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7 Reasons Multiple Gases Improve TEM Samples

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There are advantages to having multiple gas precursors available for FIB preparation of TEM samples. While a single gas can adequately meet the FIB milling protection layer and in situ lift-out welding needs for many samples, there are times when another precursor better meets the analysis needs and can make the difference between a successful or inconclusive analysis.

Here are 7 reasons why labs engaged in FIB sample preparation for TEM/STEM analysis require multiple precursors:

- 1. Reliable deposition for high throughput:** The organometallic Pt precursor methylcyclopentadienyl platinum trimethyl $((\text{CH}_3)_3(\text{CH}_3\text{C}_5\text{H}_4)\text{Pt})$ is a popular source found on many FIBs. It is one of the easiest gases to work with, because it is compatible with a wide range of beam parameters and promotes more consistent and successful deposits.
- 2. Quality high resolution results:** Low kV cleaning is commonly used after FIB milling to prepare samples for HR-TEM (aberration corrected), which are typically 50nm thin or less. Exposing Pt depositions to low kV can cause uneven erosion, resulting in curtaining which degrades the quality of the TEM sample. In this case, W or C depositions perform better and are recommended as milling mask layers for UHR TEM imaging.
- 3. Accurate metrology:** Adequate contrast between the FIB-deposited protective layer and the surface below it is required for accurate imaging. Thickness measurements depend on a clear delineation between the sample and any applied layers. C and SiO_2 sources are excellent for depositing protective layers on metals while providing contrast.
- 4. Reduced charging:** Using metals for welding an in situ lift-out sample to a grid provides a conductive path for TEM imaging. Conversely, if electrical conduction between the sample and grid is not desired, an insulator can be used for welding.

7 Reasons Multiple Gases Improve TEM Samples (cont.)

5. **Improved elemental analysis:** Having a variety of gases available improves the ability to add a layer that does not contain or have overlapping peaks with the element(s) of interest.
6. **Easier Atom Probe Analysis:** C deposition has numerous benefits. It has finer grains and reduced curtaining compared to Pt. Because it has a lower ion milling rate than Pt, it performs better as a protection layer. In addition, it is a removable protection layer. The deposited C can be completely removed by a simple “ash” process. This is especially helpful in applications such as atom probe analysis in which the surface of the sample must be available for analysis after milling.
7. **Improved in situ lift-out success yield:** Using the right welding material is crucial for an optimized lift-out process. Despite the benefits of C for deposition, it is not an ideal welding material for in situ lift-out. Regardless of C precursor used (styrene, phenanthrene, naphthalene), the final deposition is the same constituent material, C₉H₂O. This is reported for e-beam deposition and also likely true for ion beam deposition. The deposited C is particularly brittle and has high risk of fracturing during handling in situ lift-out welding or grid attachment. Pt or W are recommended for in situ lift-out welding steps.

So what's a TEM prep lab to do?

Here are 3 quick suggestions to optimize your set-up:

- Ensure that ports are a metric evaluated during the equipment selection process. There should be enough ports to grow with future needs and accommodate a variety of gases.
- If the FIB is port-limited, consider the purchase of a multiple gas injector system such as the OmniGIS[®].
- Since even the most gentle ion or electron beam deposition causes some alteration of the surface, when TEM analysis of surfaces is your primary goal, boost your chances for artifact-free preparation by buying or gaining access to sputter coaters and evaporators.

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