MESA+
INSTITUTE FOR NANOTECHNOLOGY

NanoLab Twente

TEM EXAMPLE

Ion beam induced artifacts; focus on FIB

INTRODUCTION

Ion milling with Argon gas is usually the final step in TEM specimen preparation by mechanical polishing, such as Dimple Grinding/Polishing and wedge (TriPod) Polishing. For a description, see Application 1. It involves bombarding the fragile thin specimen with energetic ions so that material is sputtered away from the specimen slice until it has reached the required thickness for optimum TEM observation. Variables that have a major effect during ion beam thinning are the ion energy and its angle of incidence.

Ion milling can also be used as a primary tool to make thin electron transparent slices out of a sample. This is achieved with Focused Ion Beam (FIB). In the standard FIB configuration a high energetic beam of Gallium ions is used to remove material (see Ref. [1]).

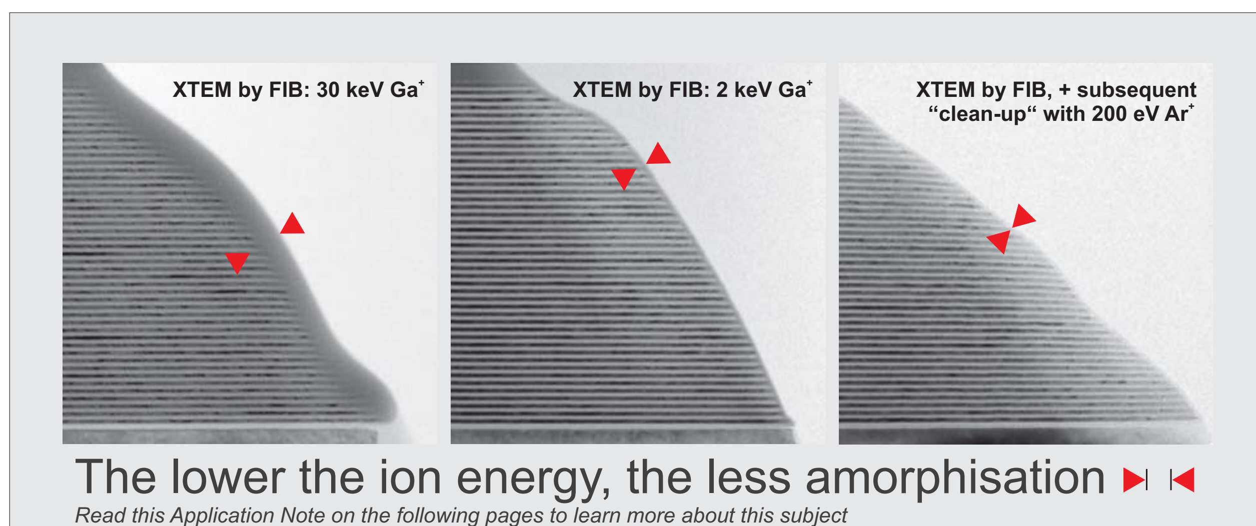
Ion-beam induced artifacts easily occur when performing sample-preparation. In general, the higher the ion energy and mass of the ions, the more damage is generated (see Ref. [2] and references therein). Amongst the surface artifacts by Ar ion sputtering or by Ga ion impact in FIB are: surface amorphisation, defects, redeposition, preferential sputtering, etc. Observable in High-Resolution TEM imaging mode by foggy contrast and identifyable in chemical analysis (e.g., EELS, EDX). The thicker the specimen is, the relatively less influence the damaged/amorphised surface layer will have on imaging quality and quantification results. Therefore, the largest deviations can be expected on the thinnest sections of a specimen, seen as apparent loss of crystallinity in crystalline samples.

This application note illustrates in an easy-to-read style how surface amorphisation of a representative XTEM specimen created by ions, and by FIB in particular, can affect TEM imaging, presenting possible methods to reduce, or even remove this unwanted artifact.

- A schematic representation of a thin electron transparent specimen when viewed in cross-sectional TEM (XTEM) is given in Figures 1 and 2 on p. 2.
- Possible methods to reduce ion beam induced artifacts are mentioned on p. 3.
- The amorphisation effect is best observed in the edge region of a typical specimen, as the example of Fig. 4 on p. 4 shows.

FOR WHO IS THIS APPLICATION NOTE?

All Newbies interested in this subject, and those wishing to see how ion beam induced amorphisation can manifest on a real-world thin TEM specimen.



