Plasma Chemistry and Kinetics in Low Pressure Oxygen Discharges: The significance of metastable states

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Outline

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A. Global (volume averaged) chemistry models
The main idea of a global model is to generate a model that encompasses a large number of reactions in order to model a processing plasma with a limited computing power by neglecting the complexity which arises when spatial variations are considered.

Thus the model does not describe spatial distribution but captures scalings of plasma parameters with control parameters.

The model allows us to investigate various phenomena, such as the effects of excited species, negative ions and particular reactions on the overall discharge.
A.1 Global (volume averaged) chemistry models – oxygen
The oxygen discharge is of significance in various materials processing applications including:

- etching of polymer films
- ashing of photoresist
- oxidation

The oxygen chemistry is complicated due to the presence of metastable atomic and molecular species.

It is in particular the two low lying metastable molecular states designated by a \(^1\Delta_g\) and \(^1\Sigma^+_g\), which are located 0.98 and 1.627 eV above the ground state, respectively.

It is well established that collisions with these metastable states have in many cases larger cross sections and thus higher reaction rates than corresponding collisions with the ground state molecule.
The global (volume averaged) model

- A steady state global (volume averaged) model was developed for the oxygen discharge
- The following species are included
  - electrons
  - the ground state oxygen molecule $O_2(X^3\Sigma^{-}_g, v = 0)$
  - The metastable oxygen molecules $O_2(a^1\Delta_g)$, $O_2(b^1\Sigma^+_g)$ and the metastable Herzberg states $O_2(A^3\Sigma^+_u, A'^3\Delta_u, c^1\Sigma^-_u)$
  - the ground state oxygen atom $O(^3\text{P})$
  - the metastable oxygen atom $O(^1\text{D})$
  - the negative oxygen ions $O^-$ and $O_2^-$
  - the positive oxygen ions $O^+$ and $O_2^+$
  - Ozone $O_3$ and its ions $O_3^+$ and $O_3^-$
- The content of the chamber is assumed to be nearly spatially uniform and the power is deposited uniformly into the plasma bulk

The global (volume averaged) model

- The particle balance equation for a species $X$ is given

$$\frac{dn^{(X)}}{dt} = 0 = \sum_i R_{\text{Generation},i}^{(X)} - \sum_i R_{\text{Loss},i}^{(X)}$$

where $R_{\text{Generation},i}^{(X)}$ and $R_{\text{Loss},i}^{(X)}$, respectively, are the reaction rates of the various generation and loss processes of the species $X$.

- The power balance equation, which equates the absorbed power $P_{\text{abs}}$ to power losses due to elastic and inelastic collisions and losses due to charged particle flow to the walls is given as

$$\frac{1}{V} \left[ P_{\text{abs}} - eVn_e \sum_\alpha n^{(\alpha)} \varepsilon_c^{(\alpha)} k_{iz}^{(\alpha)} - eu_{B0} n_i A_{\text{eff}} (\varepsilon_i + \varepsilon_e) \right] = 0$$
The global (volume averaged) model

For the edge-to-center positive ion density ratio we use

\[ h_\ell \approx \left[ \left( \frac{0.86}{(3 + \eta L/2\lambda_i)^{1/2}} \frac{1}{1 + \alpha_0} \right)^2 + h_c^2 \right]^{1/2} \]

\[ h_R \approx \left[ \left( \frac{0.8}{(4 + \eta R/\lambda_i)^{1/2}} \frac{1}{1 + \alpha_0} \right)^2 + h_c^2 \right]^{1/2} \]

where \( \alpha_0 \approx (3/2)\alpha \) is the central electronegativity, \( \eta = 2T_+/ (T_+ + T_-) \) and

\[ h_c \approx \left[ \gamma_-^{1/2} + \gamma_+^{1/2} \left[ n_\ast^{1/2} n_+ / n_-^{3/2} \right] \right]^{-1} \]

and \( n_\ast = \frac{15}{56} \frac{\eta^2}{k_{\text{rec}}\lambda_i} v_i \)

is based on a one-region flat topped electronegative profile

\[ \gamma_- = T_e/T_- \quad \text{and} \quad \gamma_+ = T_e/T_+ \]
The global (volume averaged) model

