Programa de Pós-Graduação em
Engenharia Elétrica

Setups

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Setups

3.1. ALD-I

3.2. Atmospheric Plasma Glow discharges

3.3. FlexAl

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3.5. Plasma Reactor

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3.9 The Expanding Thermal Plasma

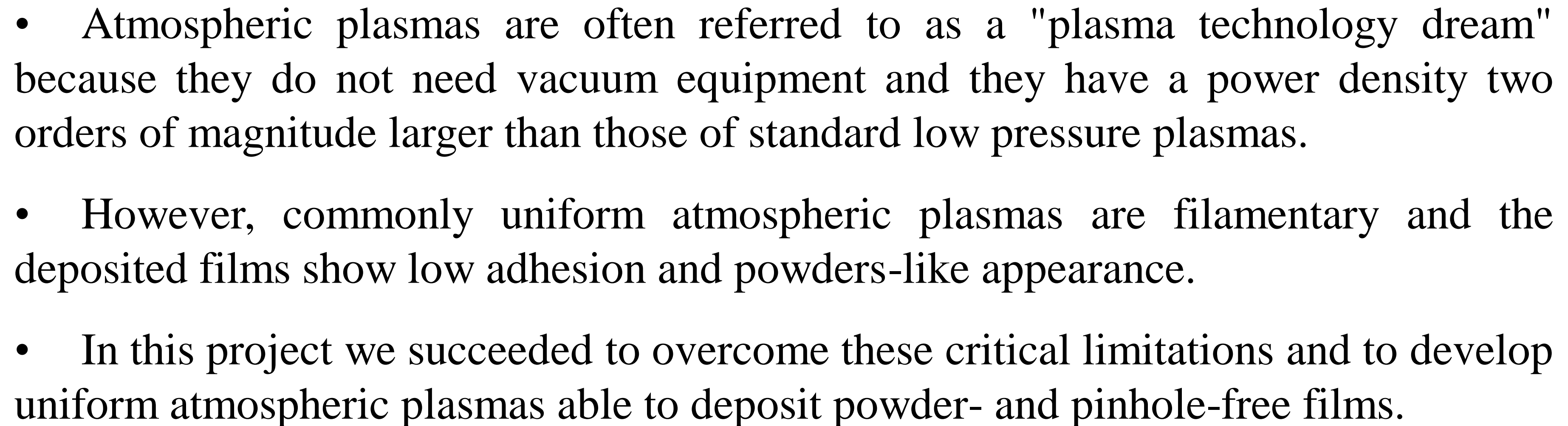
3.1. ALD-I

Diagnostics

- Home-built ALD-I system (remote plasma, 4” wafers, open-load). Experimental setup used to deposit the PA-ALD films.
- The plasma used in the ALD cycles is clearly visible. Furthermore, optical accesson the chamber allows in situ spectroscopic ellipsometry measurements to study the film growth.

3.2. Atmospheric plasma glow discharges

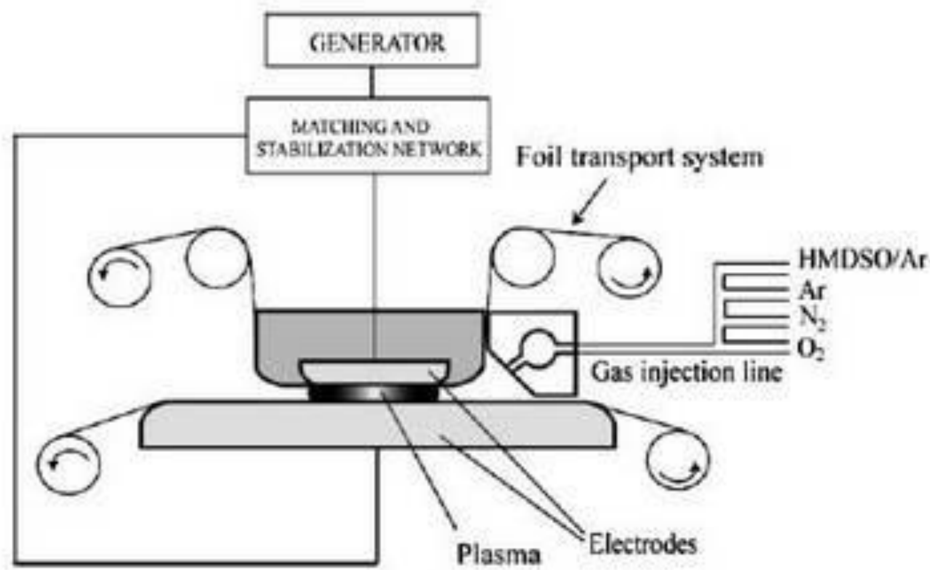
Diagnostics



- Atmospheric plasmas are often referred to as a "plasma technology dream" because they do not need vacuum equipment and they have a power density two orders of magnitude larger than those of standard low pressure plasmas.
- However, commonly uniform atmospheric plasmas are filamentary and the deposited films show low adhesion and powders-like appearance.
- In this project we succeeded to overcome these critical limitations and to develop uniform atmospheric plasmas able to deposit powder- and pinhole-free films.

3.2. Atmospheric plasma glow discharges

Diagnostics



- Fig 1. Left: Schematic of the plasma dielectric barrier glow discharge setup.

- Right: Photo of the experimental setup.

3.2. Atmospheric plasma glow discharges

Diagnostics

- The main project in our lab is the deposition of H2O vapour and O2 permeation barrier layers via a uniform and filament-free atmospheric plasma and the understanding of the deposition process.

