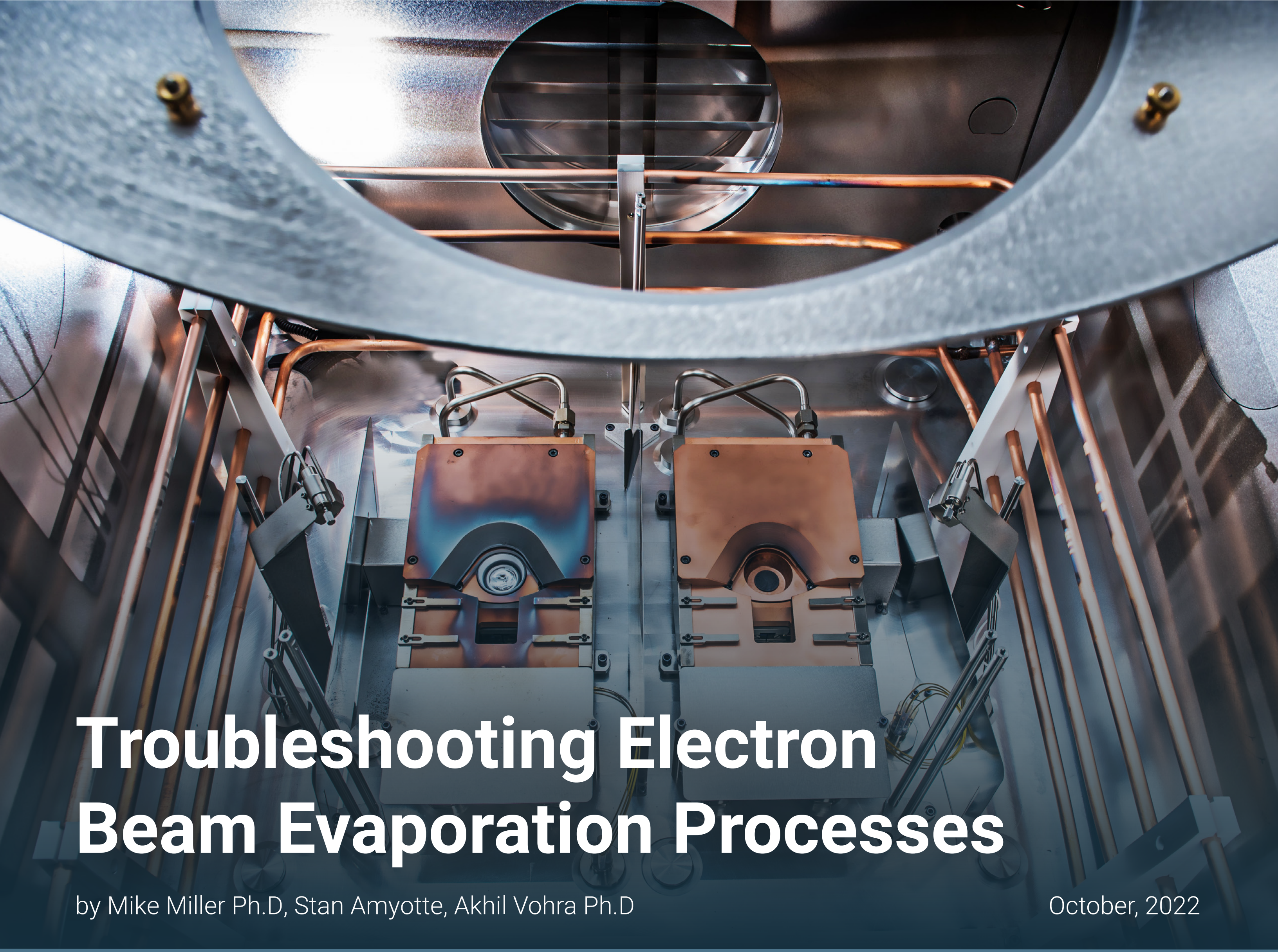

Troubleshooting Electron Beam Evaporation Processes

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INTRODUCTION

Electron beam evaporation is one of the most common and versatile deposition technologies available to material scientists today. Thin films deposited from an electron beam source are extremely pure, making them suitable for a variety of applications that are sensitive to contamination. Deposition rates from this source type can also be very high, providing a means to produce high quality films with a great deal of efficiency.

