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Application Note

Argon ion milling of FIB lift-out samples

Introduction

The high-resolution TEM and combined analytical methods became more and more important in the recent investigations of material science. As a result TEM sample preparation plays an important role in this process. For semiconductor materials the use of focused ion beam (FIB) is a common method in TEM sample preparation at present. However, FIB has some drawbacks, especially in high-resolution TEM (HRTEM) imaging. The high-energy Ga^+ ions used in FIB create amorphous layer on the sample surface limiting the information that can be obtained from the HRTEM investigations.

The Gentle Mill low-energy Ar^+ ion milling system has been developed for decreasing and/or eliminating these artifacts in the FIB prepared samples. The report provides some basic instructions for preparing TEM samples by low-energy ion milling of FIB samples.

TEM sample preparation for high-resolution TEM application

FIB sample preparation

The two most common techniques of FIB preparation are the so-called H-bar and Lift-out techniques.

H-bar samples: a slab of material is initially thinned by mechanical polishing down to a thickness of about 50µm. Then FIB is used to cut two trenches, one from each side, leaving between a thin lamella supported by two side bars on the opposite ends.

Lift-out samples: a similar approach is used, however the thin lamella is cut away from the side bars and lifted out from the trench. In-situ lift-out allows the thin lamella to be attached to a grid by FIB welding techniques.

Low-energy ion milling (Gentle Mill)

Low-energy ion milling is used for removing the damaged, amorphized surface layer generated by the high-energy Ga^+ ions during the FIB sample preparation process.

In case of H-bar samples - as a first step of the process - final trimming is performed on both sides of the FIB lamella by medium-energy (around 1000 eV) noble gas ion milling lasting for some minutes. Afterwards, by reducing the ion energy to about 200-300 eV final cleaning is done for a few tens of minutes in order to obtain good quality HRTEM samples. The entire process is completed within 1 hour depending on the parameters of the FIB processing.

Since the Lift-out samples are usually thinner than the H-bar ones, a shorter time of medium-energy ion milling in combination with low-energy cleaning may be sufficient to achieve the desired HRTEM sample quality.

In the following sections recommended low-energy ion milling/cleaning parameters are shown for some possible arrangements of FIB samples mounted on different types of grids.