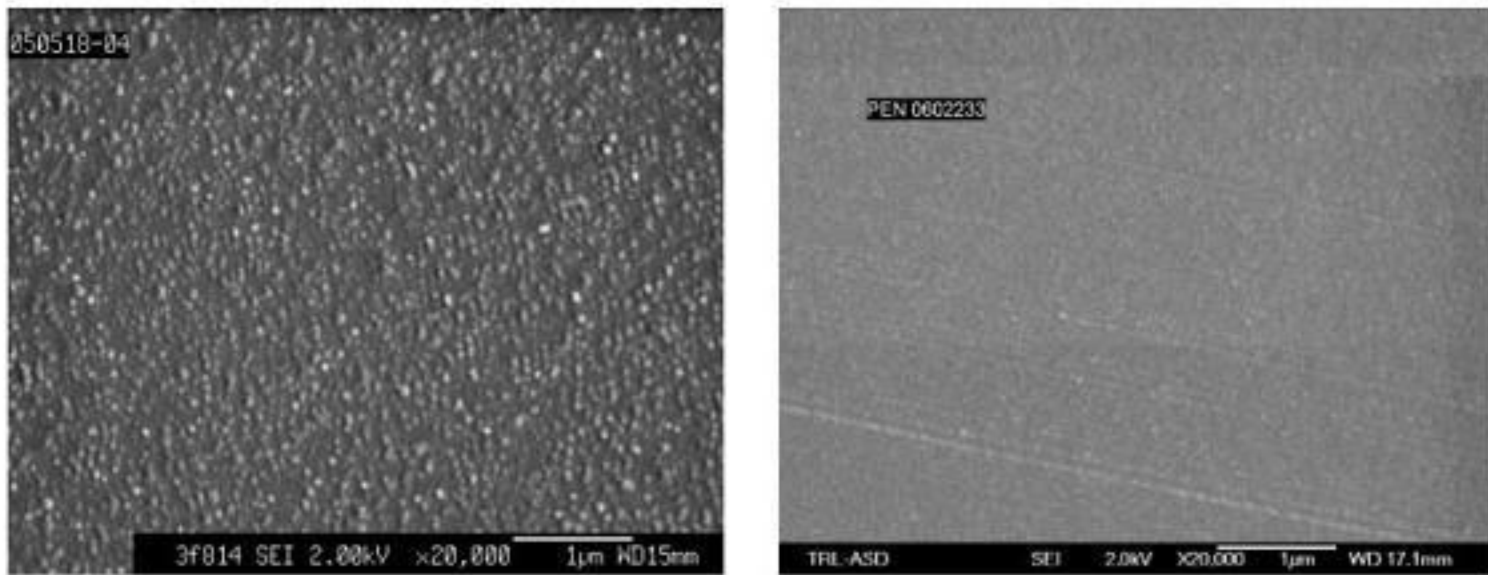


Fig 2. SEM pictures of the APGD deposited silica-like films on polymer substrates. Left: Film with embedded dust particles, right: uniform film [S. Starostine et al., Plasma Process. Polym. 4 (2007), S440].

3.2. Atmospheric plasma glow discharges

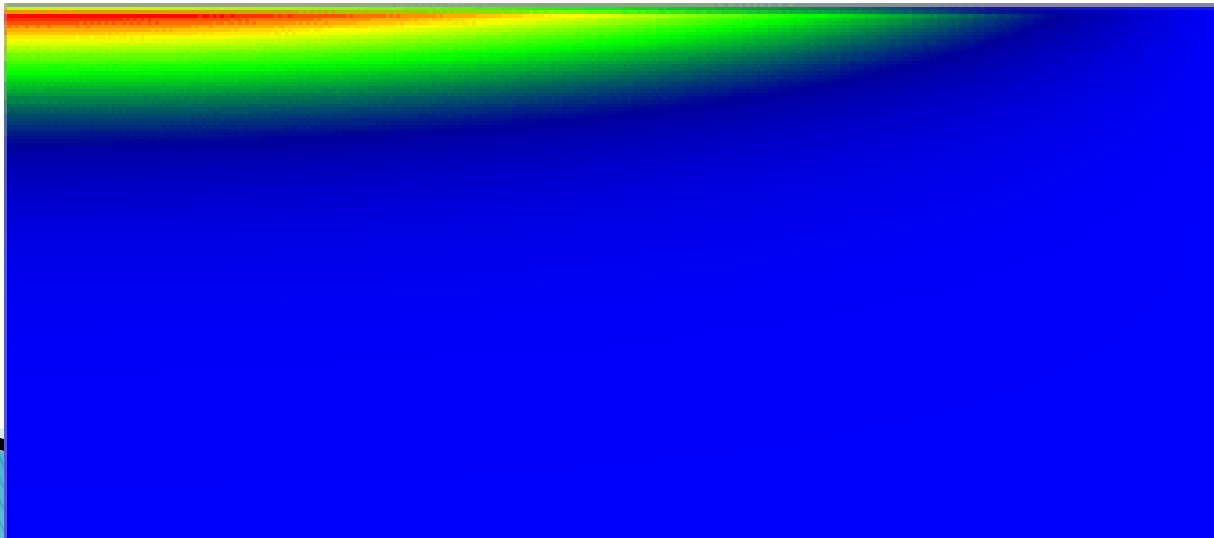
Diagnostics

- In addition to serious technological benefits, APGD represents a challenging phenomenon from the point of view of fundamental plasma physics, not yet fully investigated.
- While with our eyes we see steady laterally-uniform diffuse glow, at nanosecond scale it is a highly non-stationary transient discharge.
- During development in time, it passes several forms characterized by different plasma

3.2. Atmospheric plasma glow discharges

Diagnostics

- Fig 3. Time evolution of electron density in DBD.
- Results of numerical modeling.
- parameters: namely low current Townsend-like discharge, positive streamer and expanding glow-like discharge. In Fig. 3 one can see the evolution of the electron number density during a time interval of 600 ns, calculated for atmospheric DBD in 0.5 mm gap with a 2D cylindrical 3-moments fluid model.



