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Cryo-SEM as an effective method for avoiding contamination

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Contamination is the most common problem occurring during imaging in the scanning electron microscope (SEM). In SEM and TEM contamination causes degradation of contrast due to the electron beam induced deposition (EBID) of a hydrocarbon layer. The contamination source can be the microscope vacuum or the sample itself. For investigating samples at a ZEISS FESEM, we found that specimen-borne hydrocarbon is the main contributor for contaminating samples. The deposition process can be described by a combination of cracking of hydrocarbons on the specimen and the subsequent surface diffusion of hydrocarbons to the site being irradiated [1,2].

There are numerous techniques available to avoid contamination by cleaning the sample. Common methods such as solvent rinsing, heating, vacuum heating, and plasma etching all have limitations. Rinsing the sample with organic solvent can remove most of the organic contamination, but this method leaves behind a thin film of residue that itself contaminates the specimen. Heating and vacuum heating are limited in effectiveness and cannot be applied to heat-sensitive materials. For effective cleaning temperatures of 300 °C have to be introduced [3]. Plasma cleaning can often damage the sample or redeposit material. Cooling the sample to temperatures below -100°C decreases of mobility on the surface and results in a negligible contamination [2]. This method can be applied to sensitive samples, that would be modified by plasma cleaning or heating.

In this study we investigated silica nanoparticles. A suspension of nanoparticles in an isopropanol-based solvent is drop-casted on a substrate. Very often stabilizing agents are used as well in this suspension to avoid conglomeration of the nanoparticles. Those agents and residuals of the solvent lead to a substantial source of contamination on the sample surface. The imaging is compromised, and the measurable size of the particles is changed by the addition of the contamination layer. This can clearly be shown, especially at higher magnification. Due to the higher electron dose the contamination is becoming more prominent (Fig. 1). When cooled down to -130°C carbon contamination is not visible anymore (Fig. 2). This is expected and was reported in detailed studies of the contamination process in the SEM [2] (Fig. 3). In our contribution we demonstrate a new cryo stage design employing a Quorum 5005Z cryo stage together with the ZEISS airlock. The sample can be directly mounted on the carrier at room temperature and inserted into the airlock (Fig. 4). The carrier together with the sample can then be transferred onto the cryo stage inside the SEM chamber and then cooled down to desired temperature. A patented temperature regulation method is implemented to minimize the sample drift at cryogenic temperature.

As a short summary, the method described here can effectively avoid contamination during imaging in the SEM and maintain a high stability for high resolution imaging. This method is well suited for samples prone to contamination that does not require shock freeze and vitrification.

