Cassette-based \textit{in-situ} TEM sample inspection in the dual-beam FIB

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Abstract. A novel method is presented, combining site-specific TEM sample preparation and \textit{in-situ} STEM analysis in a dual-beam microscope (FIB/SEM) fitted with a chamber mounted nano-manipulator. TEM samples are prepared using a modified \textit{in-situ}, lift-out method, whereby the samples are thinned and oriented for immediate \textit{in-situ} STEM analysis using the tilt, translation, and rotation capabilities of a FIB/SEM sample stage, a nano-manipulator, and a novel cassette. This cassette can provide a second tilt axis, orthogonal to the stage tilt axis, so that the STEM image contrast can be optimized to reveal the structural features of the sample (true STEM imaging in the FIB/SEM). The angles necessary for stage rotation and probe shaft rotation are calculated based on the position of the nano-manipulator relative to the stage and door and the stage tilt angle. A FIB/SEM instrument, equipped with a high resolution scanning electron column, can provide sufficiently high image resolution to enable many failure analysis and process control applications to be successfully carried out without requiring the use of a separate dedicated TEM/STEM instrument. The benefits of this novel approach are increased throughput and reduced cost per sample. Comparative analysis of different sample preparation methods is provided, and the STEM images obtained are shown.

1. Introduction

Dual-beam microscopes (FIB/SEM) are commonly used for the preparation of specimens for high resolution (< 0.1 nm) transmission electron microscope (TEM) or scanning transmission electron microscope (STEM) imaging and analysis. The \textit{in-situ} lift-out technique has become the method of choice for the preparation of site-specific TEM samples. Samples for TEM/STEM inspection are required to be electron transparent (ca. < 100 nm thick) and are therefore inherently fragile. There is some small risk of damaging the sample in transferring between the FIB/SEM and a separate TEM/STEM. However, a FIB/SEM equipped with a high resolution SEM column and a STEM detector, located beneath the sample [1, 2], enables \textit{in-situ} STEM imaging to be carried out on samples prepared using the site-specific, precision focused ion-beam (FIB) milling in the same instrument. As a result, this method provides increased throughput at reduced cost per sample for failure analysis and process control applications requiring STEM analysis [3-5] but where the ultra-high-resolution of the dedicated stand-alone instrument is not required.

Lately, low voltage STEM microscopy is becoming more popular [6, 7]. There are several methods for obtaining quality STEM images in the shortest time possible, among them are the STEM in a SEM solution [8], the flip-stage system [9, 10] and the method based on the moving specimen stage and
sample grid with multiple openings [11]. Each of these methods has its advantages and disadvantages, shortly discussed below. In this paper, we describe a novel method and apparatus developed to locate, prepare and inspect samples inside a FIB/SEM using an *in-situ* probe-tip replacement system [12, 13] and a special cassette for holding these samples.

2. Cassette

The cassette for holding TEM samples is shown in figures 1-4. As can be seen from figure 1, the cassette consists of two probe-tip stations attached to the solid base. There is a special slot in each station for placing the probe-tip. Probe-tips are placed into the cassette using a nano-manipulator with a gripper mechanism which is released when the probe-tip is properly located in the selected station. The probe-tips are held inside the probe-tip station using an adhesive tape (for the best results, the special collared probe-tip, as in [11] and [12], should be used). The cassette is attached to the cassette holder (figure 2) which is, in turn, attached to the specimen stage (figures 3 and 4). There are holes in the cassette holder, located beneath the sample, permitting immediate *in-situ* STEM analysis (see figures 5c and 5d) to be carried out. A small motor can be attached to the cassette which allows tilting to obtain optimum image quality/information.

3. Method

The cassette-based *in-situ* STEM sample analysis method consists of the following steps. The *in-situ* lift-out process begins in the conventional manner in which a sample pre-form is excised from the specimen by FIB milling. Following partial thinning, the released lift-out sample (figure 5a) is properly oriented using a feature typical of FIB/SEM sample stages that allows the stage to rotate, tilt, and translate in X, Y, and Z axes. This orientation step deviates from the standard lift-out process in that, after this positioning step, the lamella face is not necessarily parallel to the lift-out plane of the manipulator. Rather, it is positioned relative to the manipulator so that, after the sample has been attached to the probe-tip and removed from its trench by the nano-manipulator, a simple rotation of the nano-manipulator shaft about the long axis of the shaft will produce an orientation of the sample which becomes effectively perpendicular to the electron beam axis, and thus, suitable for STEM analysis in a FIB/SEM (figure 5b). If necessary, the sample can be thinned further after the lift-out procedure. As shown in figures 5c and 5d, the sample is placed into a cassette in the orientation that
allows immediate STEM inspection inside a FIB/SEM when the sample stage holding the cassette is positioned and the transmitted electron detector moved in. (The transmitted electron detector is not shown in the picture). Once the cassette probe-stations are filled with the sample laden probe-tips, the cassette may be transferred outside the FIB/SEM, and the samples are ready for further investigation as necessary. If needed, the probe tip with the sample attached can be taken out of the cassette later and attached to the TEM sample grid, giving the sample proper orientation for the TEM inspection, using the ShortCut™ method [14].

![Figure 5](image_url)

**Figure 5.** Major steps of the cassette-based *in-situ* STEM sample inspection.

To obtain useful STEM images of the sample, a conversion from angle-axis representation to rotation matrix representation has been used to calculate nano-manipulator rotation ($\Theta_1$) and FIB/SEM stage rotation ($\Theta_5$) based on three pre-determined angles ($\Theta_2$, $\Theta_3$, and $\Theta_4$). $\Theta_2$ is the angle of the stage tilt relative to the X-Y horizontal plane; $\Theta_3$ is the angle about the e-beam axis on the nano-manipulator shaft relative to the door of the FIB/SEM; $\Theta_4$ is defined by the inclination of the axis of the shaft of the nano-manipulator relative to the X-Y horizontal plane of a FIB/SEM.

We have found it convenient to use the following approximate values for the angles described above for a Model 1540 CrossBeam®, manufactured by Carl Zeiss, Inc.: $\Theta_2 = 0^\circ$, $\Theta_3 = -53.4^\circ$, $\Theta_4 = 33^\circ$, thus resulting in the following computed values: $\Theta_1 = 86.1^\circ$, $\Theta_5 = 114.9^\circ$.

The low voltage STEM images taken at EHT voltages 20 kV, 25 kV and 28 kV are shown in Figures 6-8.

![Figure 6](image_url)

**Figure 6.** STEM image taken at 20 kV EHT voltage.

![Figure 7](image_url)

**Figure 7.** STEM image taken at 25 kV EHT voltage.

![Figure 8](image_url)

**Figure 8.** STEM image taken at 28 kV EHT voltage.
4. Analysis and Conclusions
A new method of cassette-based in-situ STEM sample inspection in a FIB/SEM microscope has been developed. This method allows the user to obtain useful STEM images in a shorter time, compared to the known methods described in [8-11]. The unique cassette also provides for stable placement of the sample for imaging and gives the user different options; one of which is the possibility to return the TEM sample to its initial orientation and transfer it to a dedicated TEM/STEM for further investigation. With this method, STEM sample inspection time compared to both the flip-stage [9, 10] and the method based on the moving specimen stage and the sample grid with multiple openings [11] is significantly shorter, since there is no need to weld the sample to the TEM sample grid and to cut the sample from the probe-tip. The proposed method provides quality STEM images and higher throughput. The authors expect this technique to be of great potential for applications in in-situ STEM imaging in the FIB/SEM, particularly in those requiring high-throughput and high-reliability requirements.

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