- The electron energy distribution function (EEDF) is usually assumed to be Maxwellian.
- We can also assume a general electron energy distribution

\[ g_e(\mathcal{E}) = c_1 \mathcal{E}^{1/2} \exp\left(-c_2 \mathcal{E}^x\right) \]

\[
c_1 = \frac{1}{\langle \mathcal{E} \rangle^{3/2}} \left[ \frac{\Gamma(\xi_2)^{3/2}}{\Gamma(\xi_1)^{5/2}} \right]
\text{ and } c_2 = \frac{1}{\langle \mathcal{E} \rangle^x} \left[ \frac{\Gamma(\xi_2)}{\Gamma(\xi_1)^x} \right]
\]

where \( \xi_1 = 3/2x \) and \( \xi_2 = 5/2x \)

- Here \( x = 1 \) and \( x = 2 \) correspond to Maxwellian and Druyvesteyn electron energy distributions, respectively.
The global (volume averaged) model

- The diffusional losses of the neutral oxygen atoms (ground state and metastable) to the reactor walls are given by

\[ k_{O,\text{wall}} = \left[ \frac{\Lambda_O^2}{D_O} + \frac{2V(2 - \gamma_{\text{rec}})}{A v_O \gamma_{\text{rec}}} \right]^{-1} \text{s}^{-1} \]

- \( D_O \) is the diffusion coefficient for oxygen atoms
- \( v_O = (8e T_g / \pi m_O)^{1/2} \) is the mean O velocity
- \( \gamma_{\text{rec}} \) is the wall recombination coefficient for neutral oxygen atoms on the wall surface
- \( \Lambda_O \) is the effective diffusion length of neutral oxygen atoms

\[ \Lambda_O = \left[ \left( \frac{\pi}{L} \right)^2 + \left( \frac{2.405}{R} \right)^2 \right]^{-1/2} \]

- The wall recombination coefficient \( \gamma_{\text{rec}} \) is one of the most important parameter in oxygen discharge modelling
A.2.1 Model parameters
Surface recombination

- The wall recombination probability, $\gamma_{\text{rec}}$, is a very important quantity in all low pressure molecular discharges.

- The pressure dependence on the wall recombination coefficient was achieved by fitting all the available data for stainless steel surfaces.

- The same wall recombination coefficient was used for O($^1\text{D}$) as no data is available.

Gudmundsson and Thorsteinsson, *PSST*, **16** (2007) 399

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Figure 1. The recombination coefficient of oxygen atoms at the chamber walls for stainless steel as a function of pressure. The measured data is taken from, o Singh et al [47], × Matsushita et al [90], A Mozetič and Zalar [91], □ Booth and Sadeghi [44] and * Gomez et al [46]. The solid line shows a fit to the measured data and the dotted line is a linear extrapolation from $\gamma = 0.5$ at 2 mTorr to $\gamma = 1.0$ at vacuum.

The wall recombination coefficient for oxygen atoms on stainless steel surfaces depends on pressure through

$$\gamma_{\text{rec}} = 0.1438 \exp\left(\frac{2.5069}{p}\right) \quad p > 2 \text{ mTorr}$$

$$\gamma_{\text{rec}} = -0.25p + 1 \quad p < 2 \text{ mTorr}$$
A.2.3 Particle densities
Particle densities

- The dominant species is the oxygen molecule in the ground state $O_2(X^3\Sigma_g)$ followed by the oxygen atom in the ground state $O(^3P)$
- The singlet metastable states $O_2(a^1\Delta_g)$ and $O_2(b^1\Sigma_g^+)$ and the metastable atom $O(^1D)$ are also present in the plasma in significant amounts
- A cylindrical stainless steel chamber

radius $R = 15$ cm and length $L = 30$ cm $P_{\text{abs}} = 500$ W

Particle densities

- The $\text{O}_2(b^1\Sigma_g^+)$ density overcomes the $\text{O}_2(a^1\Delta_g)$ density in the pressure range from 2.5 to 80 mTorr.
- The $\text{O}_2^+$ ions are in majority among the positive ions.
- The $\text{O}^+$ density has a sharp decrease for pressures above 4 mTorr.
- The ratio $[\text{O}^-]/[\text{O}_2^-]$ is 5.3 at 1 mTorr, and 1.3 at 100 mTorr.
- A cylindrical stainless steel chamber with radius $R = 15$ cm and length $L = 30$ cm $P_{\text{abs}} = 500$ W.