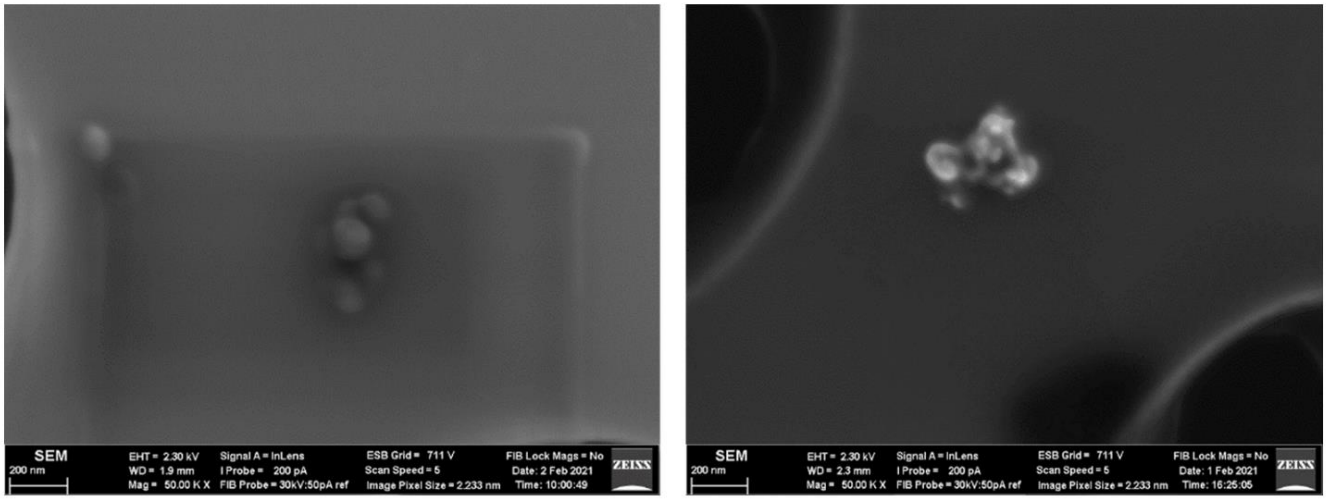

Figure 1. Nanoparticles imaged at 18°C with contamination layer and same sample imaged at -130°C with no contamination and better contrast.

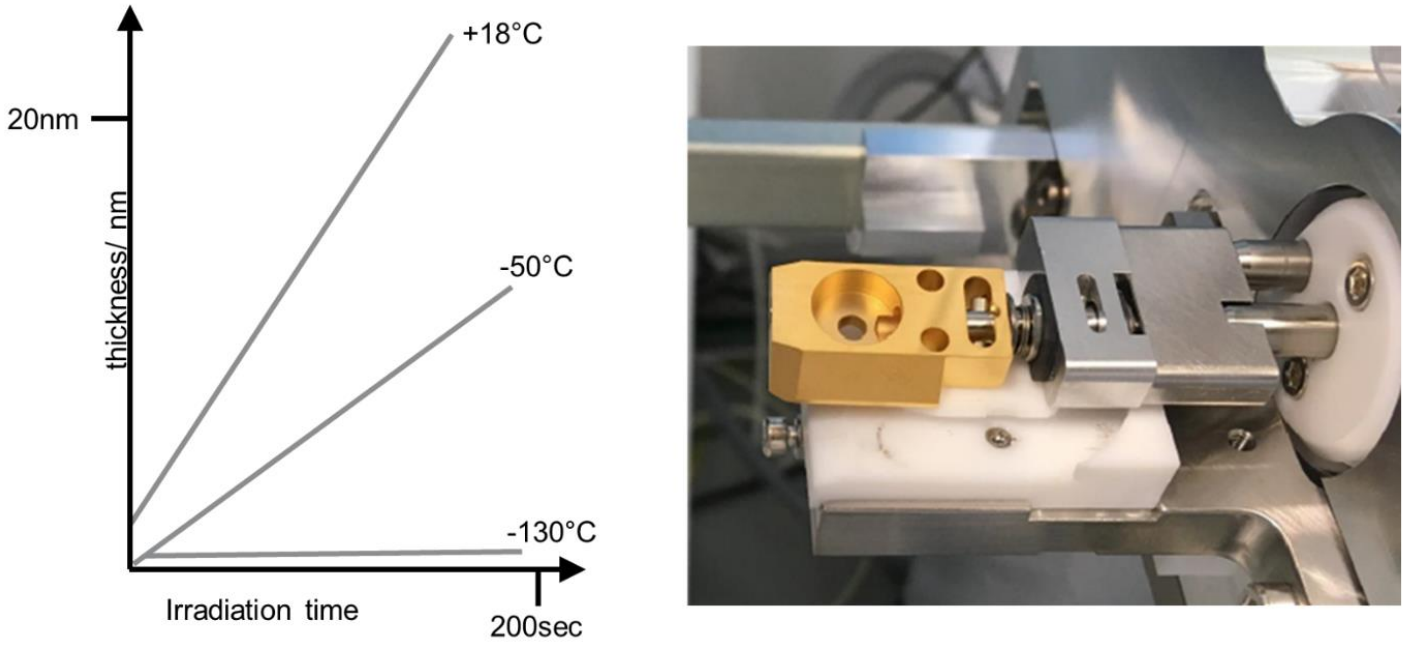

Figure 2. Dependency of conatamination layer thickness on temperature after [2] (left). Detail of the sample transfer shutlle in the SEM airlock (right).

References

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