Despite these two primary benefits, electron beam evaporation processes can also create unintended and often frustrating problems that must be resolved before realizing functional films. Summarized below is a list of common challenges associated with electron beam deposited films, and some tips and tricks to help troubleshoot these issues as they arise.

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IMPROVING ADHESION

Nearly all thin films are deposited with the intention of adhering well to the underlying substrate. Without good adhesion, nearly all of the other properties of the film become insignificant. The three most common causes of adhesion failure are **film stress, contamination, or chemical incompatibility** between the substrate material and the film being deposited.

FILM STRESS

Room temperature thin films deposited by electron beam evaporation commonly have voids in the grain structure which can lead to tensile stress. As the films become thicker, the amount of tensile stress increases and can eventually cause the film to delaminate from the substrate. In order to densify the films and reduce or eliminate tensile stress, additional energy must be imparted to the atoms as they arrive at the substrate surface.

Substrate heating, ion-assisted deposition, or plasma assisted deposition can all help add the energy needed to solve this problem.

CONTAMINATION

Good adhesion relies on a strong bond being formed between the underlying substrate and the material being deposited. Any contamination on the substrate can disrupt bond formation and lead to poor adhesion. For common substrates such as glass slides, silicon wafers, or metals, a water-drop test (or contact angle test) can be used to quickly assess the cleanliness of a test surface prior to deposition; a water drop placed on a clean surface will completely spread out, while a dirty or greasy surface will cause the water droplet to ball up.

There are a variety of methods for cleaning substrates including ex-situ methods like ultrasonic cleaning, or in-situ methods like ion or plasma cleaning.

CHEMICAL INCOMPATIBILITY

Even with clean substrates and a stress-free film, not all materials will stick to all substrates. In general, strong adhesion relies on some level of chemical interaction between the two materials and so the film/substrate architecture must be designed accordingly.

For example, gold will not adhere well to glass because it will not oxidize and form a bond to the silica surface; however, gold will adhere well to materials such as chromium or titanium because it readily forms alloys with these materials. Since chromium and titanium both oxidize easily, they will react with a silica surface and can act as an intermediate 'adhesion promoter' to allow the gold film to properly adhere to glass.

There are many other examples of adhesion promoters which – when applied appropriately – can be used to create a strong bond between most substrate/material combinations.

MAINTAINING FILM STOICHIOMETRY

An assumption often made during evaporation processes is that the composition of the deposited film will match the composition of the source material. Unfortunately, this is not always the case; when an electron beam heats up non-elemental source material, the different components may dissociate and/or evaporate at different temperatures.

For example, depositing 95:5 gold-zinc source material will not lead to an even 95:5 composition in the film; similarly, depositing from TiO_2 source material will almost certainly create a film with less than two oxygen atoms for every atom of titanium.

FOR OXIDES AND NITRIDES, the most common way to influence the oxidation state of the deposited film is to add energy (ion/plasma assistance, and/or substrate heating) along with a partial pressure of background reactive gas.

As an example, TiO_2 films can be deposited using Ti or TiO_2 source material, while introducing a background gas of oxygen and heating the substrate to facilitate the reaction. If heating of the substrate is not an option, ion or plasma assistance can accomplish the same goal.

By varying the amount of background gas and the amount of energy at the surface, the oxidation state of the deposited film can be fine-tuned.

FOR OTHER MATERIALS, such as the gold-zinc alloy in our example above, co-deposition from multiple sources is the best way to precisely control the resulting stoichiometry in the film. Alternatively, other deposition methods such as pulsed laser deposition specifically target processes that require maintenance of complex stoichiometries.

PREVENTING SPITTING

One challenge commonly associated with electron beam evaporation is the tendency for material to 'spit' large particles or globules onto the substrate.

A concentrated, high-energy beam of electrons can rapidly heat localized areas of the source material, causing it to boil violently and expel large particles up towards the surface of the substrate. While it may not be possible to completely avoid spitting, it can be mostly mitigated using a few commonly employed techniques:

If the source material is spitting, the number of particles that reach the substrate will depend directly on its distance from the source; substrates that are very close to the source will be heavily impacted while those that are further away will remain much cleaner. If possible, keep the substrate as far away as you can.