Creation of metastable $O_2(a^1\Delta_g)$ molecules

- Electron impact excitation (17)

$$e + O_2(X^3\Sigma_g^-) \rightarrow O_2(a^1\Delta_g) + e$$

has 100% contribution at 1 mTorr and 68.2% at 100 mTorr

- The role of quenching of the metastable atom $O(^1D)$ to create the metastable oxygen molecule (71)

$$O(^1D) + O_2(X^3\Sigma_g^-) \rightarrow O_2(a^1\Delta_g) + O(^3P)$$

reaches a maximum value, 20.1% contribution at 12.5 mTorr, and 10.5% at 100 mTorr

**Destruction of metastable \( O_2(a^1 \Delta_g) \) molecules**

- Electron impact dissociation (31,24)

\[
e + O_2(a^1 \Delta_g) \rightarrow O(^1D) + O(^3P) + e
\]

\[
e + O_2(a^1 \Delta_g) \rightarrow O(^3P) + O(^3P) + e
\]

are the most important channels for destruction of the metastable \( O_2(a^1 \Delta_g) \) molecules, 57.2 % contribution at 1 mTorr and 38.1 % at 100 mTorr
The most important contributor to the formation of the metastable oxygen molecule $O_2(b^1\Sigma_g)$ is reaction 72:

$$O(^1D) + O_2(X^3\Sigma_g^-) \rightarrow O_2(b^1\Sigma_g) + O(^3P)$$

which has 75.7% contribution at 1 mTorr and 93.2% at 100 mTorr.

This reaction has been suggested in the past to be a major contributor to the formation of $b^1\Sigma_g^+$ state in the atmosphere.

Destruction of metastable \( O_2(b^1\Sigma_g) \) molecules

- Wall quenching is an important loss process for \( O_2(b^1\Sigma_g) \)

- Electron impact dissociation
  \[ e + O_2(b^1\Sigma_g) \rightarrow O(1D) + O(3P) + e \]
  \[ e + O_2(b^1\Sigma_g) \rightarrow O(3P) + O(3P) + e \]
  are the most important channels for destruction along with electron impact ionization
  \[ e + O_2(b^1\Sigma_g) \rightarrow O_2^+ + 2e \]

Destruction of the negative ion $O^-$

- At low pressure that the electron impact detachment (54),

\[ e + O^- \rightarrow O(3P) + 2e \]

is the main contributor
- Ion-ion mutual neutralization is also important
- As pressure increases, the contributions of these reactions become negligible

Destruction of the negative ion $O^-$

- As the pressure increases charge exchange (113)

$$O^- + O_2(X^3\Sigma_g^-) \rightarrow O(^3P) + O_2^-$$

and detachment by collision with $O(^3P)$ (117)

$$O^- + O(^3P) \rightarrow O_2(X^3\Sigma_g^-) + e$$

have increased contribution as well as detachment by collision with $O_2(b^1\Sigma_g)$ (reaction 116)

$$O^- + O_2(b^1\Sigma_g) \rightarrow O(^3P) + O_2(X^3\Sigma_g^-) + e$$

A.2.4 Influence of chamber wall and EEDF
Influence of chamber wall and EEDF

- The parameter $x$ defines the shape of the electron energy distribution
  - $x = 0.5$ is concave or bi-Maxwellian
  - $x = 1$ is a Maxwellian distribution
  - $x = 2$ is Druyvesteyn distribution

- For anodized aluminium reactor walls, the recombination coefficient for oxygen atoms at the walls is assumed to be a constant $\gamma_O = 0.06$ based on the measurements of Guha et al. (2008)

- The chamber wall has a significant influence on the dissociation fraction
A.2.5 Chlorine dilution
Chlorine dilution – Particle densities

- The chlorine-oxide molecule ClO and its ion ClO$^+$ peak when Cl$_2$ and O$_2$ flowrates are roughly equal.
- The O$_2$(a$^1 \Delta_g$) density is about 9 – 10% of the total O$_2$ density.
- The electron density increases about 30% between pure chlorine and pure oxygen discharge.


