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Pre-Cleaning in Thin Film Physical Vapor Deposition

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For most thin film deposition applications, a pre-clean step is essential to achieving desired properties and performance requirements. There are several methods that can be used to carry out this process, which have their own specific benefits depending on what you're trying to achieve with pre-cleaning. This is a murky area of the deposition process space, and as equipment designers, we are frequently asked for the "best" solution. Unfortunately, we must answer with a resounding "it depends". There is no one best solution, and paying more will not necessarily provide performance. The key question that drives the selection is: what are you trying to remove? Sometimes the answer is well understood – "20 Angstroms of oxide", or "residual photoresist". However, all too often the answer is "whatever will keep my film from delaminating". To help bridge the gap between what is possible and what is needed, in this whitepaper we attempt to provide a general overview of some of the pre-clean methods commonly used in PVD thin film applications and when they are appropriate.

Purpose of Pre-Cleaning

Pre-cleaning removes water molecules, hydrocarbons, residue from a wet cleaning process, and other particles or contaminants from a substrate before the thin film coating is actually deposited. It can also prepare the surface of the substrate for film deposition. This process can greatly improve adhesion of the film to the substrate, leading to better performance and reliability for the coating. Better adhesion is particularly important for high stress films, since they will tend to delaminate which impacts yield and reliability.

Pre-cleaning can improve a thin film's optical or electrical properties as well, which may be adversely affected by the contaminants. A contaminant in an optical film will generally increase absorbance. This can both lower transmission and, for higher power density optics, increase surface heating when illuminated by an energetic laser beam. This can simultaneously require a higher operating power and lower the damage threshold. Similarly, electrical conductivity is often degraded by contaminants, which may then force a higher device operating power.

Certain pre-cleaning methods may also be used to micro-roughen the substrate, or to chemically activate the surface, which can improve adhesion of metallic and dielectric films. The pre-clean method chosen will depend on what you are trying to achieve or remove from the substrate.

Removal of an oxide layer requires energetic ions. Energetic ions will physically sputter etch the oxide layer. Typically, in an evaporation system, a gridded ion source is specified due to the high-energy (100s of eV) ions needed to remove the oxide layer. Etch rates are dependent on several factors: ion energy, ion flux, distance, and angle of the ion source relative to the

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substrate. In a sputter system, RF biasing of the substrate is the preferred pre-cleaning method. Etch rates are dependent on the DC bias voltage (also referred to as the self-bias voltage) that develops on the part(s) to be coated. In both cases, the gases selected for the pre-cleaning step are an important consideration. Argon is generally used for physical removal of materials. Oxygen might be mixed with Argon to chemically react with contaminants on the surface to increase the effectiveness of the pre-cleaning step. Oxygen has the added benefit of potentially activating the surface to be coated as well.

Methods of Pre-Cleaning

There are several different approaches you can take to pre-cleaning. Certain technologies, such as RF glow plates or bars, operate at a much lower energy than others. These methods are typically less cost-prohibitive and are usually a good option for simple pre-cleans, like removing hydrocarbons or moisture. They are also useful for delicate substrates that can easily be damaged by higher energy processes. However, for more difficult pre-cleaning processes on durable substrates where entire layers are being removed, a higher energy option is usually needed.

Some of the different technologies used for pre-cleaning include:

- **Heat.** A simple substrate heating is often all that is required to remove moisture and some volatile hydrocarbons. However, many substrates cannot be heated to a high enough temperature for this to be effective.
- **RF glow**, which provides low ion energies, covers a large area and has a low probability of damaging the surface if too aggressive. This can be achieved using an RF biased fixture or stage.
- **Gridded ion source**, which provides high-energy ions and a high-impact ion pre-treatment. A gridded ion source is preferred for an oxide etch.
- **Gridless end-hall ion source**, which has a broad range of ion energies and a moderate to aggressive pre-clean. It is not an effective source for an oxide etch.
- **Plasma pre-treater**, which features both high current density and high-energy ion bombardment, and may require active substrate cooling.
- **RF or microwave plasma pre-treater**, which features a low energy and moderate density plasma cleaning and is primarily used for a chemical surface pre-clean.

