

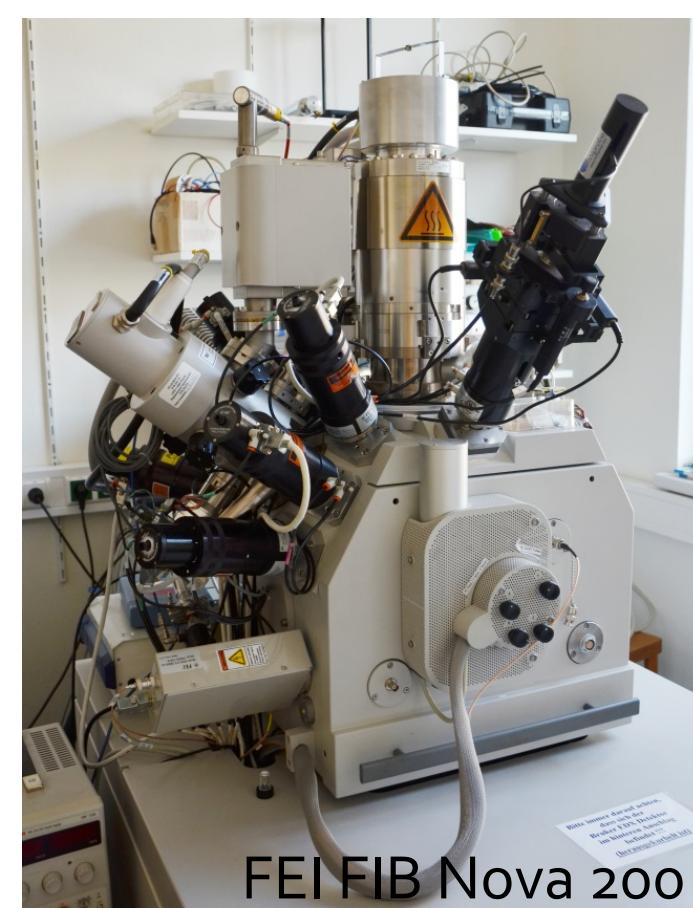
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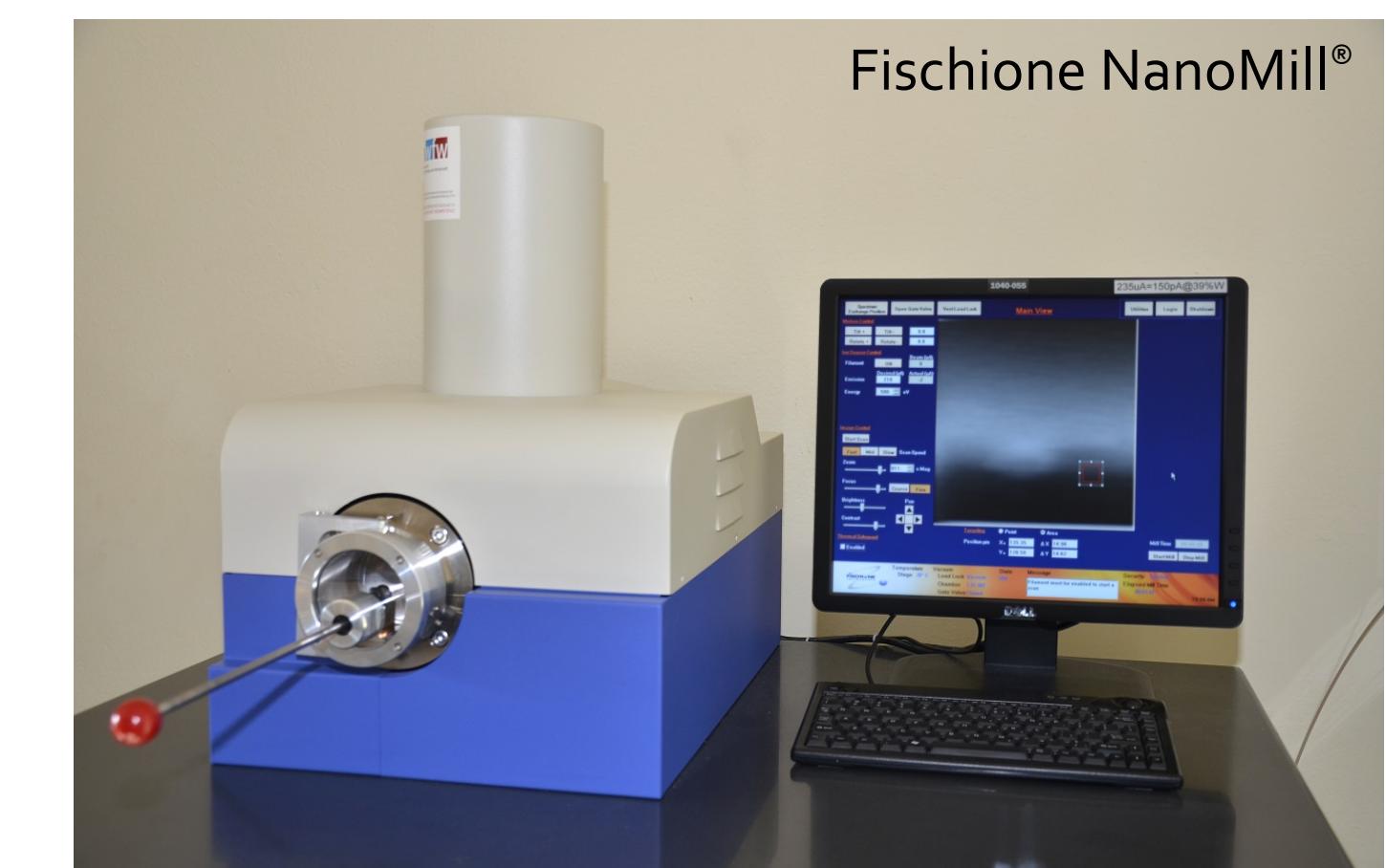
Introduction

