



بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

السلام عليكم ورحمة الله وبركاته

Instrumentation Techniques II

Topics

- 1. ITO glass-substrates**
- 2. Cleaning method of glass and ITO- glass-substrates for deposition of thin films**
- 3. Spin coating technique for deposition of thin films**
- 4. Preparation the solutions for deposition of thin films**
- 5. Using the inert atmosphere for sensitive-organic thin films (glove box)**
- 6. Thermal Evaporation System for deposition of metal thin films**
- 7. Solar cell characterization**
- 8. Spectral Response System for solar cells**

ITO glass-substrates

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What is Indium Tin Oxide (ITO) on Substrates?

- Indium tin oxide (ITO) on substrates refers to a thin film coating that combines indium oxide (In_2O_3) and tin oxide (SnO_2) deposited onto a surface.
- Indium tin oxide (ITO) is a ternary composition of indium, tin and oxygen in varying proportions.
- Depending on the oxygen content, it can be described as either a ceramic or an alloy.
- Indium tin oxide is typically encountered as an oxygen-saturated composition with a formulation of 74% In, 8% Sn, and 18% O by weight. Oxygen-saturated compositions are so typical that unsaturated compositions are termed oxygen-deficient ITO. It is transparent and colorless in thin layers, while in bulk form it is yellowish to gray. In the infrared region of the spectrum it acts as a metal-like mirror.
- ITO is highly transparent to visible light, making it suitable for applications that require transparency, like displays.
- Additionally, it has good electrical conductivity, enabling it to function as a transparent conductor. This property is crucial for touch-sensitive screens where electrical signals must pass through the transparent layer to detect touch input.
- Its ratio controls transmittance, which increases with increasing the percentage of indium.

What is Indium Tin Oxide on Substrates?

Indium Tin Oxide (ITO) substrates are used in a wide range of research applications, especially in the fields of **optoelectronics** and **micro/nanoelectronics**. Here are some common ways researchers use ITO substrates:

- **Transparent Conductive Coating:** ITO is commonly used as a transparent conductive coating for electronic devices that require a clear or opaque surface. The high transparency and electrical conductivity of ITO make it ideal for applications
- **Electrode Material:** ITO is a popular choice for use as an electrode material in various electrochemical applications, such as **biosensors**, **fuel cells**, and **batteries**. The high electrical conductivity and stability of ITO make it an attractive option for these types of applications.
- **Micro/Nano Fabrication:** Researchers can use ITO substrates as a base material for micro/nano fabrication techniques such as **photolithography** طباعة حجرية ضوئية, **electron beam lithography**, and **nanolithography**. These techniques allow researchers to create complex patterns and structures on ITO substrates that can be used for a wide range of applications.

Properties of Indium Tin Oxide

- Indium tin oxide is one of the most widely used transparent conducting oxides because of
 1. its electrical conductivity
 2. its optical transparency,
 3. the ease with which it can be deposited as a thin film, and
 4. its chemical resistance to moisture.
- As with all transparent conducting films, a compromise must be made between conductivity and transparency, since increasing the thickness and increasing the concentration of charge carriers increases the film's conductivity, but decreases its transparency.
- Its ratio controls transmittance, which increases with increasing the percentage of indium.

Fabrication of ITO Thin Films

- The deposition process for ITO involves techniques like physical vapor deposition (PVD) or sputtering, where a target consisting of indium and tin is bombarded with ions in a vacuum chamber. This results in the ejection of atoms from the target, which then condense onto the glass substrate, forming a thin, uniform layer of ITO.
- ITO-coated glass substrates offer a balance between optical transparency and electrical conductivity, making them ideal for applications where both properties are essential.
- By applying a voltage to the ITO layer, the conductivity allows for the flow of electrical current while still permitting light transmission.

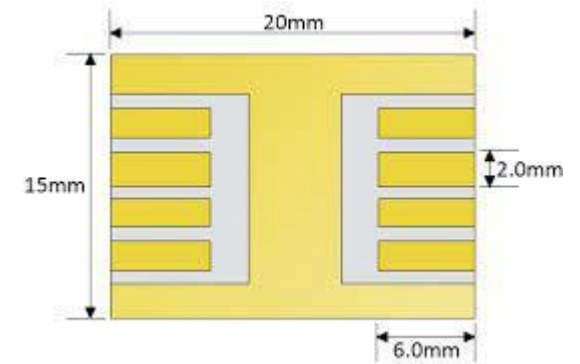
Applications of ITO

➤ It is commonly used in various electronic devices, such as:

1. flat-panel displays,
2. touchscreens,
3. and solar cells,
4. liquid crystal displays (LCDs),
5. biosensors,
6. fuel cells,
7. Batteries,
8. optical waveguides,
9. flat panel displays,
10. IR-mirrors,
11. optical filters, and
12. micro-optoelectromechanical systems (MOEMS).

Applications of ITO

- **Micro/Nano Fabrication:** Researchers can use ITO substrates as a base material for micro/nano fabrication techniques such as **photolithography** طباعة حجرية ضوئية, **electron beam lithography**, and **nanolithography**. These techniques allow researchers to create complex patterns and structures on ITO substrates that can be used for a wide range of applications.

