On the gallium accumulation at the boundaries of Al layers in FIB prepared TEM specimens

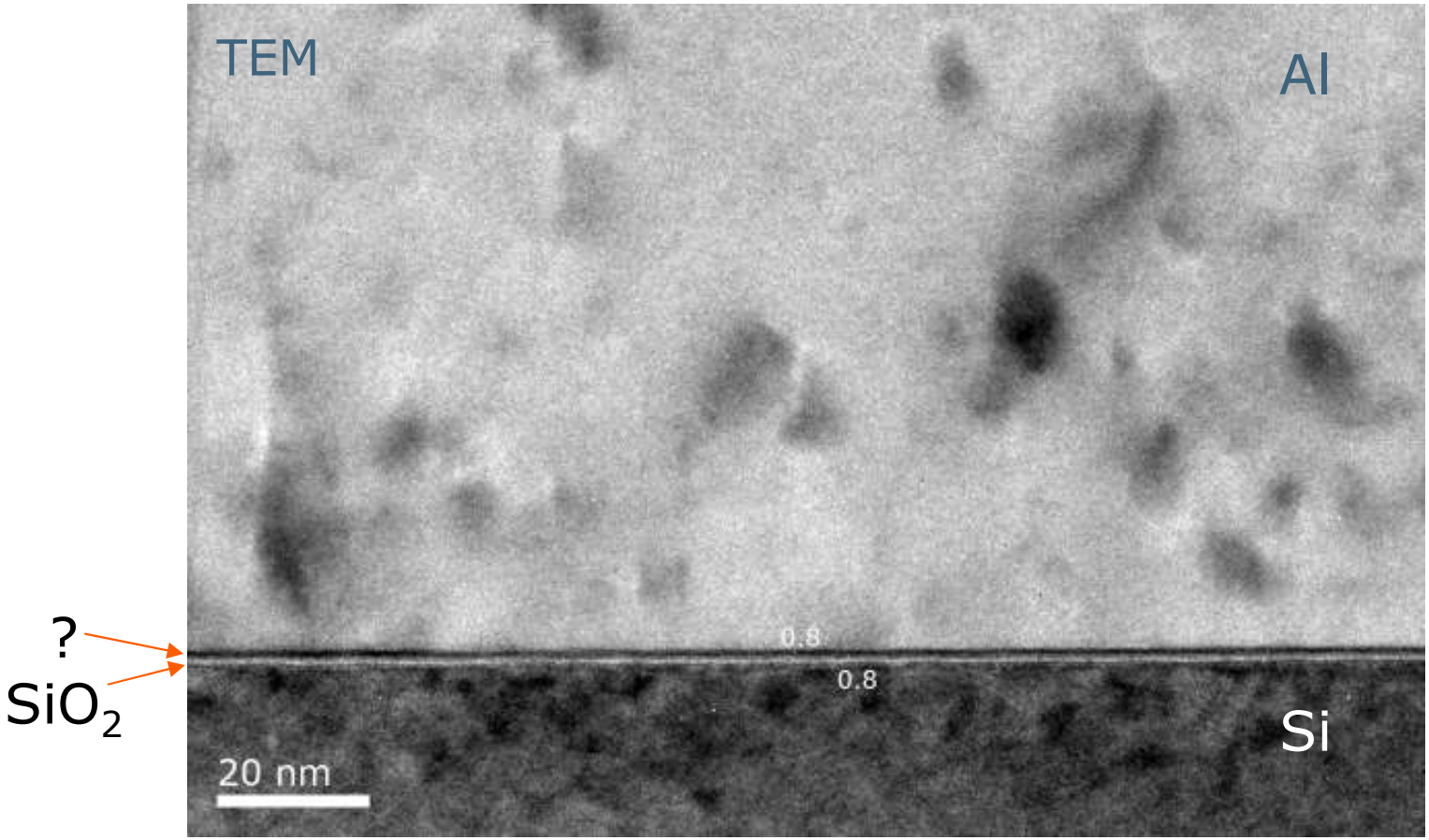
Hugo Bender
Paola Favia, Olivier Richard, Jef Geypen
Leuven, Belgium

imec

Introduction

- In many materials (Si, …) the Ga ion beam amorphises the outermost layer during FIB.
- In metals amorphisation does generally not occur. Increase of the dislocation density is reported.
- The amount of implanted Ga is estimated in literature to be in the range of 4 to 9 at % in the outer <10 nm of the thinned foil i.e. averaged over the specimen thickness only a minor Ga signal is expected in EDS analysis.
- Ga has low solubility in Al : <9at% at 26-29ºC. Grain boundary penetration of liquid Ga on Al bicrystals is extensively studied in literature and shown to be a very fast process that can lead to embrittlement of the Al.

Observation



FIB prepared specimen

Experiments

TEM specimens :

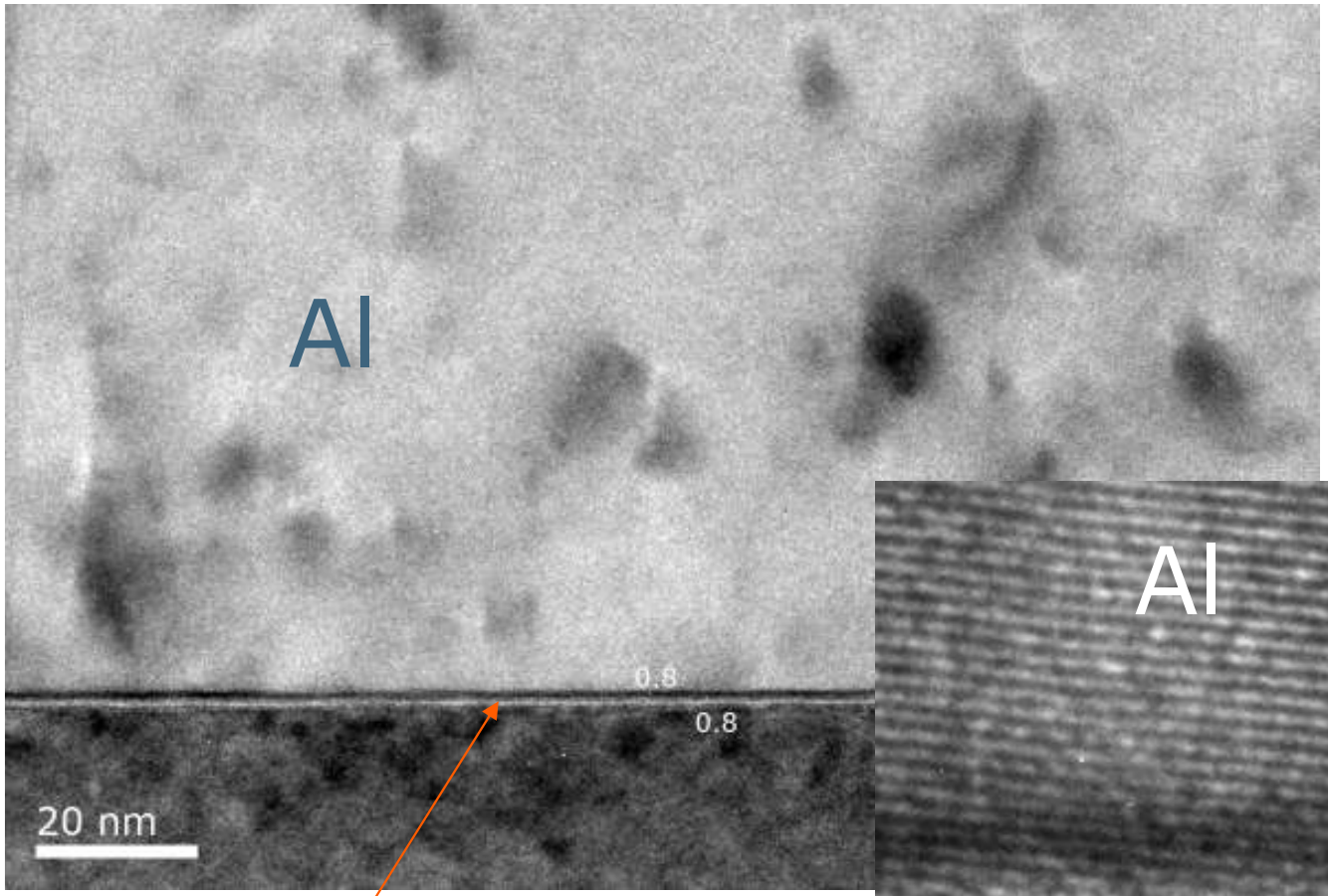
- in-situ lift-out in a dual-beam FIB/SEM, 30 keV Ga ion beam