3.3. FlexAl

Diagnostics

- Oxford Instruments FlexAL system (remote plasma, 8” wafers, loadlock). Experimental setup used to deposit the PA-ALD films.

- The plasma used in the ALD cycles is clearly visible. Furthermore, optical access on the chamber allows in situ spectroscopic ellipsometry measurements to study the film growth.

- This setup was developed in collaboration with Oxford Instruments:

3.4. OpAL

Diagnostics

- The Oxford Instruments OpAL™ system is an open-load ALD reactor suited for both thermal and remote plasma ALD. The system can handle wafer substrates with a diameter up to 200 mm (8”) and these can be loaded and unloaded in a N2 environment using an optional glovebox.

3.4. OpAL

Diagnostics

- Optical viewports can be mounted on the system such that film growth can be monitored real-time using techniques such as spectroscopic ellipsometry (as shown in the photo).

- Up to three precursors can be mounted on the system (in an extracted cabinet) and the precursor dosing system allows for heating up to 200 ºC as well as bubbling with Ar gas. In addition several plasma and reactant gases are available(O2, H2, N2, NH3, H2O).

3.5. SOLARIS
Synthesis of mOlecules from pLAsma produced Radicals Interacting with Surfaces

Diagnostics

- Short description

- This setup is designed to investigate what happens with the composition of a plasma when it interacts with a surface. By investigating the changes to the plasma at carefully chosen conditions, we aim at resolving details on the processes taking place at surfaces during plasma exposure.

3.5. SOLARIS
Synthesis of mOlecules from pLAsma produced Radicals Interacting with Surfaces

Diagnostics

- Context
- The study of the interaction between a plasma and surfaces is a hot scientific topic, for example in surface treatments of various materials:
- Deposition of thin functional layers.
- Etching.
- Modification of polymers.
- These studies mainly concentrate on the effects on the surfaces itself.

3.5. SOLARIS Synthesis of mOlecules from pLAsma produced Radicals Interacting with Surfaces

Diagnostics

- Because the processes taking place at surfaces during plasma treatment are extremely hard to investigate directly, a lot of fundamental knowledge is missing on this subject.

- In 'normal' catalysis, a special (sometimes expensive) catalyst is used to dissociate gases and form new types of (e.g. harmless) particles. In our setup, the first step is handled by the plasma and it is interesting to see what this implies for the choice of (cheaper?) catalyst materials.

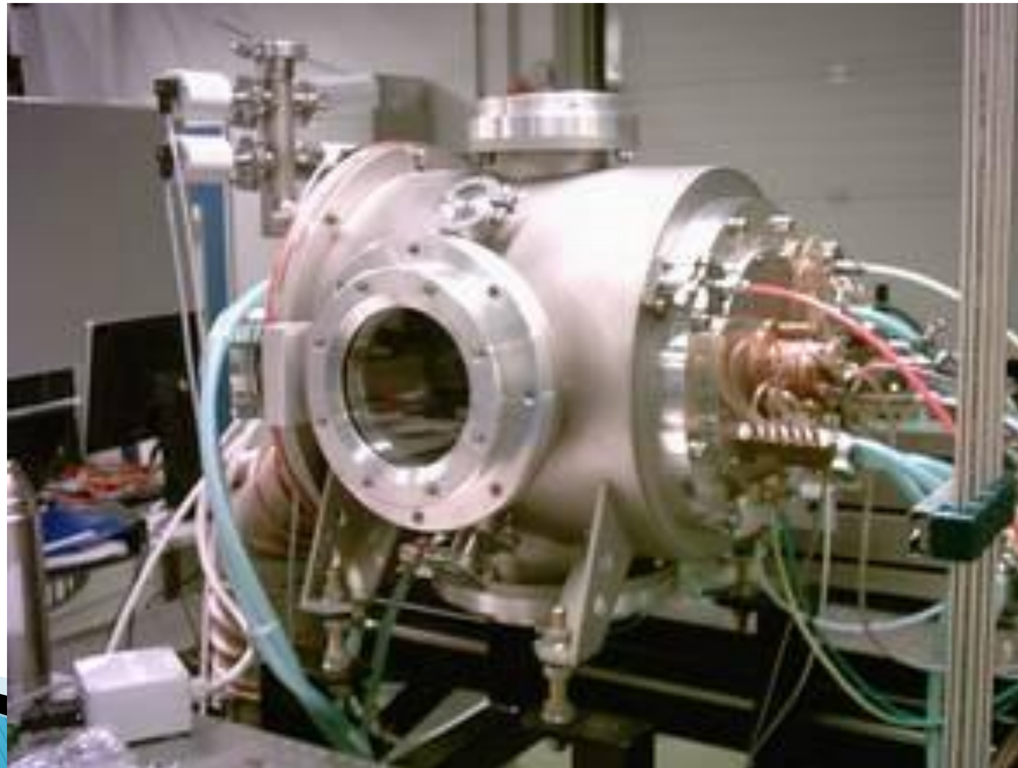
- Power of this setup

- The SOLARIS setup is small, which results in a high surface to volume ratio and has big windows, to optically investigate the plasma in detail. A mass spectrometer will be installed soon and the other diagnostic tools available within ETP can easily be connected to the setup