Considerations for Choosing a Pre-Cleaning Method

Some of the more common pre-clean requirements and options are as follows:

- **Removal of hydrocarbons** - use heat, ion source, plasma source, or oxygen glow
- **Removal of water** – use heat, ion source, plasma source, or argon glow
- **Removal of oxide layer** – use an argon ion source or a chemical etch
- **Chemically "activate" the substrate deposition surface** - oxygen ion source

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- **Removal of cross contamination from previous deposition steps** - depends on what it is!

Before you decide on a specific technology for pre-cleaning, there are certain performance, process, and budgetary factors you need to consider.

- **Deposition method**: Your deposition method of choice will greatly affect your choice in pre-cleaning method. Ion source technologies are directly compatible with evaporation systems, but not so easy to integrate with standard wafer sputtering systems.
- **Depth of cleaning needed**: You will need to consider exactly what you need to be removed from the surface of the substrate. If you're simply removing hydrocarbons and water molecules, a method providing low ion energy should be sufficient. For removing entire oxide layers, you will need an approach that offers higher density and very energetic ion energies.
- **Coverage area**: The coverage area also varies between approaches. The RF bias glow discharge and plasma pre-treater methods can cover large areas, whereas the RF or microwave pre-treaters and circular ion sources offer more limited coverage.
- **Surface sensitivity**: Depending on how sensitive your substrate material is, you could be at risk of damaging the surface through certain approaches. Typically, the higher-energy, higher-density methods are more susceptible to surface damage.
- **Cost and complexity**: It is critical to understand all of the above considerations for your application before investing in a specific pre-clean approach. Opting for an expensive high-energy ion source when your application does not require it, for example, will cost you extra money and could actually lower your yield.

Measuring Cleanliness

Just as there is no one best pre-clean method, there is no one best method for characterizing cleanliness. Measurement techniques fall into two broad classes – direct and indirect. Direct techniques are the tools of the failure analysis lab: EDS, XPS, XRF, FTIR OES, and a host of other analytical techniques aimed at identifying contaminants. These typically range from the complex to the extremely complex, and can be complicated to integrate into a production process. They often require expert advice on use and interpretation of results, but they can be invaluable when troubleshooting an out-of-control process.

Indirect techniques, on the other hand, measure a physical property that would be affected by contamination. Examples are optical transmission using a spectrophotometer, resistivity using a four-point probe, or adhesion using a tape test. There are standard methods for measuring these properties, and for an in-control process, these can be run by operators or technicians, and are very useful for monitoring film quality.