The advent of fully corrected ultra-high-resolution transmission electron microscopes (TEM) enabled access to the sub-atomic scale and has become paramount for innovations in material design relevant to science and technology. Their vastly improved performance for the characterization of materials is accompanied by the need to generate high-quality samples via sophisticated and innovative sample preparation techniques. Specimen preparation [1-3] hence is the key to obtain representative insights into morphology, structure, chemistry and functionality. Even minor preparation artefacts such as thin amorphization layers, ion implantation, selective milling or re-deposition can already prevent a deeper insight to a material [4-6]. To overcome this limitations the NanoMill® instrument is a versatile tool to improve the sample quality by a post treatment of the samples with low energy argon ions.

NanoMill® - low energy argon ion thinning and cleaning of FIB lamellae

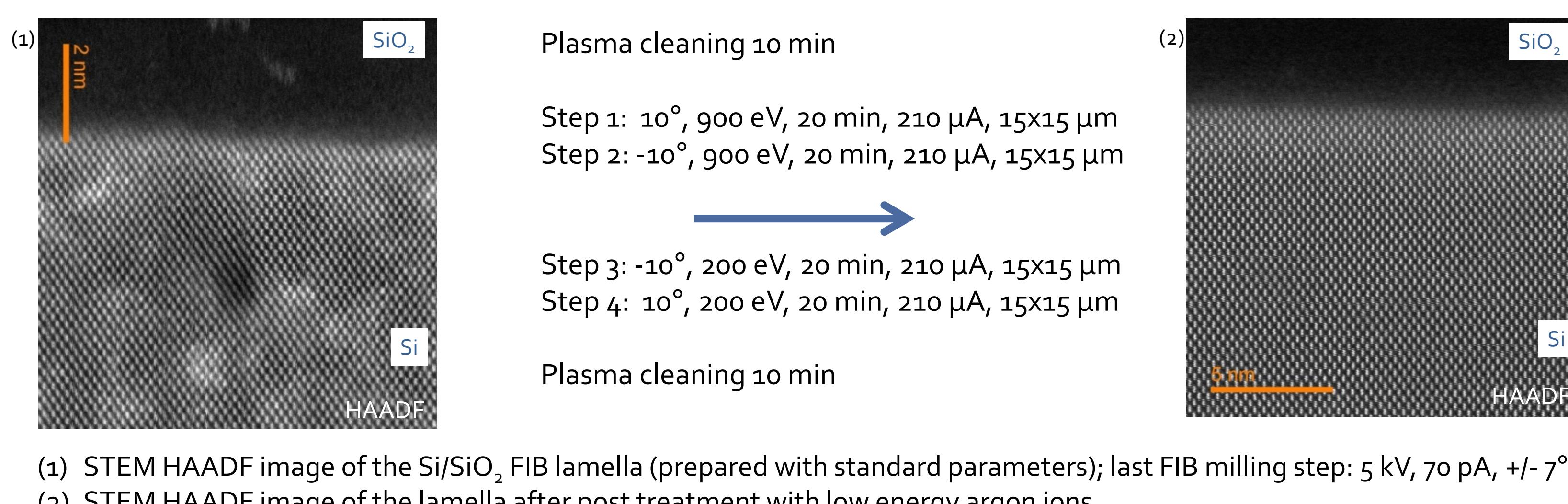


- Ion Type: Argon
- Ion Energy: 50 – 2000eV
- Ion Beam Point Resolution: 2µm at 900eV
- Stage: 360° rotation; tilt: -10° to +30°; working temperature: RT to -160°C
- ✓ Reduction of the amorphous layer to ~ 1,5 nm
- ✓ Entire removal of Ga implantation induced by FIB preparation
- ✓ Removal of preparation induced material re-deposition
- ✓ Additional milling to generate ultra thin samples for HR investigations
- ✓ Applicable for temperature sensitive materials by using liquid nitrogen cooling



