Grain Boundary segregation behavior of Bismuth in Copper

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This report is a summary of last 5 months research in this topic. This is an interim report/update as it is a work in progress.

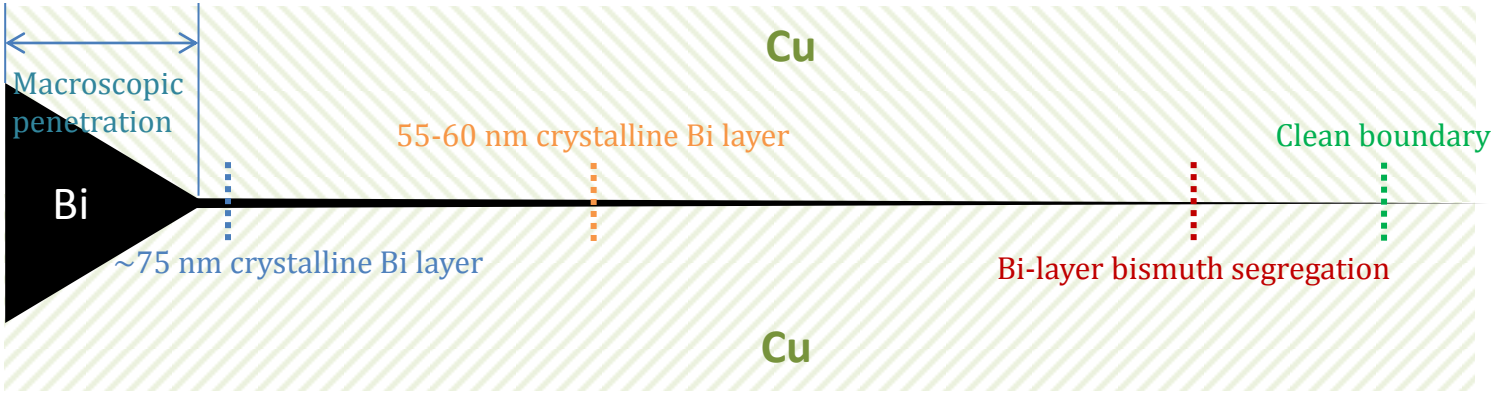
In this segment of the research the segregation behavior of bismuth in copper has been studied. The ongoing effort is focused on two samples fabricated at ***Clemson University (Jian Luo's group)***. The diffusion behavior in these samples will be measured at ***University of Illinois at Urbana-Champaign (Shen Dillon's group)***.

Additionally, copper-bismuth thin films prepared at ***University of Illinois at Urbana-Champaign (Shen Dillon's group)*** is currently at ***Lehigh University*** for electron microscopy characterization. The diffusion behavior has been studied at UIUC and the results can be found in UIUC reports.

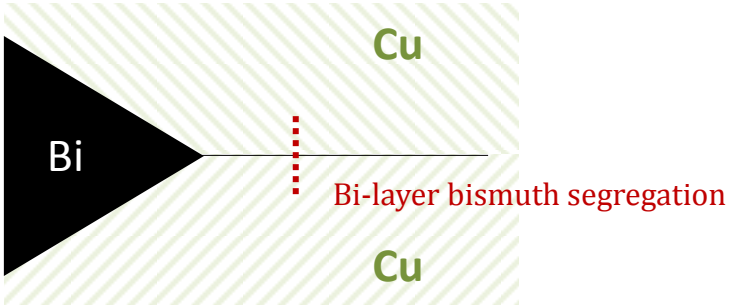
In the following slides, the significant features of two different grain boundaries from Clemson samples are presented. One of the grain boundary is from a sample that was annealed at 953°C, while the other is from a sample that was annealed at 700°C.

Summary

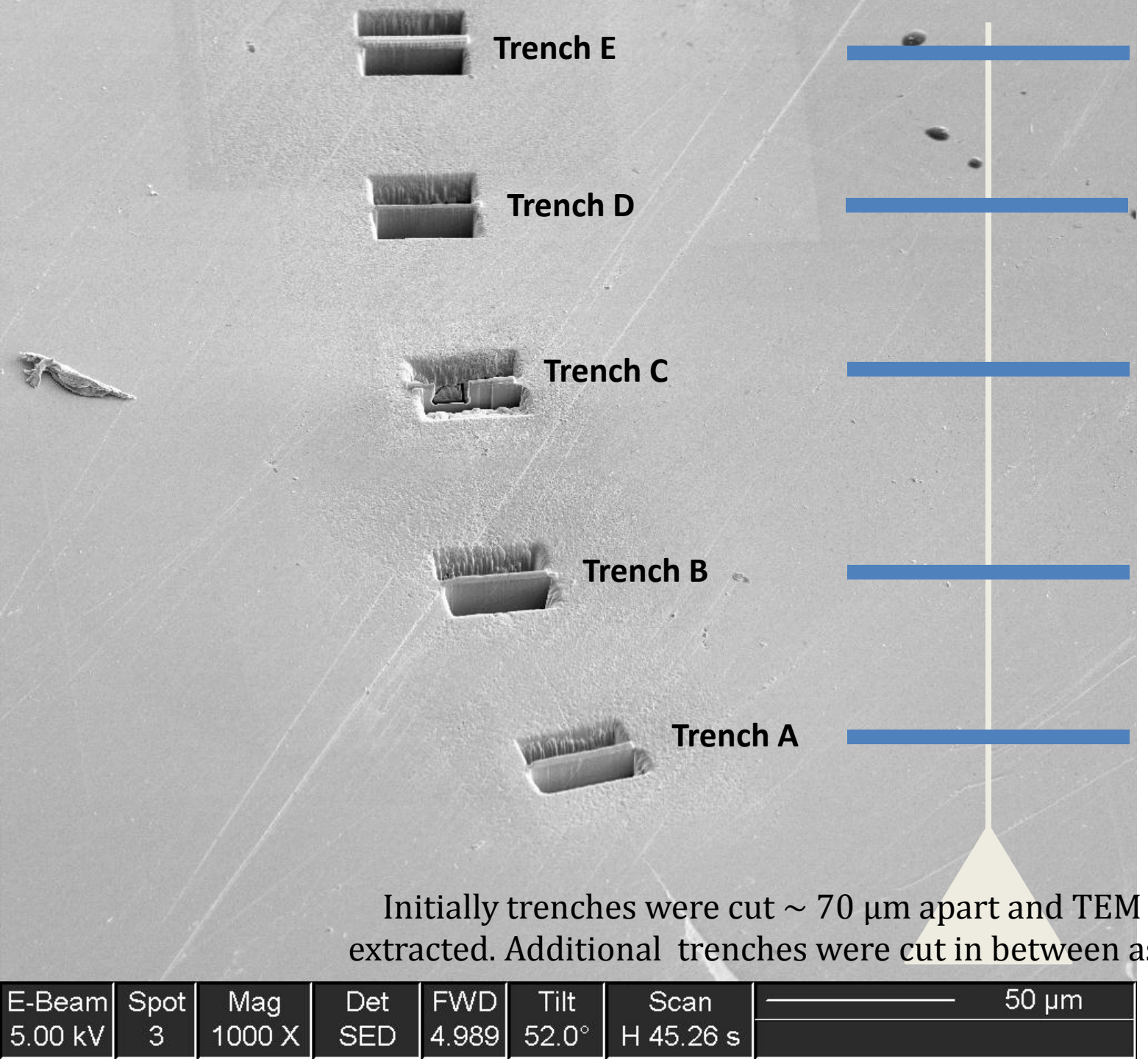
- two types of boundaries
  - Penetration depth extends for hundreds of microns beyond the apparent triple junction tip; gradually tapers off to clean boundary



  - Limited penetration of bismuth in the copper boundaries, bilayer segregation is observed at a distance 40 μm from the apparent triple junction tip.



