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Recipe for TEM sample preparation of Bi_2Te_3 samples

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The preparation of the layered three-dimensional topological insulator Bi_2Te_3 is a significant technological challenge, especially if doped with transition rare earth elements. The preparation of transmission electron microscopy (TEM) lamellas often results in dopant clustering when preparing the TEM samples by using ion-milling or focus ion beam (FIB) techniques. Here we present a novel technique for delaminating sensitive films from the substrates and for the preparation of thin TEM slices using ultramicrotomy technique.

Dy-doped thin films were grown on c-plane sapphire substrates by molecular beam epitaxy (MBE) [1]. High-quality samples with a Dy concentration of $x \leq 0.113$ were selected for high-angle annular dark field (HAADF) scanning transmission electron microscopy (STEM) and energy-dispersive X-ray spectroscopy (EDX-) STEM investigations.

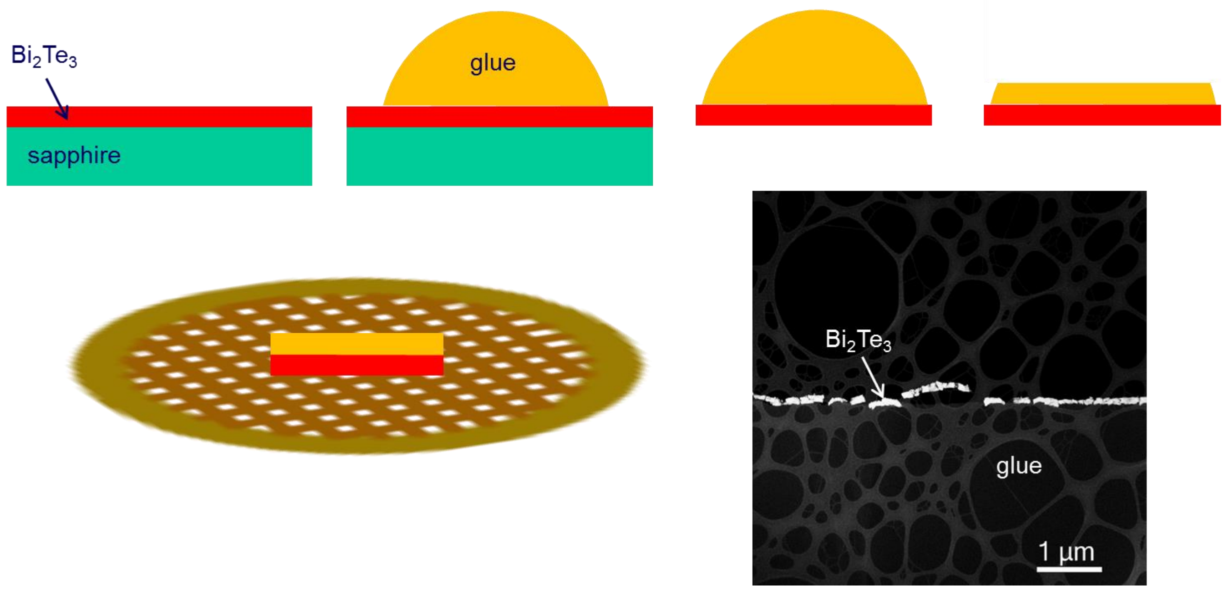

Figure 1: TEM sample preparation of (Dy0.113Bi0.887)2Te3 film. A droplet of glue was deposited on top of the film and after drying removed together with the film. Thin slices were cut using ultramicrotomy and deposited onto the Cu grids with carbon lacey film.

For TEM sample preparation the droplet of glue was first deposited onto the $(Dy_{0.113}Bi_{0.887})_2Te_3$ film and was left to dry (Figure 1). Due to the weak bonding between the film and the substrate, the film was carefully delaminated from the substrate while holding on the removed droplet of glue. Next, thin TEM lamellae were cut out of the delaminated films (attached to the glue) in cross-sectional geometry using an ultramicrotome. Thin sections were obtained using a diamond knife to cut approximately 40 nm thick slices that were captured on Cu grids covered with a lacey carbon film (Figure 1).