Si

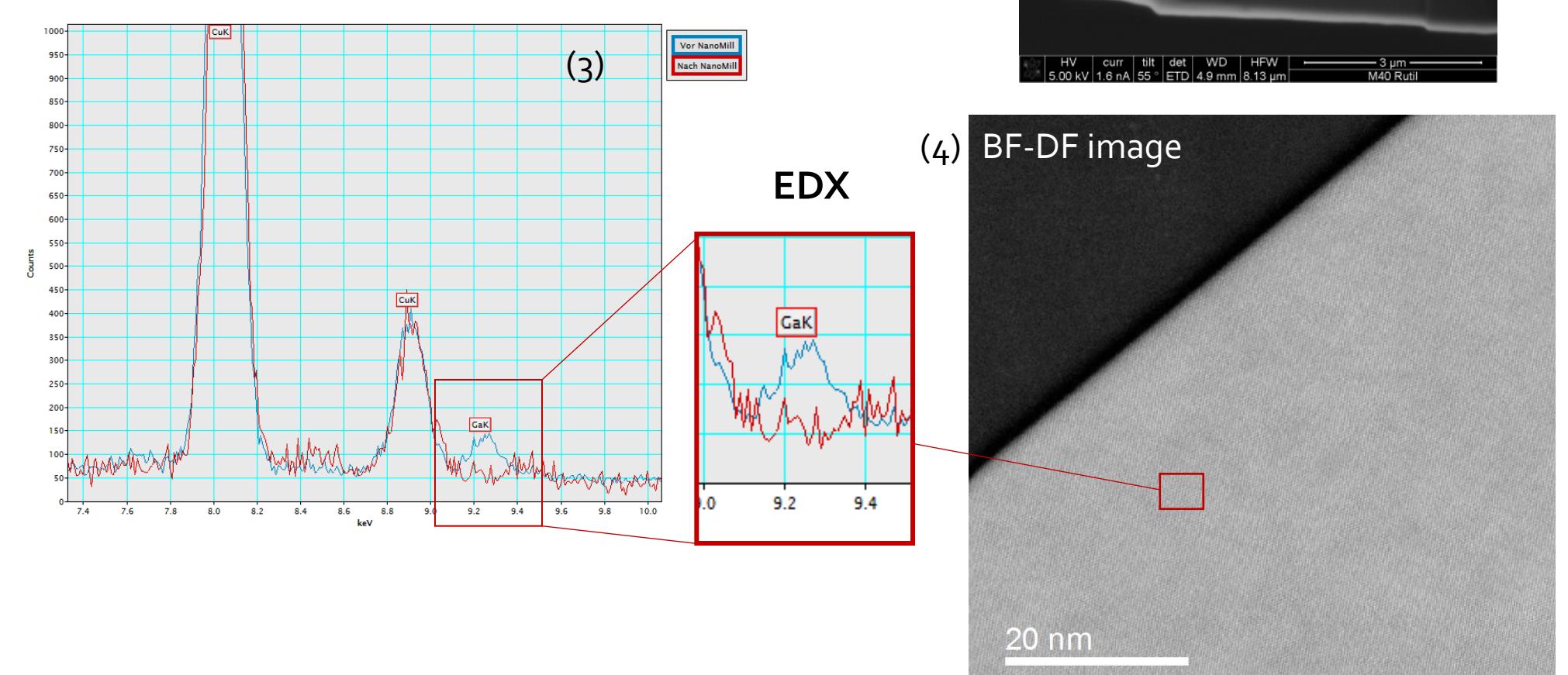