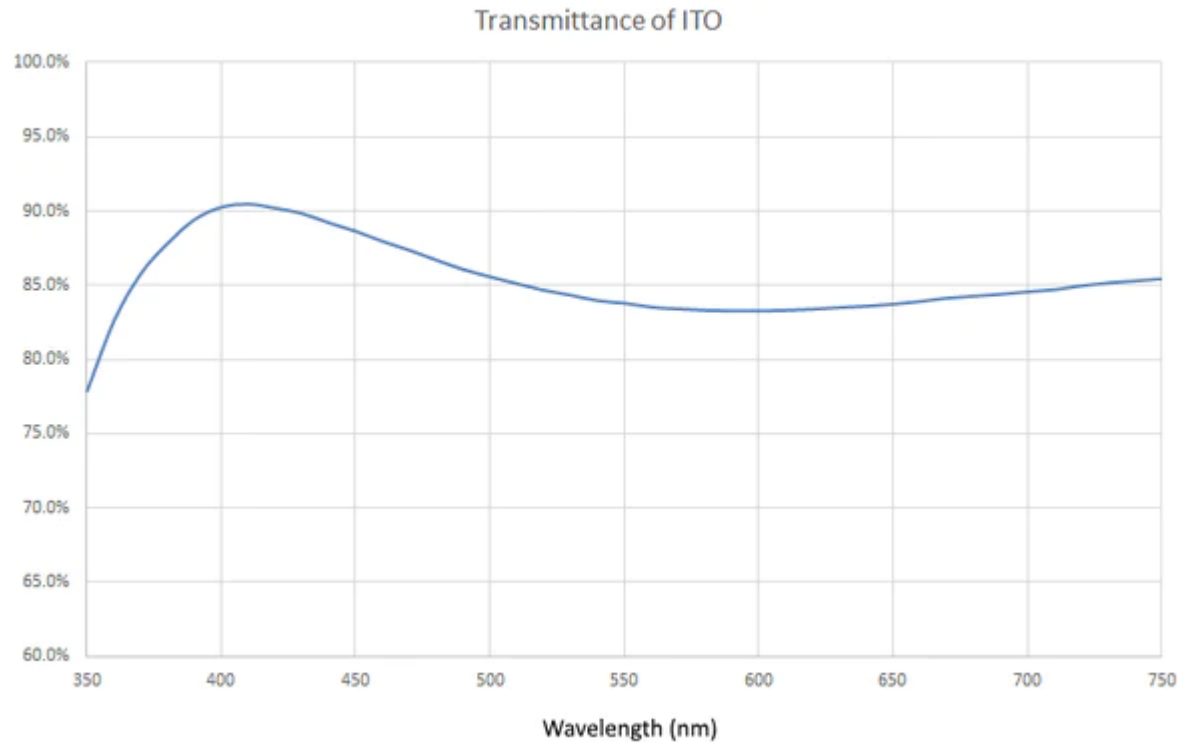


ITO patterns

Disadvantages of ITO

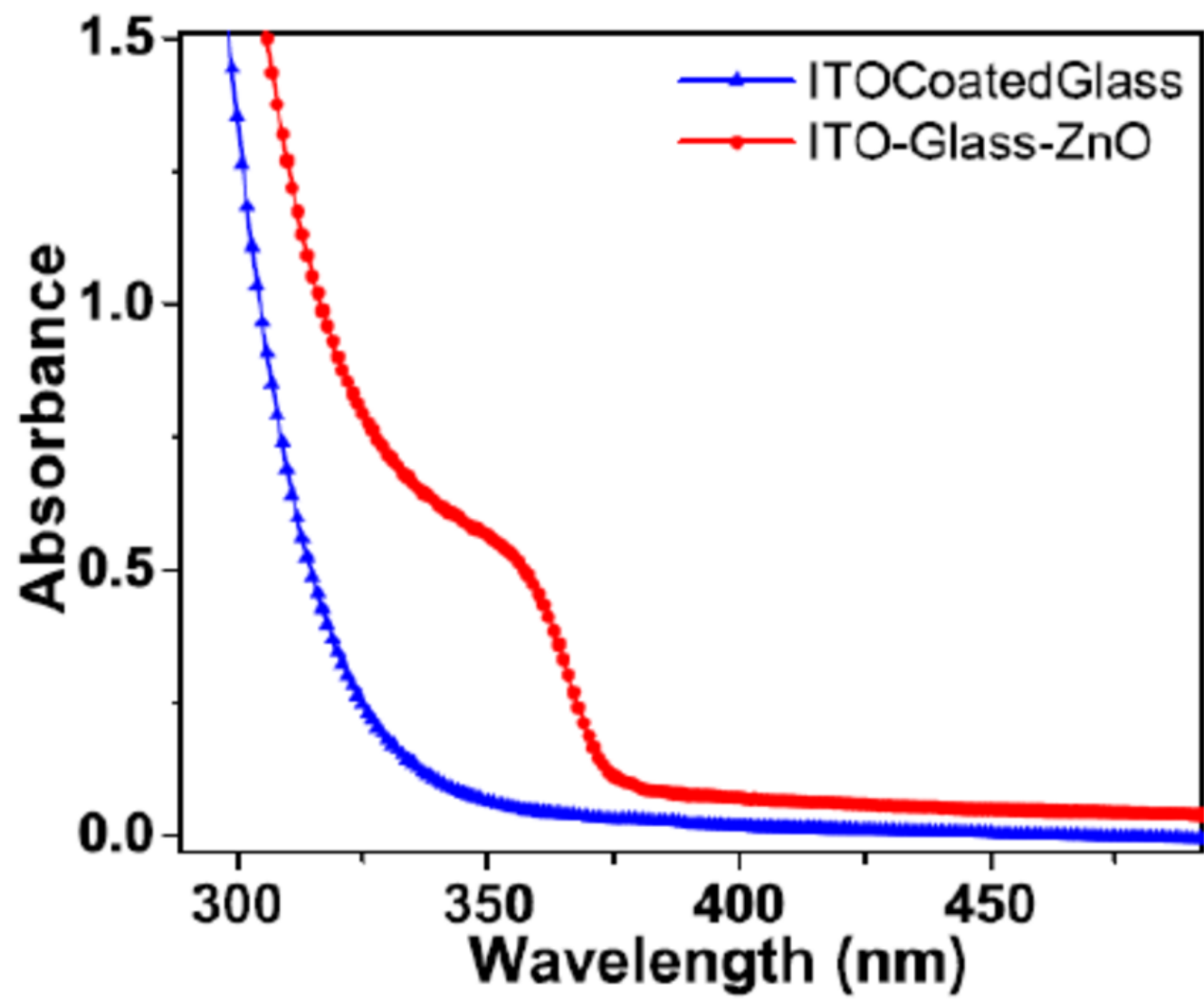
- It's worth noting that while ITO has been widely used, there is ongoing research to explore alternative transparent conductive materials that could potentially replace ITO due to its scarcity and brittleness.

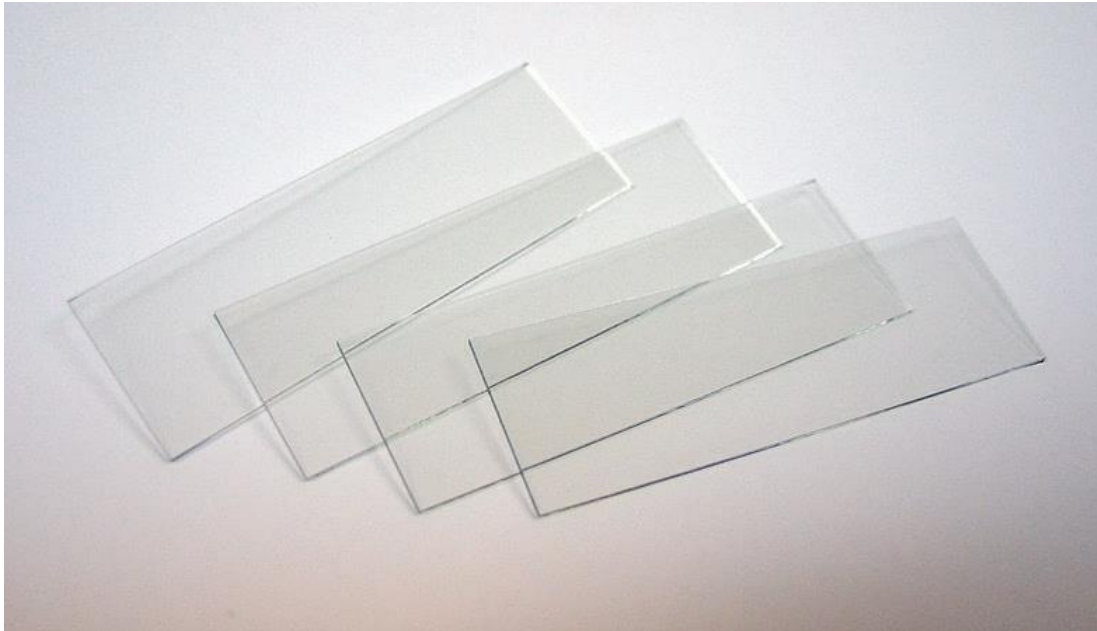
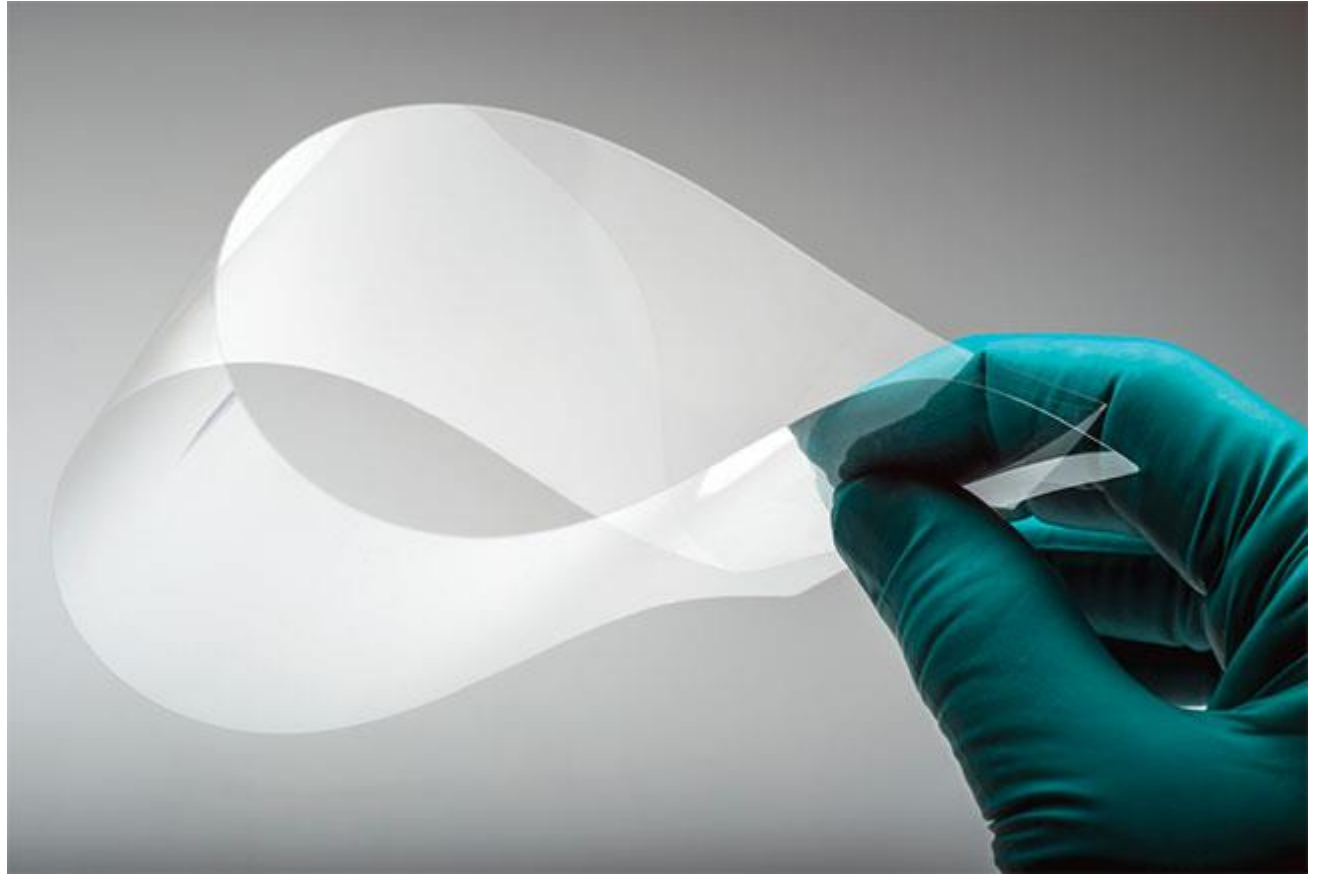
Transmittance of ITO Substrates

