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# Enhanced Preparation Technique of Plan-view Specimens for *in situ* TEM Heating Experiments based on the Synergy of Wedge-polishing and FIB

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Comprehensive characterization is of principal importance for the controlled synthesis and application of nanomaterials. *In situ* TEM, in this respect, is an excellent tool allowing the real-time observation of structural changes at the nanoscale while applying different stimuli such as heat and/or bias. This approach has been revolutionized recently by the introduction of cutting-edge MEMS-based sample carriers [1]. One of the main challenges on a way to a fruitful *in situ* TEM investigation is a suitable sample preparation by thinning the bulk material to electron transparency. It is imperative though that the applied sample preparation technique must be gentle enough to preserve the original material properties intact. This obstacle is further complicated for broad specimens where a free surface must be prepared as a plan-view specimen. It is noteworthy, that for gathering detailed three-dimensional information on the materials' structures, the commonly used cross-sectional specimens must be combined with the plan-view ones [2]. Plan-view geometry is also crucial for obtaining data on the morphological evolution of thin layers [2]. It is also crucial for assessing the areal densities of defects, orientation-related peculiarities or to map strain and functional properties such as ferromagnetism.

To make a step in solving this challenge we elaborate a novel method for sample preparation, which opens the world of *in situ* TEM heating experiments for a vast variety of fragile materials ranging from MBE-grown semiconductor nanolayers [3] to industrial coatings. It involves primary mechanical thinning of a broad sample area from the backside followed by FIB-assisted installation on the MEMS-based sample carrier (Figure 1). The synergy of the wedge-polishing technique and the advanced FIB workflow allows us to combine the advantages of both approaches minimizing invasive effects such as mechanical load and ion beam illumination. Moreover, the broad wedge-shaped specimens are well-suited for cutting multiple lamellae from the same sample giving ground to studying materials properties in a systematic fashion.

The principle and significant advantages of the proposed method are demonstrated by new insights into the thermal-induced strain relaxation and stability of Ge Stranski-Krastanov islands on Si during *in situ* TEM heating (Figure 2). We thoroughly traced the morphological and structural changes on the fly in a single heating-cooling cycle via HRTEM and SAED. To highlight the inherent strain of the SK islands the two-beam condition (TBC) has been utilized. Major strain relaxation in the islands occurs through the formation of stacking faults at 625°C induced by the different thermal expansion coefficients of the inherent materials [3]. These findings can be barely achieved without applying the elaborated method that, in particular, proves its conceptual advancement for semiconductor materials research. We provide the complete step-by-step guide and discuss the method's concept in detail making it easy to follow and adapt for diverse equipment [3, 4].

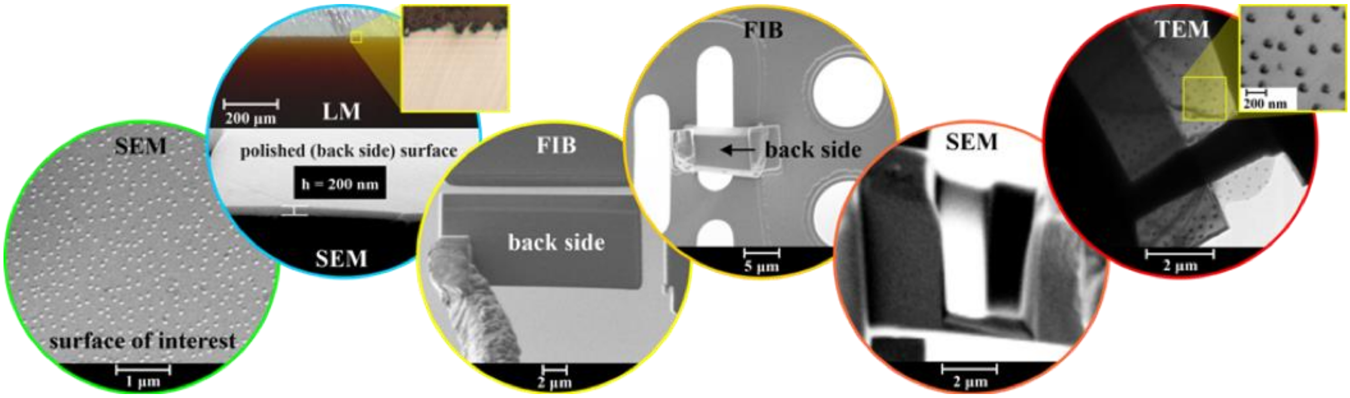


**Figure 1.** The preparation of the test plan-view MBE-grown sample on the MEMS heating nano-chip (Wildfire by DENSsolutions [1]). Left to right: SEM image of the surface before preparation of test sample; light microscopy (LM) image of a wedge polished sample and corresponding SEM image of the achieved thin edge; lift out; installation of the lamella on the chip; final thinning of the sample to electron transparency from the backside; and TEM images of the test sample on the chip.

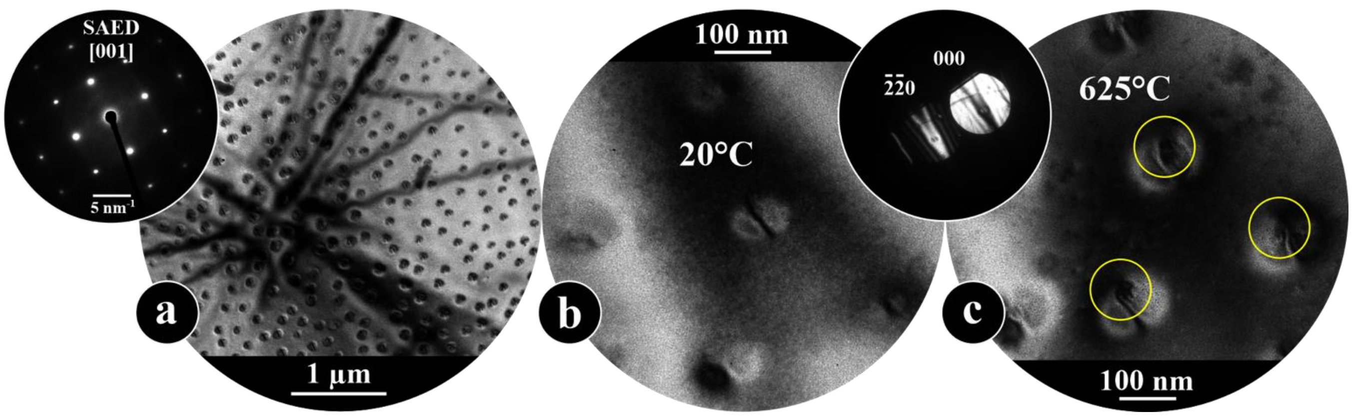


**Figure 2.** (a) – TEM image of the sample aligned along [001] and the corresponding SAED pattern (inset), (b, c) – selected snaps of the sample at different temperatures taken under two-beam condition (inset) recorded during a continuous *in situ* heating experiment. The formation of dislocations within the Ge islands at 625°C is highlighted with yellow circles.

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