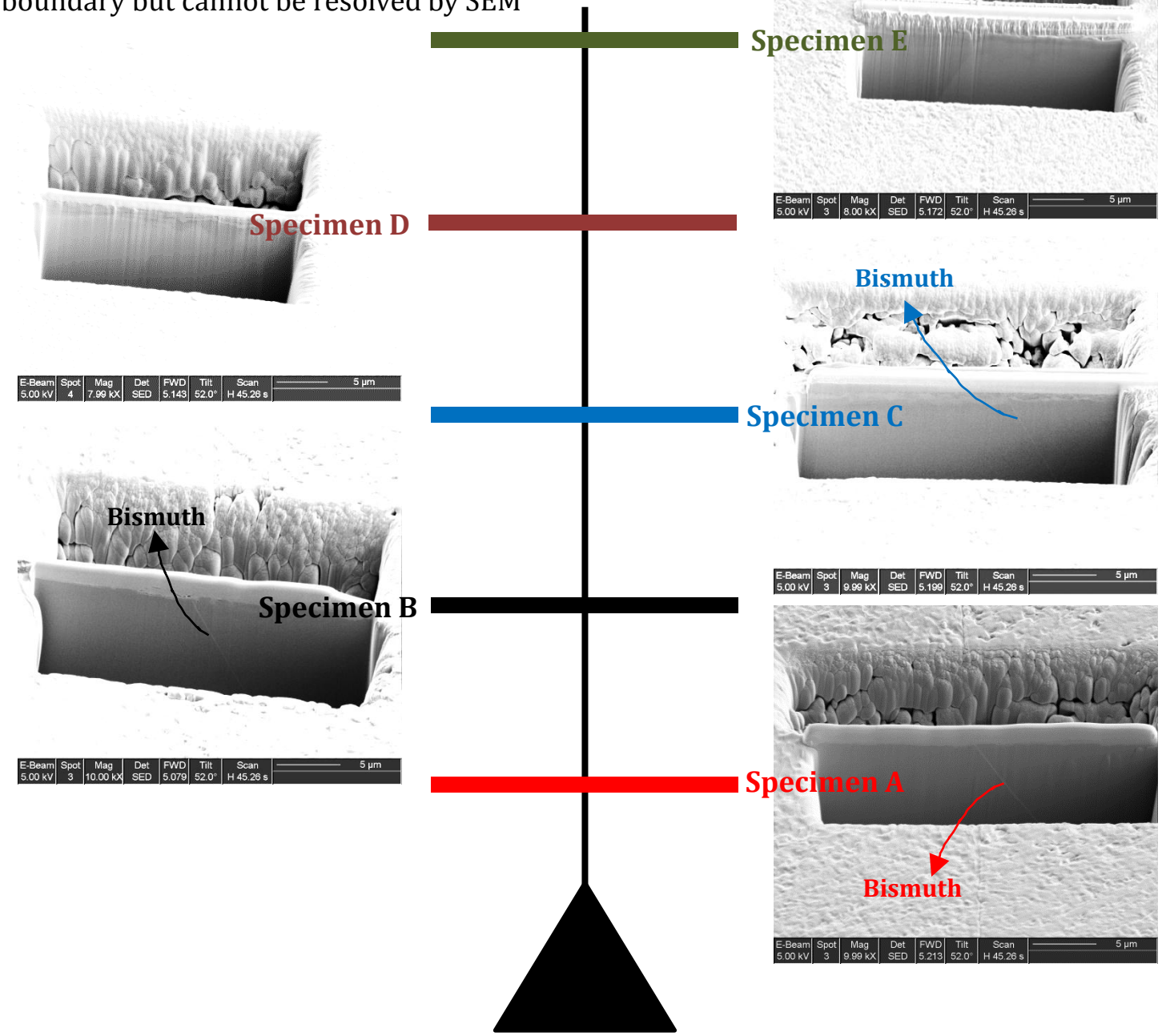
This segregation behavior is distinctively different from that in nickel-bismuth materials. In nickel-bismuth materials the macroscopic penetration extends over a larger distance. The bilayer segregation continued for hundreds of microns. In comparison, the bilayer segregation of bismuth in copper was limited to tens of microns.



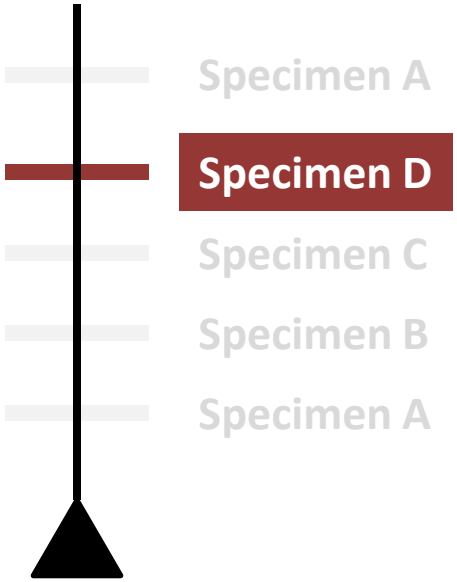
Sample A
953°C, 5h

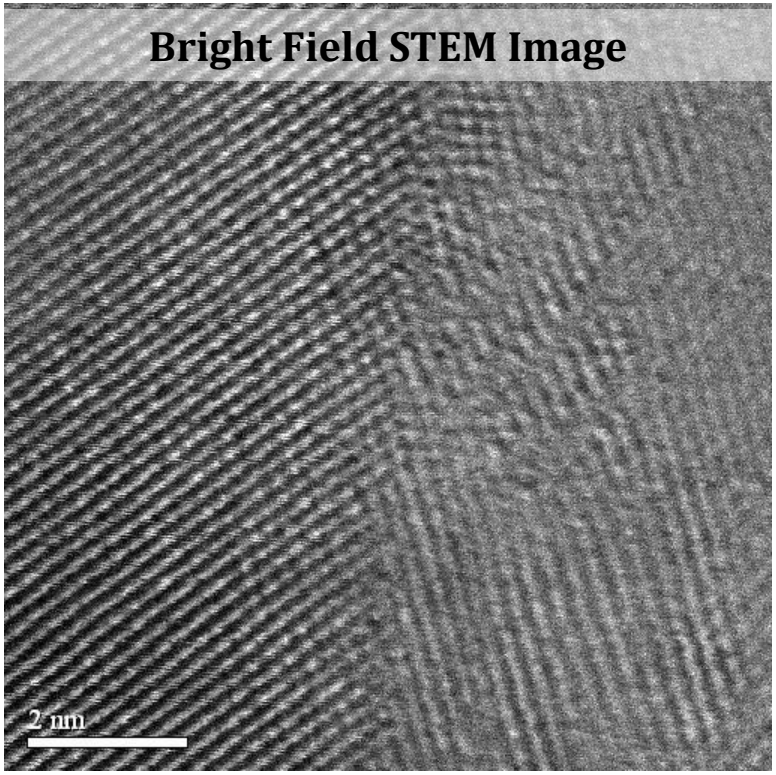
Initially trenches were cut ~ 70 µm apart and TEM specimens were extracted. Additional trenches were cut in between as found suitable.

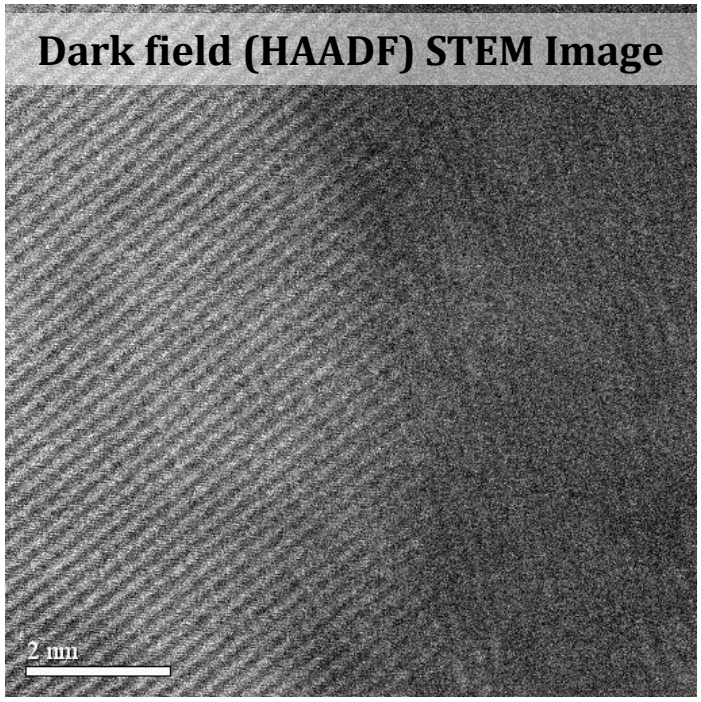
No indication of bismuth in the boundary in specimen D and E ; two possibilities-
(a) the boundary is actually clean,
(b) there is bismuth in the boundary but cannot be resolved by SEM