- The layer structures investigated are :
  - DC sputtered Al on 2-4 nm SiO_2 on Si (small samples)
  - DC/magnetron sputtered Al / native oxide / Si (200 mm wafer)
  - idem covered with 150 nm low-T CVD SiO_2 layer
  - idem with TiN/Ti barrier, after anneal : TiN/$TiAl_3$/Al

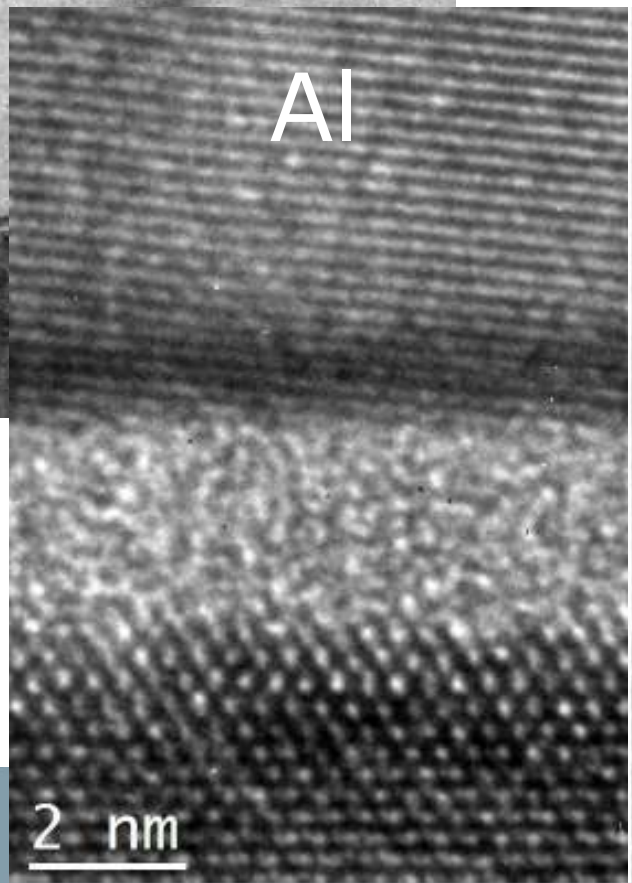
Special specimens to study the Al sidewall :

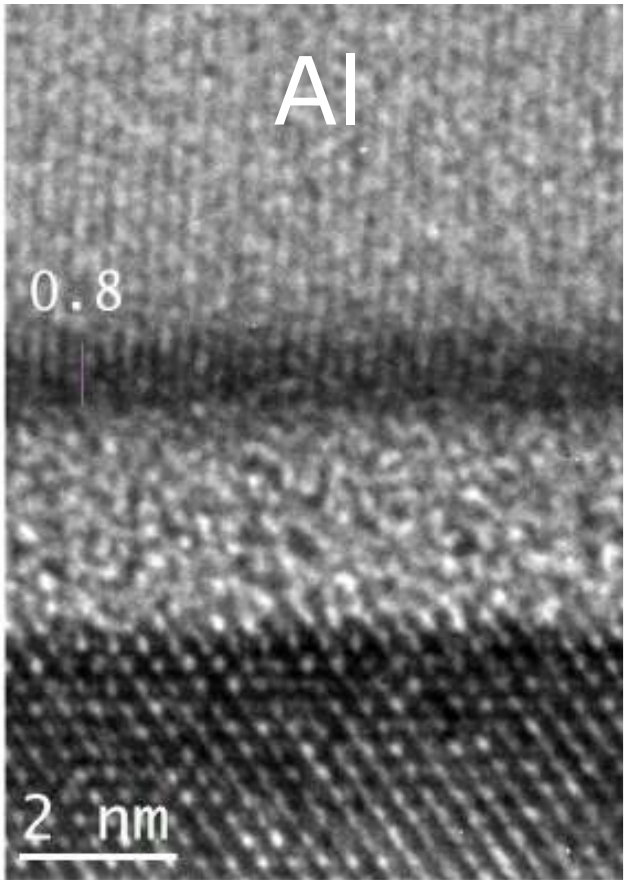
- trench milled orthogonal to the final specimen with conditions similar as for the normal specimen preparation
- filled immediately in-situ with ion-beam Pt

Al / thin oxide / Si : TEM - HREM



Al lattice is continuous in the dark layer





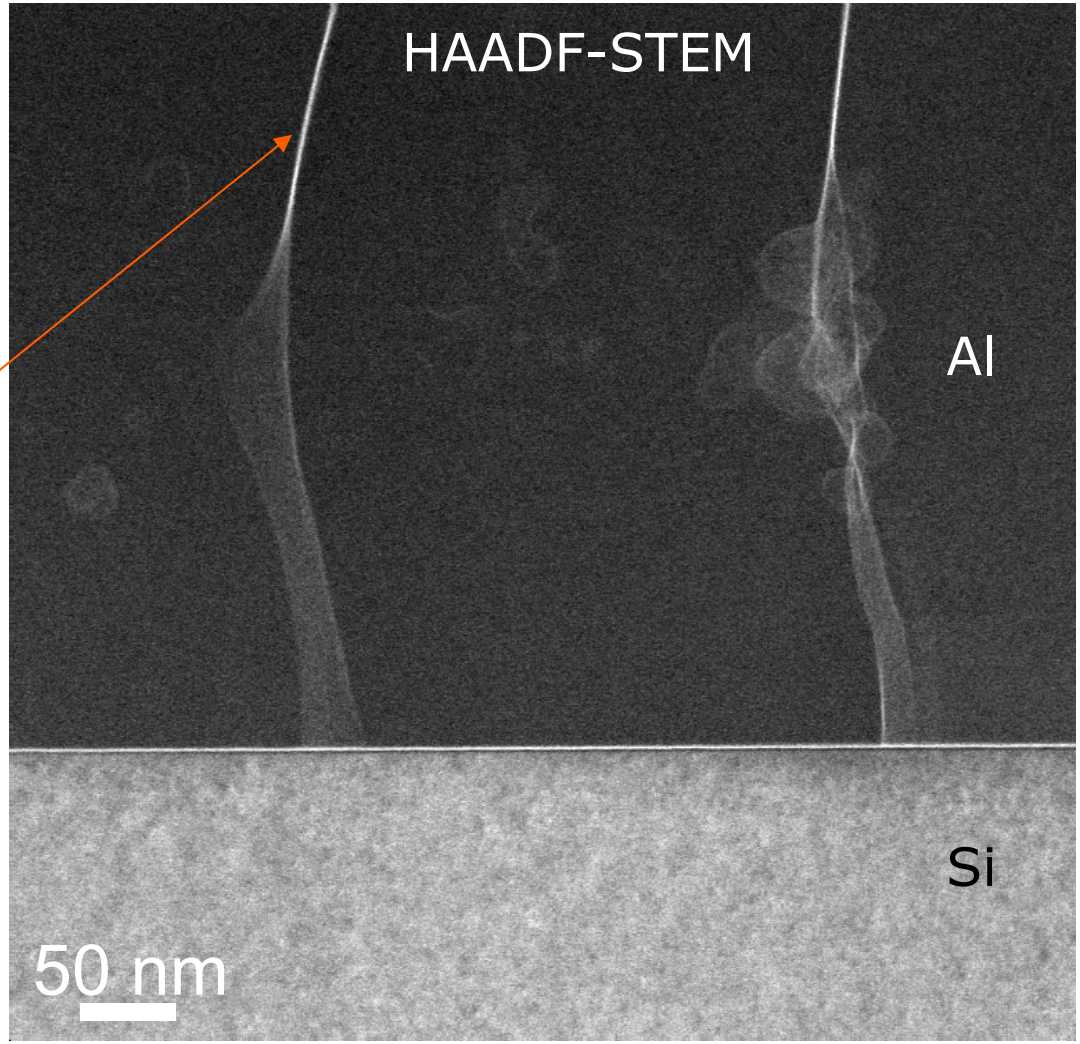
dark contrast layer in the Al near $Al/SiO_2/Si$ interface