[1] “Introduction to Focused Ion Beams: Instrumentation, Theory, Techniques and Practice“ (2005), (eds. L. A. Giannuzzi and F. A. Stevie) Springer Publishers. ISBN 978-0-387-23313-0.

TEM EXAMPLE

Ion beam induced artifacts; SCHEMATIC

Ideal electron transparent crystalline XTEM specimen ( ):

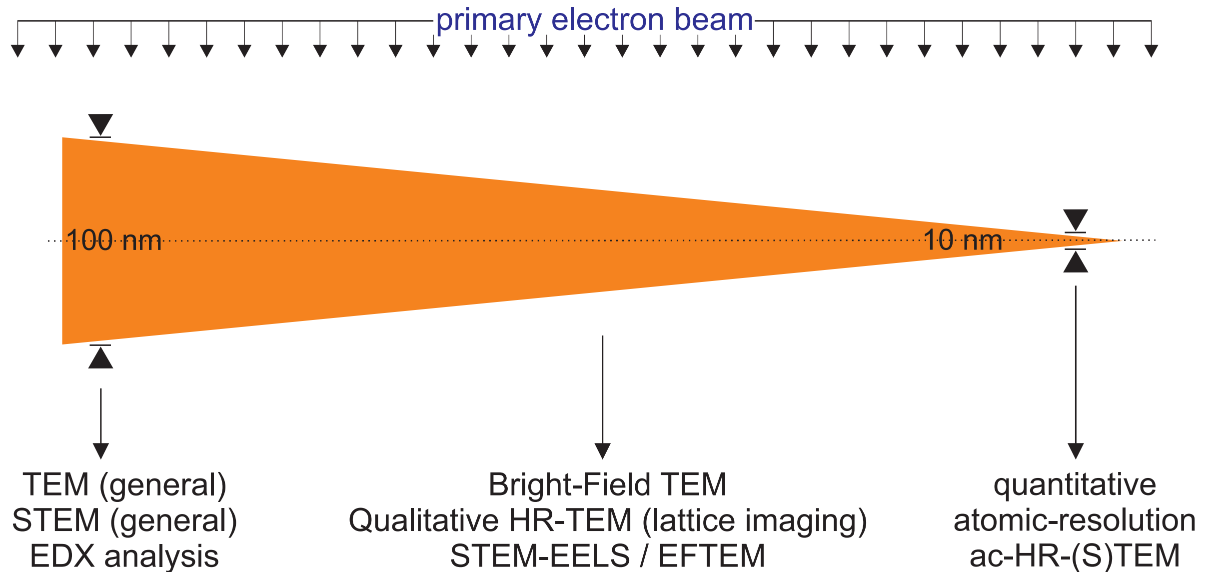


Fig. 1: Schematic depiction of the cross-section of an ideal crystalline TEM specimen in a wedge shape with optimum thickness in the range of 100 to less than 10 nm, without any damage. Such a specimen can be imaged in various TEM or Scanning TEM (STEM) modes for various purposes, and down to the thinnest section (< 10 nm) for quantitative atomic-resolution (S)TEM with aberration correction (ac), when required, including associated chemical element analysis techniques such as Energy Dispersive X-ray spectrometry (EDX) and Electron Energy Loss Spectroscopy (EELS).

Real-world crystalline (wedge) TEM specimens suffer from damage/amorphisation ( ) upon ion etching:

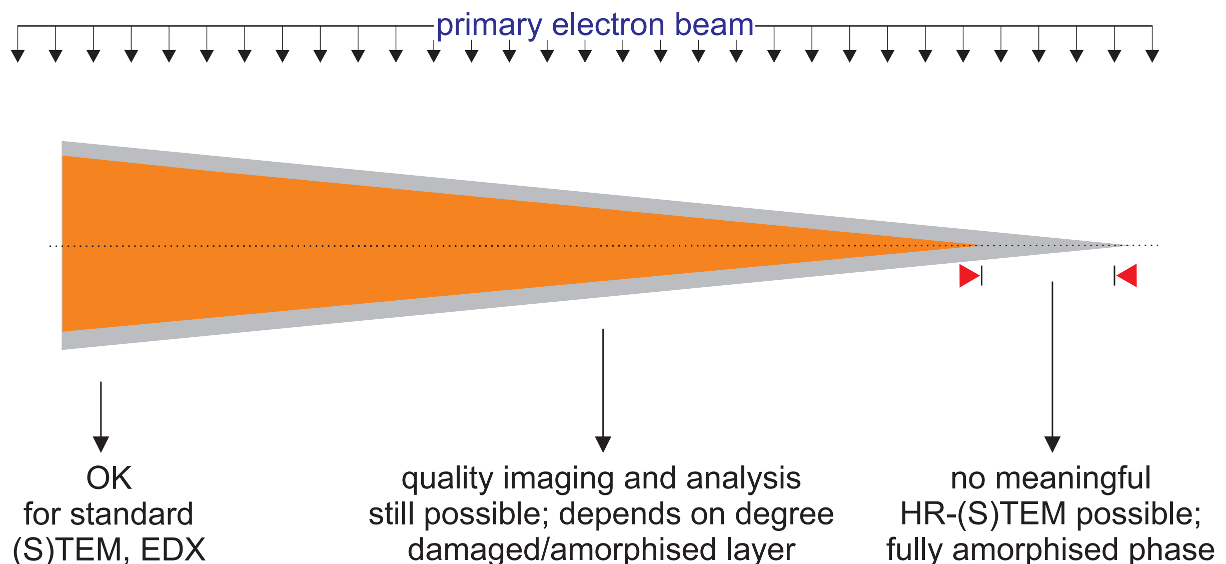


Fig. 2: Schematic representation of the same cross-section of a crystalline TEM specimen as in Fig. 1, shaped into a typical wedge slice after ion thinning. As a result of ion impact on the specimen the surface area on both sides of the thin slice is damaged. The degree of surface amorphisation depends on the mass and energy of the ions, and on the angle of incidence. In general, the higher the ion energy and mass of the ions, such as in FIB, the more damage is generated. This effect can be easily observed on the edge of a thin specimen (see p. 4).

[2] "Transmission Electron Microscopy (A Textbook for Materials Science Basics, Diffraction, Imaging, Spectrometry)", by David B. Williams and C. Barry Carter. Plenum Press, 2nd Edition, 2009: softcover: ISBN 978-0-387-76502-0

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TEM EXAMPLE

Ion beam induced artifacts; REDUCTION

Recently, it has been reported that the amorphised surface layer thickness can be reduced to as little as 1.5 nm in FIB, using a 2 keV Gallium ion beam in the final milling stages (see Ref. [3]), thus enabling the use of even the thinnest section of the specimen for HRTEM observation (see Figs. 1, 2).

The amorphised surface layer of a thin TEM slice created by FIB can, however, be reduced even further down to essentially zero (0) nm, as we experienced, using Post-Processing with low energy Argon ions. Turn to page 4 to learn how this can be accomplished.

Alternatively, the (co-)use of He or Ne ion beams, as generated in instruments with a Gas Field Ion Source, such as the Helium ion Microscope (HIM), - in conjunction with a traditional Ga ion beam within the same instrument -, looks to be a very promising method for improved quality XTEM sample preparation and sectioning (see, e.g., Ref. [4]) .