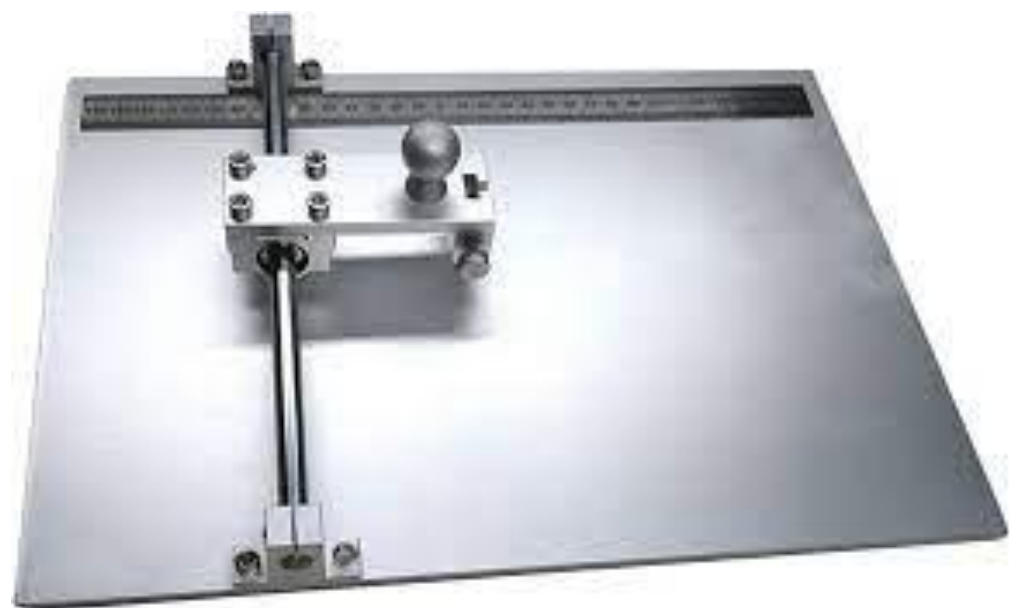


Specifications

Substrate sizes	20mm x 15mm 25mm x 25mm 25mm x 75mm (Microscope Slide)
Thickness	1.1 mm
Glass type	Polished soda lime, float glass
Substrate coating	Fully oxidized ITO
ITO thickness	100 nm
ITO quoted resistance	$20 \Omega \cdot \square^{-1}$
ITO measured resistance*	14 - 16 $\Omega \cdot \square^{-1}$
Glass roughness	1 nm RMS (by AFM)
ITO roughness	1.8 nm RMS (by AFM)







Because of its excellent conductivity and transparency, indium tin oxide has become a popular material for the production of thin films. It is also used for glass windows, EMI shielding, and solar cells. Its ratio controls transmittance, which increases with increasing the percentage of indium. When applied to an electronic device, ITO films are particularly useful. These materials are used in electronics. In addition to being an excellent transparent electrode, indium tin oxide film is effective for solar cells and other devices.

Indium tin oxide is a ternary compound of indium and tin, and is a semiconducting material. Its optical properties make it an ideal choice for high-tech applications such as flat panel displays and IR-mirrors. Its low surface resistance and high film thickness makes it a great material for optical devices. Its conductive qualities make it a popular material for electronic and scientific products.

Indium tin oxide is an excellent transparent conductor and is used in many types of electronic and scientific devices. This tertiary alloy is composed of indium and tin, and is therefore colourless, transparent, and yellow. Its excellent properties make it a highly popular material. It also has excellent electrical and thermal conductivity, and is used in high-performance electronics and in electronics.

What is indium tin oxide? This material is a transparent conducting metal that is both electrically and optically transparent. It is a highly versatile material, which makes it a useful material for electronic devices. It is also a valuable metal in electronics. In addition to being a good conductor, indium tin oxide also has many other uses in the electronics industry. In addition to this, indium tin oxide is a high-quality source of copper, which can help save money on electricity.

Its high transparency is beneficial in many applications, including medical devices. It allows visible light to pass through while reflecting ultraviolet rays. It is used in glass windows, and is also used in aeroplane windshields. In addition to electrical conductivity, indium tin oxide is a versatile material. In addition to conductive electronics, indium tin oxide is used in organic light-emitting diodes and electroluminescent lamps.

Indium tin oxide is an important metal, containing tin, indium, and oxygen. Its high transparency makes it a very desirable material for electronics. Its high optical properties make it an important metal for many other applications. It is a widely used material in liquid crystal displays, touch screens, plasmas, and LCD monitors. Further, it is used as an anti-static coating.

ITO is an alloy of indium, tin, and oxygen. Its electrical conductivity makes it an important material in the optoelectronics industry. It is used to produce displays and other products that use indium tin oxide. It is also used in solar cells and heated defrosting coatings. Its transparent structure makes it an excellent material for LEDs and other electronic equipment.

Indium tin oxide is used in optics and microelectronics. Its optical properties make it an important material for high-performance solar cells. Its name is derived from the Latin word *indicum*, which means "violet". The material is a popular material in optical systems and semiconductors. The market for indium tin oxide is expected to grow at a rate of 2.8% between the years of 2021 and 2026.

Overall, the high electrical conductivity, optical transparency, and other desirable physical properties of ITO make it a versatile material that researchers can use in many different ways, both in fundamental research and in applied technology.

At What Temperature Do ITO Glass Wafers Start Degrading?