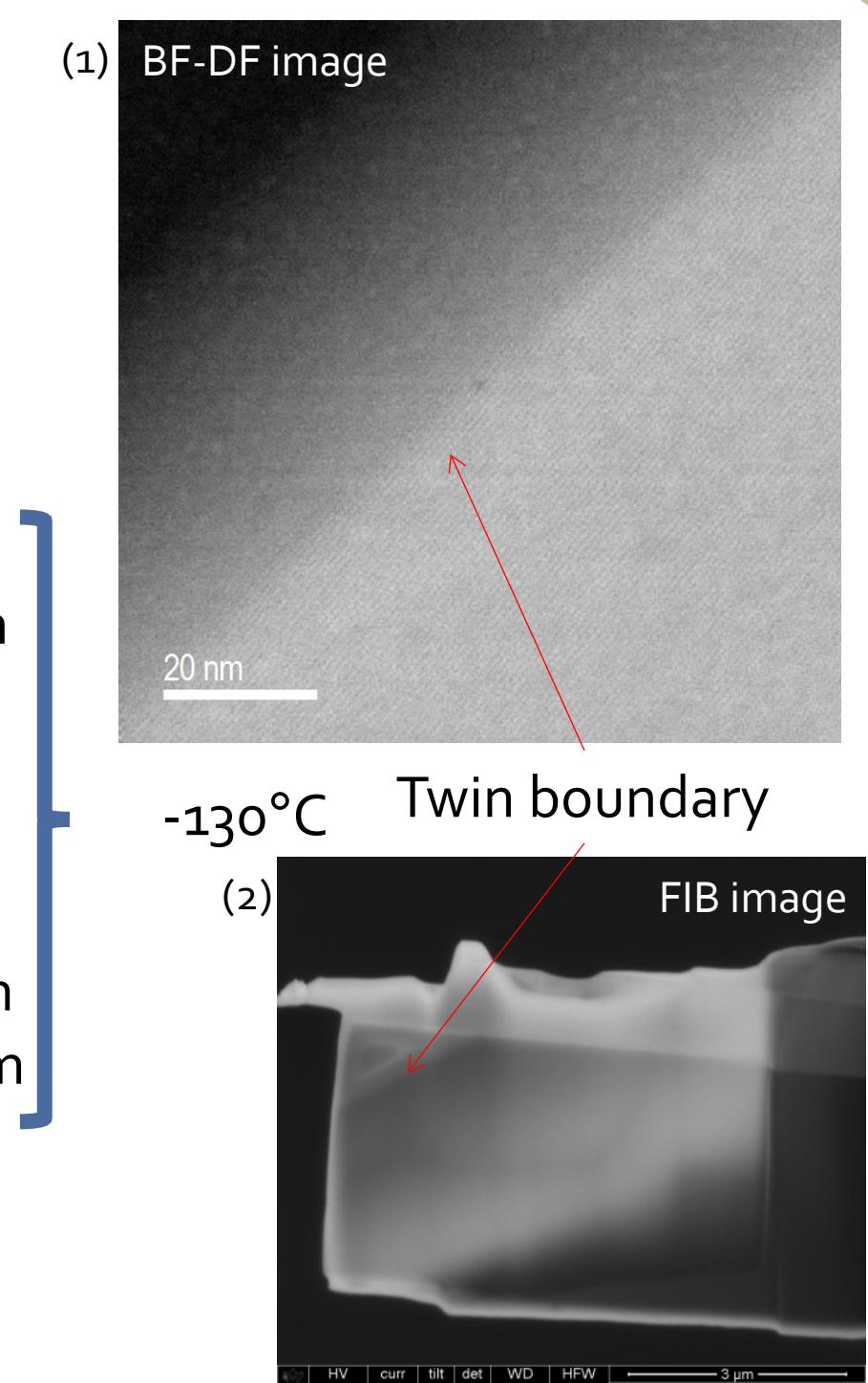
Amorphous surface layer



Rutile (TiO₂)