3.5. SOLARIS Synthesis of mOlecules from pLAsma produced Radicals Interacting with Surfaces

Diagnostics

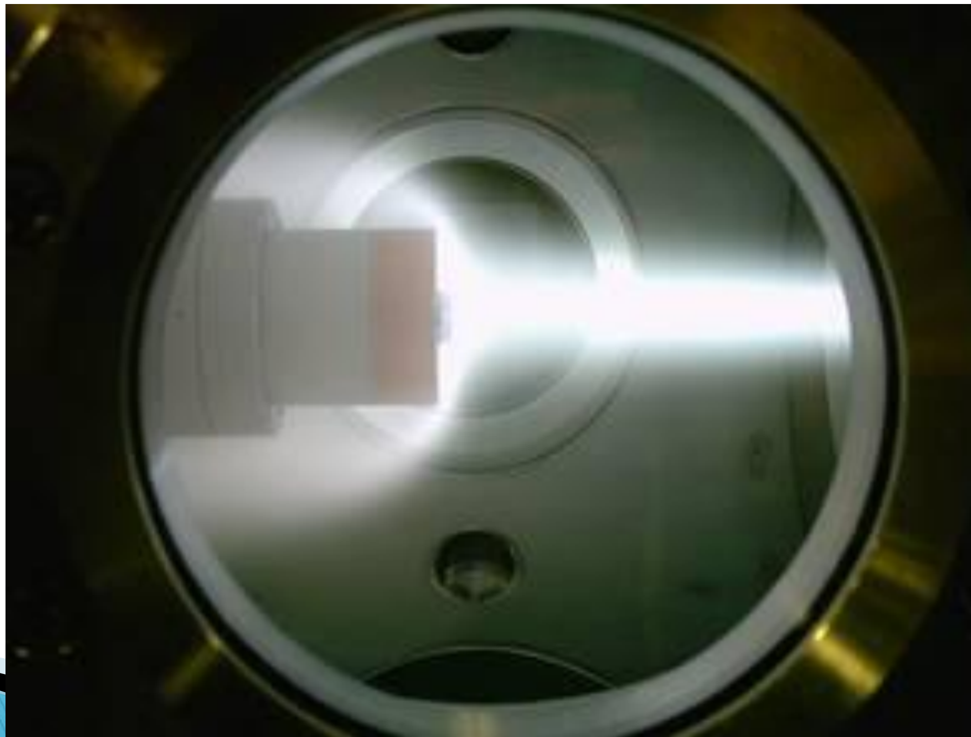
- Setup of the first plasmas created with the fully automated plasma reactor.



3.5. SOLARIS
Synthesis of mOlecules from pLAsma produced Radicals Interacting with Surfaces

Diagnostics

- An expanding argon plasma (40 scc/s Ar, p = 1.0 mbar and a current of 45A)



3.5. SOLARIS
Synthesis of mOlecules from pLAsma produced Radicals Interacting with Surfaces

Diagnostics

- expanding nitrogen plasma (40 scc/s N2, p =1.0 mbar and I = 45 A)

3.5. SOLARIS Synthesis of mOlecules from pLAsma produced Radicals Interacting with Surfaces

Diagnostics

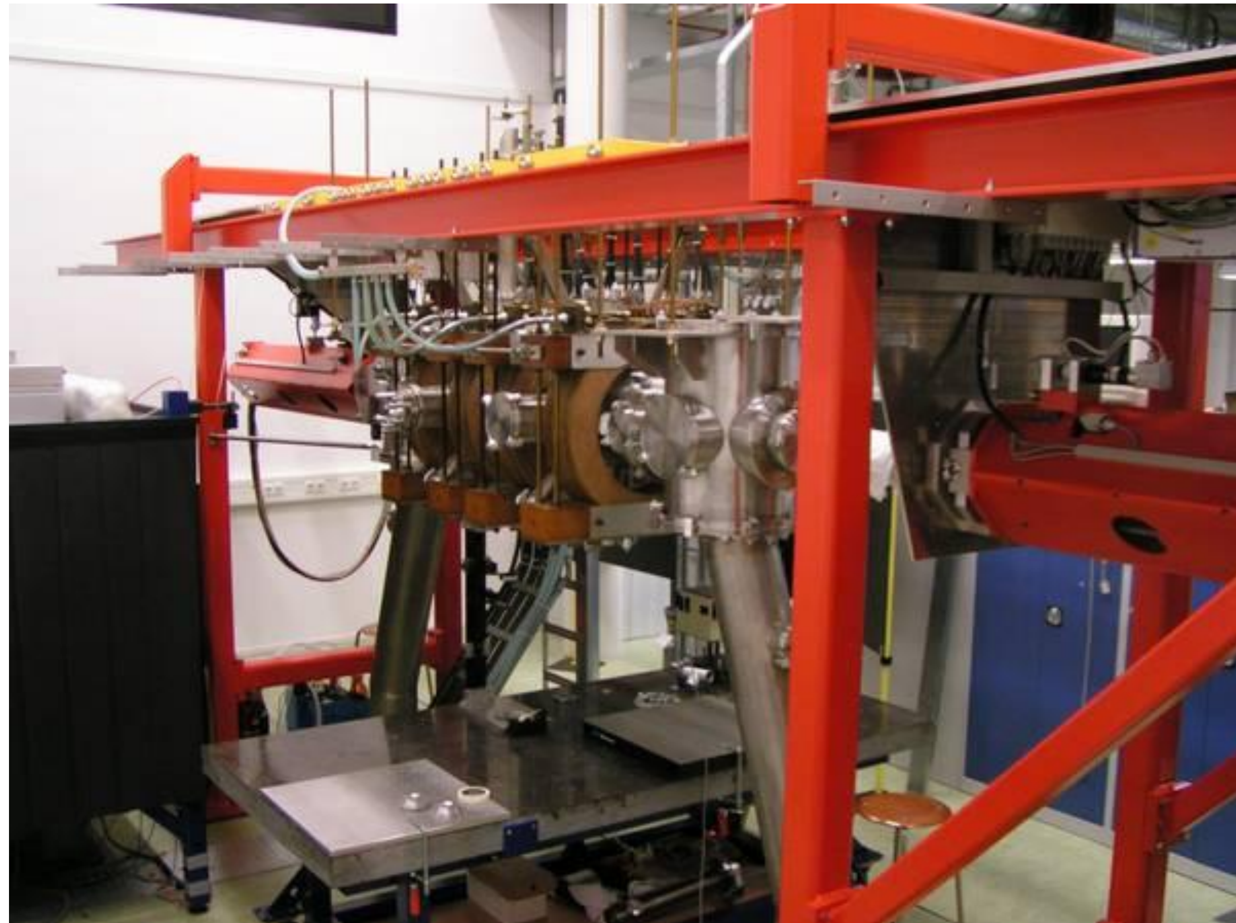
- an expanding argon plasma (40 scc/s ar, p = 1.0 mbar and I = 45 A) to which 10 scc/s nitrogen is admixed.