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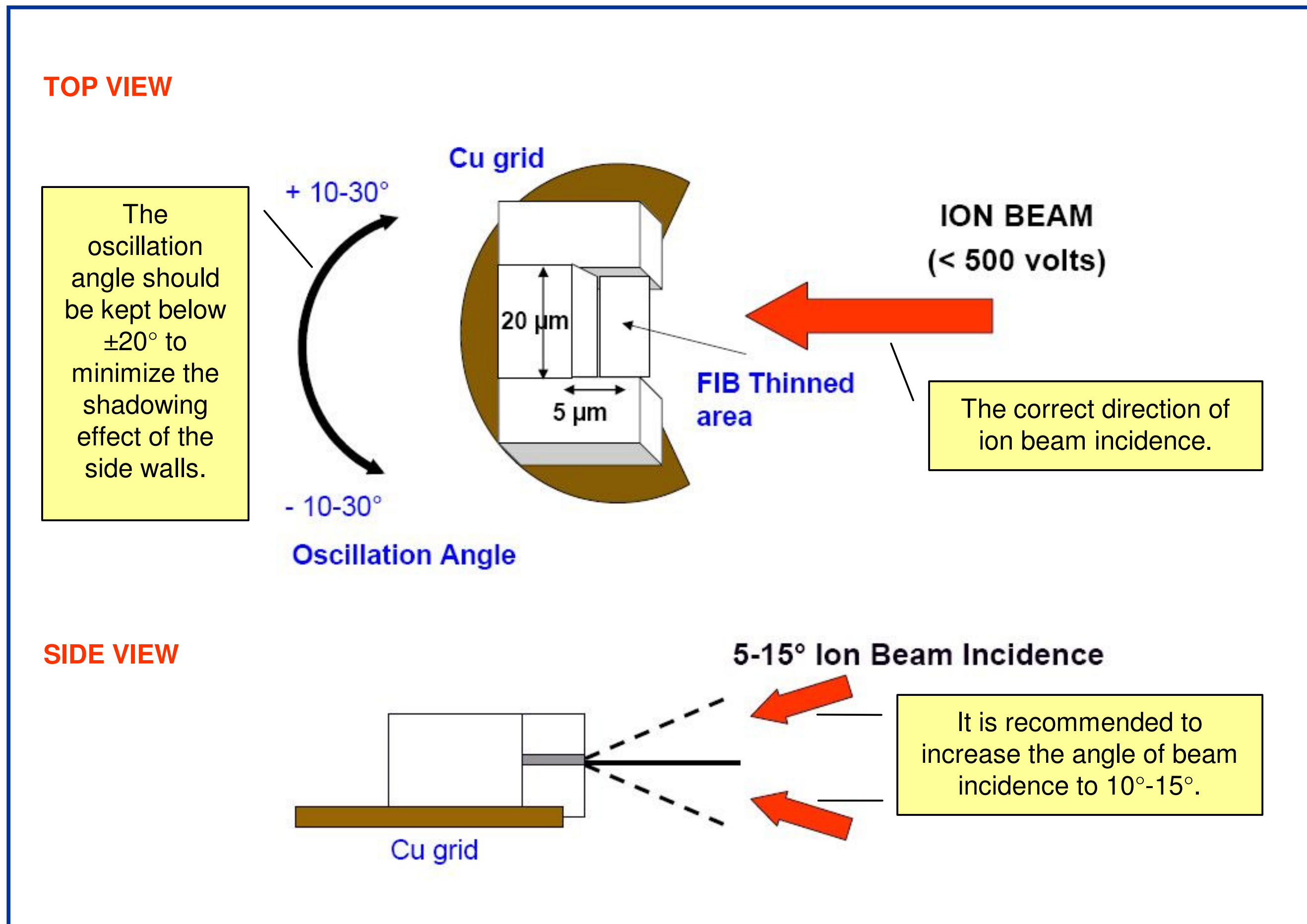
H-bar samples



Lift-out samples

Illustration of the lift-out technique:
The FIB lamella is welded (fixed) to a grid before further processing. Once the FIB welding process is completed and the lamella is properly fixed to the grid, medium- and low-energy ion milling is performed on the sample using the Technoorg Gentle Mill equipped with a dedicated low-energy ion source.

In order to avoid any re-deposition or sample contamination proper noble gas ion milling conditions should be set. The following parameters should to be taken into account in order to find the proper milling conditions for a given arrangement:

- the geometry and the material of the grid,
- the size and the material of the lamella,
- presence of any protective layer on the lamella and the material of the protective layer,
- the way of sample mounting on the grid.

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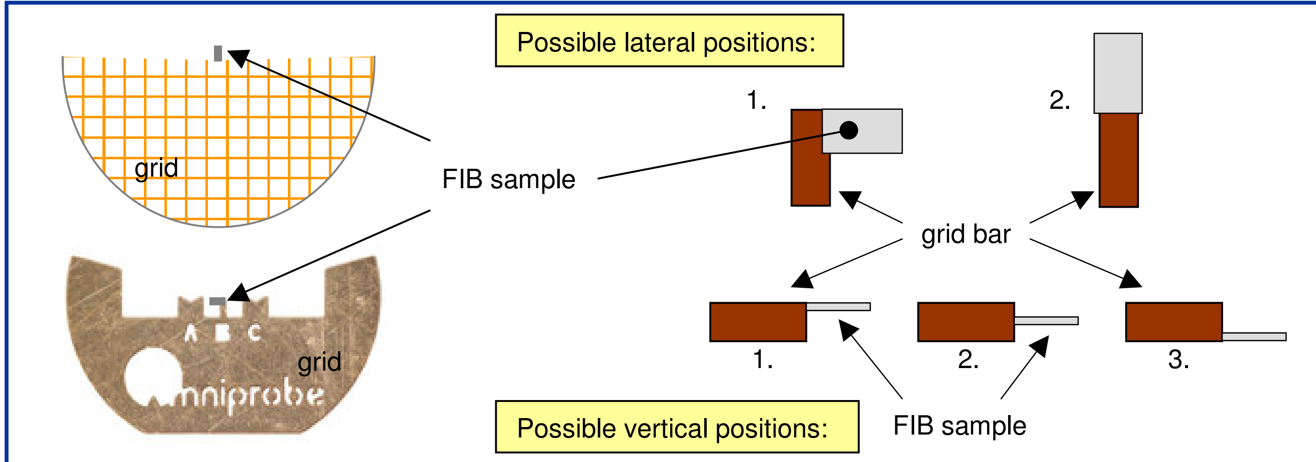
Lift-out samples