Al / thin oxide / Si : HAADF-STEM

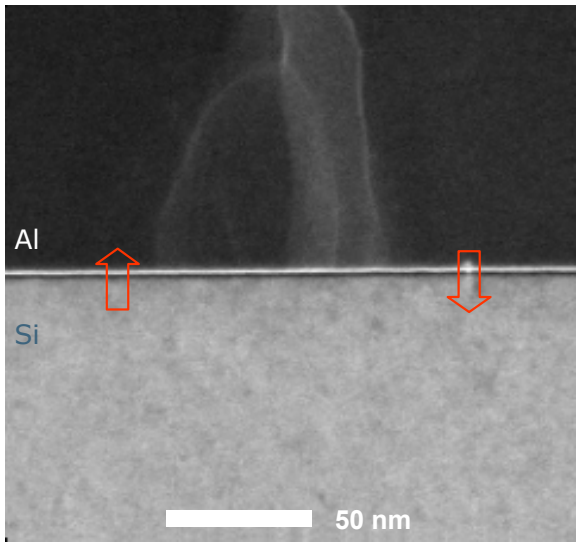
bright contrast layer

at Al-Al grain boundaries

in the Al near Al/SiO_2/Si interface

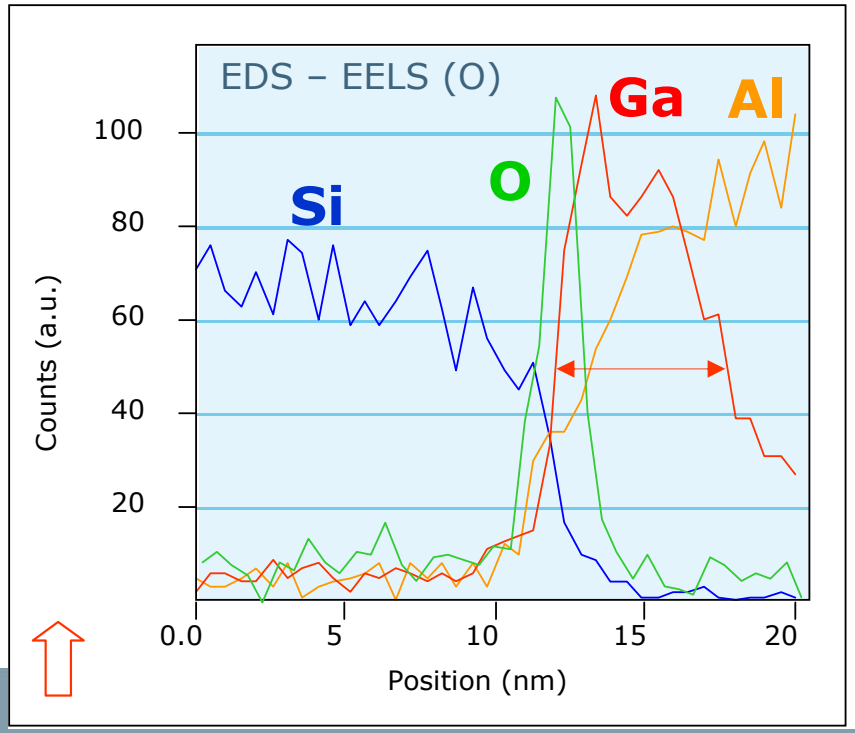


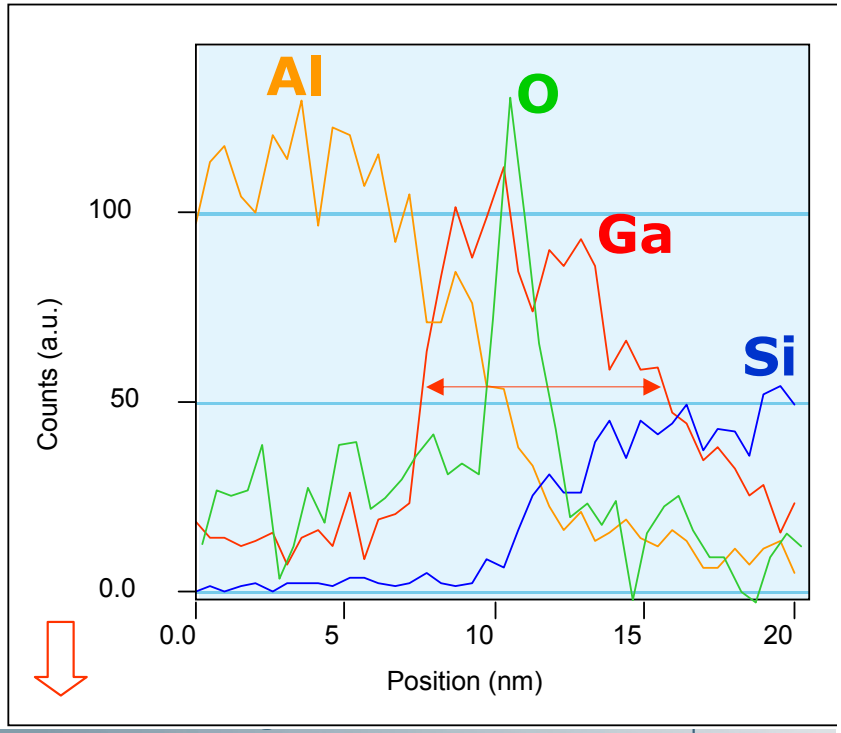
Al / thin oxide / Si : HAADF-STEM – EDS/EELS



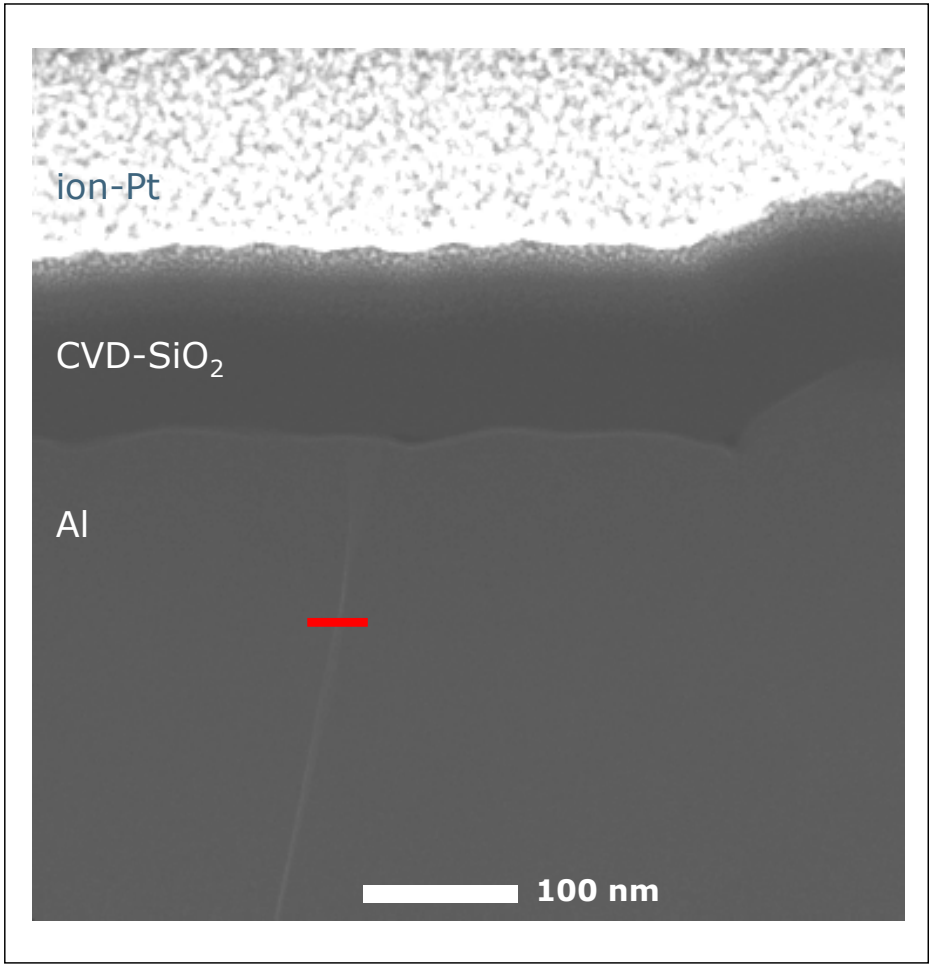
EDS/EELS :