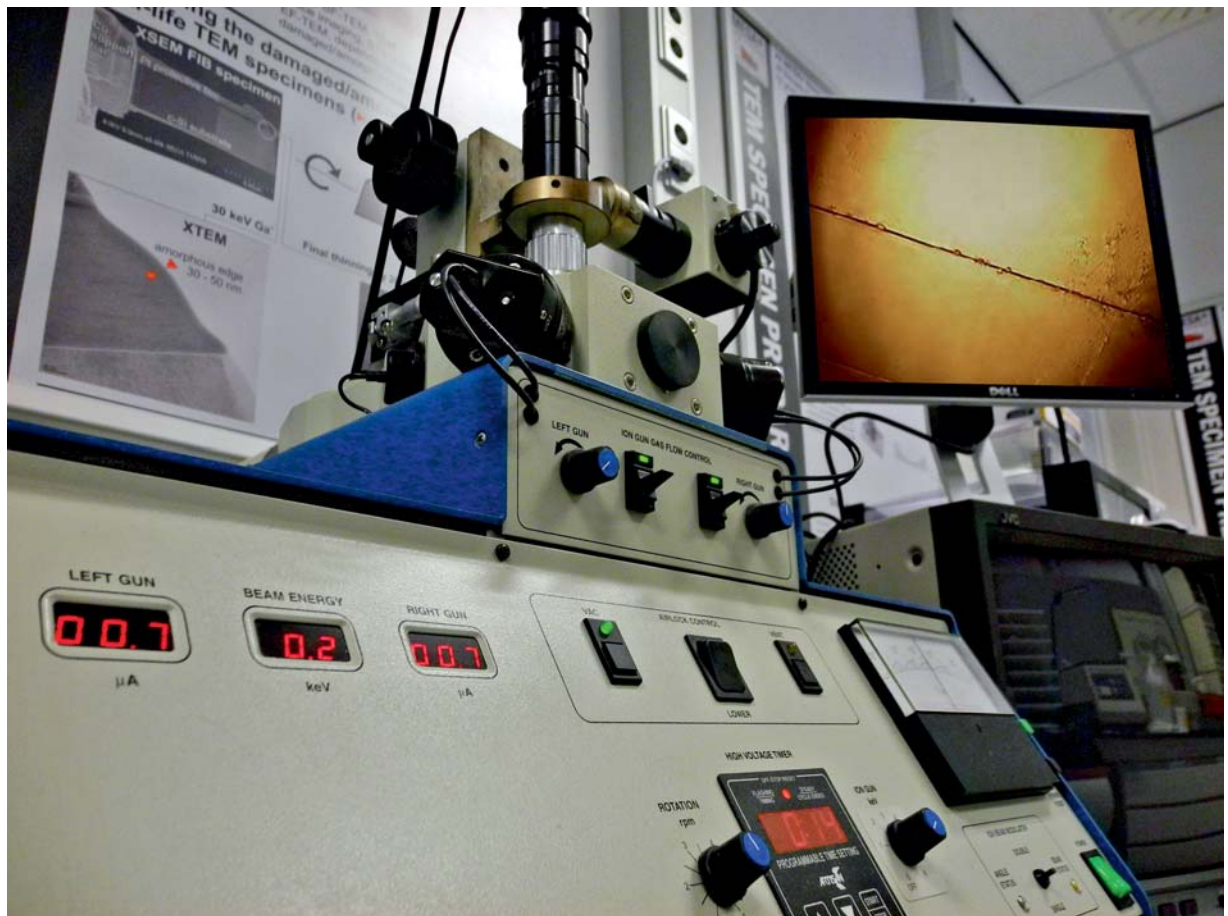

Fig. 3: GATAN model 691 dual-beam ion miller in action at MESA+ NanoLab. The Argon ion beam energy can be adjusted in the energy range of 6 - 0.1 keV. The PIPS, as the instrument is commonly named, is used to obtain final electron transparency of TEM specimens pre-thinned by mechanical polishing techniques, such as Dimple Grinding / Polishing (see Application 1). With a bit of practice, however, the PIPS can also be used to remove (part of) the amorphous layer on a thin specimen created by FIB. Alternatively, one may consider the use of Fischione's NanoMill (see Ref. [5]), an instrument that is dedicated for this single task alone (see the example on page 4).

Additional information regarding the "clean-up" method of lift-out TEM specimens from FIB instruments by low energy Argon ion beams [5] can be found on the web-site of GATAN:

http://www.gatan.com/files/PDF/products/app_notes/AppNote_PIPS_II_Argon_Ion_Polishing_FL1.pdf
http://www.gatan.com/files/PDF/knowhow/Know_How_summer_2013_web_copy.pdf

[3] "Latest Developments in FIB Technology and Applications", Lucille A. Giannuzzi, Microsc. Microanal. 16 (Suppl 2), 2010 © Microscopy Society of America 2010, p. 166, 167. doi:10.1017/S143192761006126X

Ion beam induced artifacts; EXAMPLE

Reducing the damaged/amorphised top layer(s) of real-life TEM specimens (►| |◄) by lowering the ion energy:

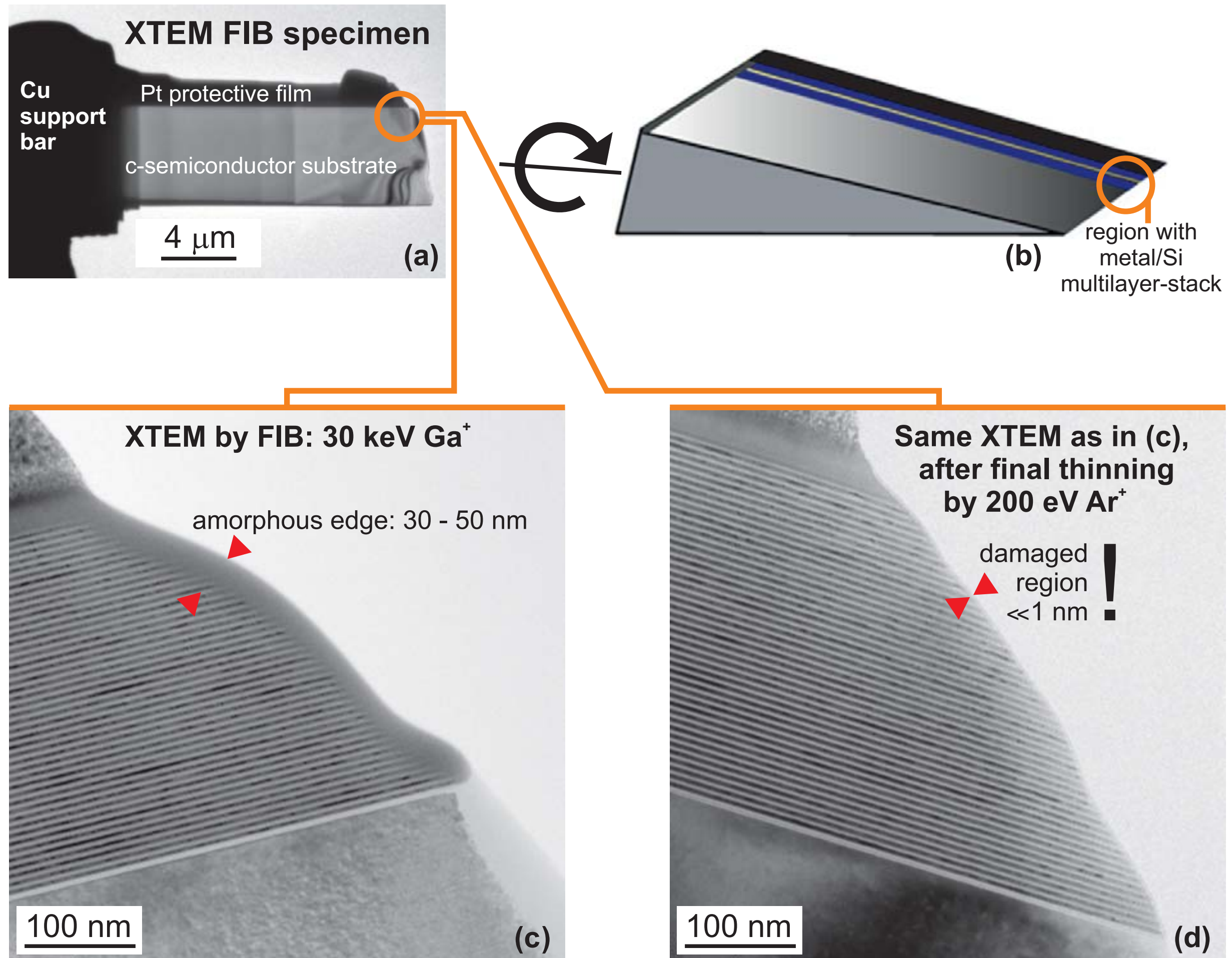

Fig. 4: FIB was applied to prepare a thin TEM slice out of a test sample composed of a metal/Si multilayer stack on a crystalline semiconductor substrate. An overview of this slice by XTEM is displayed in (a). It shows the typical components of a FIB specimen: a thick Pt layer to protect the precious sample surface from the impact of the high-energy heavy Gallium ions, and the Cu support bar, mounted onto a suitable TEM grid. A schematic representation of this XTEM FIB specimen is shown, - rotated -, in (b). The XTEM images shown in (c) and (d) are the zoomed-in areas of the encircled parts in (a) and (b). The TEM image in (d) is the same as in (c), after Post-Processing with low-energy Ar ions. See below for an explanation.

A beam of 30 keV Ga ions in a typical FIB configuration can cause roughly 20 nm damage per specimen side in Si (Refs. [1, 3]), observable as an amorphised edge of ~ 30 - 50 nm width (c). When the Ga ion beam energy is reduced to 2 keV, our results indicate a total damaged layer thickness of the order of 3 - 7 nm (see middle photo in showcase display on page 1). Almost complete removal of this damaged layer could, however, be achieved when the specimen in (c) was exposed to a beam of Ar ions at very low energy (200 eV), as evidenced by the XTEM recording in (d), using Fischione's NanoMill instrument (Ref. [5]).

<u>Summarising</u>: the quality of XTEM specimens made electron transparent by FIB milling can be improved significantly by lowering the FIB accelerating voltage, Post-Processing with ultra-low energy Argon ions, resulting into even better results.

[4] „Multi-Beam Ion Microscopy", by David C. Joy, MicroscopyToday 20(5)2012. doi:10.1017/S1551929512000569.
[5] Fischione's NanoMill (see http://www.fischione.com/) was tested by us, using our FIB thinned XTEM specimens (Fig.4).