A cylindrical stainless steel chamber $L = 10$ cm and $R = 10$ cm
$p = 10$ mTorr and $P_{\text{abs}} = 500$ W
Oxygen dilution – Particle densities

- The total rate for creation and loss of ClO molecules is at maximum when the oxygen content is 65%.
- Wall recombination of Cl molecules, is the dominating pathway for creation of ClO molecules.
- The bulk processes and recombination of ClO$^+$ ions at the wall account for roughly 33–43% of the total rate for ClO creation, combined.

A.3 Summary
Summary

- A global model of O\textsubscript{2}, and Cl\textsubscript{2}/O\textsubscript{2} discharges has been applied to explore the creation and destruction of the singlet metastable molecules O\textsubscript{2}(a\textsuperscript{1}\Delta\textsubscript{g}), O\textsubscript{2}(b\textsuperscript{1}\Sigma\textsubscript{g}+).

- The singlet delta state O\textsubscript{2}(a\textsuperscript{1}\Delta\textsubscript{g}) is created mainly by electron impact excitation

\[ e + O\textsubscript{2}(X\textsuperscript{3}\Sigma\textsubscript{g}−) \rightarrow O\textsubscript{2}(a\textsuperscript{1}\Delta\textsubscript{g}) + e \]

- The singlet delta state O\textsubscript{2}(b\textsuperscript{1}\Sigma\textsubscript{g}+) is created mainly by

\[ O(\textsuperscript{1}D) + O\textsubscript{2}(X\textsuperscript{3}\Sigma\textsubscript{g}−) \rightarrow O\textsubscript{2}(b\textsuperscript{1}\Sigma\textsubscript{g}) + O(\textsuperscript{3}P) \]

- At low pressure that the electron impact detachment

\[ e + O\textsuperscript{−} \rightarrow O(\textsuperscript{3}P) + 2e \]

is the main contributor to the loss of O\textsuperscript{−}.

- The ClO molecule is mainly created by recombination at the discharge wall.
B. 1D particle-in-cell/Monte Carlo collision simulation
Outline

- The 1D particle-in-cell/Monte Carlo collision simulation
  - The oxygen discharge
    - Capacitively Coupled Oxygen Discharge at 13.56 MHz – Voltage Source – pressure dependence
  - Summary
The 1D particle-in-cell/Monte Carlo collision simulation
In particle-in-cell simulation the plasma is represented as a collection of macroparticles.

Each macroparticle is a charged “cloud” representing many real charged particles.

Each macroparticle has the same charge-to-mass ratio \((q/m)\) as the real charged particle.

Equations of motion are solved for each macroparticle.

The electric and magnetic fields are calculated self-consistently using charge densities and currents produced by the macroparticles.
The oopd1 1d-3v PIC/MCC code

- We use the oopd1 (objective oriented plasma device for one dimension) code to simulate the discharge.
- The oopd1 code was originally developed at the Plasma Theory and Simulation Group at UC Berkeley.
- It has 1 dimension in space and 3 velocity components for particles (1d-3v).
- The oopd1 code is supposed to replace the widely used xpdx1 series (xpdp1, xpdcl and xpds1).
- It is developed to simulate various types of plasmas, including processing discharges, accelerators and beams.
  - Modular structure
  - Includes relativistic kinematics
  - Particles can have different weights.

B.1. The oxygen discharge
The oxygen discharge

We consider a discharge that consists of:

- electrons
- the ground state oxygen molecule $O_2(X^3\Sigma_g^-)$
- the metastable oxygen molecule $O_2(a^1\Delta_g)$
- the metastable oxygen molecule $O_2(b^1\Sigma_g)$
- the ground state oxygen atom $O(^3P)$
- the metastable oxygen atom $O(^1D)$
- the negative oxygen ion $O^-$
- the positive oxygen ions $O^+$ and $O_2^+$

We apply a global model\(^1\) beforehand to calculate the partial pressure of the various neutrals

\(^1\) Thorsteinsson and Gudmundsson, *Plasma Sources Sci. Technol.*, **19** 055008 (2010)
The oxygen discharge

- The reaction set for the oxygen is comprehensive and for this study includes 67 reactions