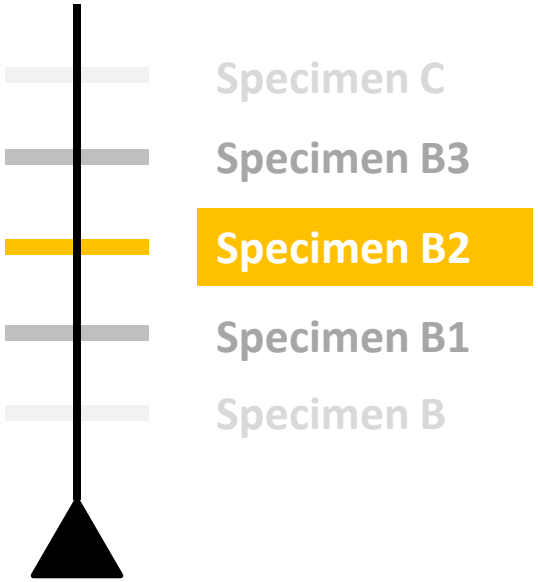
SPECIMEN D





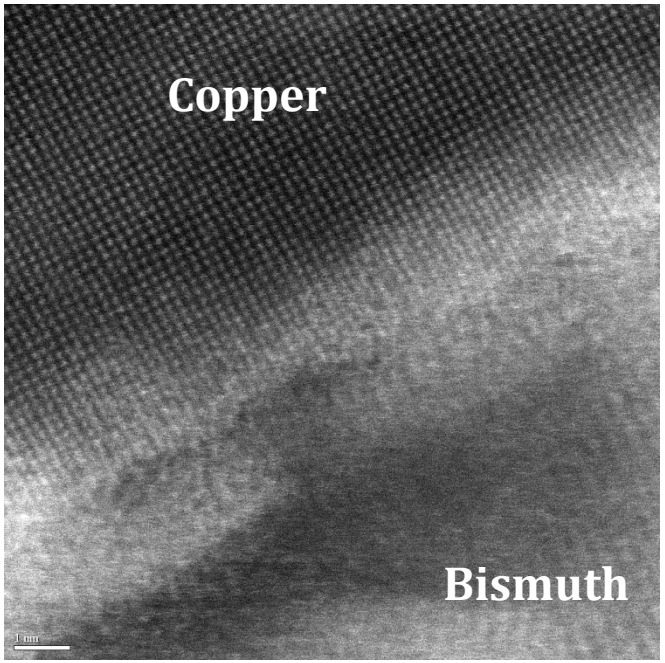


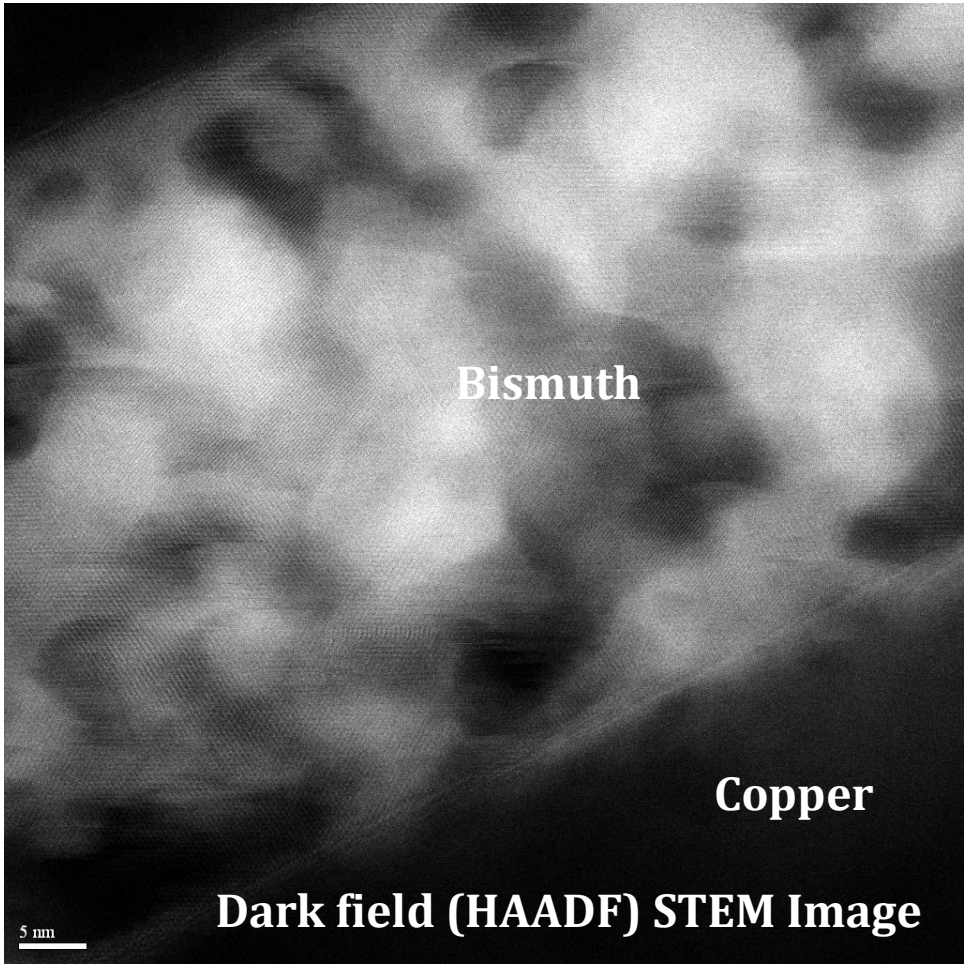
Clean Boundary !



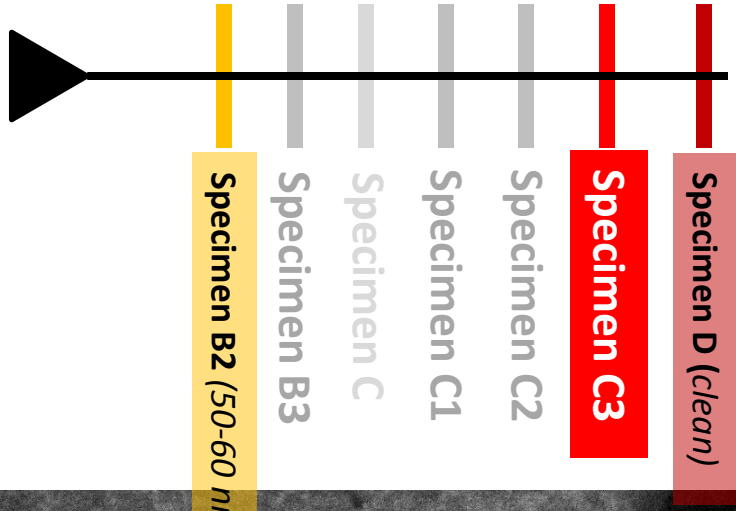
SPECIMEN B2

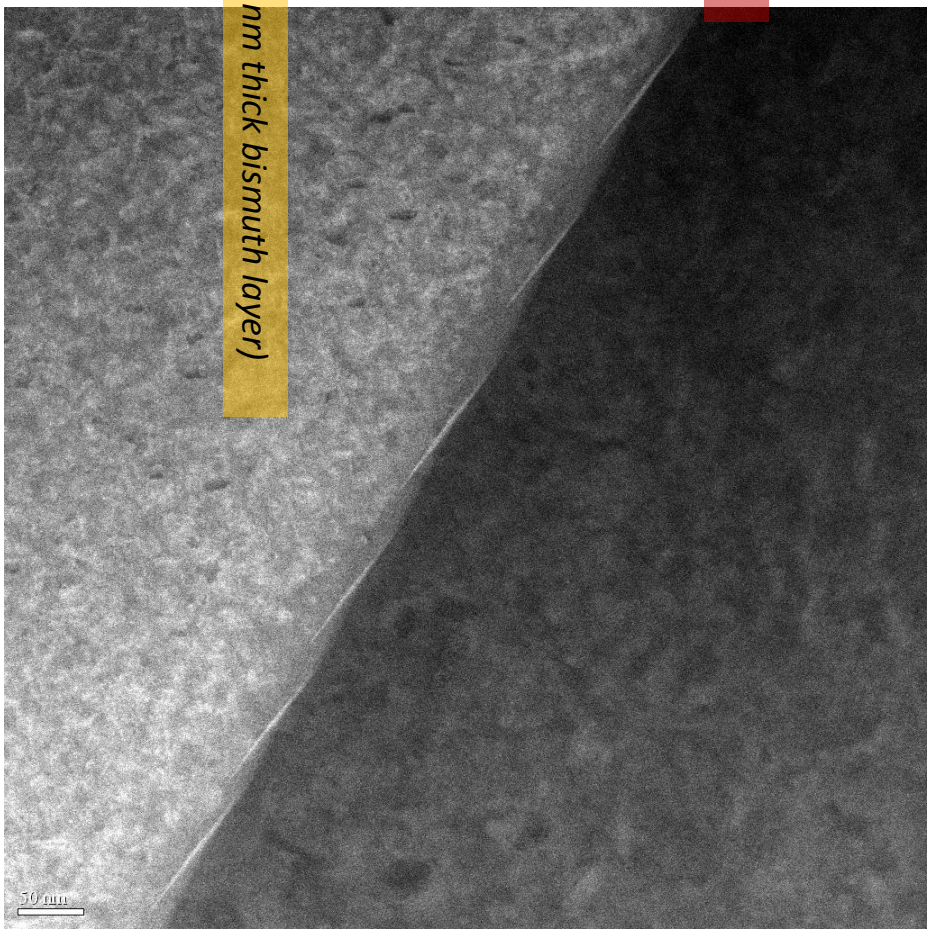
55-60 nm thick crystalline bismuth layer