Ga implantation

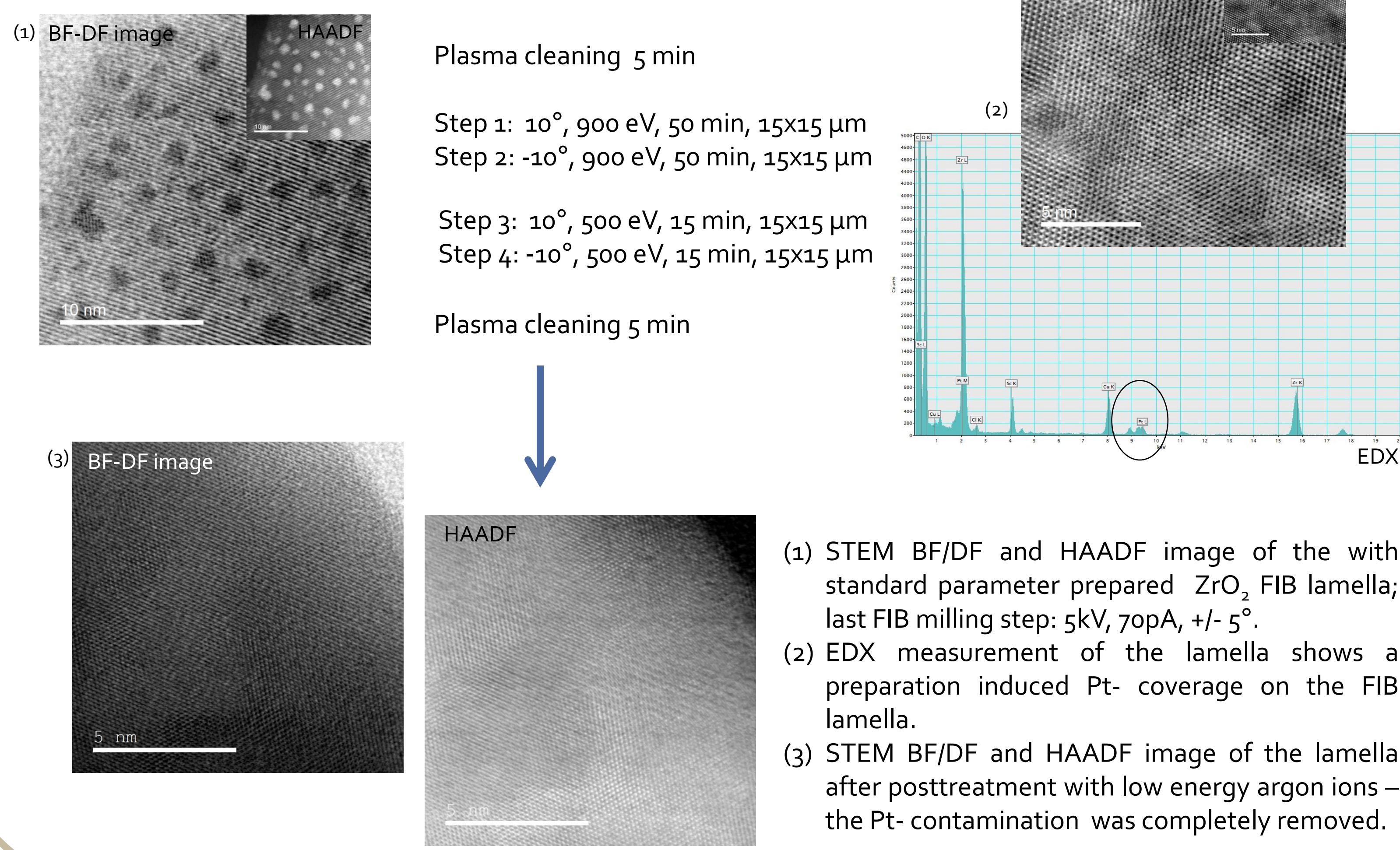
- Plasma cleaning 5 min
- Step1: 900 eV, 10°, 5 min, 15x15µm
- Step2: 900 eV, -10°, 5 min, 15x15µm
- Step3: 500eV, -10°, 5 min, 15x15µm
- Step4: 500 eV, 10°, 5 min, 15x15µm
- Step5: 200eV, 10°, 10 min, 15x15µm
- Step6: 200eV, -10°, 10 min, 15x15µm
- Plasma cleaning 5 min



- (1) STEM dark field image of a twin boundary;
- (2) FIB image of TiO₂ sample (prepared with standard parameters);
- (3) Comparison of the implantation of Ga by EDX measurements before and after improved preparation procedure;
- (4) STEM dark field image of the lamella after improved sample preparation procedure.

ZrO₂

Pt contamination



Conclusion

A post treatment of FIB lamellae with the NanoMill®, adapted to the respective sample system, has proved to be very useful for the reduction and removal of FIB-induced preparation artifacts. This allows to prepare a lot of different material systems and to produce the best possible samples for HRSTEM imaging and analysis.

Acknowledgements

„We kindly acknowledge financial support by the Austrian Research Promotion Agency (FFG) (project 850220/859238).“

and



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