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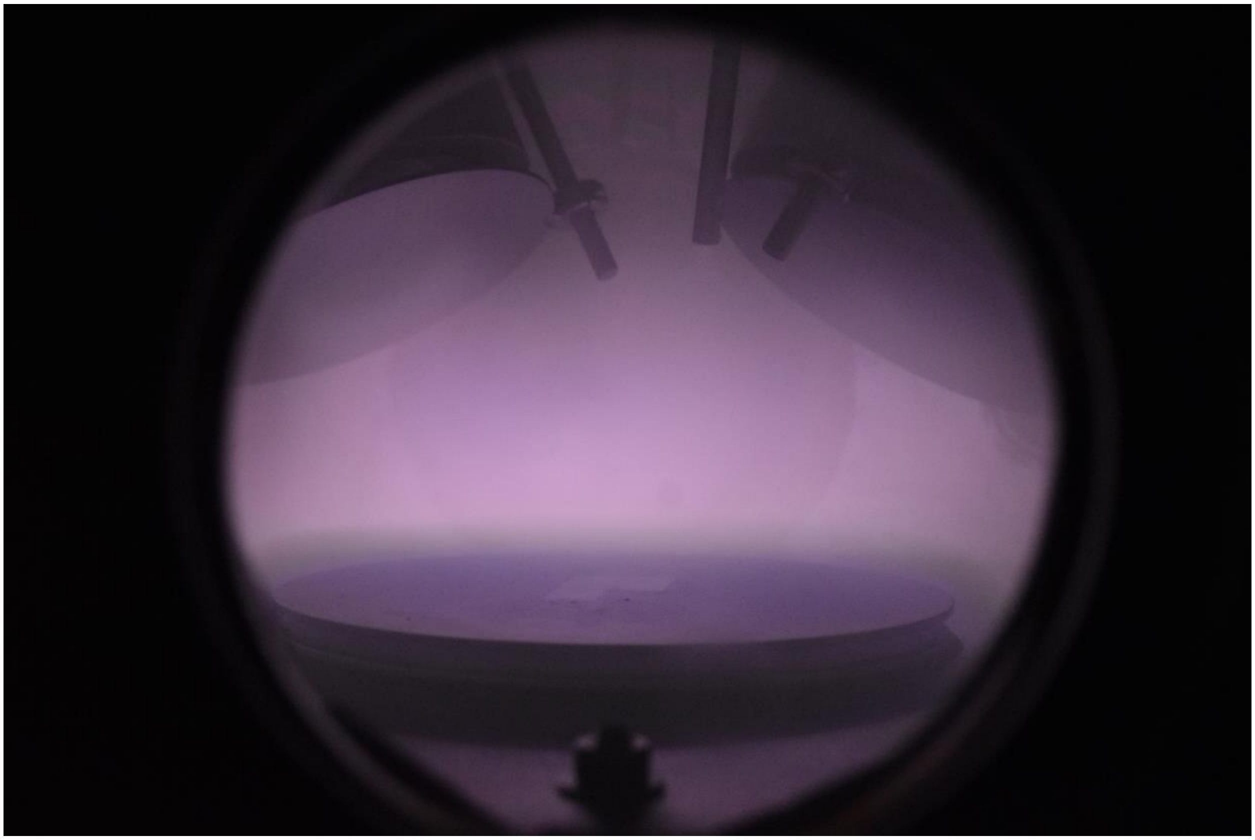
A detailed discussion of the various methods is beyond the scope of this whitepaper, but may be the subject of a future paper if there is sufficient interest.

Examples

By way of illustration, let's consider a few examples. First, consider Denton's Discovery 200 sputter module. This can be configured as a standalone module with a load lock, or as part of a cluster tool. This system has a 300mm cathode directly sputtering down onto a 200mm substrate, with a typical source-to-substrate distance on the order of 100mm. A typical thermal limit is that a patterned wafer must remain below 150°C to avoid damaging the photoresist. Here the options are much more limited. Regardless of what needs to be removed, the only real option is an RF biased chuck for a gentle plasma clean. Since ion sources require line-of-sight to the substrate being cleaned, there is no way to have one installed along with the cathode. Stronger pre-clean would need to be done outside the Discovery module.



