

What EBID can tell us about Contamination ?

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Electron beam induced contamination has been a familiar problem in electron microscopy since the earliest days of the technique, because it constitutes an unwanted modification to the sample, although it was also the earliest and easiest technique of patterning a specimen on the nanoscale. More recently much attention has been paid to Electron Beam Induced Deposition (EBID), a technique in which an electron beam is employed to deposit some chosen material on to a specimen surface by deliberately initiating a reaction in a precursor gas (such as WF_6) which then results in deposition on to the surface. Clearly these two effects are closely related and so it is possible to take what has been learnt about optimizing EBID and to use that data instead as a cautionary guide to help to minimize or avoiding contamination.

There are several steps involved in material deposition by EBID [1]. Molecules capable of being decomposed by beam induced radiolysis land on the specimen surface. Some fraction of these remain long enough to interact with electrons passing through the surface plane (either as incident, or as backscattered or secondary electrons). Depending on the parameters of the incident electron beam – its current and energy, and whether or not it is a point, or a broad beam, stationary or is being scanned – material then begins to deposit at some rate ultimately forming a physical feature nanometers or thicker in size. The detailed studies which have been made the kinematics of EBID have shown that this process is limited by mass transport of the active species on the sample surface and the time that each molecule spends on the surface. These insights now make it possible to identify which factors will most affect contamination.

Firstly, deposition requires a reservoir of the material with which the electrons will interact and for both casual contamination and EBID this will be the gas or vapor phase around the sample. If the concentration of the active ingredient in the vapor phase is too low then the rate of deposition falls very rapidly because the reaction is typically mass transport limited. The reduction of the hydrocarbon content of the residual gases in the vacuum chamber is therefore the obvious first step in minimizing contamination.

Secondly, deposition is often a competitive process. For example, if there is also water vapor present in the residual gases in the vacuum system then, although carbon deposition still occurs, the water vapor may give rise to beam induced etching which attacks - and can remove - the deposited carbon, the equilibrium between etching and deposition processes depending on the amount of water vapor present. This is the situation that is usually exploited in a variable pressure SEM (VPSEM) where minimal or

even zero contamination rates are achieved even though such instruments typically only employ simple, oil based, pumps.

Third, detailed experimental and theoretical work has demonstrated that a key factor in the growth rate of a deposit is the residence time of the target molecules on the sample surface. This is because the electron-molecular reactions occur only on the surface and not in the vacuum phase. The longer the surface residence time then the more opportunity there is for interactions to occur and to lay down the material. Measurements show that the binding energy for typical molecules (e.g. hydrocarbons, or precursors containing metal) is of the order of 100-150meV and consequently the residence time is a very sensitive function of the surface temperature T when this is in the region $250\text{K} < T < 350\text{K}$. Lowering the temperature from 20C to -20C increases the residence time by more than one order of magnitude, with a corresponding increase in the deposition rate for a given beam dose while raising the temperature by a similar increment lowers the deposition rate. Hence contamination can be minimized by warming the target surface slightly above room temperature. This advice does not conflict with the usual precaution of using a liquid nitrogen cold finger because, although this might slightly chill the specimen, its main function is to trap hydrocarbon etc, on to the finger and so reduce the local concentration in the vapor phase.

Fourth, because deposition requires mass transport to position molecules under the electron beam, contamination is fed by charging and is strongly affected by the scanning behavior of the beam. When a surface acquires a charge, which under most conditions will be negative in polarity, an electrostatic field is created with field lines pointing towards the incident beam point. This electrostatic field also enhances the surface mobility of molecules already on the surface and drags them into the electron beam where they react and deposit carbon. As long as the charge field is maintained then fresh material can be transported across the surface and exposed to the beam. This effect is maximized when the beam is held stationary on a point ("Spot mode"), but is reduced in severity when the beam is scanned. This is because the motion of the probe leads to a thin layer of the deposit which both depletes the reservoir of molecules available on the surface, and reduces the ability of any charging field to pull material under the beam. The classical technique of pre-exposing a surface at low magnification prior to examination at high magnification exploits this behavior and can reduce contamination rates considerably [2,3]

References

- [1] Rack P D et al , (2003), *Appl..Phys.Lett.* **82**, (14), 1
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