The properties of nano-scale defects such as bubbles in materials are extensively studied for both current and potential future purposes. Those range from the mechanical effects of alpha-particle irradiation in nuclear reactor walls, to the study of plasmonics and fluids at the nanometric scale. The creation of He bubbles in Si and other semiconductors is particularly interesting for their potential applications for electronics, such as their ability for gettering or the Smartcut™ process.

Our purpose is to improve the understanding of the processes governing the evolution of those bubbles during thermal annealing by studying their inner fluid pressure and density, which are predominant factors in their behavior during growth.

So far, spatially-resolved EELS has been shown to be a powerful tool for elemental quantification. But the intense probe used for this technique results, for our systems, in the desorption of He, making consecutive experiments on any single bubble difficult. Furthermore, this forbids studying bubbles during in situ annealing. Here the method uses EFTEM instead of STEM for spectrum acquisition, in order to greatly reduce the local irradiation intensity and to facilitate sample drift detection as well (see Figs. 1-3). This, combined with the measurement of the density-related He \(1s\to2p\) transition-energy shift (Fig. 3), provides a means to determine the density in the bubbles.

In our He-implanted Si samples, bubbles range from approximately 20 to less than 5 nanometers in diameter (Fig. 1). Initial results from EFTEM have allowed us to establish conditions and procedures for the acquisition, treatment and extraction of data from the samples. Specifically, optimal parameters were determined for signal quality versus acquisition time. The energy filtering aberrations and sample drift can now be corrected simultaneously, by a procedure which has been coded in-house and shows good results on both aspects. Noise reduction is necessary, and a statistical treatment is applied to the data (MSA) for further signal improvement. Finally, the spectra are deconvolved for multiple scattering, and the He K-edge is extract and fitted (Fig. 3). Pixel per pixel, density and pressure maps can now be obtained over several bubbles simultaneously (Fig. 4), and mean pressure and size can be extracted for each one. While this procedure was being implemented, in situ thermal annealing experiments were performed, clearly showing bubbles emptying between room temperature and 800°C, and movement and shape alteration in the same range. Acquisitions were performed with various annealing temperature and time steps, allowing for the detailed study of the bubbles’ behavior relative to those parameters as well as their proximity with one another.
Fig. 1: Filtered image at 17+/-0.5eV, around Si plasmon, with largest bubbles clearly visible.

Fig. 2: Filtered image at 23+/-0.5eV, around He K-edge, same area as Fig. 1.

Fig. 3: Single spectrum acquired via EFTEM, and post-treatment extracted signal. He K-edge clearly visible at ~23eV, behind Si plasmon at ~17eV.

Fig. 4: Density map obtained from same area as Fig. 1 showing density variations between and across bubbles. Data truncated below 90nm-3 for clarity.