Different mounting positions of the lamella on the grid:



Sample 1

Standard approach

TOP VIEW and BOTTOM VIEW

α, β

sample

grid

SIDE VIEW

α

β

Ion beam incidence at minimum access angle – standard approach

$\alpha \ll \beta$

Low-energy milling directions

Advanced milling scheme

TOP VIEW

10°

±30°- 40°

BOTTOM VIEW

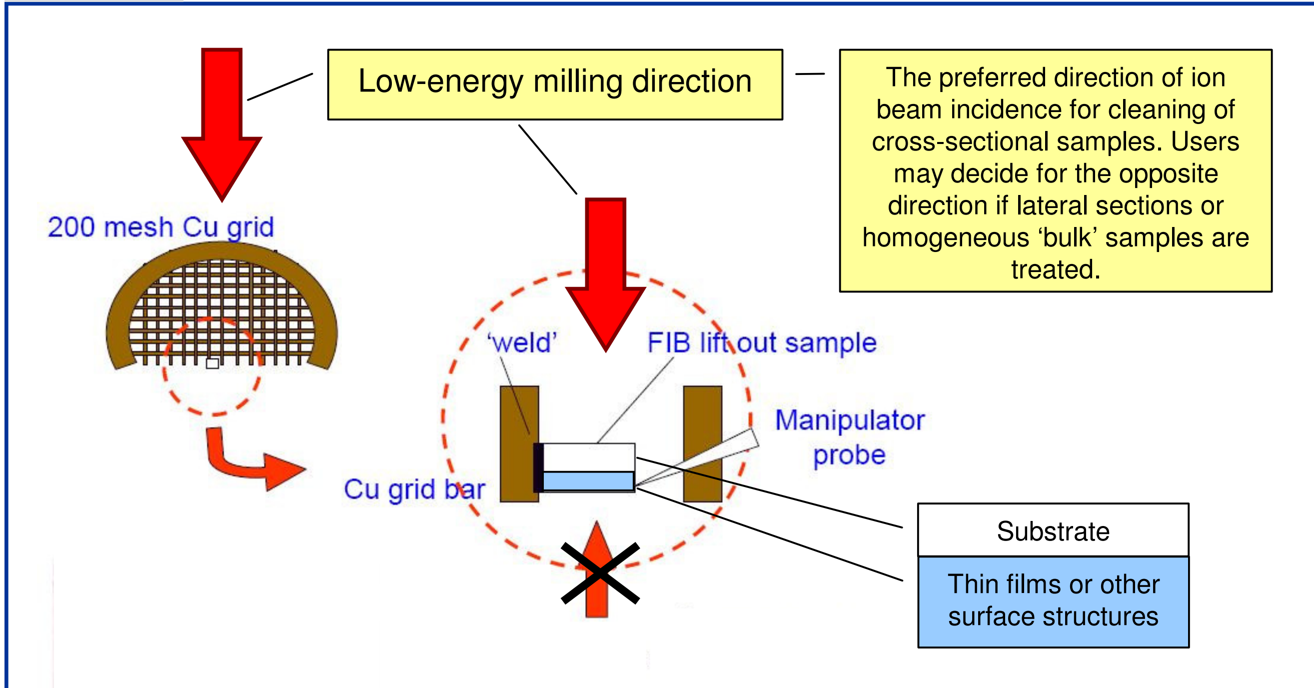
10°

±10°

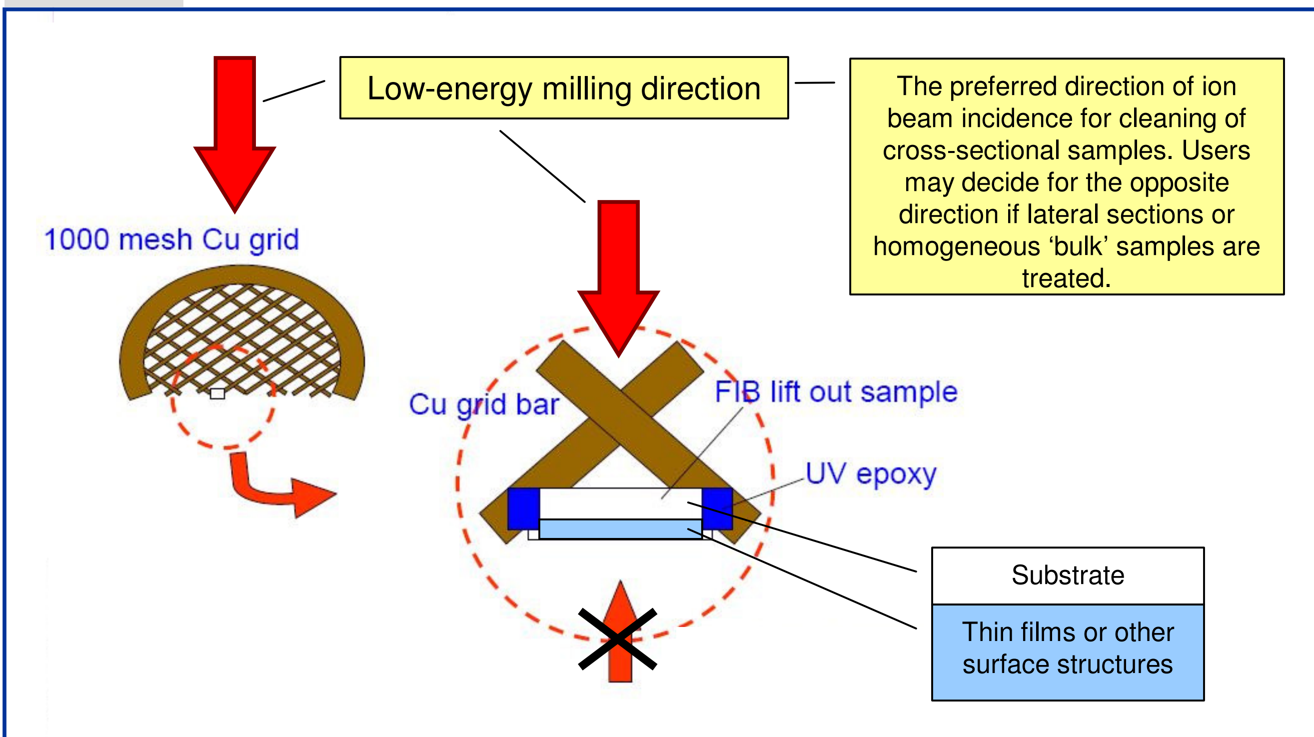
Recommended accelerating voltage: 200-300 eV
Angle of beam incidence:
top:10° bottom: 10°
Angle of oscillation:
top:±10 ° bottom: ±30°-40°