3.6. PLEXIS (PLasma EXpansion in Interaction with Surfaces)

- The PLEXIS setup (PLasma EXpansion in Interaction with Surfaces) is fully equipped to study a plasma expansion, originating from a cascaded arc. As you can see in the picture below, the setup is relatively big: the internal volume is 550 liter. Many optical access ports are available at different parts of the reactor.
- A plasma can be created from Ar, N2, H2 or mixtures of these gases; these and various other gases can be admixed into the reactor. A double pumping system is used to pump the vessel down to a pressure of usually 20 Pa. A Nd:YAG laser combined with a dye laser can be used for LIF, VUVLIF and TALIF measurements on various species in the plasma reactor. Also OES and a mass spectrometer are installed permanently on the reactor.

3.6. PLEXIS (PLasma EXpansion in Interaction with Surfaces)

- Special features are the moveable plasma source and substrate, to investigate e.g. density profiles of various species at fixed settings of optical diagnostics. Also magnetic coils (brown in the picture below) can be used to constrict a H2 expansion.

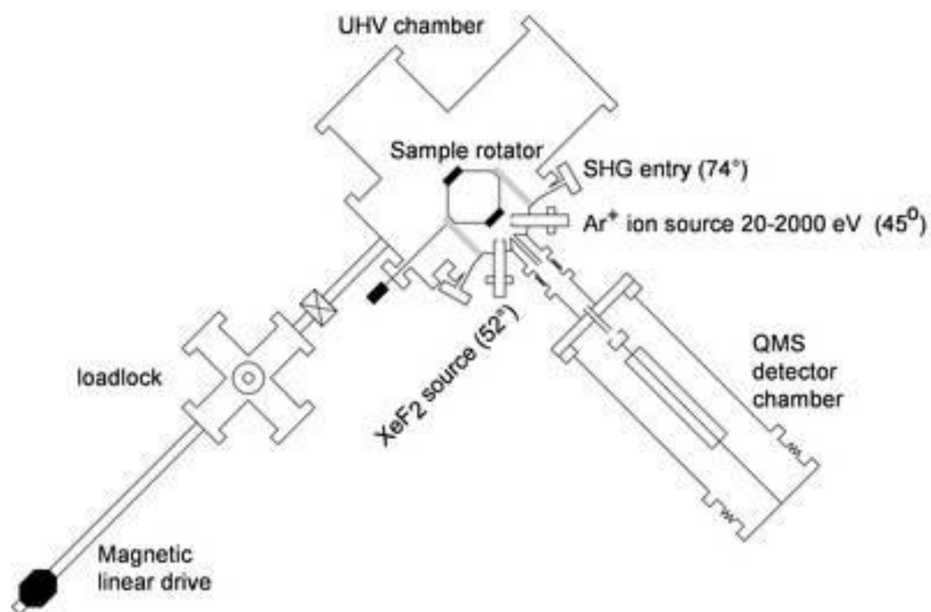


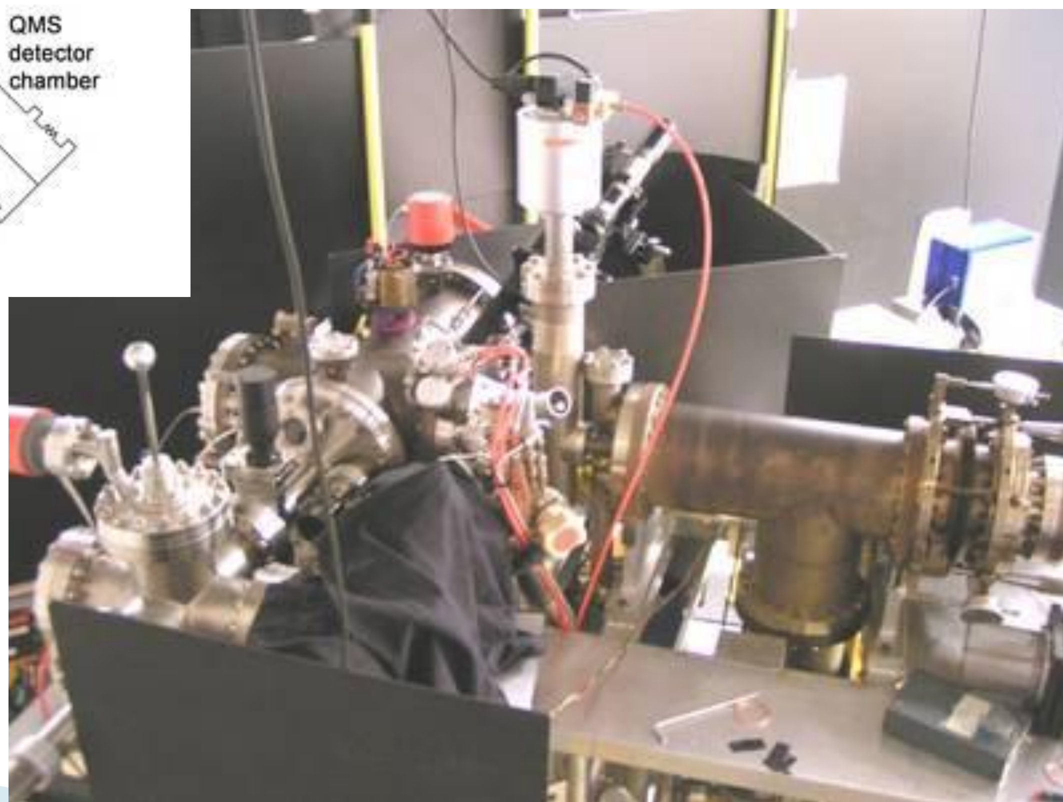
3.7. Beam etching experiment

- Scepter is an acronym for Surface Chemistry Experiment for a Physical Theory of Etch Reactions.
- It is a beam setup consisting of a 10-2000 eV Ar+ ion beam (custom-build Nonsequitur Technologies) and a XeF2 radical beam.
- The main chamber has a base pressure of below 10-8 mbar and the samples can be loaded from a load lock which can store up to 6 samples at a time.
