts with the surface after which the reaction product is volatile. Due to the purely chemical mechanism, this process is highly selective, but is isotropic as the movement of the etching species has no preference direction. In this process, the role of the plasma is merely the creation of the etching species.
- (III) Ion enhanced etching. In this process, neutral reactive etchants chemically react with the surface only when enough energy is supplied by ions bombarding the target. Here, an anisotropic mechanism is combined with the selectivity of chemical etching.
- (IV) Inhibitor ion enhanced etching uses the anisotropic movement of ions in combination with chemical etching. In this case, however, an inhibitor species is added which forms a protective layer on the substrate. Etching only takes place where ion bombardment removes the protective layer, allowing a quick etchant to etch the unprotected area on the substrate.

When macroscopic particles (typically 10 nm to 100 μ m) are immersed in a plasma, they acquire a negative charge. Due to this negative charge, the particles can be confined by the electric fields which are typically present in the plasma sheath [10]. The position on which the forces on a particle (besides the aforementioned electric force, mainly gravity and the ion drag force are dominant) cancel each other provides insight in the plasma dynamics in the plasma sheath [17]. This allows the use of microscopic particles as probes in gas discharges.

Our previous measurements in [15] have been performed with a contaminated argon RF discharge, thus containing a small fraction of oxygen, due to a non-zero base pressure as is always the case in laboratory and industrial plasmas. By actively adding oxygen to a pristine argon (as is done in the current work) discharge, among others (e.g. oxygen ions) the etchant O is produced and the etch mechanism can be further clarified right at the confinement position of the particle. It is

Figure 2. Schematic view of the setup, cross sections from the top and from the side. (a) Vacuum vessel with three viewports. (b) Plasma. (c) Microparticle. (d) Laser. (e) Polariser, polarising the light perpendicularly to the scatter plane. (f) Laser beam. (g) Collector rotating around the plasma. (h) Camera. (i) Photodiodes. (j) Pillars between electrodes. (k) Detection boundaries. (l) Electrodes. (m) Stepper motor to rotate the collector around the plasma. (n) Particle dispenser. (o) Modification of electrodes for particle confinement. (p) Spectrometer. (q) Optical fiber. (r) Connection to pumping system. (s) Gas inlet. Adapted from [15]. (a) Top view. (b) Side view.

known that oxygen is able to etch polymer particles (like MF) [23]. A picture of a confined particle is shown in figure 1.

3. Methods

The experimental setup used for the current investigation is similar to that described in detail in a previous article [15]. In this section, an overview of the most important aspects of the

used setup is provided. Figure 2 gives a schematic representation of the setup. For additional details of the setup the reader is directed to [15].

A radio frequent (13.56 MHz) discharge is created between two horizontally aligned parallel plates at a pressure of 16 Pa. The stainless steel electrodes have a diameter of 140 mm and are aligned 44 mm apart, while the bottom one is powered and the top one is grounded. The plasma power dissipation is fixed at 10 W.

A single spherical melamine formaldehyde (MF) particle is confined in the plasma sheath above the bottom electrode. Particles are stored in a particle container which is situated above the plasma. By shaking the container, particles will fall through a hole in the container's bottom and fall further through a larger hole in the top electrode after which the particles are confined in the plasma. The amount of particles leaving the particle suspender can be regulated by shaking the container more or less strongly. By tuning this process, it is possible to immerse a single particle. A full description of the workings of this particle dispenser is given in [24].

The bottom electrode is modified to allow the confinement of a single particle. As described in section 2, gravity and the different plasma forces acting on the particle allow particle confinement in the direction perpendicular to the electrode, which is parallel with the gravitational acceleration. The bottom electrode is designed with a circular indent to also allow horizontal confinement of the particle. This indent is 3 mm deep and has a radius of 5 mm. Due to this indent, a net horizontal electric force is acting on the particle when it is out of the center of the indent, keeping the particle in the center. According to the supplier [25], the used particles have a radius of $2.35 \mu\text{m}$ and a refractive index of 1.68.

The particle size is measured by measuring laser light scattered from the particle angularly resolved. A diode laser emitting light with a wavelength of 532 nm, which is polarised perpendicularly with respect to the scatter plane, is used as light source for the incident beam. A detector is placed inside the vacuum chamber and can rotate around the plasma so that it measures the light scattered from the particle in the angle range from 5° to 160° degrees.

The oxygen content is controlled by argon and oxygen flows by two separate flow controllers. The vessel has been pumped down to a base pressure of $5 \cdot 10^{-6}$ mBar.