Lift-out samples

Sample 2

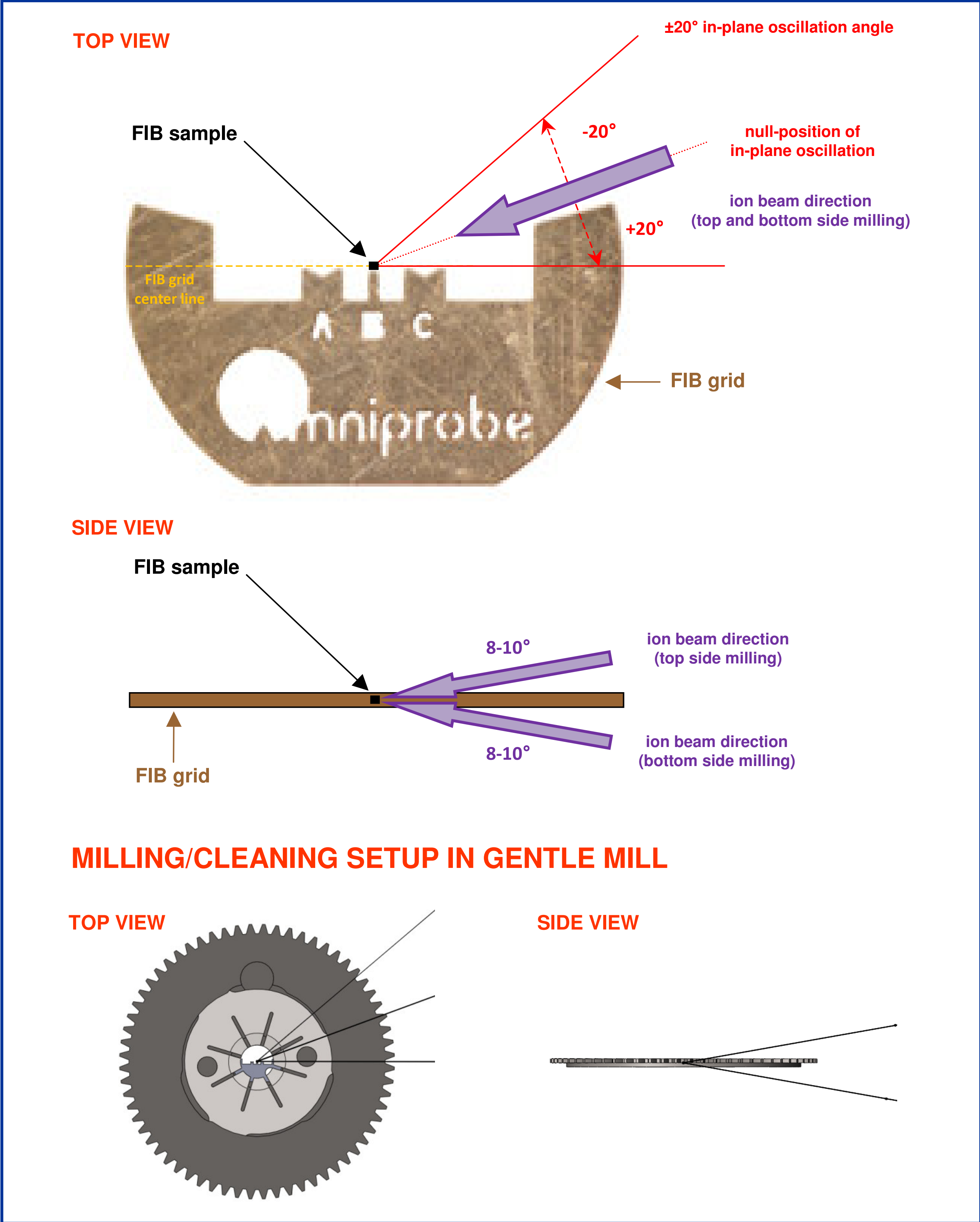


Sample 3



Lift-out samples

Sample 4



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