B. 1. 1. Capacitively Coupled Oxygen Discharge at 13.56 MHz – pressure dependence – including $O_2(a^1\Delta_g)$
We apply a voltage source with a single frequency

\[ V(t) = V_{\text{rf}} \sin(2\pi ft) \]

The electrodes are circular with a diameter of 14.36 cm

The gap between the electrodes is 4.5 cm

We set \( V_{\text{rf}} = 222 \) V and \( f = 13.56 \) MHz

The neutrals (\( \text{O}_2 \) and \( \text{O} \)) are treated as background gas at \( T_g = 300 \) K with a Maxwellian distribution

The dissociation fraction and the metastable fraction is found using a global model

The pressure is varied from 10 – 500 mTorr
Capacitively Coupled Oxygen Discharge at 13.56 MHz

For a parallel plate capacitively coupled oxygen discharge at 50 mTorr with a gap separation of 4.5 cm by a 222 V voltage source at 13.56 MHz

- $\text{O}_2^+$-ion density profile
- $\text{O}^+$-ion density profile
- $\text{O}^+$-ion density profile
- $\text{O}^-$-ion density profile
- electron density profile

Gudmundsson and Ventéjou (2015) JAP 118 153302
The sheath width decreases as the pressure is decreased in the pressure range from 50 mTorr to 10 mTorr.

The sheath widths are largest at 50 mTorr.

As the pressure is increased from 50 mTorr up to 500 mTorr the sheath width decreases.

This agrees with what has been observed experimentally in the pressure range 40 – 375 mTorr.

Mutsukura et al. (1990) JAP 68 2657 and van Roosmalen et al. (1985) JAP 58 653
The electron heating profile $J_e \cdot E$.

In the pressure range 50 - 500 mTorr the electron heating occurs almost solely in the sheath region.

As the pressure is decreased the Ohmic heating contribution in the plasma bulk increases and sheath heating decreases.

Gudmundsson and Ventéjou (2015) JAP 118 153302
Capacitively Coupled Oxygen Discharge at 13.56 MHz

- At 10 mTorr excluding the metastable states in the simulation has very small influence on the heating mechanism.
- At 50 mTorr the metastable states have a significant influence on the heating mechanism.
- The role of the metastables is even more significant at 200 mTorr.
At low pressure the EEPF is convex, the population of low energy electrons is relatively low.

As the pressure is increased the number of low energy electrons increases and the number of higher energy electrons (> 10 eV) decreases.

Thus the EEPF develops a concave shape or becomes bi-Maxwellian as the pressure is increased.

Gudmundsson and Ventéjou (2015) JAP 118 153302
Capacitively Coupled Oxygen Discharge at 13.56 MHz

Our results agree with the measurements of Lee et al. (2010) which explored experimentally the evolution of the EEPF with pressure in a capacitively coupled oxygen discharge in the pressure range 3 – 100 mTorr.

They find that the EEPF became more distinctly bi-Maxwellian and the density of low energy electrons increases as the gas pressure is increased.
The effective electron temperature drops as the pressure is increased when the metastable singlet oxygen molecule $O_2(a^1\Delta_g)$ is added to the discharge model. This is particularly true in the electronegative core due to detachment by the metastable $O_2(a^1\Delta_g)$ molecule:

$$O^- + O_2(a^1\Delta_g) \rightarrow \text{products}$$
At low pressure the EEPF is convex and develops a concave shape or becomes bi-Maxwellian as the pressure is increased.

These results contradict what is commonly found for the capacitively coupled argon discharge where the EEPF evolves from being concave at low pressure to being convex at high pressure.