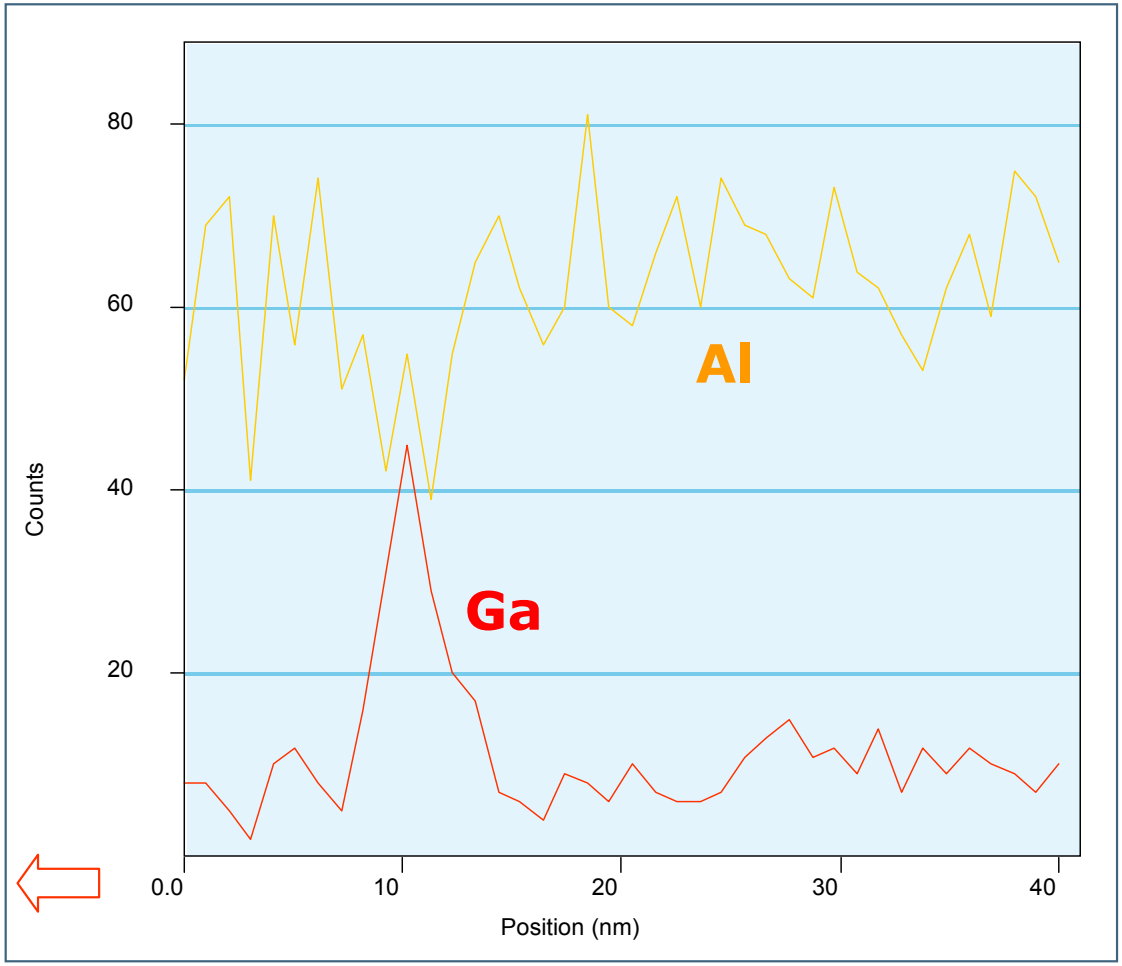
- accumulation of Ga in Al near the interface
- width of the Ga profile is much larger than on the images
- width depends on the sense of the linescan