- Attached to the setup is a modified Balzers Quadrupole Mass Spectrometer and a single wavelength ellipsometer operated by a HeNe laser.

3.7. Beam etching experiment

Diagnostics





Diagnostics

3.8. Substrate Bias Voltage

- Energy and flux of ions impinging the substrate are of key importance for surface processes such as ion-assisted film growth or etching.
- The most frequently used technique to manipulate both the ions energy and their flux towards the substrate is based on the generation of a negative potential, socalled bias potential, on the substrate.
- Positive ions crossing the potential fall are gaining energy up to -e(Vb-Vp), where Vb is the bias potential, Vp the plasma potential and e the elementary charge, which enhances ion-induced surface effects such as atomic scale heating, or at a higher energieseven resputtering (etching) of the growing film.

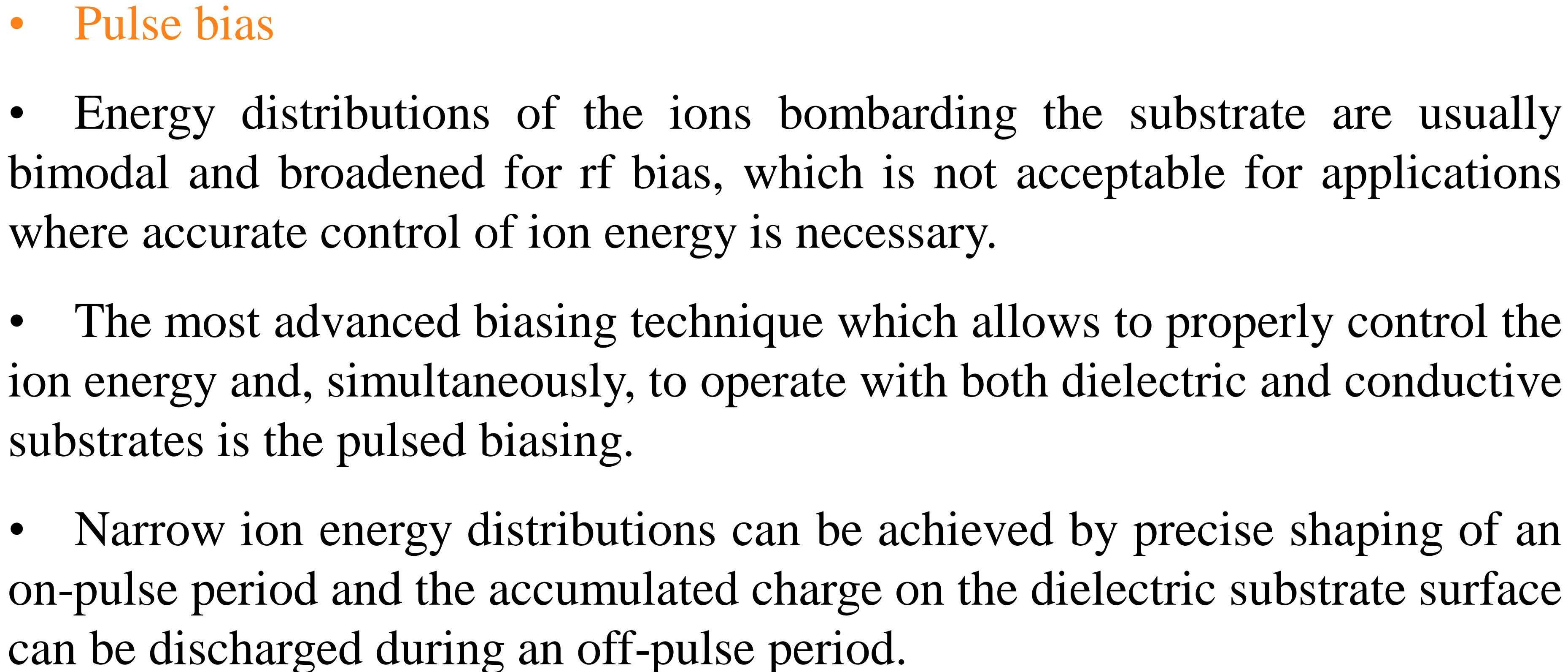
3.8. Substrate Bias Voltage

- Dc and rf bias

- Dc bias is suitable for applications when a conductive material is deposited, but not when dielectric material is deposited. The dielectric layer on the substrate behaves as a capacitor which prevents a direct current to flow. This difficulty can be solved by radio frequency (rf-) bias voltage.

- Due to a higher mobility of electrons compared to ions, a self-rf negative bias potential is developed by connecting the rf power either on the dielectric substrate or on the conductive substrate coupled with a blocking capacitor.