Glow Discharge in Sputter Module, usin RF biased chuck

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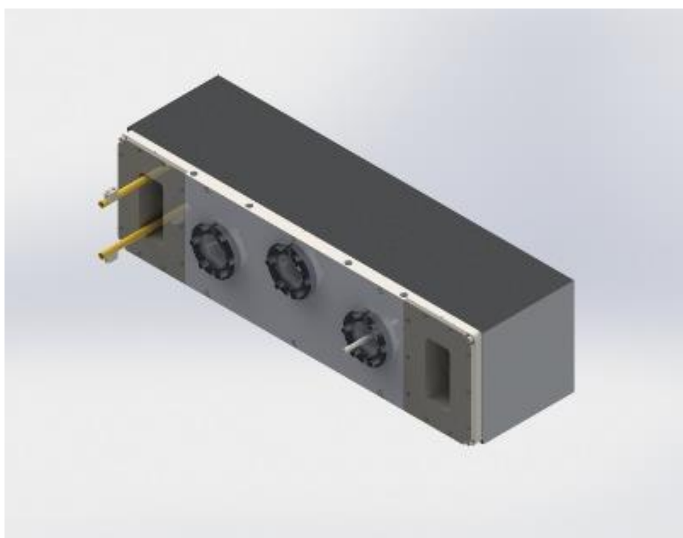
Next, let's look at oxide removal in a drum sputter coater. In this configuration, rectangular cathodes sputter "in" onto a rotating cylinder with rectangular substrate holders. For removing an oxide layer we need an energetic argon ion source. However, this is a large-area configuration, so typical circular ion sources are often impractical due to uniformity, cost, and space considerations. In this case, a linear source such as Denton's unique linear plasma ion source, currently under development, is ideal. It is able to cover large areas and provide the energetic argon ions required for oxide removal.

Finally, another application for the linear plasma source: surface preparation of hard-coated polycarbonate to improve adhesion of optically clear diamond-like carbon (DLC) for display applications. The DLC coating is both wear- and fingerprint-resistant; however, it is a high-stress film. Without proper preparation of the polycarbonate, the DLC is prone to both delamination and crazing. The plasma ion source is able to remove loosely-adhered oligomers and activate the surface. This surface pretreatment effectively eliminates delamination and crazing.



Denton Vacuum's Linear ion source

Conclusion

As we've shown, there are a number of options for pre-clean, and careful consideration is required to match a pre-clean method with a particular thin film requirement. The table below is a more detailed listing of the characteristics of some of the typical technologies we've discussed, and applications where they are appropriate. Hopefully the information below and the discussion above can help guide a pre-clean method selection that is appropriate for your process. If you would like further discussion, just drop us a note by email or visit our website.

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Features & Considerations

Technology	Features	Considerations
RF Glow (Plate or biased chuck)	• Capable of supporting Ar/O2 plasma discharges over a wide pressure/power range. • Low ion energies, ~ 15-30 eV range. • Medium/low plasma densities • Generates afterglow to continuously interact with substrate even when not in proximity of plate.	• Supports both Ar ion soft "etching" of surface and O2 radical/ion chemical cleaning. • Low probability to damage surface. • Low probability of metal contamination. • Large area coverage.
Gridded Ion Source	• "High" energy ions, 1-3 kV accelerating voltages, bombardment of surface. • Typically, Ar is preferred treatment gas.	• "High" impact plasma pre-treatment. • Argon sputter clean of surface. High energy ions can damage sensitive surfaces. • Requires line-of-sight between source and substrate. • Operation is sensitive to re-sputtering of substrate on source anode. • Expensive and complex to integrate. • Small coverage areas.
Gridless end-Hall Ion Source	• Broad range of ion energies, 50-100s of eV. • Compatible with Ar and O2. However, Ar pre-clean is most typical.	• Moderate to aggressive Ar ion physical pre-clean. • Long source to substrate distances typically required. • Small coverage areas. • Array required for large area substrates. Non-uniform treatment anticipated. • Complex and expensive to install and maintain.

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Plasma pre-treater (inverted magnetron type)	• High voltage (>+1kV) DC low current inverted magnetron type ion sources • DC magnetron based plasma pre-treatment – magnetron devices for high power pre-treatment of plastic web.	• High energy ion bombardment of substrate. • Large-area sources (up to 3 m long available)
RF or Microwave plasma pre-treater	• Low energy (< 50 eV), moderate density plasma cleaning. • Large area afterglow generated.	• Active substrate cooling may be required. • Small source to substrate distances preferred. Highly localized plasma discharge. • High maintenance costs. • Primarily a chemical surface clean. • Very Expensive & complex due to RF or microwave power supplies & engineering.