VAMAS

Quantitative Microstructural Analysis
Technical Work Area 37

Call for Participation

Project 7

Evaluation method of surface layer quality of TEM specimen prepared by focused ion beam processing

Objectives:

The aim of this Interlaboratory Comparison (ILC) is to develop a method for evaluating damage layers introduced by FIB fabrication on the surface of transmission electron microscopy (TEM) specimens. Such damage layers are commonly affected by the acceleration voltage conditions at the finishing processing stage. A single-crystal Si is provided, and the thickness of the amorphous layer introduced as a results of one of the ionic damages is determined by TEM observation.

Background:

In the case of semiconductor Si devices, an amorphous layer typically forms on the surface during FIB fabrication; in order to evaluate device properties, it is necessary to carry out a finishing process to reduce this amorphous layer. In metals, these finishing processes can introduce fine lattice defects on the surface, which are deleterious for TEM observations. Attempted methods for removing the damage layer have included lessening the acceleration voltage during FIB fabrication, and removal of the damage layer by Ar^{+} ion milling. However, a well-defined method for evaluating the area of the damaged layer in FIB fabricated samples has not yet been established.

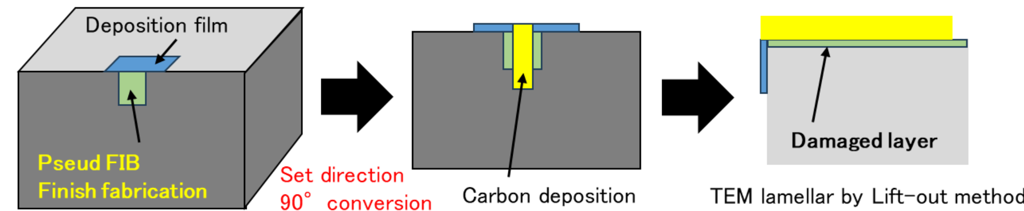
Standardization needs:

In order for TEM specimens prepared by FIB fabrication techniques to accurately reflect the desired material microstructure, the damaged layer must be removed. It is necessary to establish a procedure to objectively evaluate the thickness of the damage layer introduced in various TEM specimens.
Since the thickness of the amorphous layer introduced as a damage layer in Si specimen varies depending on the acceleration voltage and other conditions during FIB processing, a common criterion for evaluating this thickness should be developed. The sample distributed is (001) single crystal Si, and the FIB fabrication conditions are reported in this ILC.. Verify that differences in damage layers caused by differences in FIB equipment and finishing conditions can be evaluated by TEM observation as differences in amorphous layer thickness.

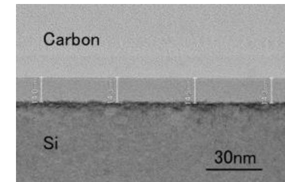
Relevant Standards:

ISO/FDIS 17297 FIB Vocabulary
ISO/TC202/SC1 - in progress

ISO/CD 16887 Guidelines for TEM specimen preparation using FIB
ISL/TC202/SC3 - in progress



I. FIB finishing fabrication has been demonstrated at edge area of [001] single Si crystal.
II. The sample is taken out of the FIB equipment and tilted at 90° to protect the processing surface by C deposition.
III. TEM lamellar is picked up and the thickness of amorphous region at the top of Si wafer is measured.



Work Programme:

1. **June 2025:** Specimens will be delivered to participants along with the protocols.
2. **June/Aug 2025:** FIB processing of thin lamellae and successive TEM observation will be done by the participants in accordance with the protocols provided.
3. **September 2025:** Data submission and analysis
4. **October 2025:** First draft of ISO/PSI will be established.
5. **January 2026:** A peer-review publication is prepared on next step.

Deliverables and Dissemination

- Results disseminated in a peer-reviewed scientific journal.
- Evaluation method of damaged layer of TEM specimen made by FIB will be established by the ISO in the TC 202/SC3.

Funding:

Participants fund their own involvement in the project.

Status: The project is due to start in March 2025 for a duration of up to 24 months, additional participants are welcome.

For more information:

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