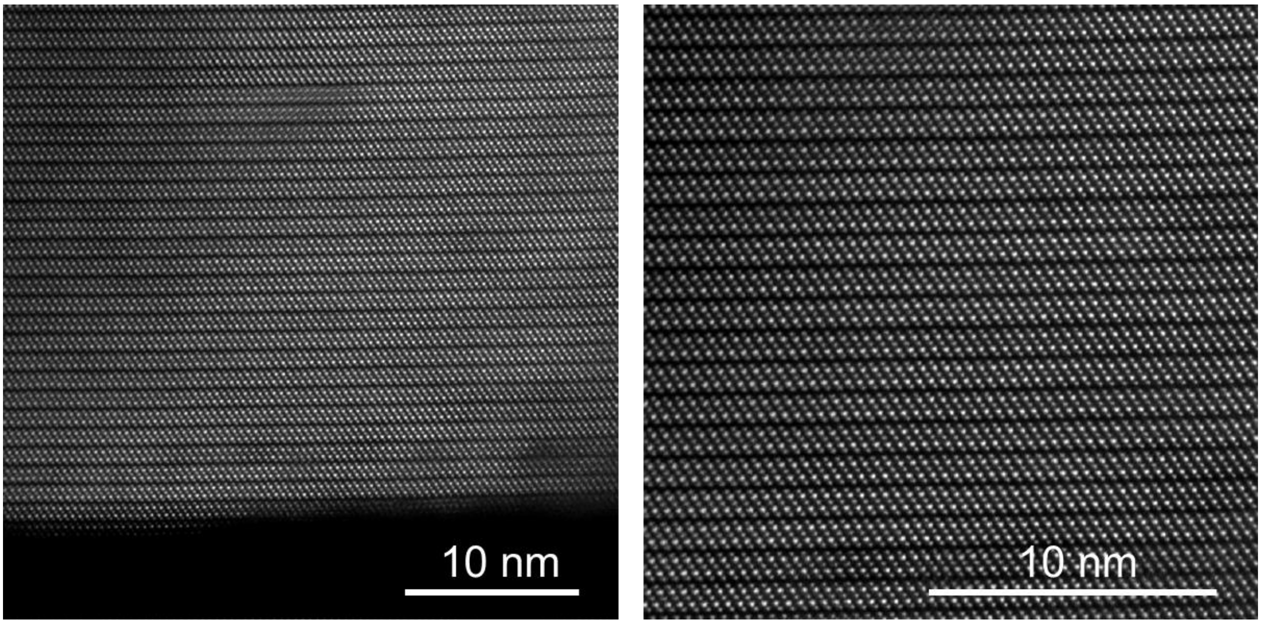

Figure 2: Low-magnification HAADF-STEM images of the high crystallinity (Dy0.113Bi0.887)2Te3 film.

Low-magnification HAADF-STEM images of the high crystallinity $(Dy_{0.113}Bi_{0.887})_2Te_3$ thin films acquired at 60 kV are shown in Figure 2, showing defect free samples. The characteristic crystal structure formed by the stacked quintuple layers separated by the van der Waals gaps is clearly visible. A higher magnified HAADF-STEM image with the overlaid structural model is presented in Figure 3 (left). EDX line-scans were acquired along the Bi lattice planes and traversing the van der Waals gap between the adjacent quintuple layers (see arrows in Fig. 3). The corresponding intensity profiles of Bi-M, Te-L and Dy-L X-ray emission lines along the arrows are presented in Figure 3 (right).

The intensity of the Dy-L signal is exactly following the Bi-M signal, indicating the substitutional incorporation of Dy atoms on Bi sites and the absence of Dy in the van der Waals gaps. Our measurements show no evidence for cluster formation or local phase segregations [2].

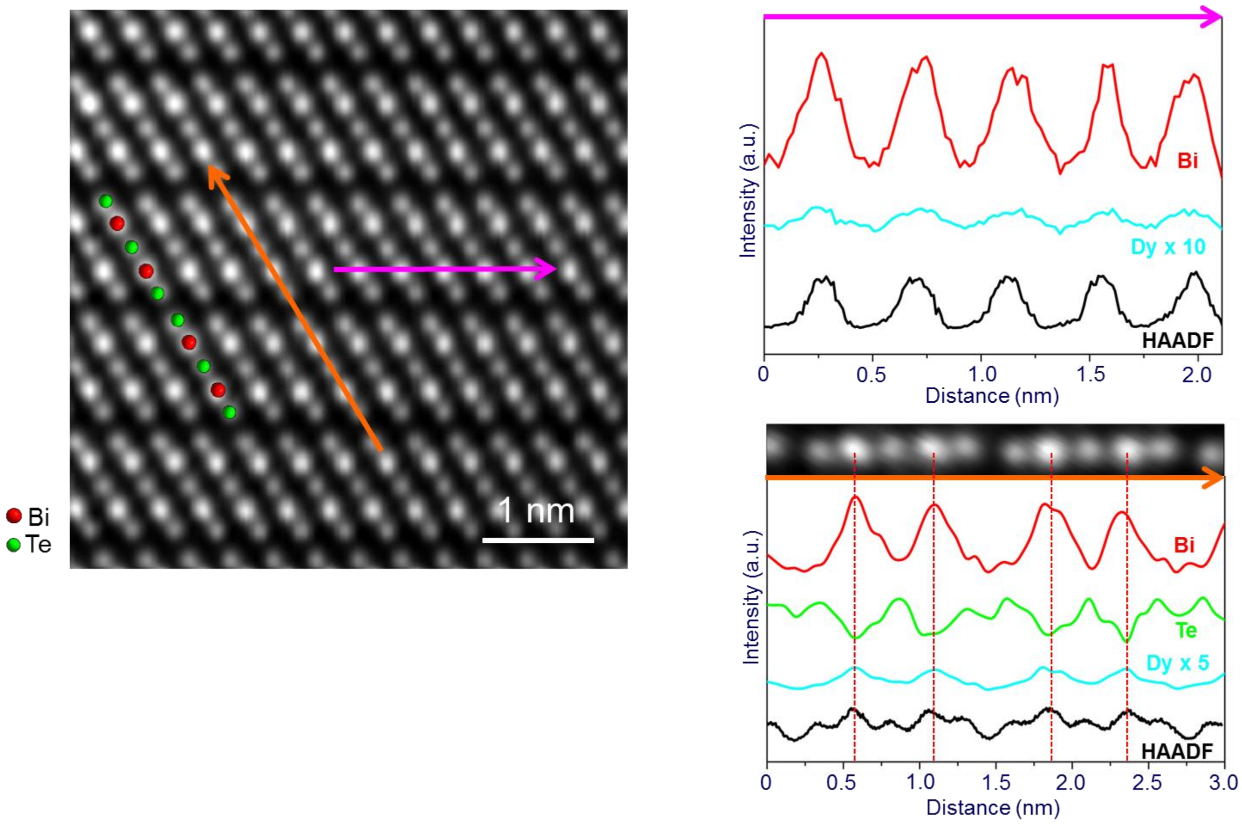

Figure 3: HAADF-STEM image with corresponding EDX line profiles acquired along Bi lattice planes (violet) and across the van der Waals gap (orange).

References:

[1] SE Harrison et al., *J Phys: Condens Matter* **27** (2015), 245602.

[2] SE Harrison et al., *Sci Rep* **5** (2015), 15767.