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Focused Ion Beam Induced Nanoscale Phase Transitions in Layered Structures

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Specimen preparation for Transmission Electron Microscopy (TEM) using Focused Ion Beam (FIB) is a common method [1]. While it offers many advantages over other methods, like site specific specimen preparation, it also suffers from many disadvantages. Artifacts induced by FIB range from Ion implantation to thermal effects [2,3].

In this work, we investigate thermal effects induced by FIB milling in chalcogenide-based 2D layered Sb₂Te₃ thin films covered by Cu layers. Sb₂Te₃ thin layers are epitaxially grown on p-type Si (111) substrates and polycrystalline samples grown on SiO₂ using pulsed laser deposition [4]. Dependent on beam current used during FIB lamella preparation and Sb₂Te₃ layer thickness, hole formation in the Cu layer, thickness change in the Sb₂Te₃ layer and nanoscale modifications are observed (Fig 1.). The structural changes are confirmed by in situ X-Ray Diffraction heating. The introduction of a separation layer (e.g. Pt) between the Cu and Sb₂Te₃ layers hinders thermal induced structural changes by FIB (Fig 2.). Moreover, Cr - Sb₂Te₃ and a Cu – GeTe layer systems show no modifications during preparation.

[1] T. Ishitani, H. Tsuboi, T. Yaguchi, H. Koike; Transmission electron microscope sample preparation using a focused ion beam; *Microscopy* (1994), 322.

[2] J. Mayer, L.A. Giannuzzi, T. Kamino, J. Michael; TEM sample preparation and FIB-induced damage; *MRS bulletin* (2007), 400.

[3] R. Schmied, J. E. Fröch, A. Orthacker, J. Hobisch, G. Trimmel, H. Plank; A combined approach to predict spatial temperature evolution and its consequences during FIB processing of soft matter; *Physical Chemistry Chemical Physics* (2014), 6153.

[4] H. Bryja, J.W. Gerlach, A. Prager, M. Ehrhardt, B. Rauschenbach, A. Lotnyk; Epitaxial layered Sb₂Te₃ thin films for memory and neuromorphic applications; *2D Materials* (2021), 045027.

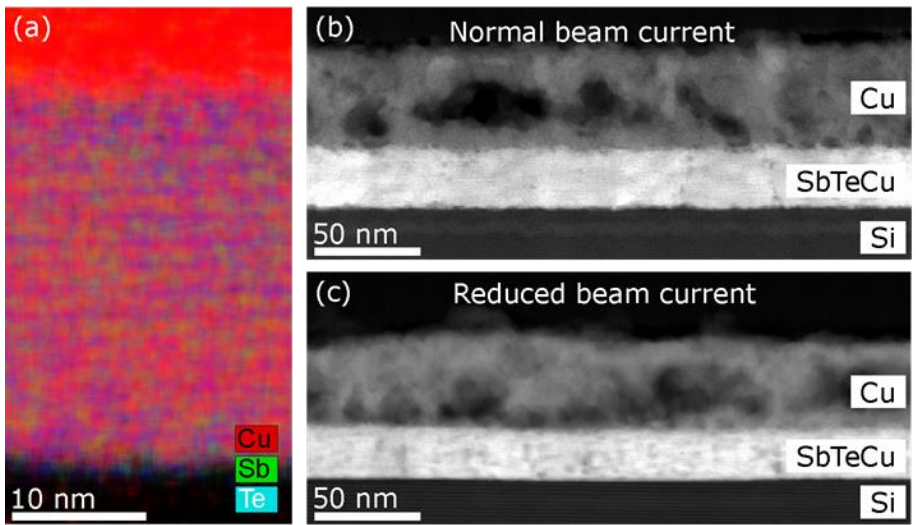


Fig. 1: (a) EDX Map of a lamella prepared with FIB, (b-c) Overview HAADF-STEM images of specimen prepared with normal and reduced FIB beam currents.

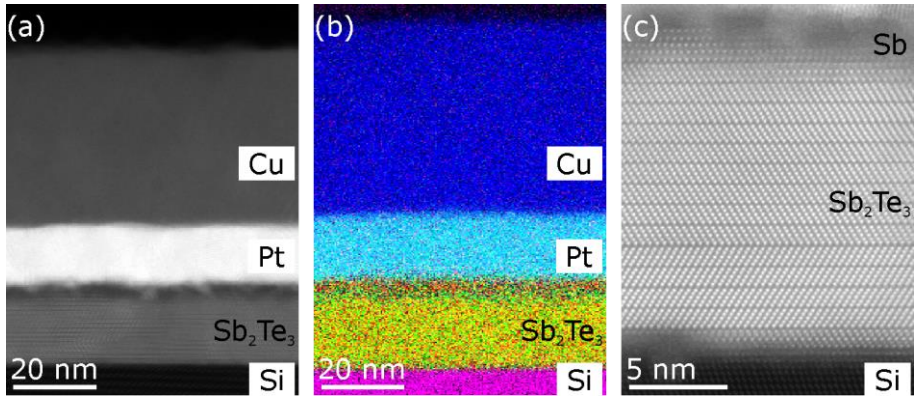


Fig. 2: Cu/Pt/Sb₂Te₃ layer stack. (a) Overview HAADF-STEM image. (b) Overview EDX elemental map. (c) Atomic-resolution HAADF-STEM image, showing initial Sb₂Te₃ structure and no redeposition of Cu.