3.8. Substrate Bias Voltage



- Pulse bias

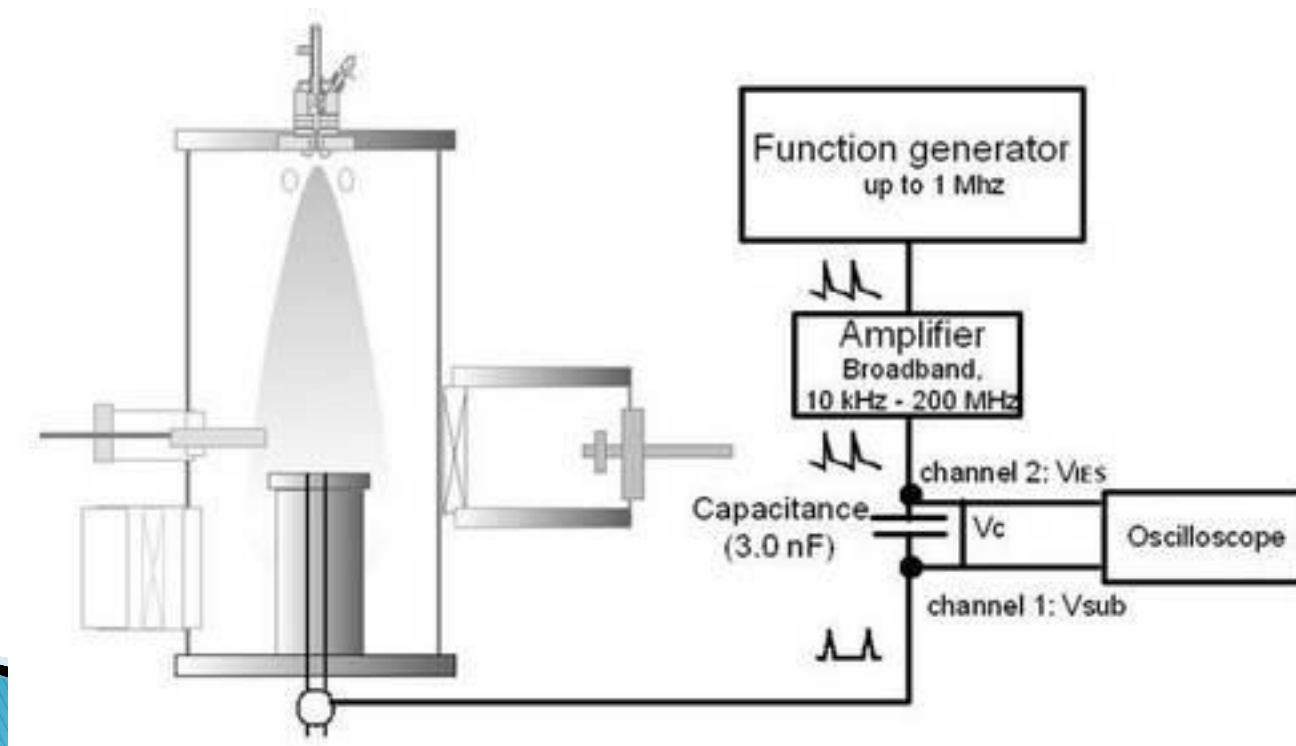
- Energy distributions of the ions bombarding the substrate are usually bimodal and broadened for rf bias, which is not acceptable for applications where accurate control of ion energy is necessary.

- The most advanced biasing technique which allows to properly control the ion energy and, simultaneously, to operate with both dielectric and conductive substrates is the pulsed biasing.

- Narrow ion energy distributions can be achieved by precise shaping of an on-pulse period and the accumulated charge on the dielectric substrate surface can be discharged during an off-pulse period.

3.8. Substrate Bias Voltage

- Pulse bias
- Schematic diagram of the Pulse bias setup.



3.9. The Expanding Thermal Plasma (ETP) technique

- Basics

- The Expanding Thermal Plasma (ETP) technique has been developed in our group at the Eindhoven University [1].

- The ETP setup consists of a high-pressure plasma source and a low-pressure chamber. The plasma source is a cascaded arc. It is operated at high flows (tens of sccs/several slm) of non-depositing gases (Ar, Ar-H2, Ar-N2, H2, N2, etc.) leading to pressures of 0.4 bar (300Torr) when the plasma is ignited. In this plasma source reactive species are created that can be used for downstream precursor gas dissociation for, e.g., plasma deposition of materials or surfacetreatment.

- The discharge in the cascaded arc is current controlled by a dc power supply and thepower dissipated is typically within the 2-5 kW range.

Diagnostics

3.9. The Expanding Thermal Plasma (ETP) technique

- Basics

- The plasma in the arc is thermal with an electron density of ~1022 m-3 (1016 cm-3) and with an electron and heavy particle temperature of both ~1 eV. The high heavy particle temperature leads to almost full dissociation of molecular gases when these are injected in the arc.

- The plasma emanates from the cascaded arc source through a nozzle and expands into the deposition chamber which is typically at a pressure of ~0.2 mbar (~0.15 Torr).

- Due to the large difference in pressure between the arc and the chamber, the plasma is accelerated leading to a supersonic expansion. At a few centimeters from the arc outlet there is astationary shock, after which the plasma expands subsonically.

Diagnostics

3.9. The Expanding Thermal Plasma (ETP) technique

- Basics

- In the supersonic expansion and shock, the electron temperature is reduced to ~0.1-0.3 eV and the electron density to ~1017-1019 m-3 (1011-1013 cm-3). The directed velocity after the shock is typically 1000 m/s.

- Depending on the gas mixtures used, the cascaded arc can deliver large flows of several types of reactive ionic and atomic species such as Ar+, H, N, etc.

3.9. The Expanding Thermal Plasma (ETP) technique

- Fig 1: picture of the Expanding Thermal Plasma setup.

Diagnostics

3.9. The Expanding Thermal Plasma (ETP) technique

- The picture is of the CASCADE system used for the deposition of thin film a-Si:H solar cells and has no coils for the creation of a magnetic field. Typical dimensions of the low-pressure chamber are a length of ~60-80 cm and a diameter of 32-50 cm. An injection ring for precursor gases is located at ~5 cm from the cascaded arc outlet while a substrate holder is typically placed 35-50 cm from the outlet.