Uniform heating of the source material can prevent localized boiling and reduce spitting. This can be accomplished by manipulating the sweep profile of the beam, or by providing thermal isolation between the crucible and the water-cooled hearth. It is worth noting that uniform heating will help with spitting, though it may cause the crucible to heat up and potentially create contamination in the film; while this is uncommon, it is worth considering for processes and applications that are highly sensitive to contamination.

ACHIEVING REPEATABLE FILM THICKNESS

Nearly all thin film deposition processes have a targeted thickness that will impact the performance of that particular material/layer within its final application. Control of deposition rate and film thickness during electron beam evaporation processes is commonly managed using **quartz crystal microbalances (QCMs)**.

A **QCM** is able to measure the instantaneous deposition rate and total deposited thickness during a process, though it is important to note that these measurements are typically made at a position in the vacuum chamber other than where the substrate resides; the system must be calibrated in order to correlate the rate and thickness at the QCM position to the position of the substrate itself. Many process variables will influence this calibration factor (often called a 'tooling factor'), and consequently the ability of the system to repeatably deposit the desired film thickness.

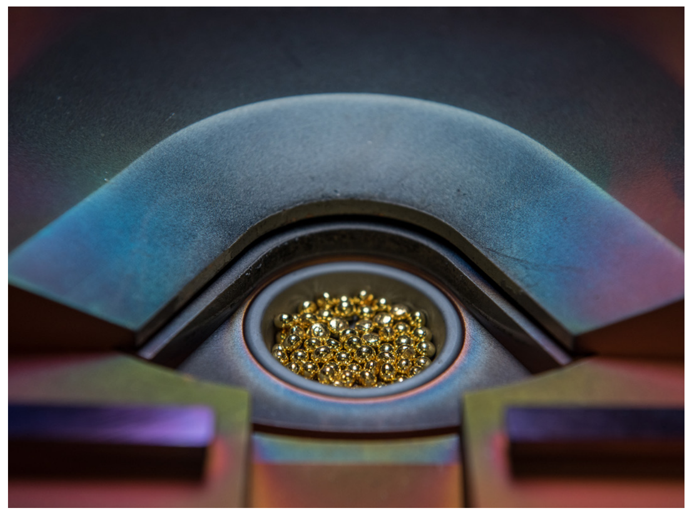
- Maintaining a consistent material fill level within the crucible will ensure that the line of sight to the **QCM** does not change significantly from one deposition to the next. If the fill level starts to drop, the crucible itself may shadow the view of the source material from the **QCM** which will impact the tooling factor and change the calibration.

- A consistent position and sweep pattern of the electron beam within the pocket will also contribute to having a stable, repeatable deposition plume.

- A stable deposition is much more likely to be consistent between the measurement and substrate locations than an unstable one.

  Some tips on keeping a stable deposition rate can be found above in the section on 'preventing spitting'.

- Other variables such as the deposition rate, process pressure, and **QCM** lifetime can all contribute to run-to-run variability in a deposition; if any of these are changing from one run to the next, the system will fall out of calibration (or require unique, independent calibrations for each scenario in order to be accurate).



A gold-filled crucible within an electron beam source demonstrating an appropriate 80% fill level.

A dual quartz crystal microbalance often used for measuring instantaneous rate and thickness of electron beam deposited films.

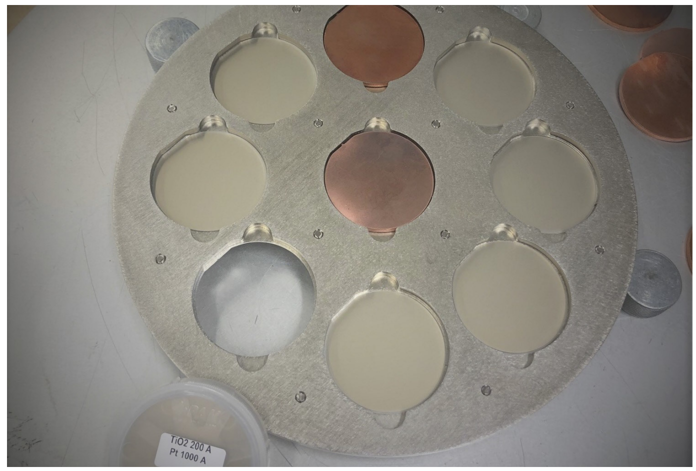
PREVENTING HEAT DAMAGE

Electron beam evaporation is commonly used to deposit films onto substrates that may be sensitive to heat, or which have already been coated with materials like photoresist, electron beam resist, or soft/organic molecules that cannot survive temperatures much higher than 80 °C.