The particle size and refractive index are determined based on the measured angular scatter profile. Given the size relative to the wavelength and the refractive index of a spherical particle, Mie theory [26] is valid under these conditions. The inverse method for determining the relevant particle parameters is applied here. Because measuring the scatter profile typically takes an hour, the etch rate of a single particle can be determined next to the refractive index and the particle size. This method comes down to fitting the position of peaks and valleys in the profile to those in Mie theory, using three fit parameters (refractive index, initial particle size and etch rate). A full description of this method is described in [15].

Figures 3–14 show the measured scatter profiles which are fitted with the described method. The oxygen content is varied and is given in the caption of the figures. The positions of peaks and values which are used in the algorithm are marked by the grey rings. In the angle domain marked in grey, visual access to the particle is blocked in the setup. Note that during the angular profile scan the particle size is decreasing due to etching and that the analysis method incorporates this feature self-consistently. The angular scatter profile is used to determine the size, the refractive index and the etch rate of the confined particle.

Figure 3. Oxygen content is 0%.

Figure 4. Oxygen content is 0.5%.

Figure 5. Oxygen content is 1%.

4. Results

The etch rate of a single particle is measured as a function of the oxygen content in an argon/oxygen admixture. After the results are presented, the etch mechanism is shortly discussed. For each measurement with a different amount of oxygen a new particle is injected, so that the different measurements are comparable. Note that deviation in the initial particle size between different measurements is taken into account. The refractive index of the particles is hypothesized real, as is motivated in [15].

Figure 15 shows the etch rate of particles as function of the oxygen content. The dashed line is placed to guide the eye

Figure 6. Oxygen content is 2%. **Figure 9.** Oxygen content is 5%. **Figure 7.** Oxygen content is 3%. **Figure 10.** Oxygen content is 10%. **Figure 8.** Oxygen content is 4%. **Figure 11.** Oxygen content is 15%.

and show similarities with the model later in this section. It is clear that the fraction of oxygen influences the etch rate significantly. In a pure argon plasma, minimal etching takes place. Adding a small amount as only 0.5% of oxygen, immediately increases the etch rate to about 2 nm min^{-1} . Further increasing the oxygen content raises the etch rate further, but it is leveled at about 4.7 nm min^{-1} for oxygen contents higher than 20%.

The dependence of the etch rate on the fraction of oxygen gas is not uncommon for polymer etching of bulk material, but it is the first time it is found for a plasma confined microparticle. With respect to etching of bulk materials, Murillo *et al* have found a similar sub-linear trend in the etch rate of polymers as fraction of the oxygen content in an argon admixture

[11]. Joubert *et al* found that the etch rate of sample polymers saturates for increasing atomic oxygen density in oxygen/nitrogen admixtures [21]. In both of these investigations, it was found that the etch rate increased with the provided ion energy.

To give an explanation for the leveling of the etch rate at higher oxygen content in figure 15, the etch process itself is discussed in more detail. Assuming that atomic oxygen in synergy with ion bombardment is responsible for etching [11], the full mechanism can be explained in three sequential steps. Step 1: adsorption. Atomic oxygen is created in the plasma by dissociation of molecular oxygen and is adsorbed at the particle's surface. Step 2: etch reaction. Ions impacting on the particle provide energy for the adsorbed etchant to undergo

Figure 12. Oxygen content is 20%.

Figure 13. Oxygen content is 35%.

Figure 14. Oxygen content is 50%.

the etch reaction and create etch products. Step 3: desorption. The etch products leave the particle by desorption [13].

These steps are described qualitatively by the etch model of Bray and Rhinehart [27]: at the surface, a certain amount of sites for etching are present which may be in different states. The vacant sites (S_v) are available for adsorption of an etchant species or etch product, the other sites are occupied by either adsorbed etchants (S_O) or adsorbed etch products (S_p). The sum of these states gives the total amount of sites $S = S_v + S_O + S_p$. In a stationary situation, the amount of occupied sites is given by:

$$\frac{dS_O}{dt} = r_a - r_r = k_a \Gamma_O S_v - k_r \Gamma_i S_O = 0, \quad (1a)$$

Figure 15. Etch rate as function of oxygen content in the discharge. For all measurements the pressure is 16 Pa and the power 10 W. The dashed line is placed to guide the eye; it is a fit through the data points following the function $Y(X) = a + \frac{bcdX}{c+dX}$, neglecting the data points at 4% and 15% of oxygen.