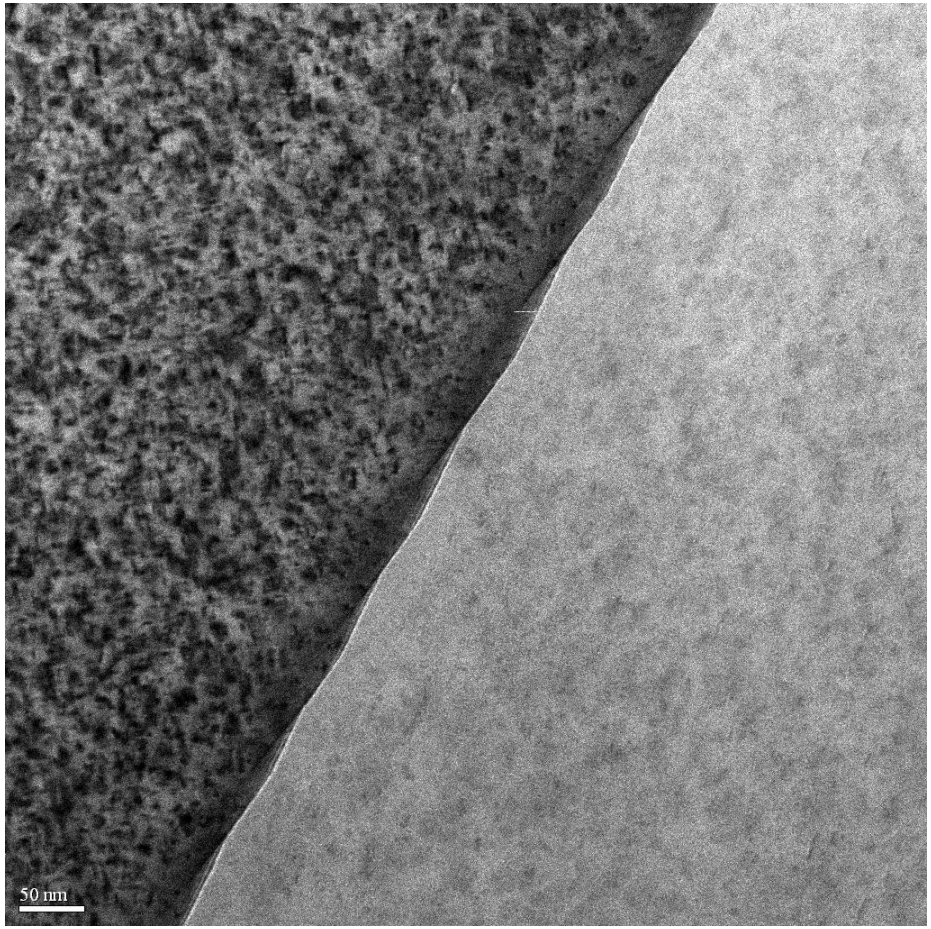




SPECIMEN C3

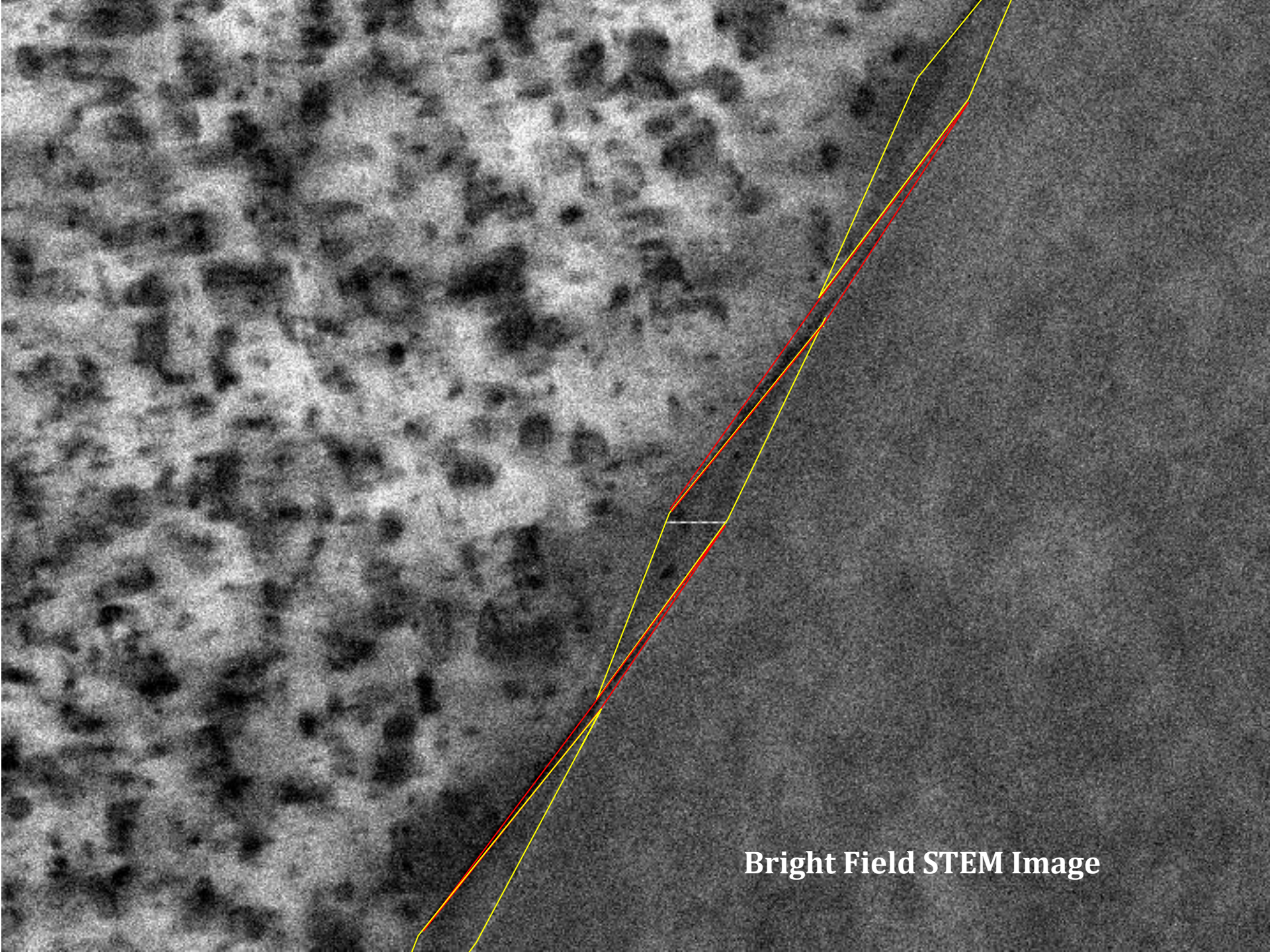


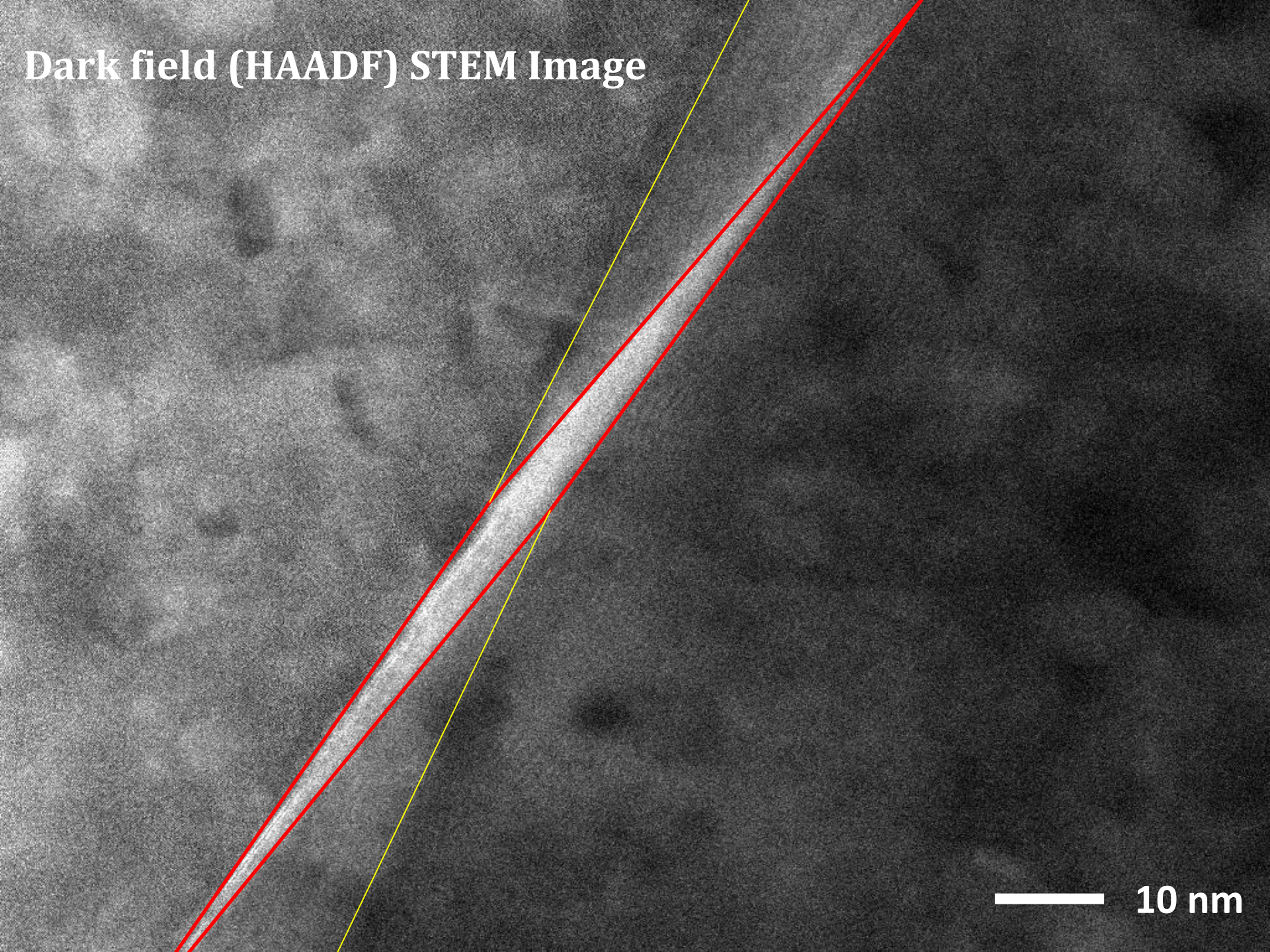


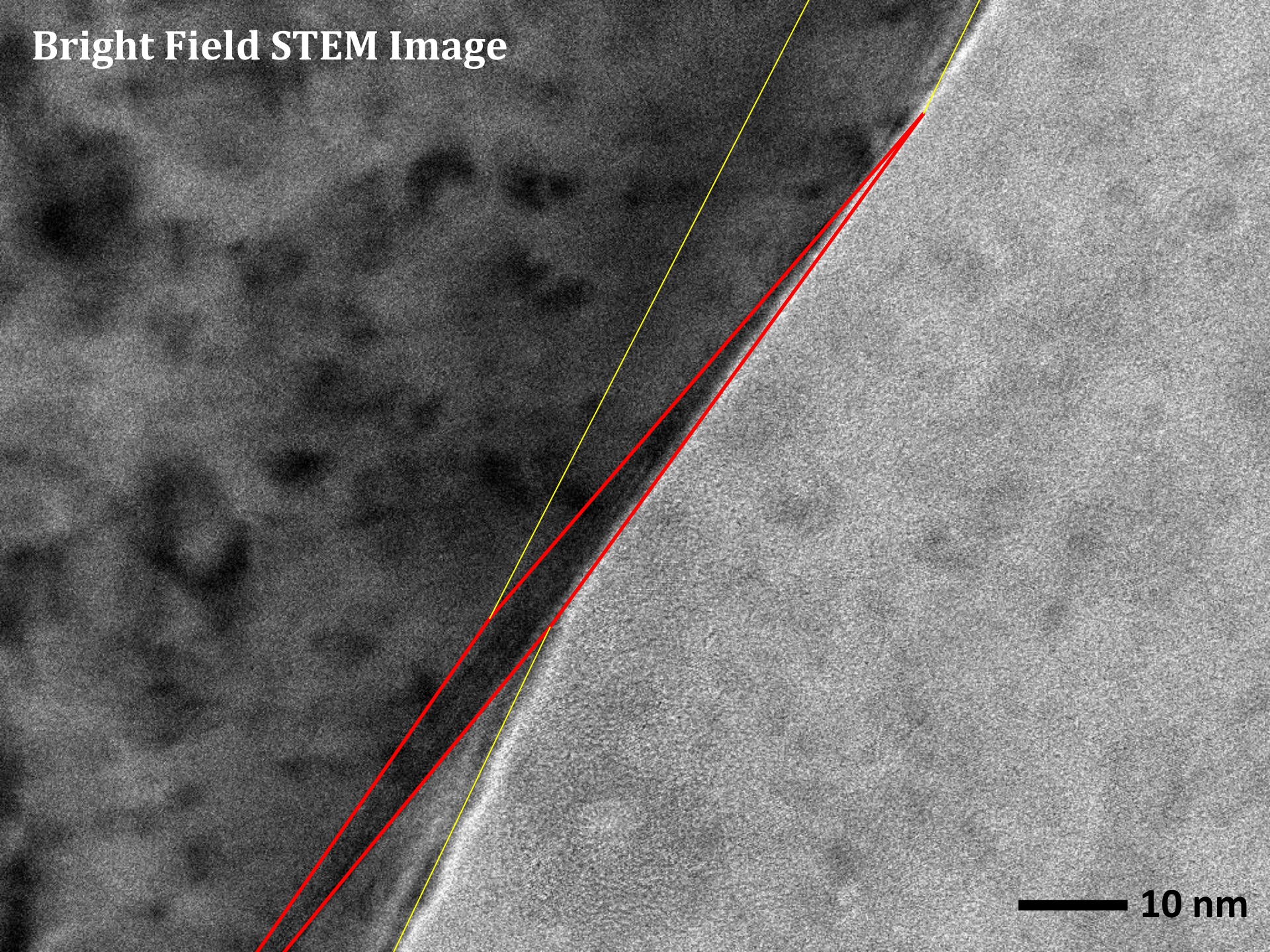


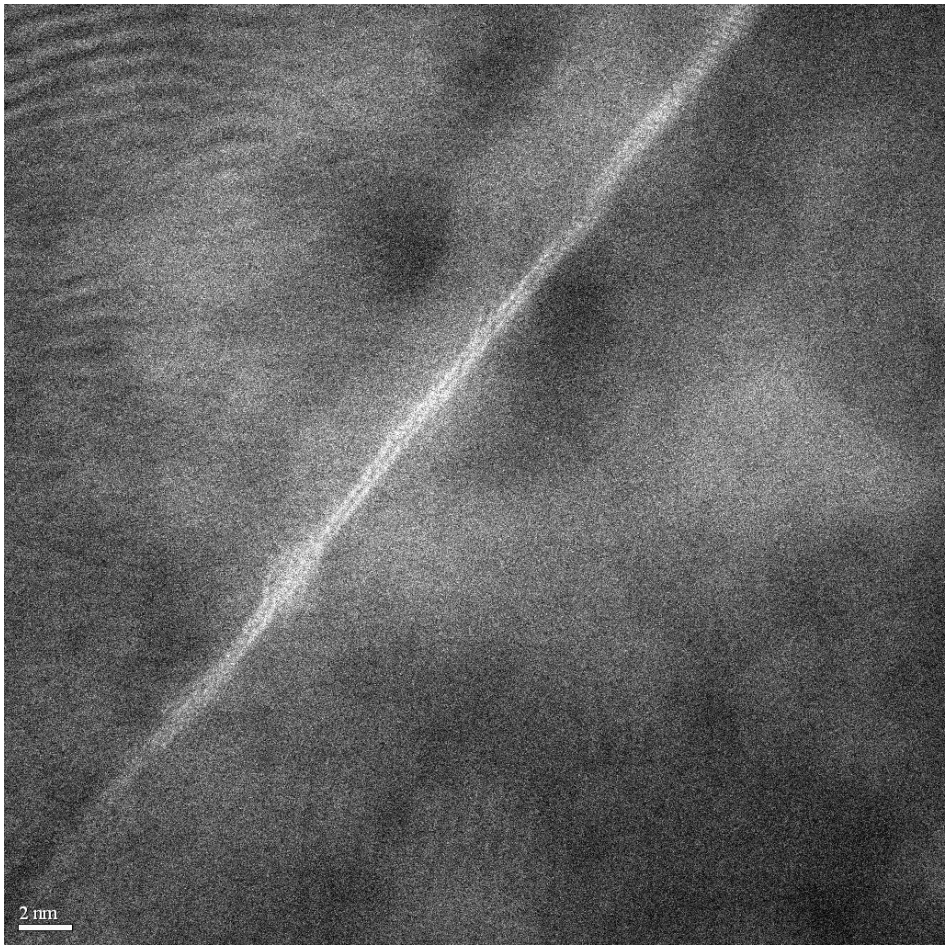
Faceted boundary; bismuth is apparent only one type of facets in the grain boundary. The other facets might have bismuth as well; needs very thorough analytical and imaging routines.

The boundary is tilted in both axes. Appears to be parallelogram in projection: close ups next slide.