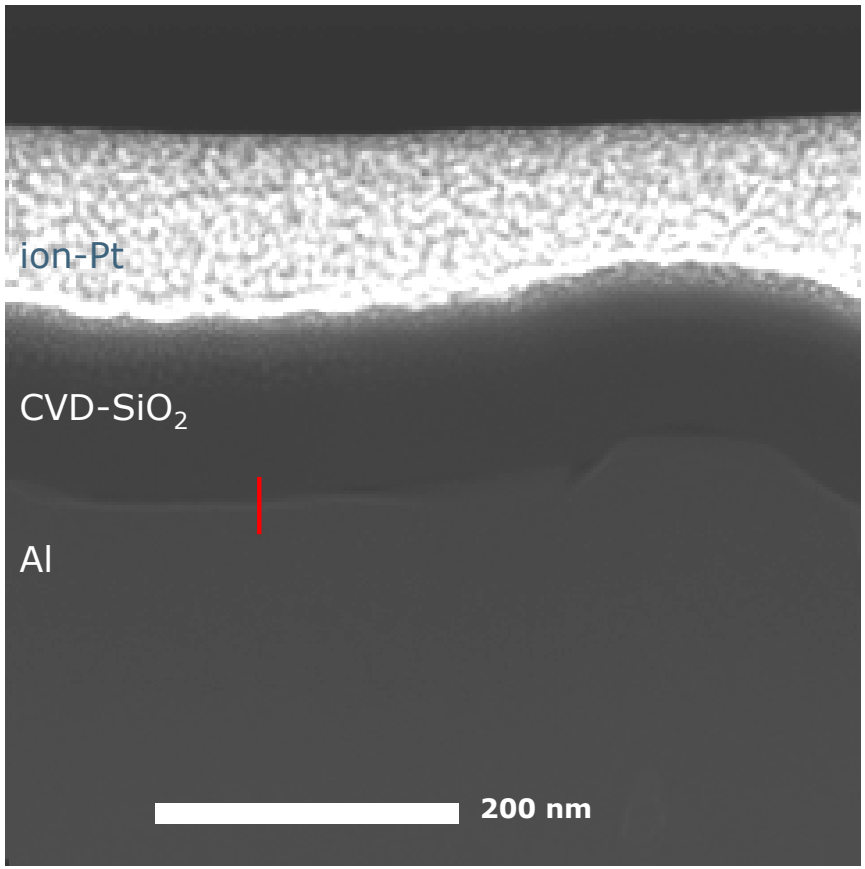
Al-Al grain boundary : HAADF-STEM – EDS

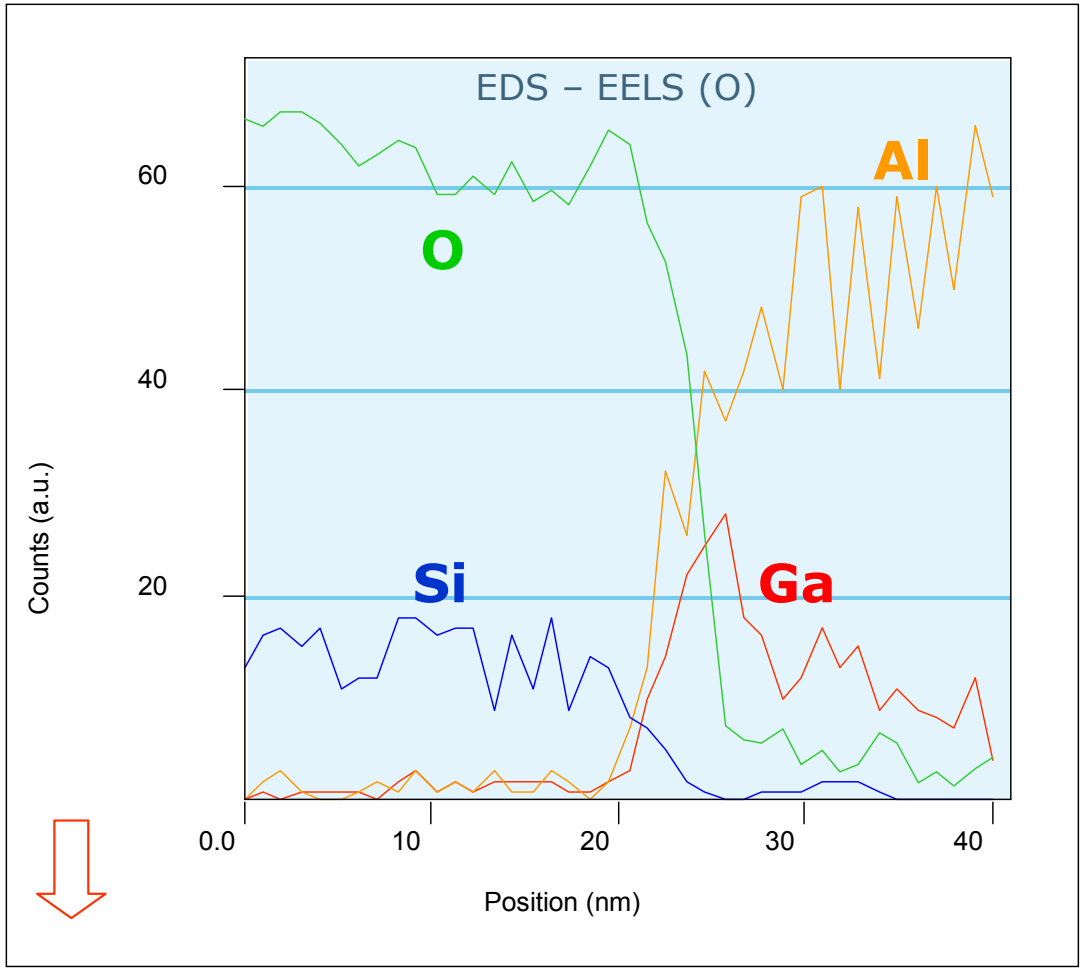




EDS : accumulation of Ga at the Al grain boundaries

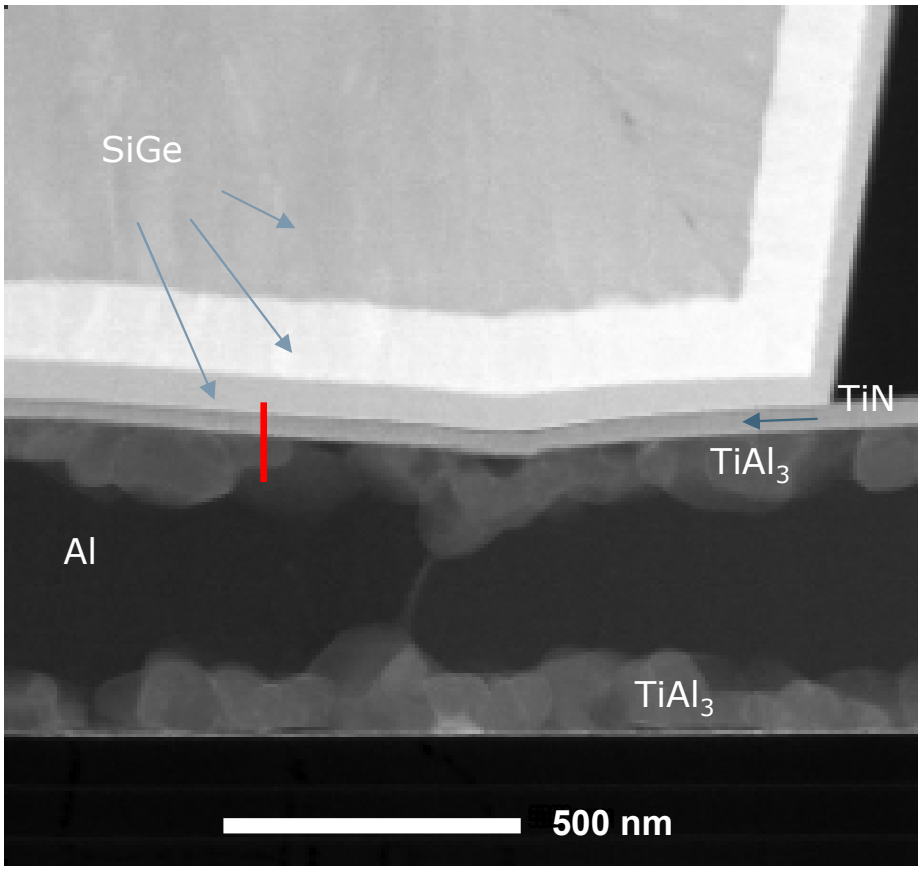
SiO_2 / Al : HAADF-STEM – EDS/EELS

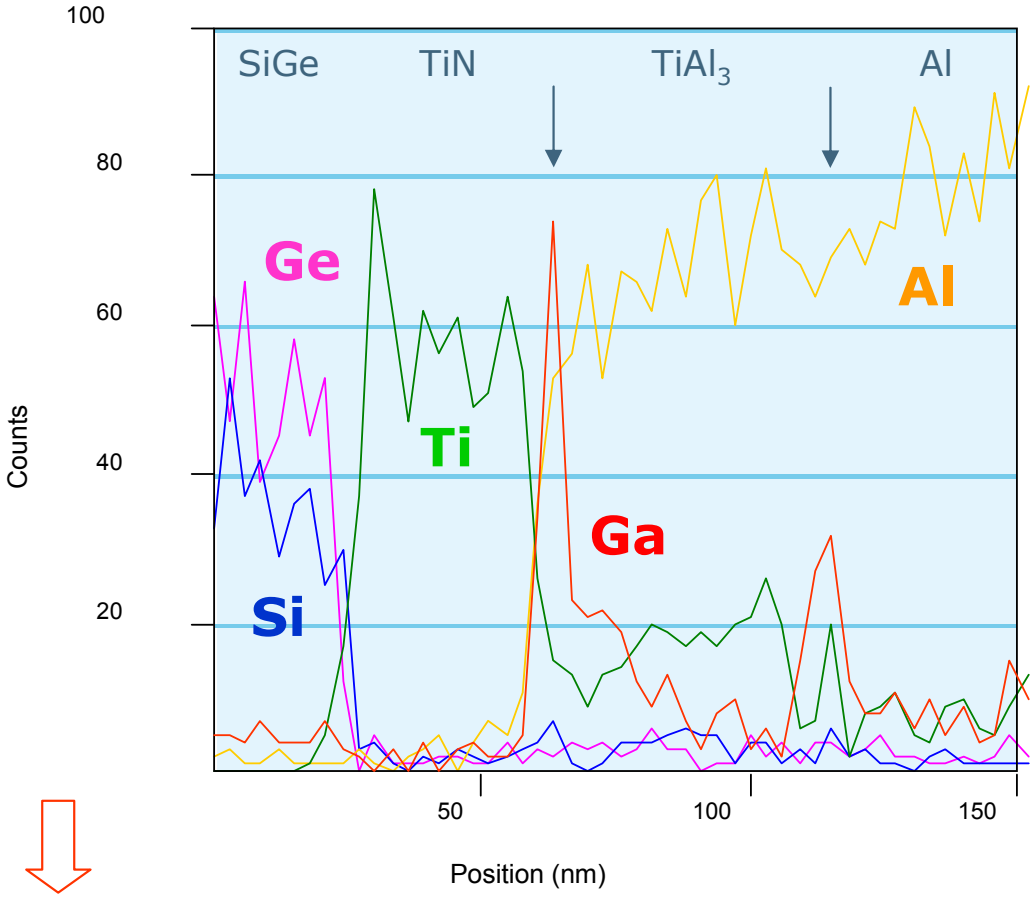




Ga enrichment at the SiO_2/Al interface

TiN / $TiAl_3$ /Al : HAADF-STEM – EDS





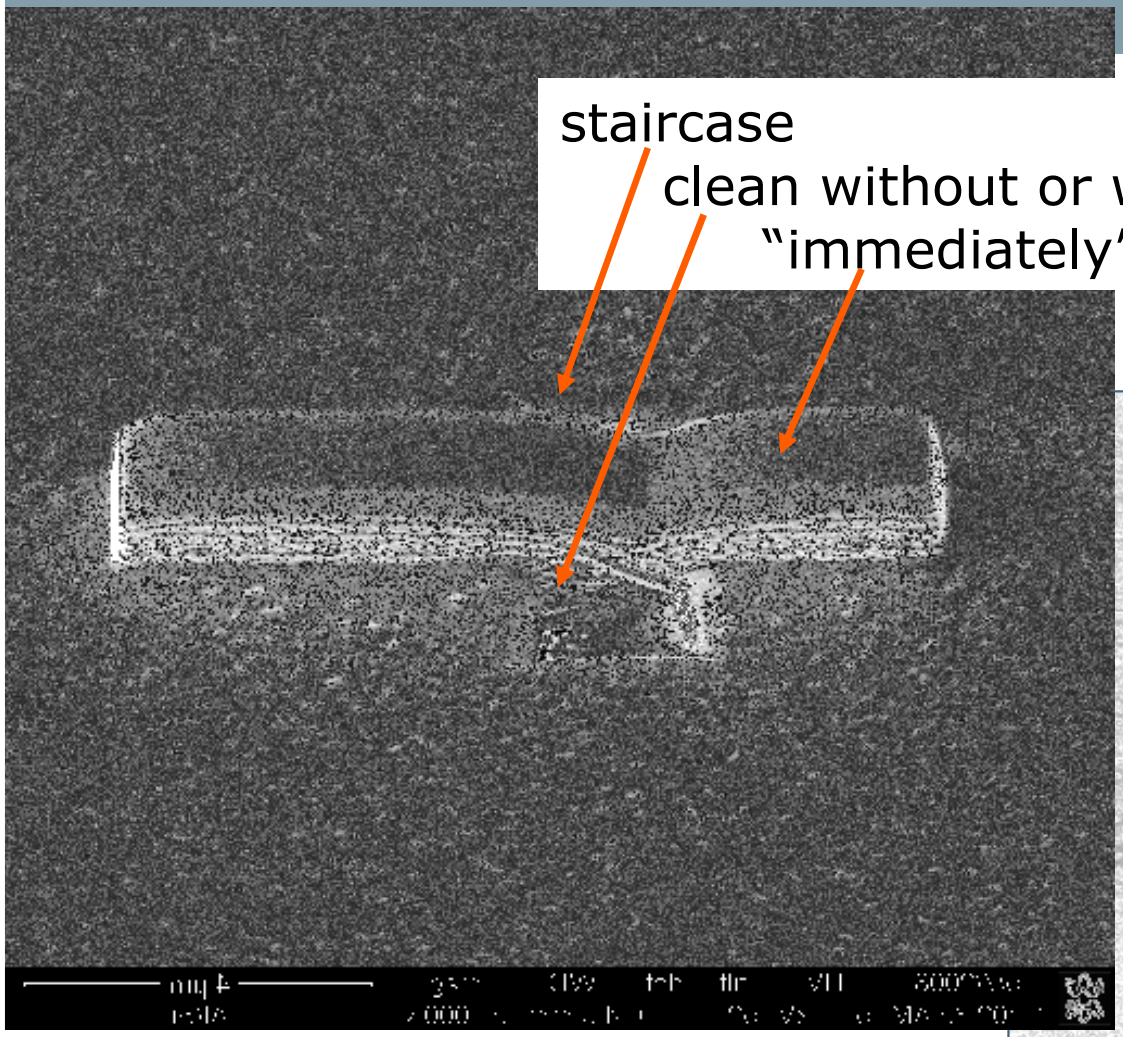
accumulation of Ga at TiN/$TiAl_3$ and $TiAl_3$/Al interfaces

