



## TEM observation of blisters in thin films

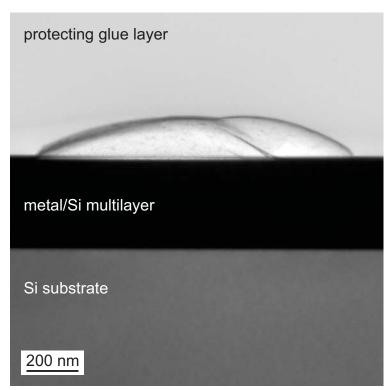


Fig. 1: TEM cross-section revealing a fully intact double-blister with diameter of the order of 1.5  $\mu$ m. The specimen thickness at this stage is estimated to be at least 1.5  $\mu$ m, allowing to observe the fully intact hemispherical half-dome of the double-blister though. Individual layers are however, barely visible.

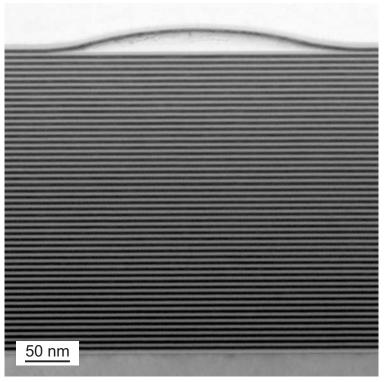


Fig. 2: TEM cross-section revealing a fully intact blister with diameter of the order of 300 nm. The specimen thickness at this stage is estimated to be of the same order of magnitude. At this specimen thickness the individual Metal and Si layers comprising the multilayer become distinguishable.

The surface of a metal/Si multilayer was purposely exposed to a harsh external source. Following this treatment AFM measurements indicated the formation of blisters with strongly varying diameters in the range of several tens of nm, up to approx. 2  $\mu$ m (see Ref. [1] for full background info on this subject).

To study these surface effects in more detail, a suitable cross-sectional TEM (XTEM) specimen was made by Dimple Grinding / Polishing and Argon ion Milling.

It is essential that as many areas as possible are included in the XTEM study to provide sufficient statistical data. Where possible from areas with the appropriate TEM thickness (approx. 100 nm), but in general the majority of the analyses were in this study explicitly performed on thick (>> 100 nm) to extremely thick (0.5 – 2 µm) areas.

An important advantage of TEM analysis on relatively thick areas is that, in many cases, the entire blister can be observed fully intact, that is, in the direction of the projection of the 3D structure into a 2D image. Disadvantage is however, a progressive deterioration in TEM image quality, in particular for areas with thickness far exceeding 100 nm.

An added benefit of exploring relatively thick specimen areas is that any possible detached metal/Si film fragments are well preserved by the protecting (GATAN G-1) glue layer, allowing extended electron beam exposure for TEM analysis.

The TEM images shown here were recorded after successive Ar+ milling stages (Fig. 1 - 4, resp.), yielding a progressively thinner specimen.



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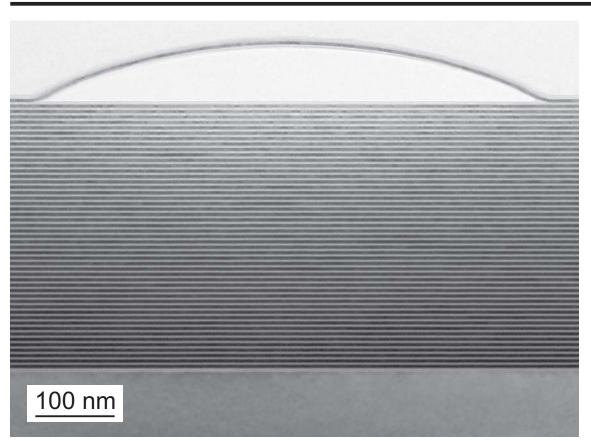


Fig. 3: TEM cross-section recorded at an advanced argon ion milling stage, yielding the appropriate specimen thickness for general Bright Field TEM imaging. The BF TEM image now shows a thin slice of the hemispherical halfdome of a single blister, such as shown in Fig. 2.

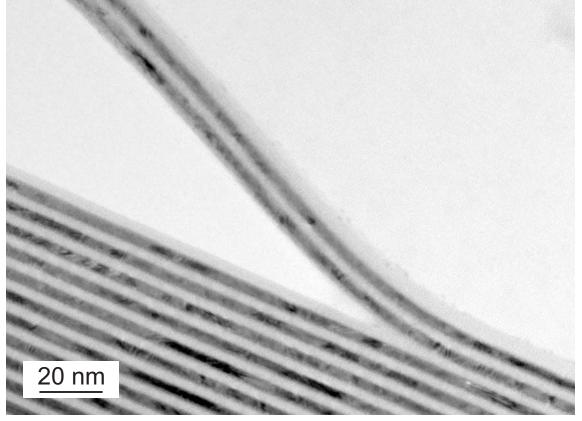


Fig. 4: TEM cross-section, recorded at the final argon ion milling stage following the stage in Fig. 3, showing a close-up of a double-bilayer detachment point. TEM specimen thickness is 50 - 80 nm, allowing high-quality TEM imaging.