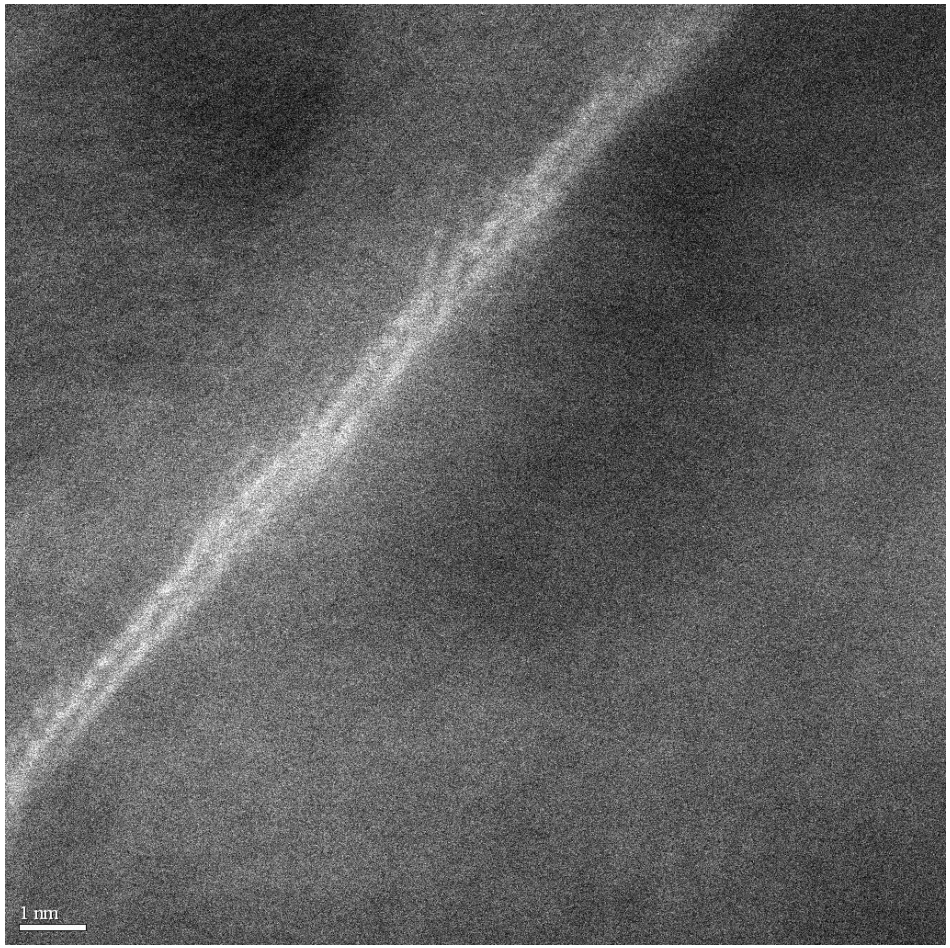

Bright Field STEM Image


Dark field (HAADF) STEM Image
10 nm


Bright Field STEM Image
10 nm

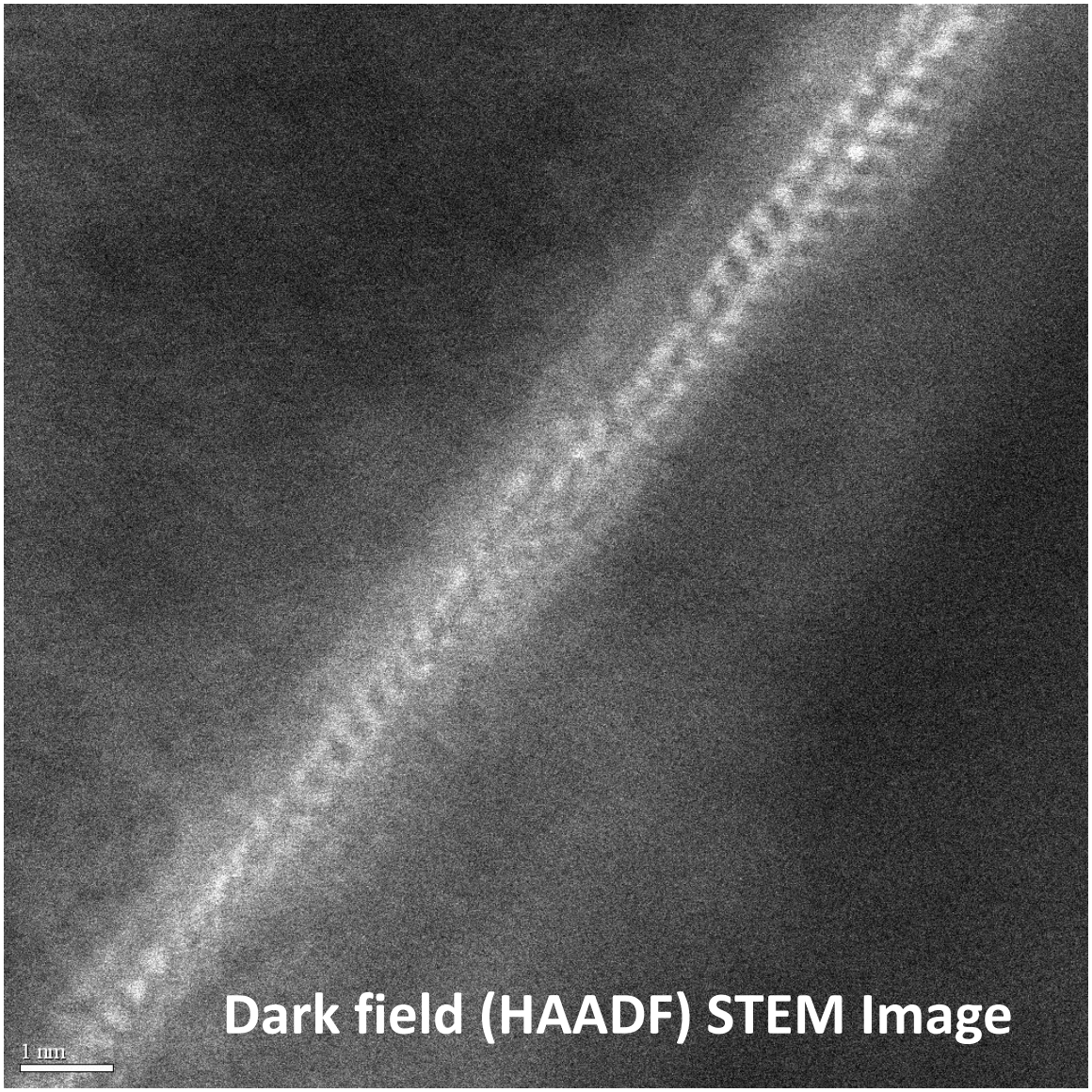


Dark field (HAADF) STEM Image

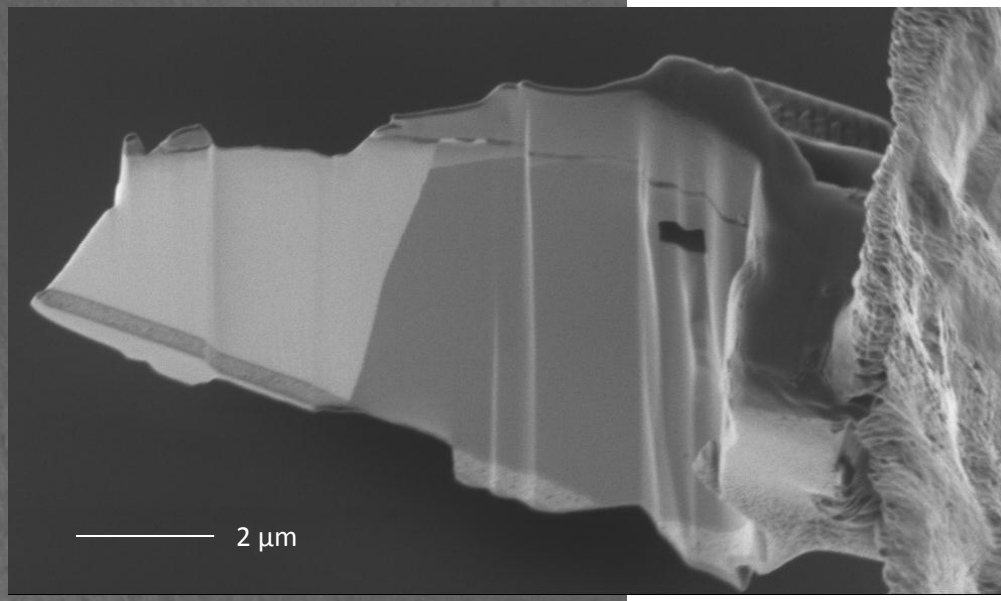


Dark field (HAADF) STEM Image

The faceted boundary with bismuth when tilted very close to edge on appears to have a bilayer segregation of bismuth in the boundary



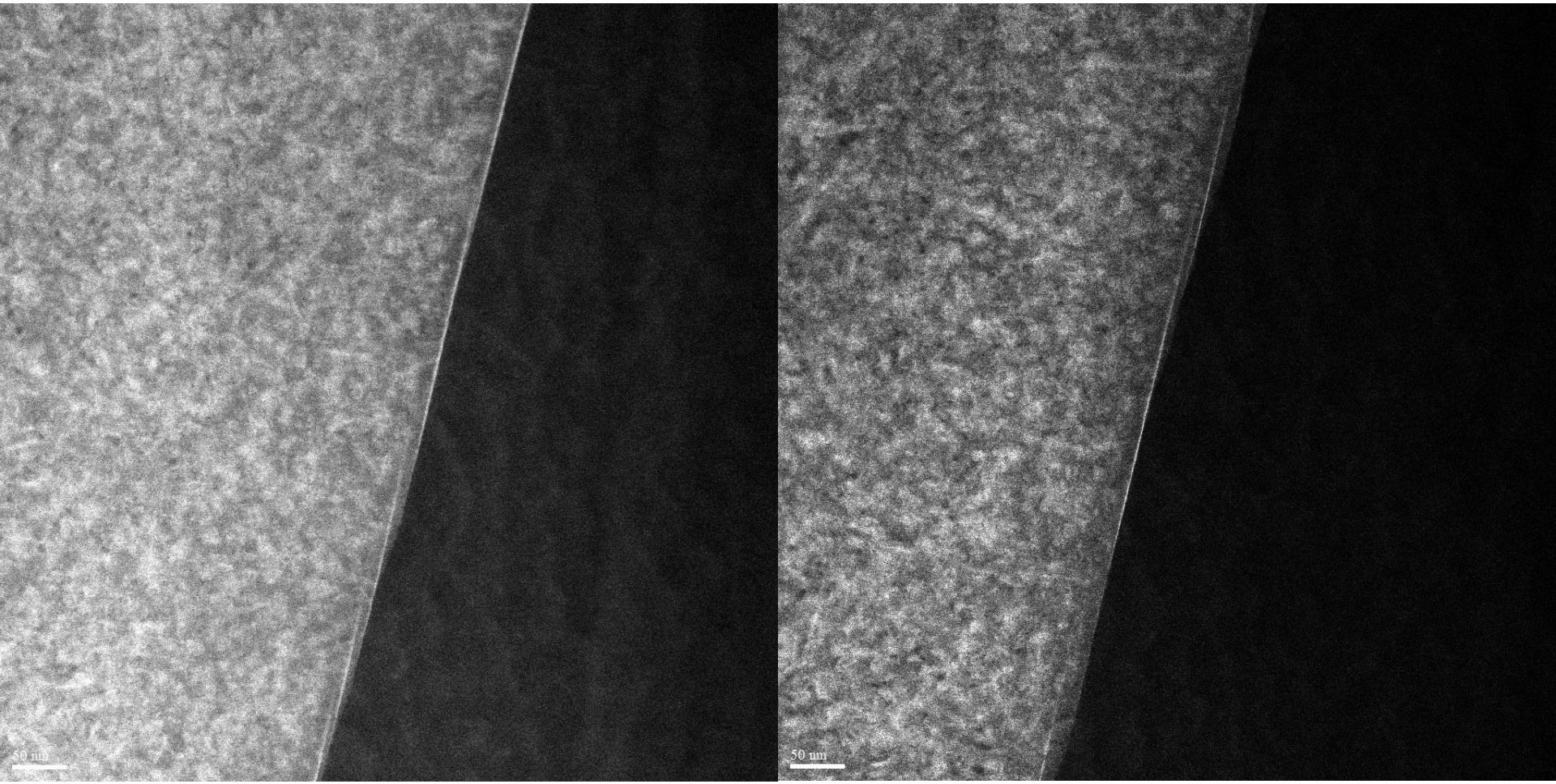
The faceted boundary with bismuth when at a different tilt. The segregation of bismuth occurs in a periodic fashion.