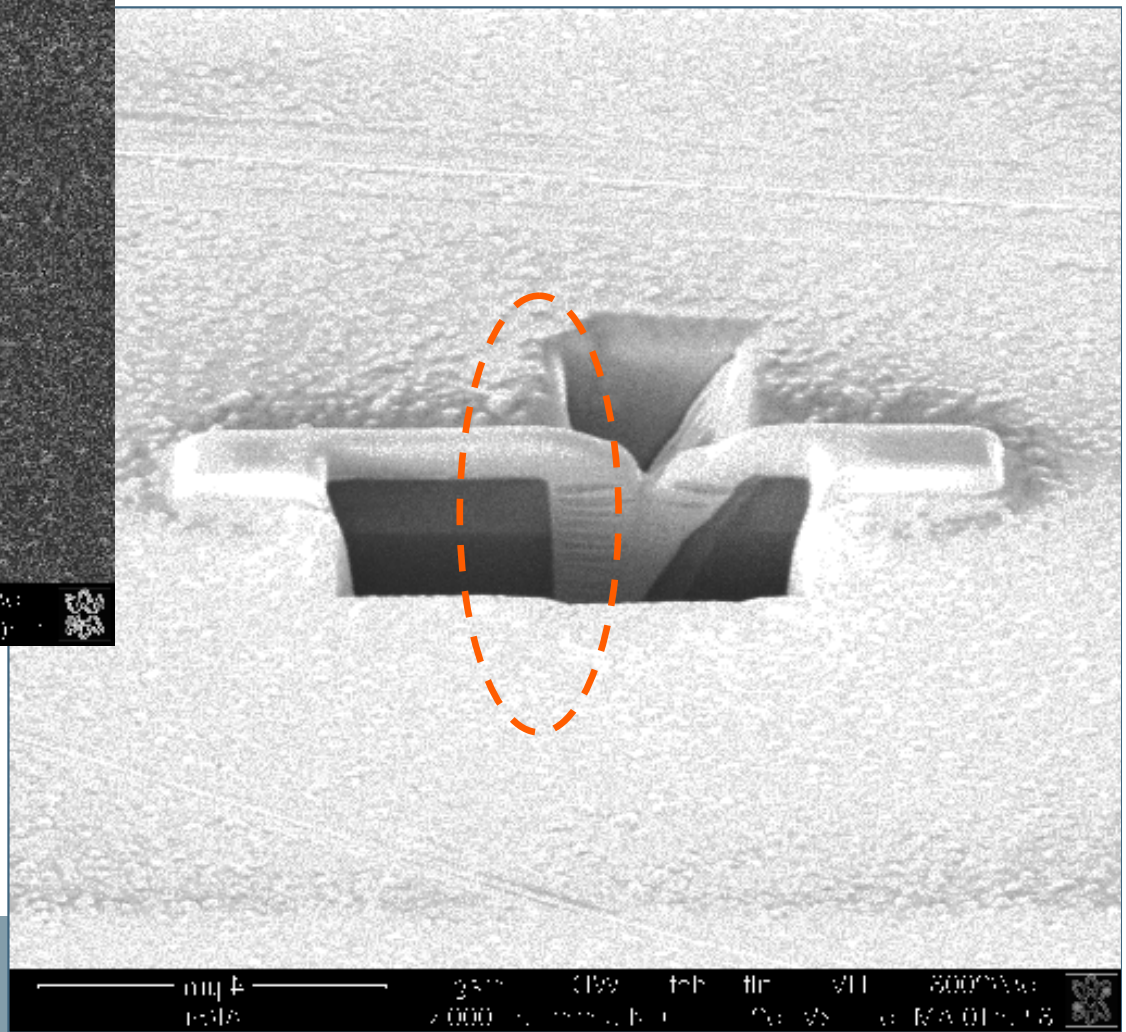
Al / ion-beam deposited Pt : preparation