There are several ways to help prevent and minimize this type of damage, some of which are more intuitive than others.

To prevent overheating of a substrate, it is best to locate it as far away from the deposition source as possible during evaporation. It is also important to ensure the substrate is in good thermal contact with a heat sink (active or passive) to help dissipate the radiative process heat. In a vacuum environment, simply placing or clipping the substrate to a heat sink or cooled plate is not enough to overcome the thermally insulating properties of vacuum. Vacuum compatible, compliant materials with good thermal conductivity are available (such as a thermal silicone sheet or a pyrolytic graphite sheet) and can be placed between the substrate and the heat sink to ensure good conduction. **This is one of the most important factors for managing unintentional substrate heating and should not be overlooked.**

An electron beam evaporation system demonstrating a long source-to-substrate distance, appropriate for temperature sensitive substrate materials.



A substrate carrier demonstrating the use of thermally conductive silicone to improve heat transfer to a copper backing plate.

Unlike magnetron sputtering, the amount of total heat exposure during an electron beam evaporation process is much larger for processes with low deposition rates compared to those with high deposition rates. While this might not seem intuitive, increasing the deposition rates for an electron beam process does not require a linear increase in power; this means that a higher rate will decrease the time of exposure at a faster rate than it increases the amount of exposure. **For many processes, increasing the deposition rate by 2-10 times is all that is needed to prevent overheating of the substrate.**

Electron beam sources are water-cooled to protect the source and concentrate the heat of the process to the material being deposited. Ensuring proper water flow/temperature, as well as good thermal contact between the crucible/material and the water-cooled hearth are also important factors in minimizing the total amount of heat being radiated into the vacuum chamber.

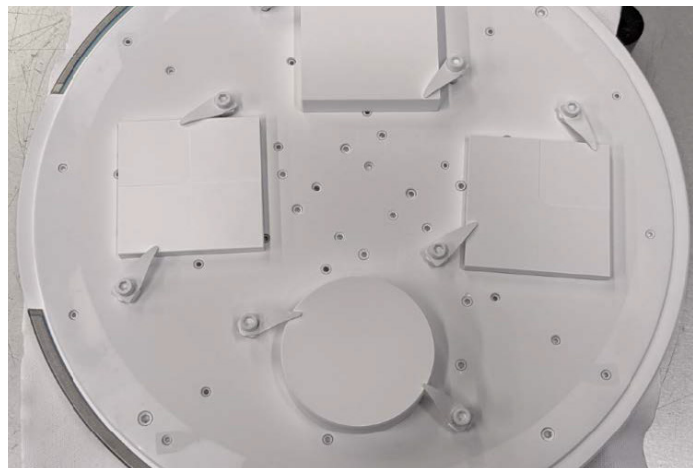
IMPROVING RESISTIVITY AND REFLECTIVITY OF METAL FILMS

Metal films deposited using electron beam evaporation can be extremely pure and should have resistivity and reflectivity values that approach (or even sometimes improve upon) that of the bulk material. Metal films that are hazy or resistive can often be a sign of problems with the process, or the vacuum chamber itself.

Both reflectivity and resistivity correlate with the purity of the deposited film. Any contamination from within the process chamber (vacuum environment, high partial pressure of water/oxygen, or outgassing of the crucible material) can lead to hazy, resistive films.

As an example, aluminum films deposited in a vacuum chamber with a base pressure of 10^{-6} Torr and from a graphite crucible may be hazy and highly resistive because of film oxidation and carbon contamination.

The same films deposited at a base pressure of 10^{-8} Torr and directly from the copper cooled hearth will be highly reflective and conductive, making it much more suitable for use in most thin film applications.



A hazy aluminum film showing clear signs of oxidation during an evaporation process without the appropriate level of high vacuum.

Resistivity of metal films can also be influenced by the number and type of grain boundaries in the film, both of which are impacted by the deposition rate, substrate temperature, growth angle, and the presence of any other ion/plasma assistance.

We hope that this guide – while not comprehensive – provides a ‘cheat sheet’ for helping you troubleshoot the next electron beam evaporation process that gets out of line.

If you have further questions or need some additional advice. **Get in touch with us**.

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