Sample B
700°C, 5h

E-Beam	Spot	Mag	Det	FWD	Scan	
30.0 kV	3	3.50 kX	SED	5.036	H 22.63 s	20 µm

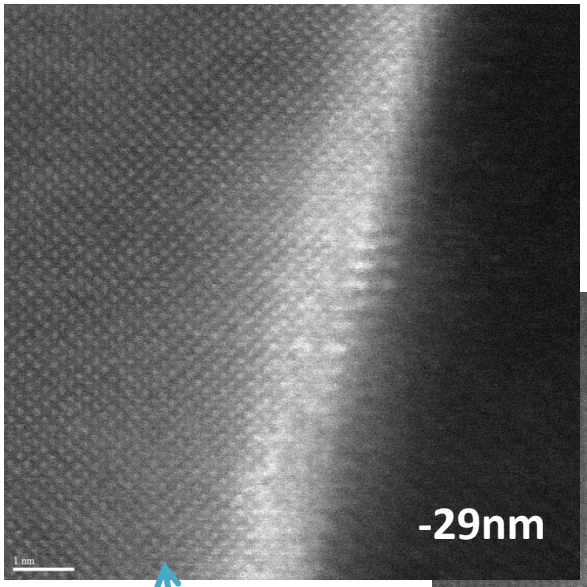
Dark Field STEM Image

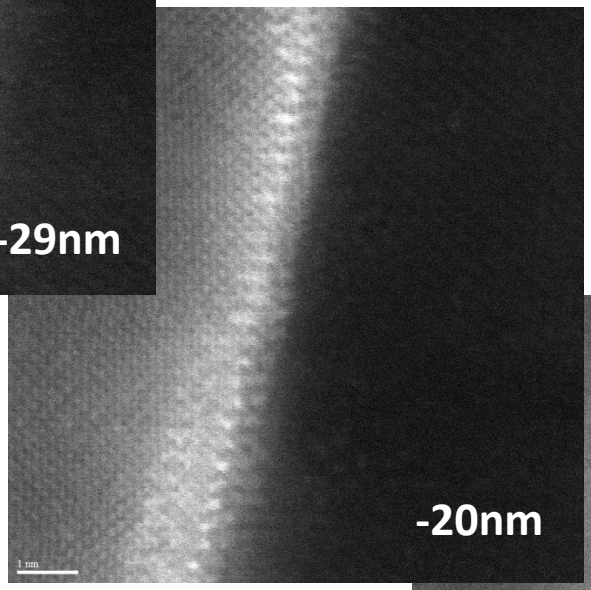


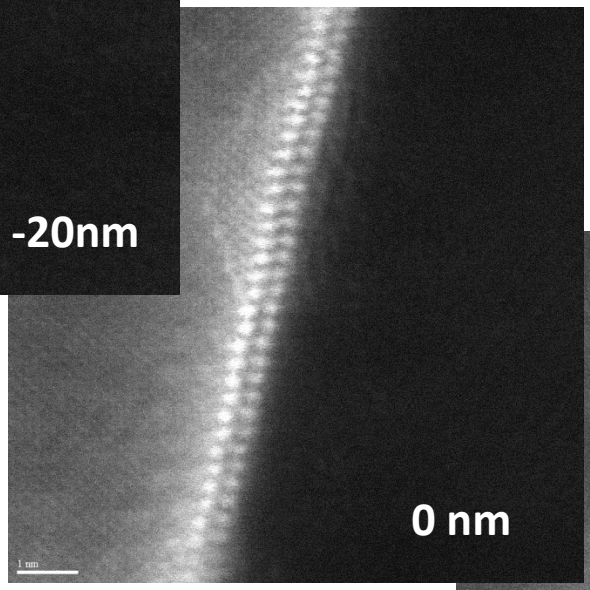
Different segments of the boundary.
The presence of bismuth can be observed in the steps.

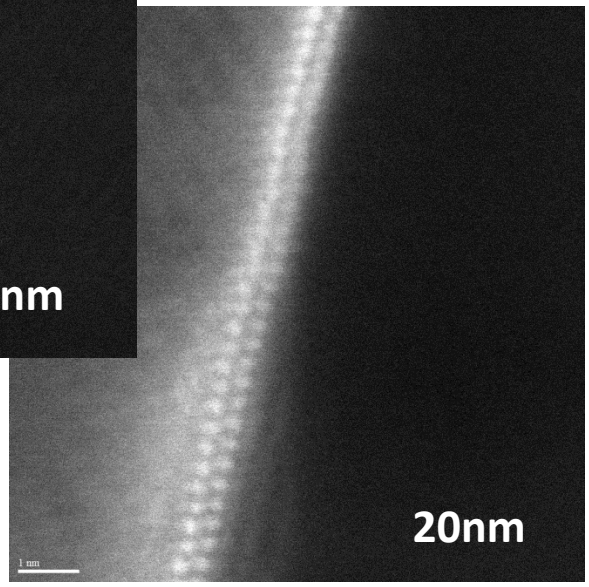
Dark Field STEM Image

Defocus series of a small segment of the boundary





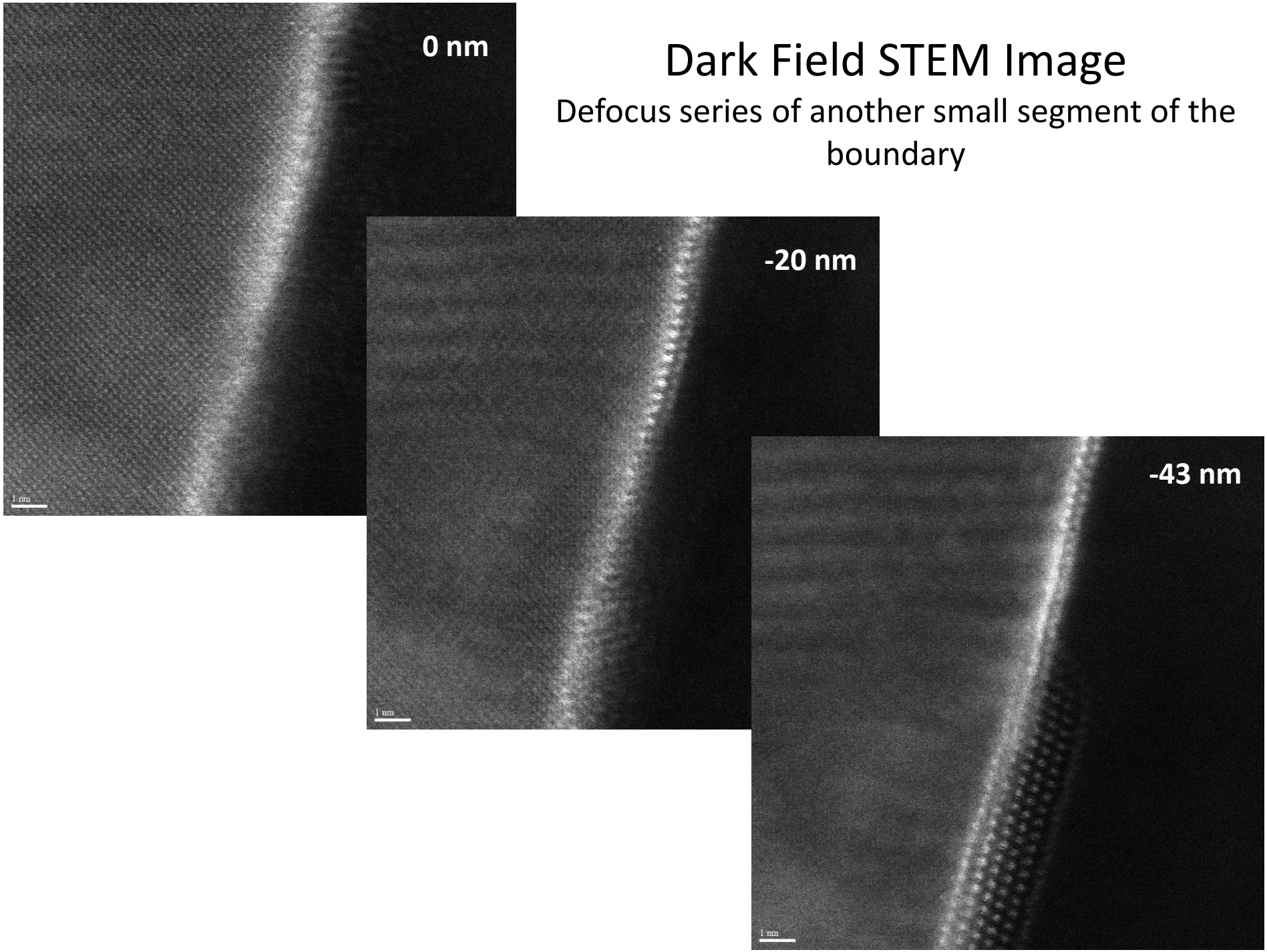




The grain on the right side is at zone axis.