staircase

clean without or with 2º extra tilt

"immediately" Pt deposition

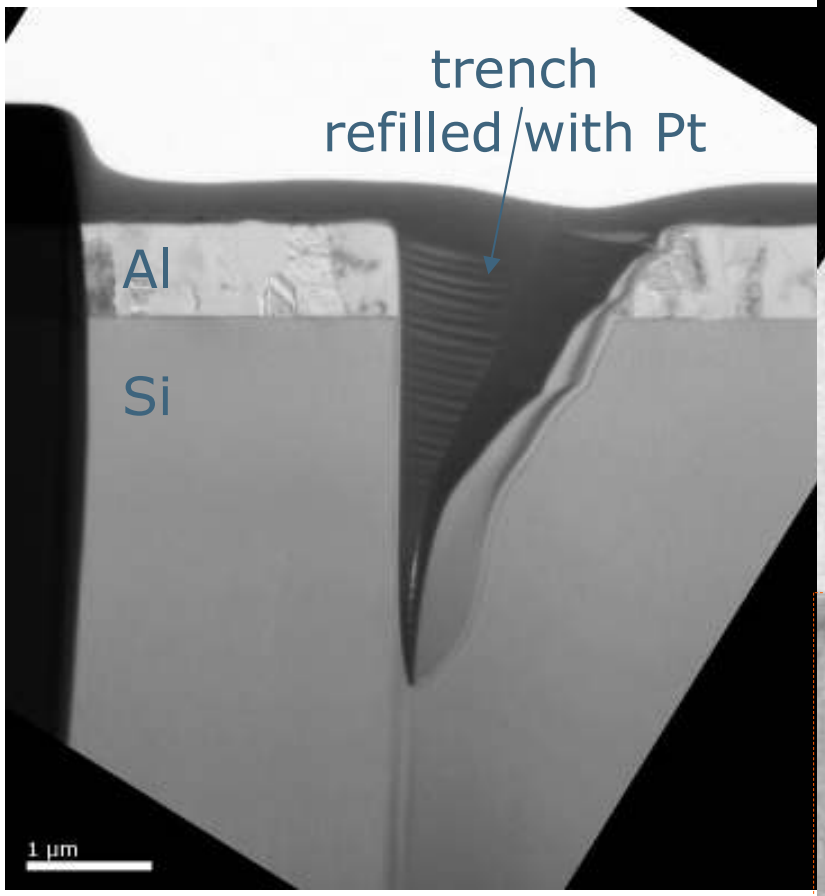


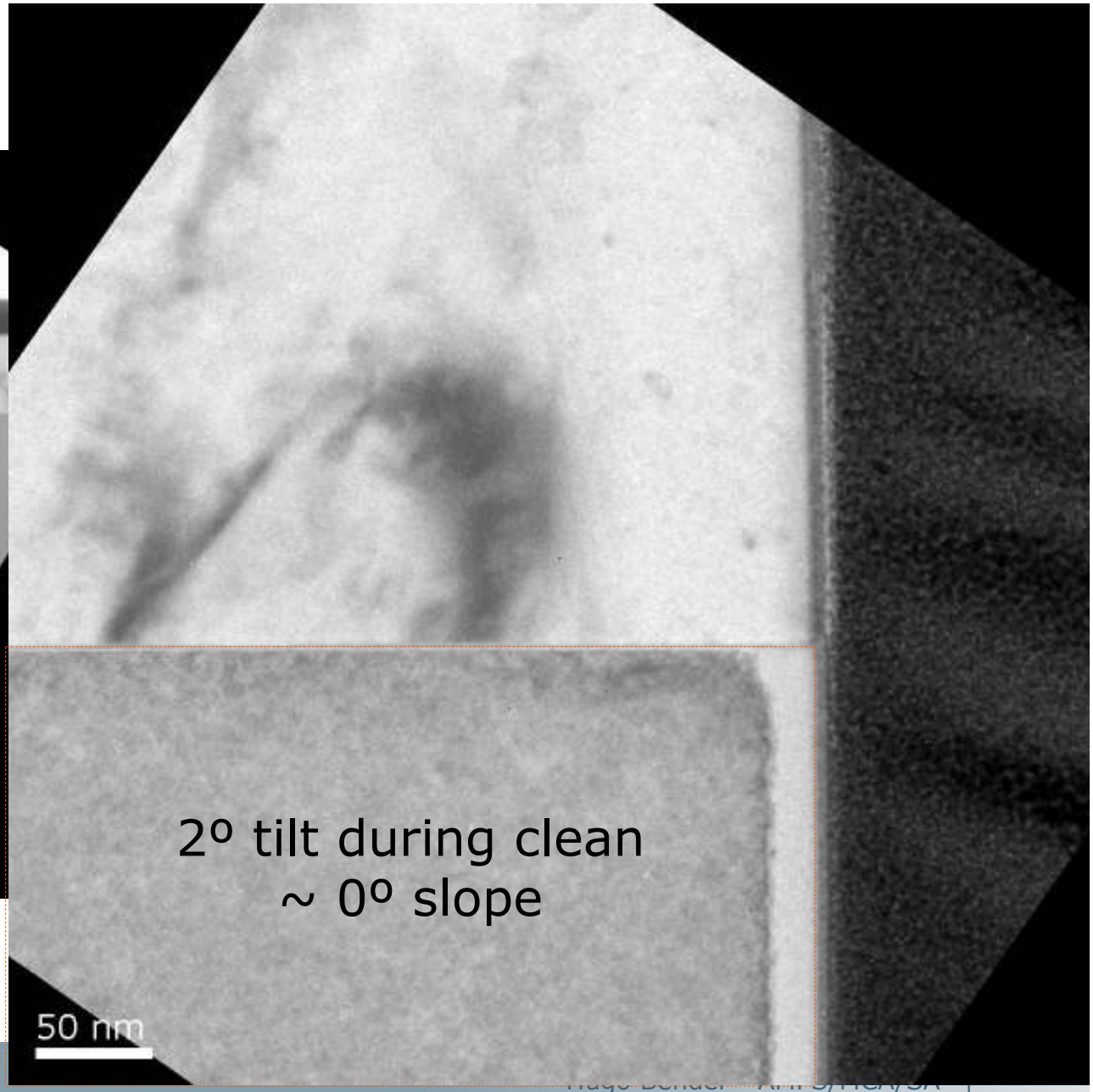


standard in-situ lift-out preparation

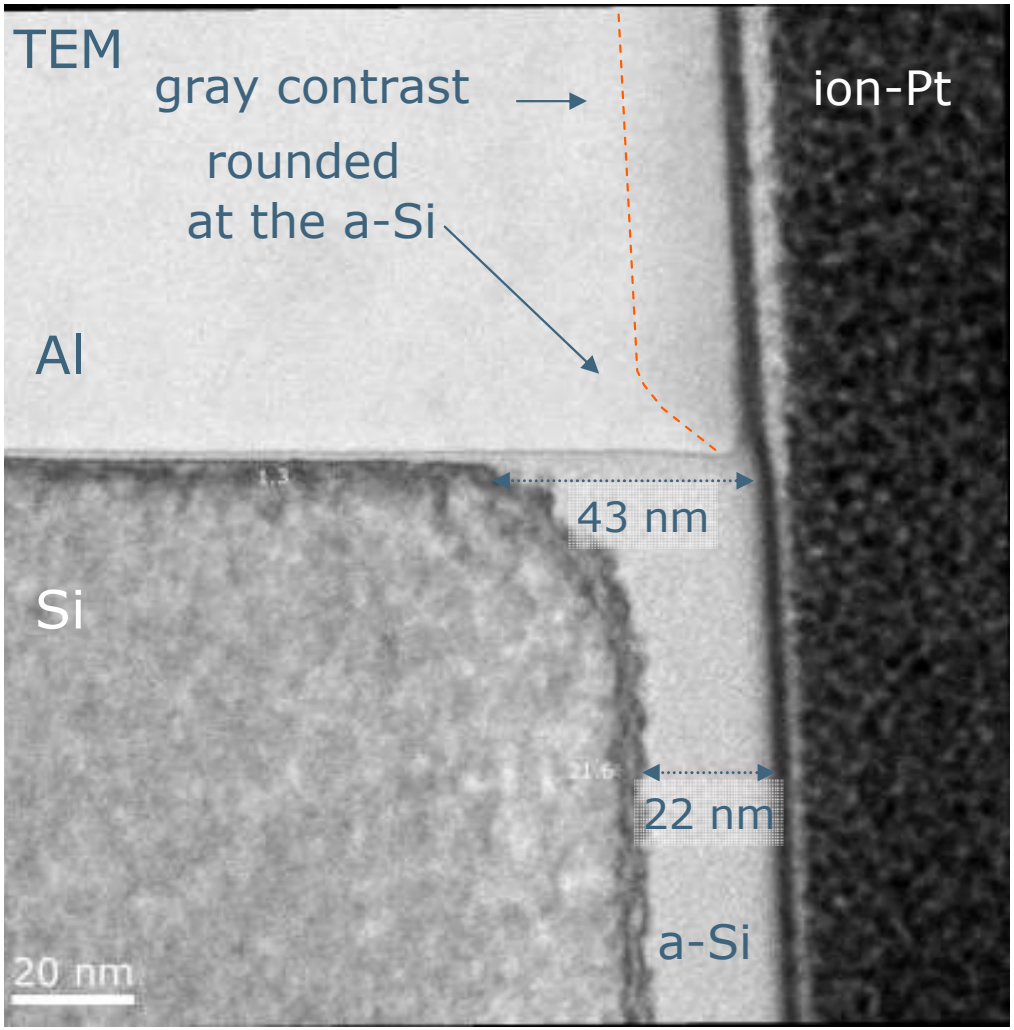
Al / ion-beam deposited Pt – trench sidewall