Gudmundsson and Ventéjou (2015) JAP \textbf{118} 153302
B. 1. 2. Capacitively Coupled Oxygen Discharge at 13.56 MHz – pressure dependence – including $O_2(a^1\Delta_g)$, $O_2(b^1\Sigma_g)$ and $\gamma_{see}(E)$
It has been known for decades that the metastable oxygen molecule $O_2(b^1\Sigma_g)$ plays an important role in the oxygen discharge. Thompson (1961) *Proc. Royal Soc. A* 262(1311) 519

Recent global model study indicates there is a significant density of $O_2(b^1\Sigma_g)$ in the oxygen discharge. Toneli et al., *J. Phys. D*, 48 325202 (2015)

The $O_2(b^1\Sigma_g)$ is mainly created through

$$O_2(X^3\Sigma_g^-)+O(^1D) \rightarrow O_2(b^1\Sigma_g)+O(^3P)$$
Capacitively Coupled Oxygen Discharge at 13.56 MHz

- The density profiles of charged particles and fast neutrals comparing including \( \text{O}_2(a^1 \Delta_g) \) (left) and \( \text{O}_2(a^1 \Delta_g) \) and \( \text{O}_2(b^1 \Sigma_g) \) (right) at 50 mTorr
Capacitively Coupled Oxygen Discharge at 13.56 MHz

- The number of cold electrons increases as $O_2(b^1\Sigma_g)$ is added to the discharge model.
- The electron heating in the bulk drops to zero.
- The EEPF is roughly independent of the partial pressure of $O_2(a^1\Delta_g)$ and $O_2(b^1\Sigma_g)$ while both are included.
- Adding secondary electron emission ($\gamma_{\text{see}} = 0.2$) leads to a high energy tail in the EEPF.
Capacitively Coupled Oxygen Discharge at 13.56 MHz

- We have compiled experimental data from the literature on secondary electron emission yields for the species $\text{O}_2^+$, $\text{O}^+$, $\text{O}_2$ and $\text{O}$ bombarding various metals and substances.
- A fit was made through the available experimental data.
The $\text{O}_2^+$, $\text{O}^-$ and electron density profiles for $\gamma_{\text{see}} = 0.0$, $\gamma_{\text{see}} = 0.2$, and energy dependent secondary electron emission yield

- The electron density increases with increased secondary electron emission yield
Increased secondary electron emission yield increases the electron heating rate in the sheath region and the sheath region becomes narrower.
Comparison to experimental findings:

- \( \gamma_{\text{see}} = 0.0 \),
  4.4 % \( \text{O}_2(\text{a}^1\Delta_g) \)

- \( \gamma_{\text{see}} = 0.0 \),
  4.4 % \( \text{O}_2(\text{a}^1\Delta_g) \) and 4.4 % \( \text{O}_2(\text{b}^1\Sigma_g) \)

- \( \gamma_{\text{see}} = \gamma_{\text{see}}(E) \),
  4.4 % \( \text{O}_2(\text{a}^1\Delta_g) \) and 4.4 % \( \text{O}_2(\text{b}^1\Sigma_g) \)

Experimental findings by Keckar

(Ph.D. Thesis, Dublin City University, January 2015)
Summary

- We demonstrated particle-in-cell/Monte Carlo collision simulation of a capacitively coupled discharge.
- In an oxygen discharge at low pressure the EEPF is convex and develops a concave shape or becomes bi-Maxwellian as the pressure is increased.
- These results contradict what is commonly found for the capacitively coupled argon discharge where the EEPF evolves from being concave at low pressure to being convex at high pressure.
C. Overall Summary
Overall Summary

- A global (volume averaged) model can be used to understand the plasma chemistry
  - Which particles are important
  - Which reactions are important
  - How do the plasma parameters scale with the control parameters – power, pressure, discharge dimensions
- Particle-in-cell/Monte Carlo collision simulations can be used to explore the plasma kinetics
  - To find the electron energy distribution function
  - To find the ion energy distribution (IED) and the ion angular distribution (IAD)
Overall Summary

- There is clearly a need for basic experimental work on capacitively coupled oxygen discharges
  - Langmuir probe measurements, EEDF, $T_{\text{eff}}$, $n_e$ versus pressure and applied voltage
  - Electronegativity for various pressures and applied voltage
- The rate coefficient for detachment by $O_2(b^1\Sigma_g)$ has to be reevaluated
- What is the role of electron impact dissociative attachment from vibrationally excited molecular states?
- What is the role of the metastable Herzberg states $O_2(A^3\Sigma_u^+, A'^3\Delta_u, c^1\Sigma_u^-)$?
- Measurements of the wall quenching coefficient of $O_2(b^1\Sigma_g)$ are needed
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The slides can be downloaded at
http://langmuir.raunvis.hi.is/~tumi/ranns.html

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