$$\frac{dS_p}{dt} = r_r - r_d = k_r \Gamma_i S_O - k_d S_p = 0, \quad (1b)$$

where r_a , r_r and r_d are the rates at which respectively the adsorption, etch reaction and desorption steps take place. k_a , k_r and k_d represent coefficients for the respective processes. Γ_O , Γ_i and Γ_p give the flux to the particle for respective etchants O, ions i, and etch products p. Backwards reactions (e.g. desorption of etchants for step (1)) are neglected. Solving equations (1a) and (1b) for S_O yields:

$$S_O = \frac{k_a \Gamma_O S}{k_r \Gamma_i + \frac{k_a k_r}{k_d} \Gamma_O \Gamma_i + k_a \Gamma_O}. \quad (2)$$

Assuming that the etch reaction step is the limiting step and the desorption step to be very fast ($k_d \gg k_r$ [27]), the etch rate r_r can be written as:

$$r_r = k_r \Gamma_i S_O = \frac{k_a k_r S \Gamma_i \Gamma_O}{k_r \Gamma_i + k_a \Gamma_O}. \quad (3)$$

This expression can be clarified by taking the limits of the atomic oxygen flux and the ion flux. For constant coefficients, taking the ion flux goes towards infinity, the etch rate is determined by the atomic oxygen flux and vice versa.

This model can be used to explain the results presented above in figure 15. Apparently, the increase in flux of etchant species towards the particle for increasing oxygen content dominates the total etch rate only for low values of the oxygen contents. For oxygen contents roughly above 5%, creation of additional neutral etchants leaves the particles etch rate unaltered apparently. This indicates that the etch rate for high oxygen contents is limited by the limited energy flux provided by the impinging ions.

The initial particle size and the refractive index of the particle vary for each particle. These values are given, together with the etch rate, in table 1. Figure 16 gives a graphical representation of the variation in initial particle size and refractive index and the difference to the supplier specified values (square marker). The deviation from the specified value is relatively large compared to the spread of the data points for both the refractive index and the particle size.

Table 1. The found fit parameters for each scan together with corresponding error. m_k represents the refractive index, $\frac{da}{dt}$ gives the etch rate and a_0 is the initial particle size.

[O ₂] (%)	m_k	$\frac{da}{dt}$ (μm)	a_0 (nm min ⁻¹)
0.0	1.639 ± 0.005	0.15 ± 0.3	2.149 ± 0.006
0.5	1.642 ± 0.007	2.2 ± 0.4	2.061 ± 0.009
1.0	1.643 ± 0.002	2.2 ± 0.2	2.057 ± 0.005
2.0	1.646 ± 0.006	2.2 ± 0.3	2.146 ± 0.007
3.0	1.634 ± 0.007	2.9 ± 0.3	2.154 ± 0.007
4.0	1.634 ± 0.004	3.9 ± 0.5	2.16 ± 0.012
5.0	1.637 ± 0.005	3.3 ± 0.09	2.172 ± 0.002
10.0	1.631 ± 0.004	3.8 ± 0.3	2.169 ± 0.007
15.0	1.634 ± 0.013	3.1 ± 0.4	2.167 ± 0.009
20.0	1.631 ± 0.004	4.6 ± 0.2	2.176 ± 0.005
35.0	1.630 ± 0.013	4.9 ± 0.5	2.211 ± 0.011
50.0	1.631 ± 0.002	4.7 ± 0.09	2.122 ± 0.002

Figure 16. Graphical representation of the found initial particle size and refractive index. The square marker represents the specified values.

5. Conclusions

The etch rate of a microparticle in an oxygen containing discharge is not proportional with the oxygen content in the background gas, for oxygen content higher than 20% the etch rate reaches a maximum of 4.7 nm min^{-1} . This effect can be explained by the contribution of the positive ions, assuming that the density of atomic oxygen increases with the oxygen content in the background gas. For small amounts of oxygen, the atomic oxygen flux dictates the etch rate. The etch rate is dictated by the ion flux when oxygen is abundant.

Additionally, this research shows that data on the particle radius more accurate than given by the supplier is essential for quantitative utilisation of the microparticle as probes for the plasma sheath. This is because small variations on particle radius have a significant influence on the plasma—particle dynamics under investigation (mainly the electric field, particle charge and ion drag force). It is shown in this work that measured particle properties are different from the values provided by their suppliers. Consequently, the radius of MF particles measured by angularly resolved Mie scattering is found

approximately 10% lower than specified. Also the refractive index is measured to be lower than specified.

The influence of gas contamination in the discharge should not be underestimated in time consuming measurements. Small amounts of contamination (e.g. oxygen, water) could enhance etching of mainly polymer particles. When this is the case, the particle size and mass are not constant. Mass losses should be determined and taken into account for correct interpretation of the measurements, especially for measurements that take a relatively long time.

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