- During processing the system is pumped by a stack of roots blowers (pumping capacity 500-1500 m3/hr), and overnight it is pump by a turbo pump reaching a typical base-pressure of $\sim 10^{-6}$ mbar (~10-6 mTorr).

3.9. The Expanding Thermal Plasma (ETP) technique

Diagnostics

- Fig 2: The supersonic expansion region of the expanding thermal plasma for an Ar-H2 plasma. At approximately 5 cm from the arc outlet the precursor injection ring is visible.

3.9. The Expanding Thermal Plasma (ETP) technique

- The cascaded arc plasma source
- The cascaded arc is a dc plasma source operated at non-depositing gases and at high pressures (typically ~400 mbar or ~300 Torr). It is current controlled at 25-90 A with a corresponding operating voltage of 70-250 V, depending on the gas mixture used.
- The discharge is sustained in a narrow plasma channel (diameter typically 3-4 mm) between three cathodes and a grounded anode.

3.9. The Expanding Thermal Plasma (ETP) technique

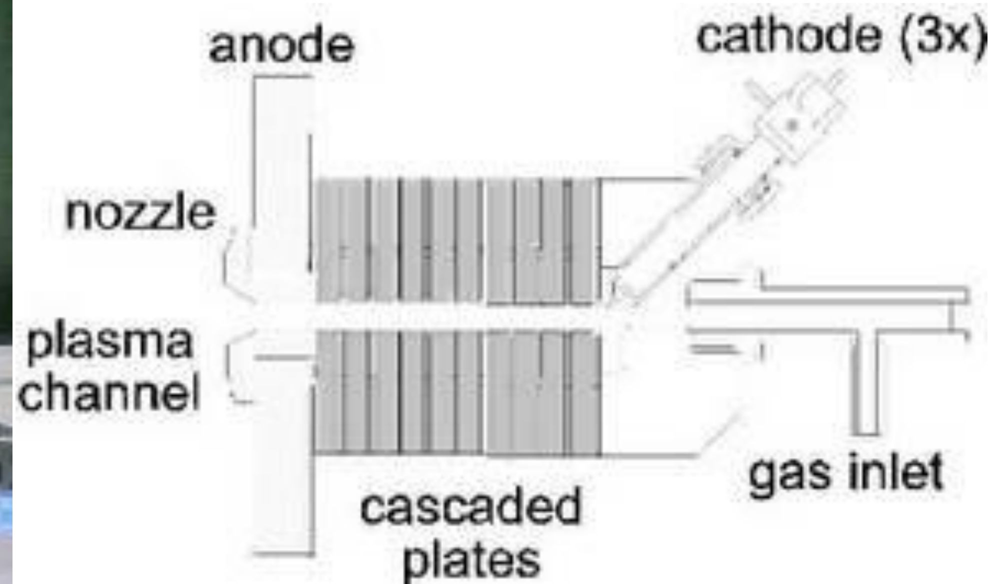
- The cascaded arc plasma source

- The electrically insulated cascaded plates lead to a gradual potential drop. All parts are of copper, except for the cathode tips (tungsten with 2% lanthanum), the PVC and boro nitride spacers between the cascaded plates, and the boro nitride cover of the cathode feed through.

- The arc is vacuum-sealed with O-rings and the cathode, anode, and plates are all water-cooled. The length of a cascaded arc with ten plates is typically 10 cm. The companies OTB Solar and General Electrichave developed commercial versions of this plasma source, using, e.g., molybdenum instead of copper.

3.9. The Expanding Thermal Plasma (ETP) technique

- Figure and picture of a cascaded arc plasma source.



3.9. The Expanding Thermal Plasma (ETP) technique

- Plasma processing with the ETP technique

- One of the main applications of the ETP technique so far is plasma deposition of thin films of several materials. This is usually done by injecting precursor gases by means of an injection ring 5 cm from the cascaded arc outlet.

- The injected precursor gas is ionized and dissociated by the reactivespecies emanating from the plasma source. Deposition can subsequently take place by the growth precursors at a temperature-controlled yoke and substrate holder (-50 ^{o}C - 500 ^{o}C).

Diagnostics

3.9. The Expanding Thermal Plasma (ETP) technique

- Plasma processing with the ETP technique

- The substrates are accurately temperature-controlled by means of a He back flow. The substrates and substrate holder are loaded into the deposition chamber from a loadlock system by means of a magnetic transfer arm. Currently, 4 ETP setups are available in our group: 2 for fundamental plasma studiesand 2 for deposition (Depo 1 and Depo 2) and related plasma studies.

- Two other ETP deposition setups are also directly used by our group, one in cooperation with DIMES at the Delft University of Technology (the CASCADE system) and one at the physics department of TNO in Eindhoven (TNOTPD).

3.9. The Expanding Thermal Plasma (ETP) technique

- Plasma processing with the ETP technique

- Furthermore, some ETP setups are currently used in industry, e.g., by General Electric and OTB Solar (with commercial ETP-based systems shipped to solar cell manufactureres such as Shell Solar and Sunpower). An overview of the materials deposited by the ETP is given in the table below.

Deposition systems

Materials	Gas mixture	Deposition system
a-C:H	Ar-C2H2(CH4/C2H2)	Depo 1
a-CNX	Ar-N2-graphite / Ar-N2-C2H2	Depo 1
SiOX	Ar-O2-HMDSO/D4	Depo 1
a-Si:H/nc-Si:H	Ar-H2-SiH4 (Si2H6)	Depo 2 and CASCADE
a-SiNX:H	Ar-H2-N2-SiH4 / Ar-H2-NH3-SiH4	Depo 2
ZnO:Al	Ar-O2-DEZ-TMA	TNO-TPD
Sieteling	Ar-SF6	Depo 1