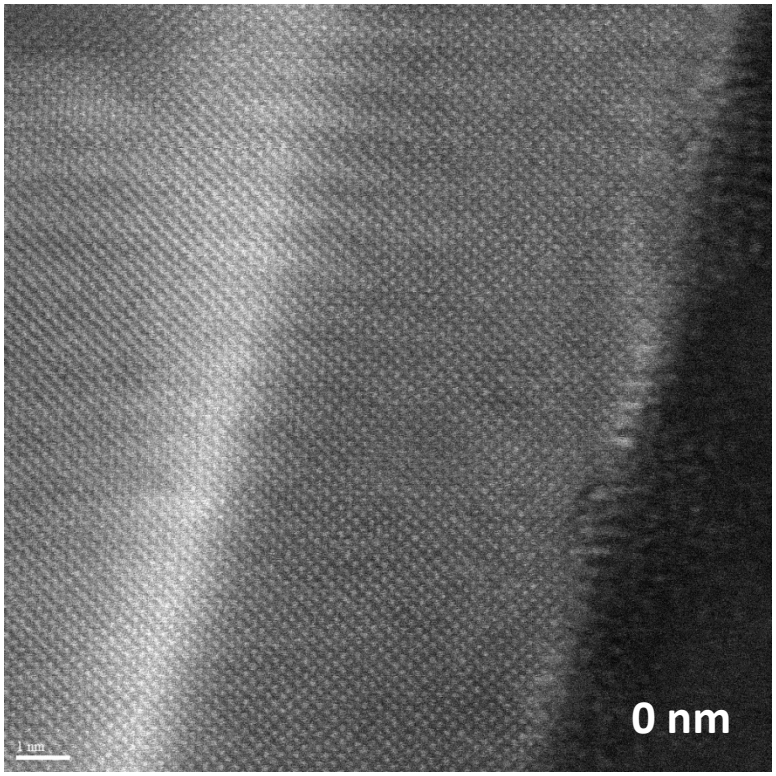
Dark Field STEM Image

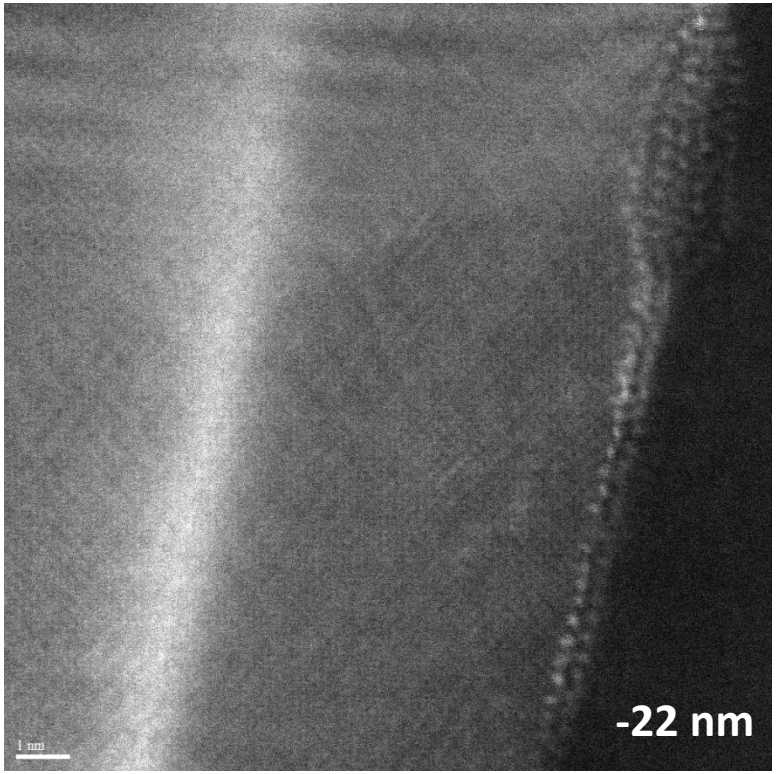
Defocus series of another small segment of the boundary



Dark Field STEM Image

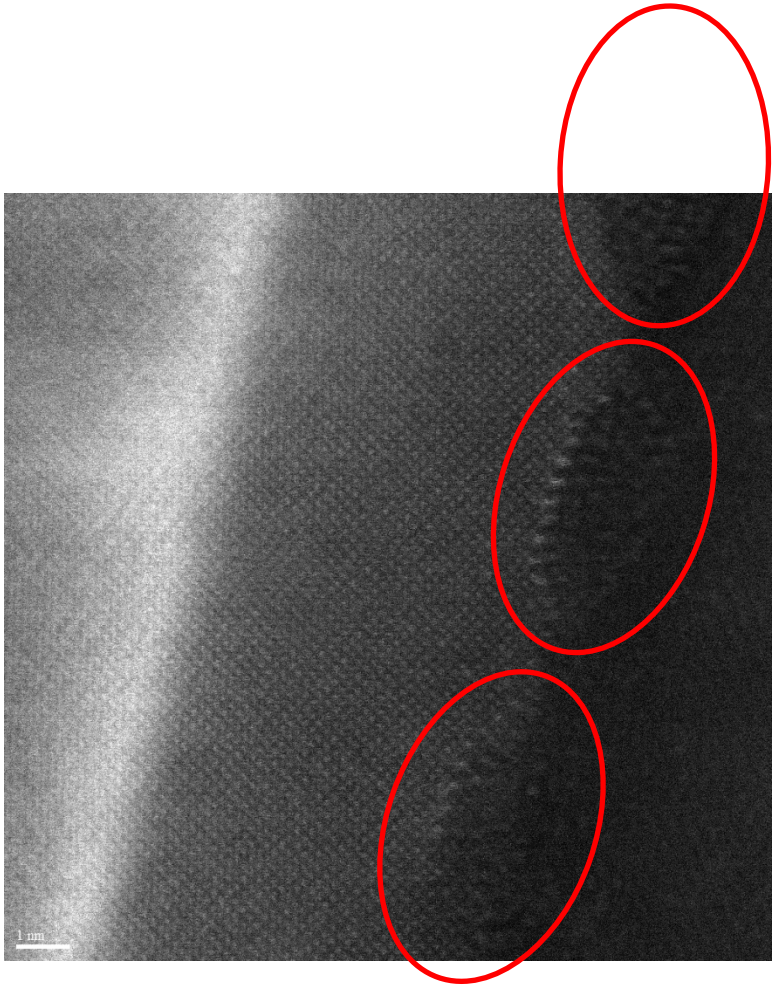
Through focal pair of a unique segment of the boundary; a lens shaped segregation can be observed on the right hand top corner of the images. Additionally a bright contrast can also be observed in the grain on the right side of the image



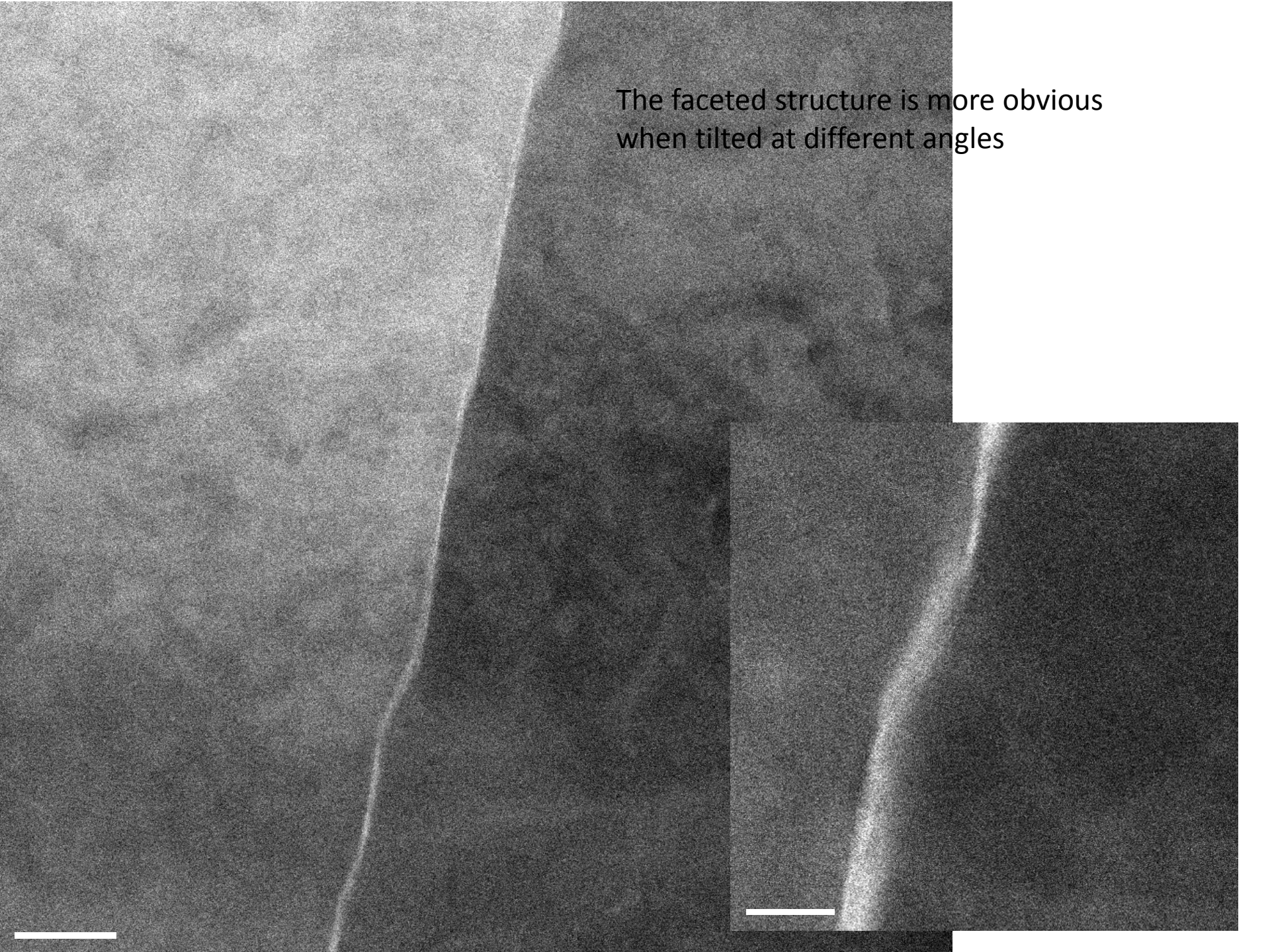


Dark Field STEM Image

Similar feature was observed at other segments as well



Lens


The faceted structure is more obvious
when tilted at different angles