A Phd candidate asked for the following quote:

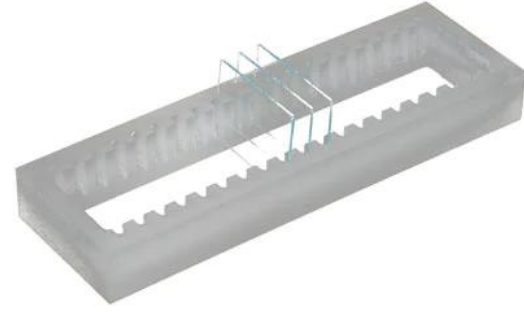
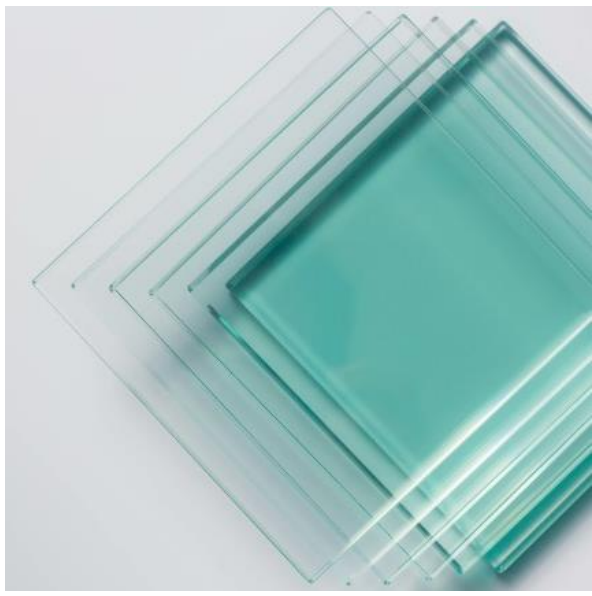
"ITO Glass take a temp of more than 90C?"

Indium tin oxide (ITO) glass can withstand temperatures up to around 200°C, but its electrical and optical properties can degrade above 90°C. The exact temperature limit for ITO glass depends on various factors such as the composition, thickness, and deposition process, among others. It is best to consult the manufacturer's specifications for more information on the temperature tolerance of a specific ITO glass product.

Cleaning method of glass and ITO- glass-substrates for deposition of thin films

ITO glass supplier (Xinyan Technology, www.xinyan.hk) suggest the below cleaning procedures:

- 1. ITO is swabbed under a diluted detergent (e.g. Deconex or FPD detergent, Alconox or soap).**
- 2. Rinse with DI water.**
- 3. Sonication with DI water for 10min.**
- 4. Sonication with acetone for 10min.**
- 5. Sonication with ethanol for 10min,**
- 6. Nitrogen blow dry**
- 7. UV ozone or Oxygen-plasma treatment for 20 min.**



Cleaning of ITO-Glass Substrates

Cleaning inside Fume Hood

Flores Valles

INSTRUCCIONES DE MANEJO DE LA VENTANA DE GASES
FUME HOOD OPERATING INSTRUCTIONS

- 1 Compruebe el indicador de caudal para verificar que la vitrina funciona correctamente.
Check air flow monitor to verify that the fume hood is operating properly.
- 2 Before de la vitrina todo el material de laboratorio que se usará en el experimento.
Remove all materials not needed for the immediate work.
- 3 Use appropriate safety precautions in emergencies.
Use basic cabinet for reagent storage.
- 4 Limpie inmediatamente cualquier derrame.
Clean spills immediately.
- 5 Mantenga la gualletina totalmente cerrada cuando no trabaje en el interior de la vitrina.
The hood should remain closed when the hood is not in use.
- 6 Siempre que sea posible, utilice la gualletina como pantalla protectora.
Use the hood as body shield whenever appropriate.
- 7 Si suena la alarma, cierre totalmente la gualletina y pida ayuda.
If alarm sounds, close completely the hood and contact safety representative immediately.

Remote Dispenser
for Type I Water

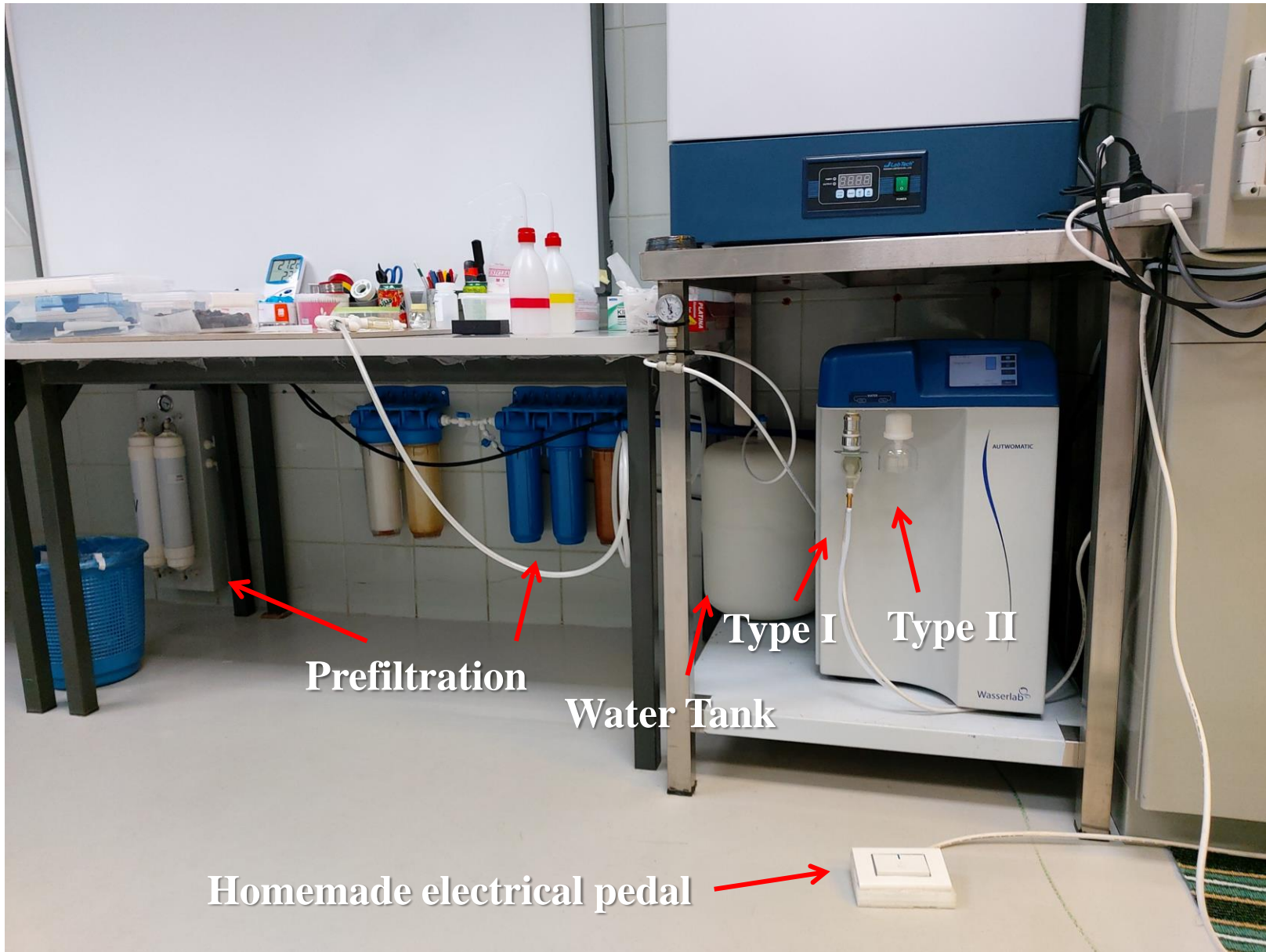
Ultrasonic
Cleaner

Digital Hotplate
Stirrer

Ethanol &
Methanol



Water Purification System (for Type I & II water)