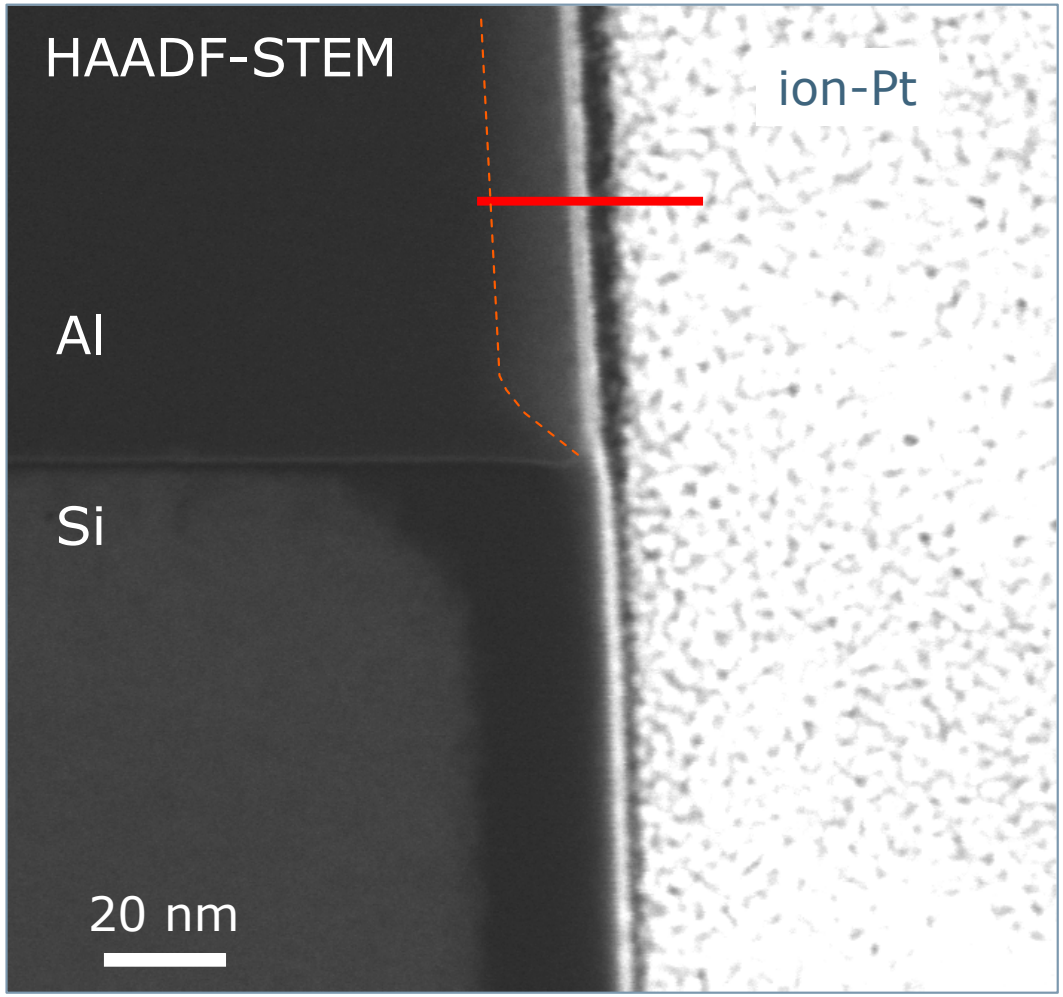
in-situ lift-out preparation



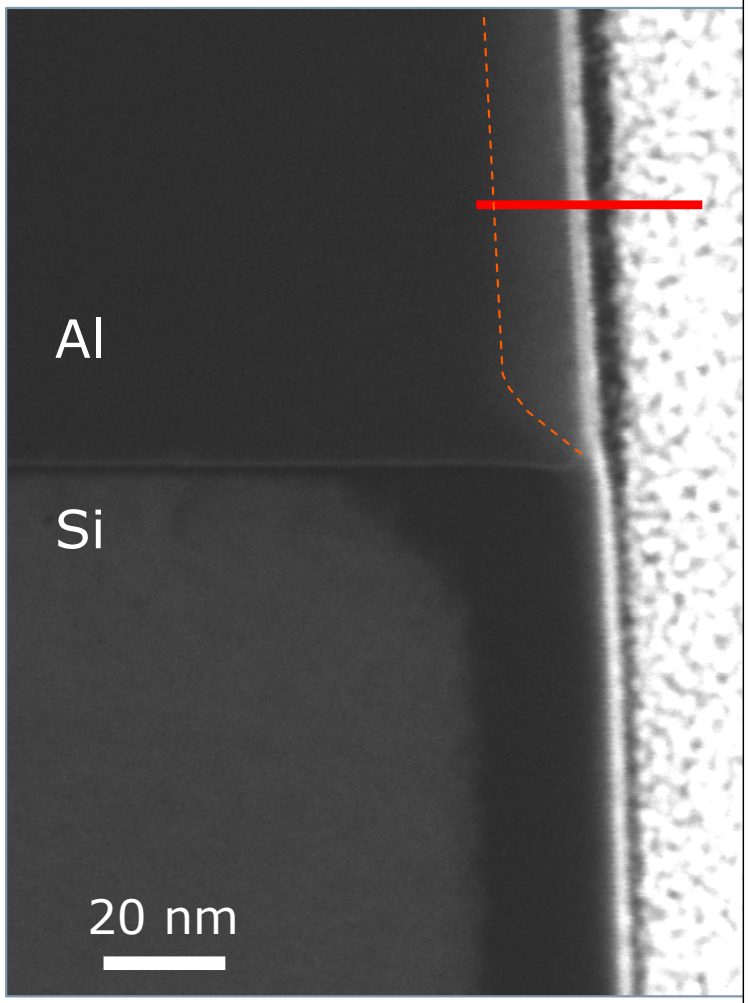


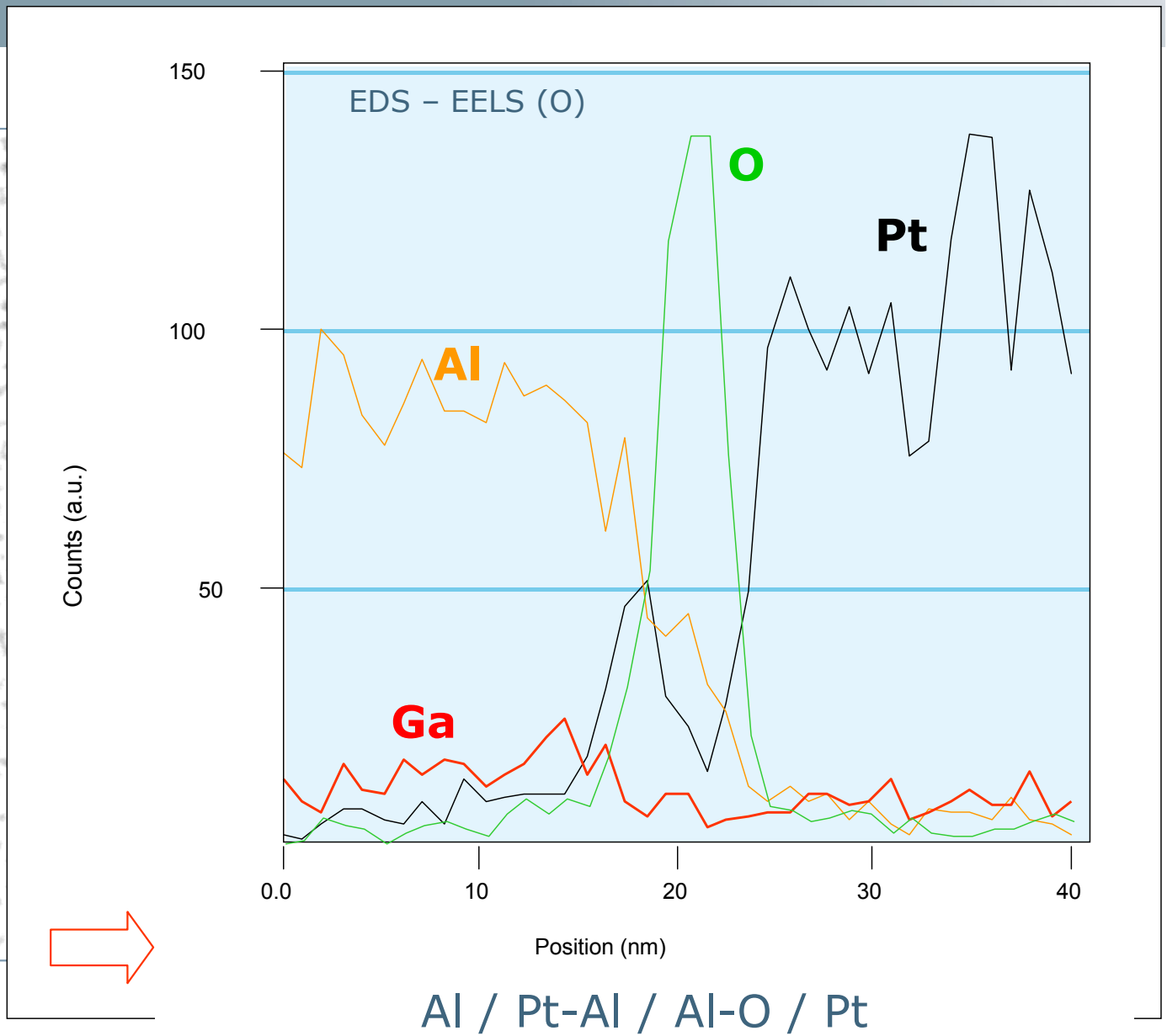
Al / ion-Pt – trench sidewall : TEM / HAADF-STEM





Al / ion-Pt – trench sidewall : EDS/EELS





Al / Pt-Al / Al-O / Pt
Ga profile in the bright region
(Al-O from plasma clean TEM specimen ?)

Summary

- Accumulation of Ga in the Al near all kinds of interfaces :
  Al/SiO_2 (interfacial oxide or CVD capping layer)
  $Al/TiAl_3$
  $TiAl_3/TiN$
  Al/Al grain boundaries

- Ga-rich layer in TEM (STEM) images : ~ 1 nm
  Al lattice planes are continuous

- Larger width of the Ga rich layer on EDS/EELS STEM line-profiles :
  Ga-diffusion under the electron beam

- In Al bulk and other materials the Ga signal is generally below detection limit

- On the Al sidewall : Ga incorporation in Al in ~ 15 nm zone
  Near the Si interface : Ga diffused to the interface
  On front/backside of the specimen : similar effects will form the Ga-enriched layer

- To be done : lower energy Ga milling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