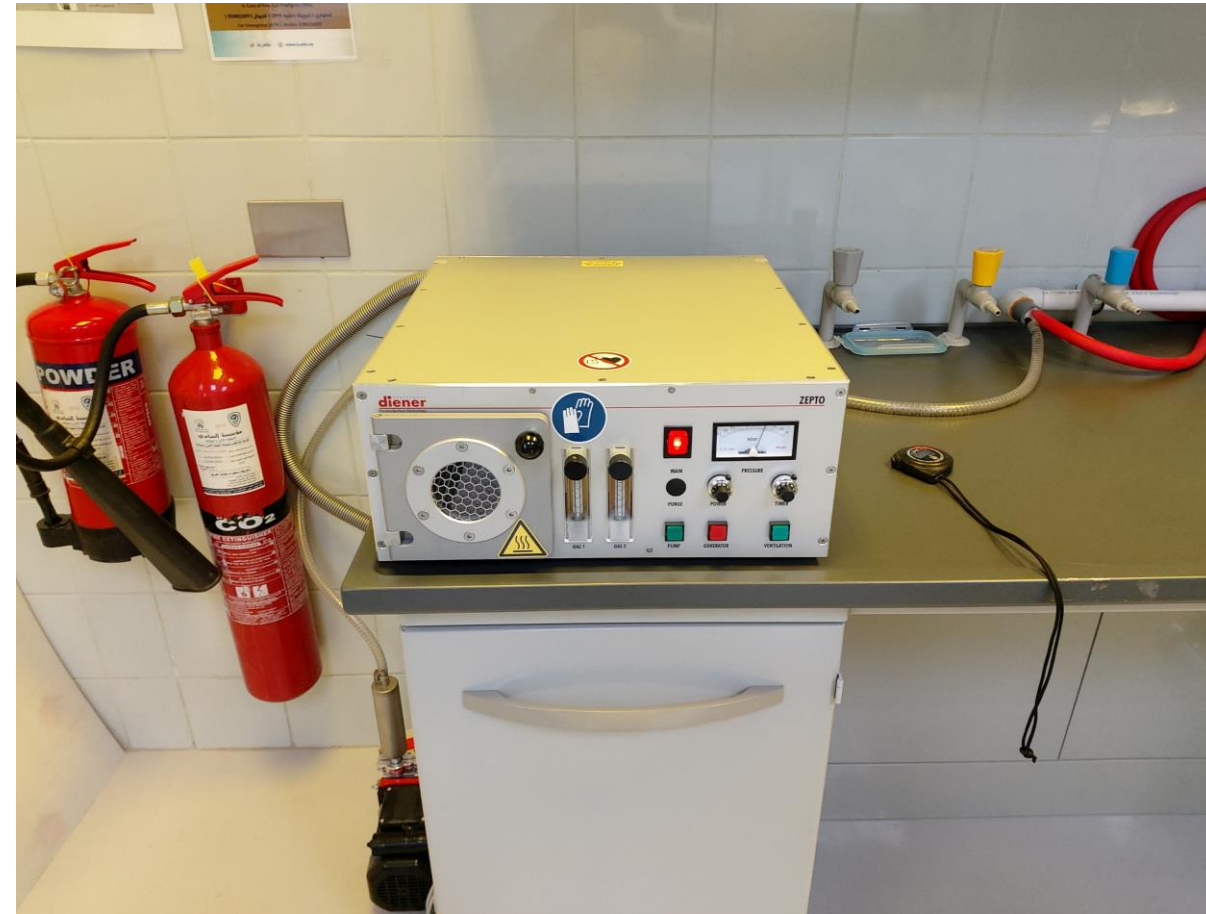


Cleaning of ITO-Glass Substrates

Vacuum Oven



Plasma Cleaner



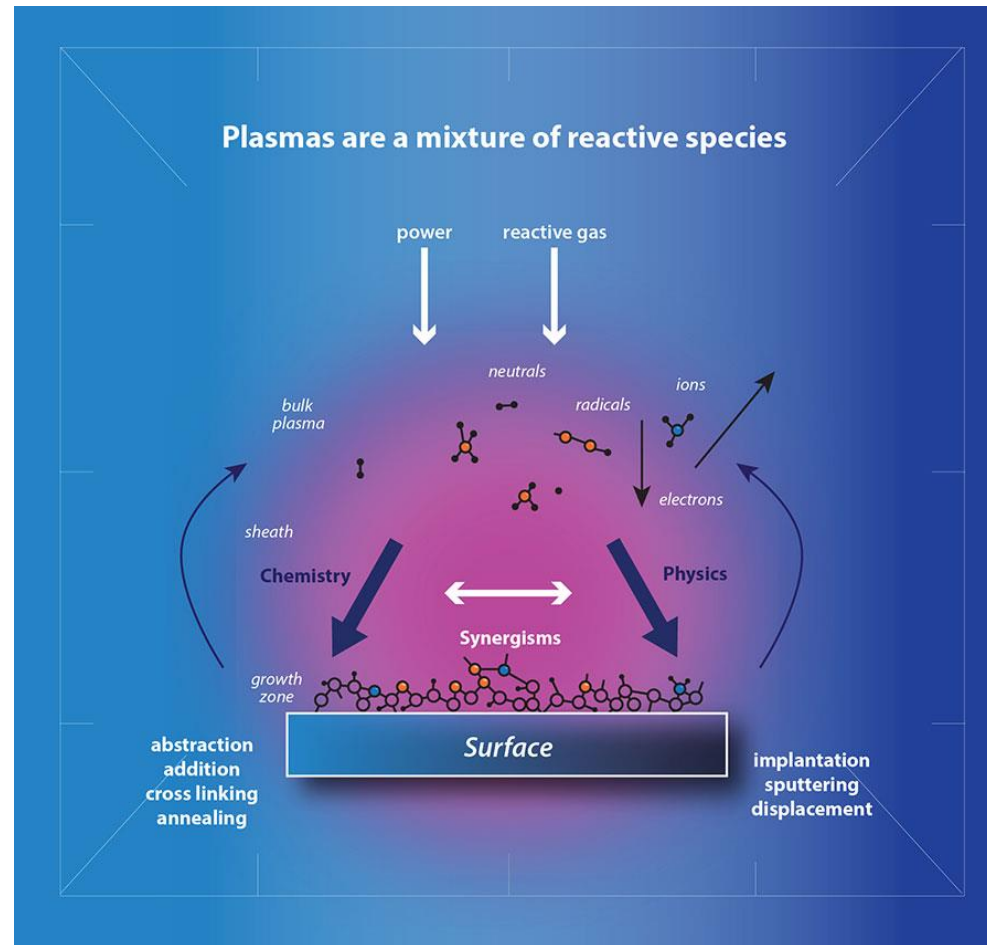
Fume Hood for Cleaning of ITO-Glass Substrates



Chemicals for Cleaning

What is plasma?

Plasma can be described as “the fourth state of matter” because it is not a liquid, a solid, or a gas. Plasma exists in the form of ions and electrons. It is essentially an ionized gas that has been electrified with extra electrons in both negative and positive states.



- A plasma contains positive ions, electrons, neutral gas atoms or molecules, UV light and also excited gas atoms and molecules, which can carry a large amount of internal energy (plasmas glow because light is emitted as these excited neutral particles relax to a lower energy state).
- All of these components can interact with the surface during plasma treatment. By choosing the gas mixture, power, pressure etc. we can quite precisely tune, or specify, the effects of the plasma treatment.

What is Oxygen-plasma treatment?

- It is the process by which gas (oxygen O_2) is ionized by electric field in a vacuum chamber to form plasma and alter the surface of ITO.
- Plasma treatments are used to alter the surface properties of a wide range of materials to make them easier to bond, glue and paint. By treating substrates, we both clean and activate the surface, improving their adhesion characteristics.

Advantages:

- The Oxygen-plasma treatment improves bonding, printing, coatings and primers; this is also known as increased wettability.
- Organic surface contaminants are invisible to the casual observer, but they greatly impact an object's ability to interact with other materials.
- When plasma be treated a surface, we are able to remove these contaminants. This increases the bond strength of a solder or glue, increases wettability.
- The plasma treatment is considered to be the most effective way because it can increase the work function of ITO, produce the dipole layer, and improve the electrical characteristics

The applications:

- It is commonly used in the manufacturing of electronic devices, medical devices, textiles, plastics, rubbers, and more. Nearly any dry material can be treated in a plasma chamber

What is Oxygen-Plasma?

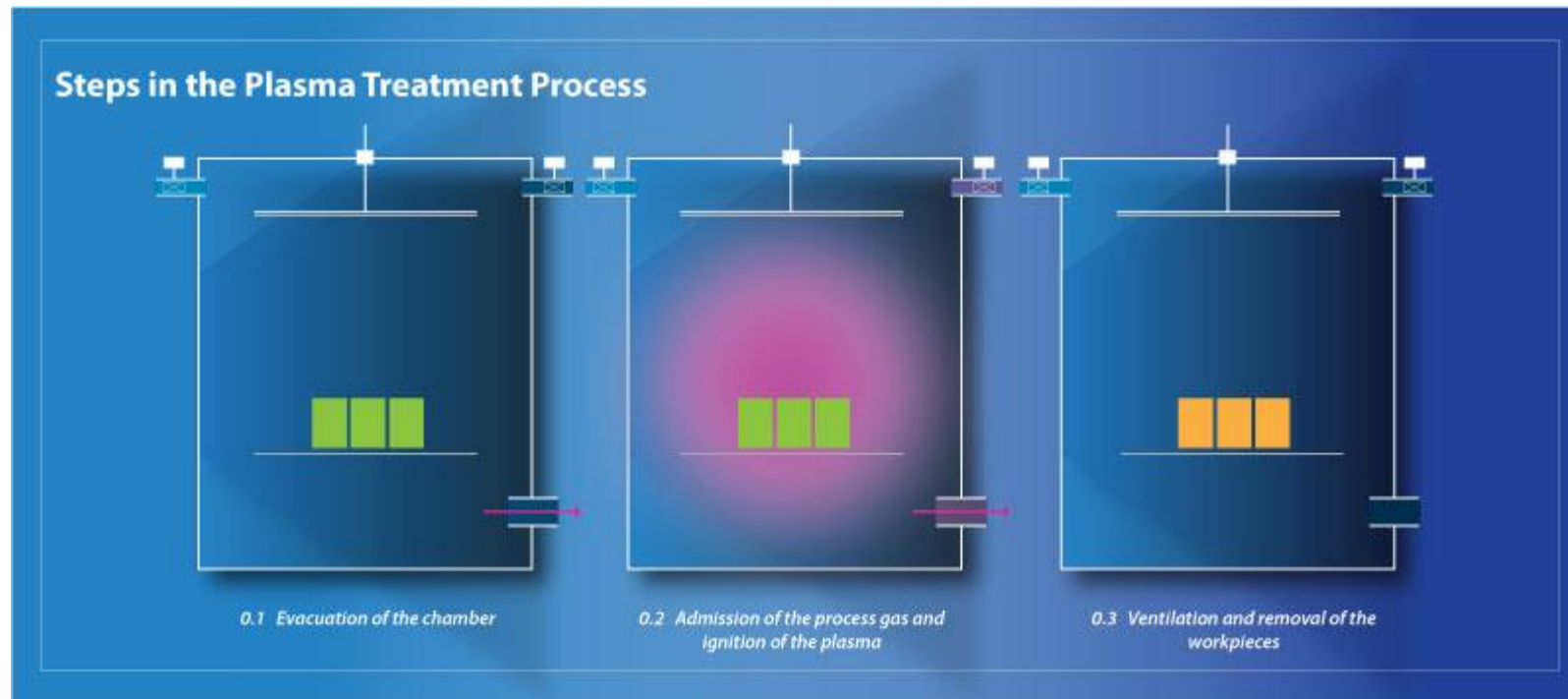
- Oxygen plasma refers to any plasma treatment performed while introducing oxygen to the plasma chamber.
- Oxygen is often used to clean surfaces prior to bonding. It may also be combined with other gases to etch a variety of materials such as plastic and rubber.
- Oxygen is the most common gas used in plasma cleaning technology due to its low cost and wide availability.
- Oxygen plasma is created by utilizing an oxygen source on a plasma system.
- An oxygen cylinder or generator can be used in the lab.
- Oxygen gas is commonly used to clean non-metal materials such as glass, plastics, and Teflon.
- Like other forms of plasma, oxygen cleans organics and is capable of surface modification.
- O₂ plasma will clean the surface of a plastic sample and increase its wettability.
- Oxygen plasma can also be used to clean metal surfaces if it's mixed with Argon.
- Argon gas (as an oxidation preventing gas) strips the oxygen molecule away from the metal to prevent oxidation.

Plasma Ashing with Oxygen

- **Plasma ashing is the process of removing carbon from products during the manufacturing process. Plasma ashing is always performed with oxygen plasma in a high frequency plasma system.**
- **Generally, the goal of plasma ashing is to completely remove organic matter, including volatile carbon oxides and water vapor. All removed contaminants are pumped out of the chamber by the vacuum system.**

How the plasma treatment process works

- ✓ Plasma treatment can be performed in an evacuated enclosure or chamber. The air is pumped out and gas is allowed to flow in at low pressure before the energy in the form of electrical power is applied.
- ✓ It's important to note that the plasma treatment is actually a low-temperature process, meaning that heat-sensitive materials can be processed quite readily.



Common everyday plasmas include:

Fluorescent lights 'Neon' signs, and Plasma televisions of course

➤ COMMON INDUSTRIAL USES, MATERIALS & APPLICATIONS OF PLASMA TREATMENT INCLUDE:

Industries:

- Aerospace
- Automotive
- Contact lenses & optics
- Filter media
- Medical
- Microscopy
- PCB/Electronics assembly
- O-rings
- Plastic/Loyalty cards
- Print
- R & D Academic
- Textiles & Fabrics

Materials most commonly plasma-treated

- Plastics, including PEEK, PTFE, Polypropylene, Polyethylene Composites
- Engineering polymers
- Microfluidics PDMS
- Glass, metals & ceramics

Common results of plasma treatment

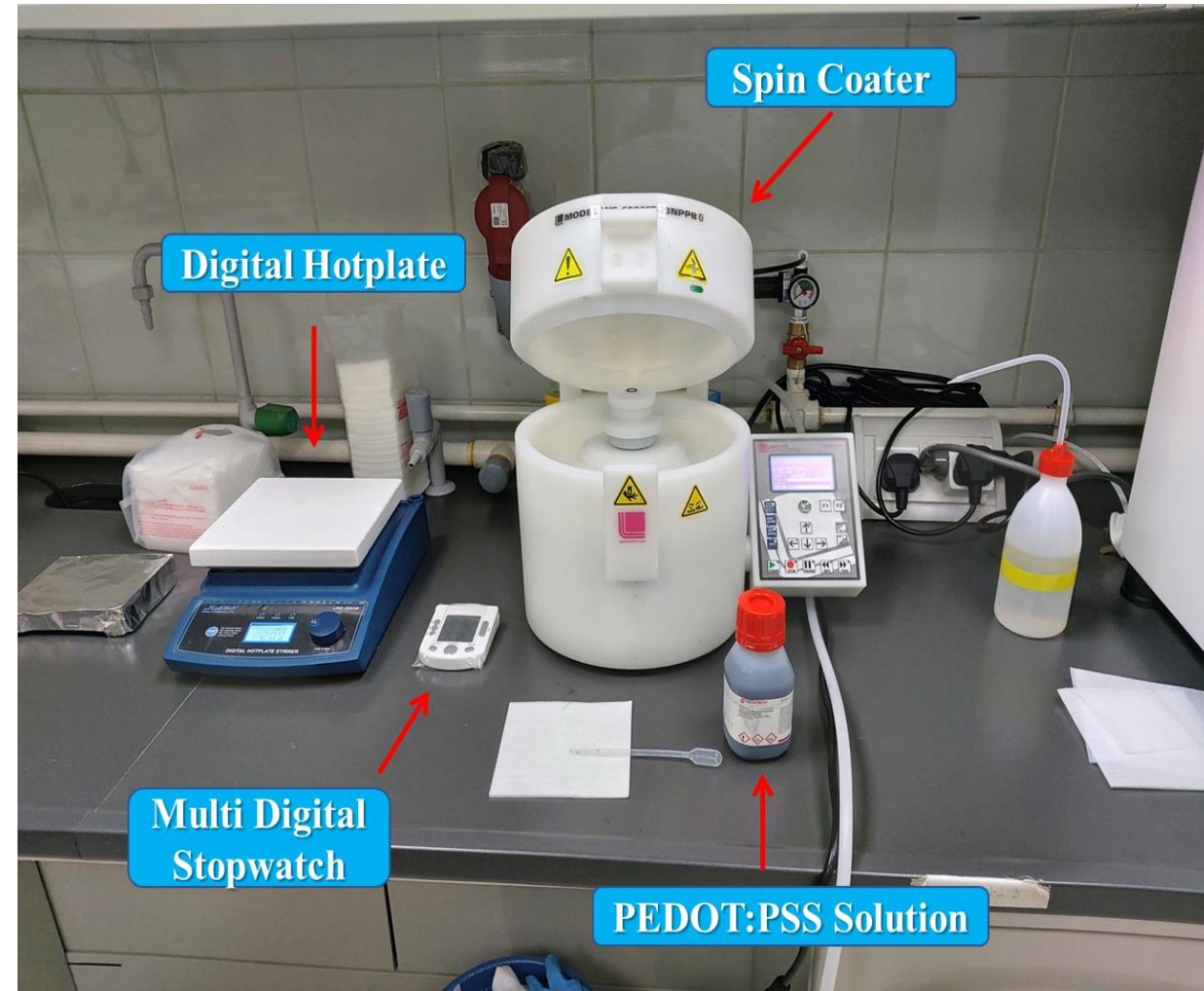
- Plasma surface cleaning
- Plasma surface activation to improve adhesion
- Plasma coatings, hydrophobic & hydrophilic
- Plasma etching

Plasma Treatment Effects

- Plasmas are a 'soup' of energetic, highly reactive species that are able to interact with any surface which they contact. By choosing the right configuration and processing parameters we can produce specific effects upon the surface.
 - ❑ Plasma Treatments can be categorised as
 - ❑ Plasma Cleaning,
 - ❑ Plasma Surface Activation,
 - ❑ Plasma Coatings and
 - ❑ Plasma Etching.

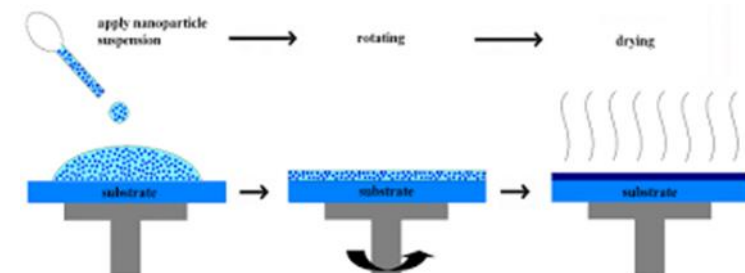
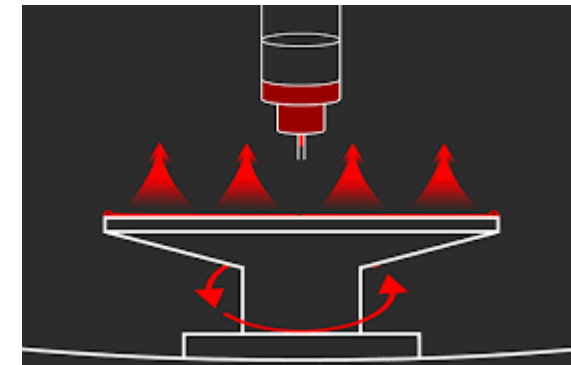
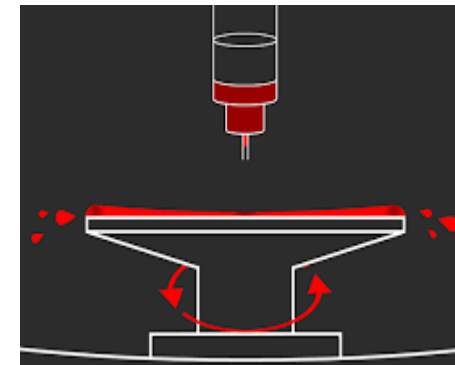
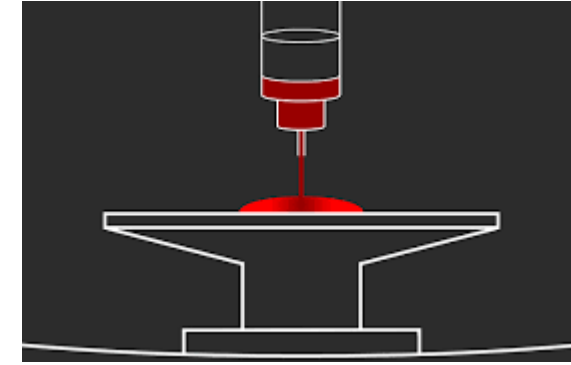
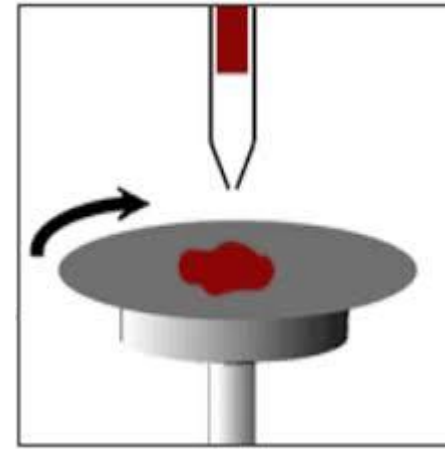
Spin coating technique for deposition of thin films

- **Spin coating** is a procedure used to deposit uniform thin films onto flat substrates.
- Usually, a small amount of coating material or solution is applied to the center of the substrate. The substrate is then rotated at speeds up to 10,000 revolutions per minutes (rpm) to spread the coating material by centrifugal force.
- A machine used for spin coating is called a spin coater, or simply spinner.



Spinning Mechanism

- In general, a cleaned substrate is kept on the chuck of the spin-coating machine. It generates a vacuum below the substrate to fix it in position (labeled as vacuum chuck) and rotates the substrate at the setting speed.
- Due to high centrifugal forces, the solution tends to spread out on the substrate and coat it completely
- The solution or suspension material is dropped from above into the rotating substrate.
- Spin coating utilizes centrifugal forces to deposit the required material on the substrate.



Spinning Mechanism

- Rotation is continued while the fluid spins off the edges of the substrate, until the desired thickness of the film is achieved.
- The applied solvent is usually volatile, and simultaneously evaporates.
- The higher the angular speed of spinning, the thinner the film.
- The thickness of the film also depends on the viscosity and concentration of the solution, and the type of solvent.
- The exact thickness of a film will depend upon the solution concentration and solvent evaporation rate (which in turn depends upon the solvent viscosity, vapor pressure, temperature and local humidity)

The steps involved with spin-coating technique are:

1. Deposition of the precursor solution of the desired material on the substrate.
2. Spin up the substrate at a certain fixed speed for a few seconds.
3. Spin off the substrate.
4. Dry the film layer by evaporating the solvent.
5. Repeat the above process (1)-(4) to get the film of desired thickness.

Spin coater Components

Dispenser for solutions



Sample holder



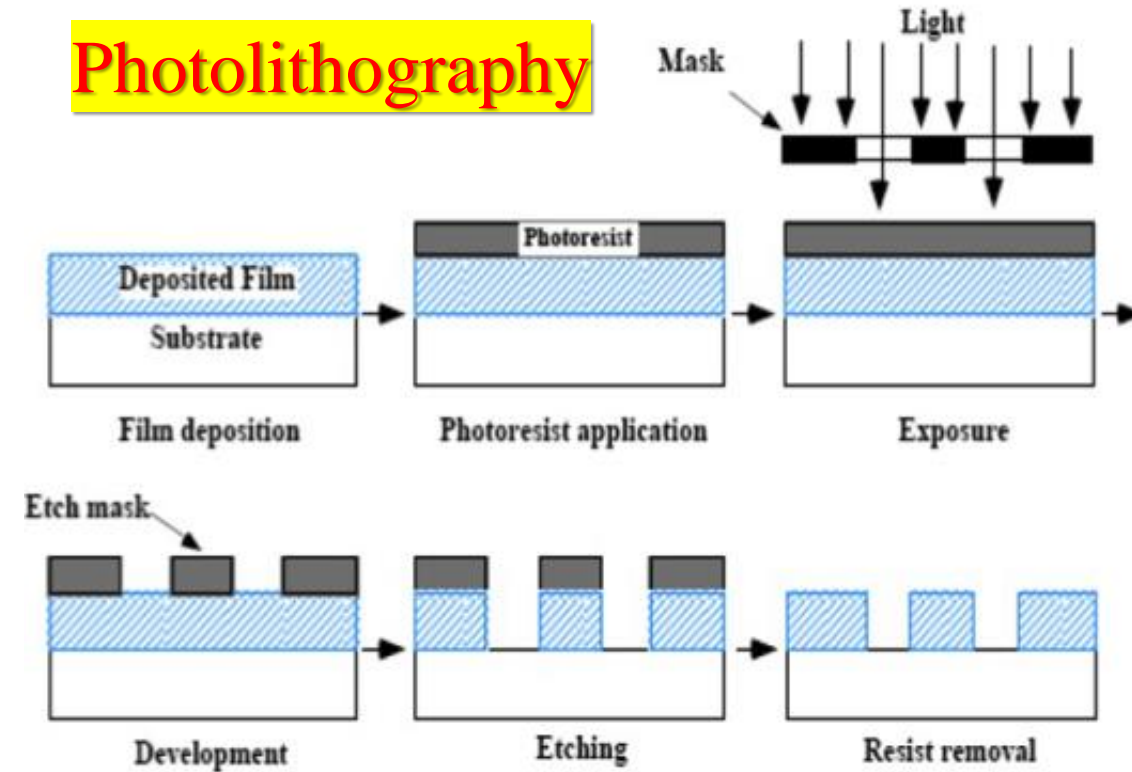
Spin coater

Vacuum pump to hold the substrates in place

Usage

- Spin coating is widely used in microfabrication of functional oxide layers on glass or single crystal substrates, where it can be used to create uniform thin films with nanoscale thicknesses.
- It is used intensively in photolithography, to deposit layers of photoresist about 1 micrometer thick. Photoresist is typically spun at 1200 to 4800 rpm for 30 to 60 seconds.
- It is also widely used for the fabrication of planar photonic structures made of polymers.
- Spin coating thicker films of polymers and photoresists can result in relatively large edge beads whose planarization has physical limits.

Photolithography

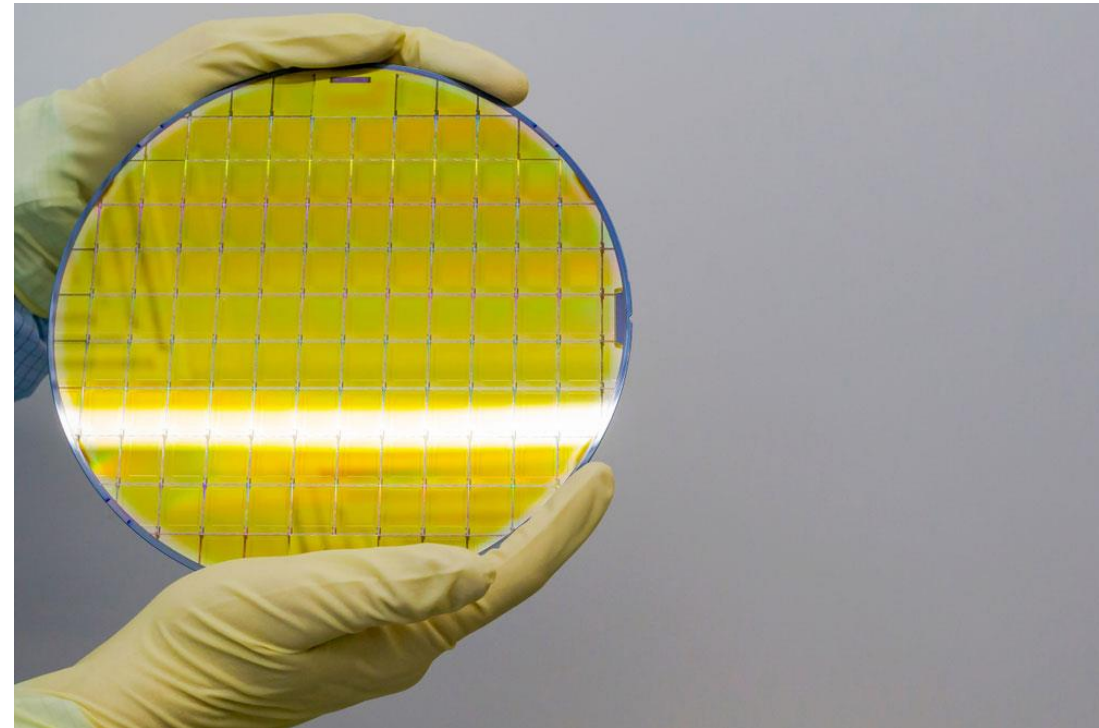


A single masking level.

- Spin-coating technique is currently used by the microelectronics industry since it allows the deposit of smooth films of polymer, thicker than those obtained by LB technique.

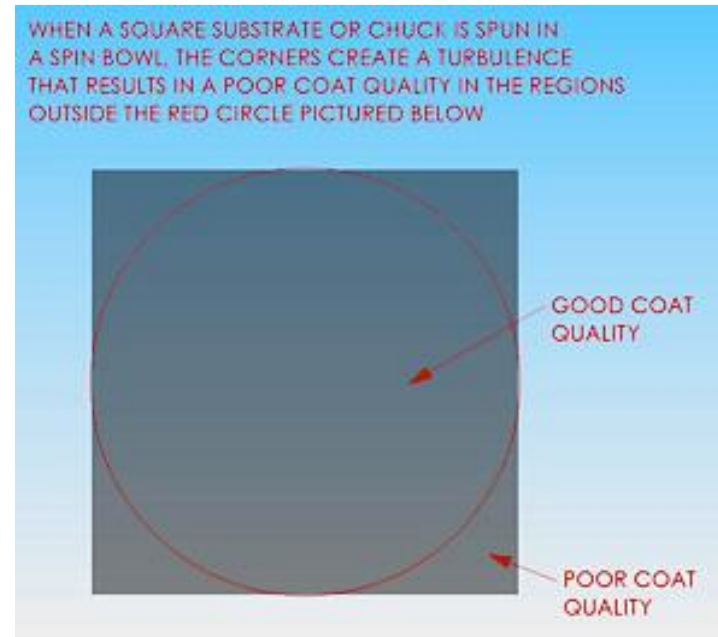
Spin Coating Applications

- Spin coating is extremely widely used and has a varied range of applications.
- The technique can be used to coat anything from small substrates measuring only a few millimetres squared, up to flat panel displays which might be a meter or more in diameter.
- It is used for coating substrates with everything from photoresistors, insulators, organic semiconductors, synthetic metals, nanomaterials, metal and metal oxide precursors, transparent conductive oxides and many more materials.
- In short, spin coating is ubiquitous throughout the semiconductor and nanotechnology R&D and industrial sectors.



Advantages

- One advantage to spin coating thin films is the uniformity of the film thickness with nanoscale thicknesses.
- Owing to self-leveling, thicknesses do not vary more than 1%.
- Spin-coating is a solution-based process developed for low-cost deposition of thin films of materials ranging from polymers (as organic photoresists) to functional inorganic films including amorphous and crystalline chalcogenides.
- The thickness of films produced in this manner may also affect the optical properties of such materials. This is important for electrochemical testing, specifically when recording absorbance readings from Ultraviolet-visible Spectroscopy, since thicker films have lower optical absorbance and typically do not allow light to shine through in comparison to thinner films allowing light to go through before the optical density of the film becomes too low.



Disadvantages

- Spin coating is an inherently batch (single substrate) process and therefore has a relatively low throughput compared to roll-to-roll processes.
- One of the main disadvantages of spin coating is the size of the substrate. As the size increases, the high-speed spinning becomes difficult, because film thinning becomes difficult.
- The material efficiency of spin coating is very low. In general, 95%–98% of material is flung off and disposed of during the process and only 2%–5% of material is dispensed onto the substrate
- The fast drying times can also lead to lower performance for some particular nano-technologies

Spin Coating Thickness Equation

- In general, the thickness of a spin coated film is proportional to the inverse of the square root of spin speed as in the below equation where ω is angular velocity/spin speed and h_f is final film thickness.
- This means that a film that is spun at four times the speed will be half as thick. A spin curve can also be calculated from this equation such as shown below.

$$h_f \propto \frac{1}{\sqrt{\omega}}$$

