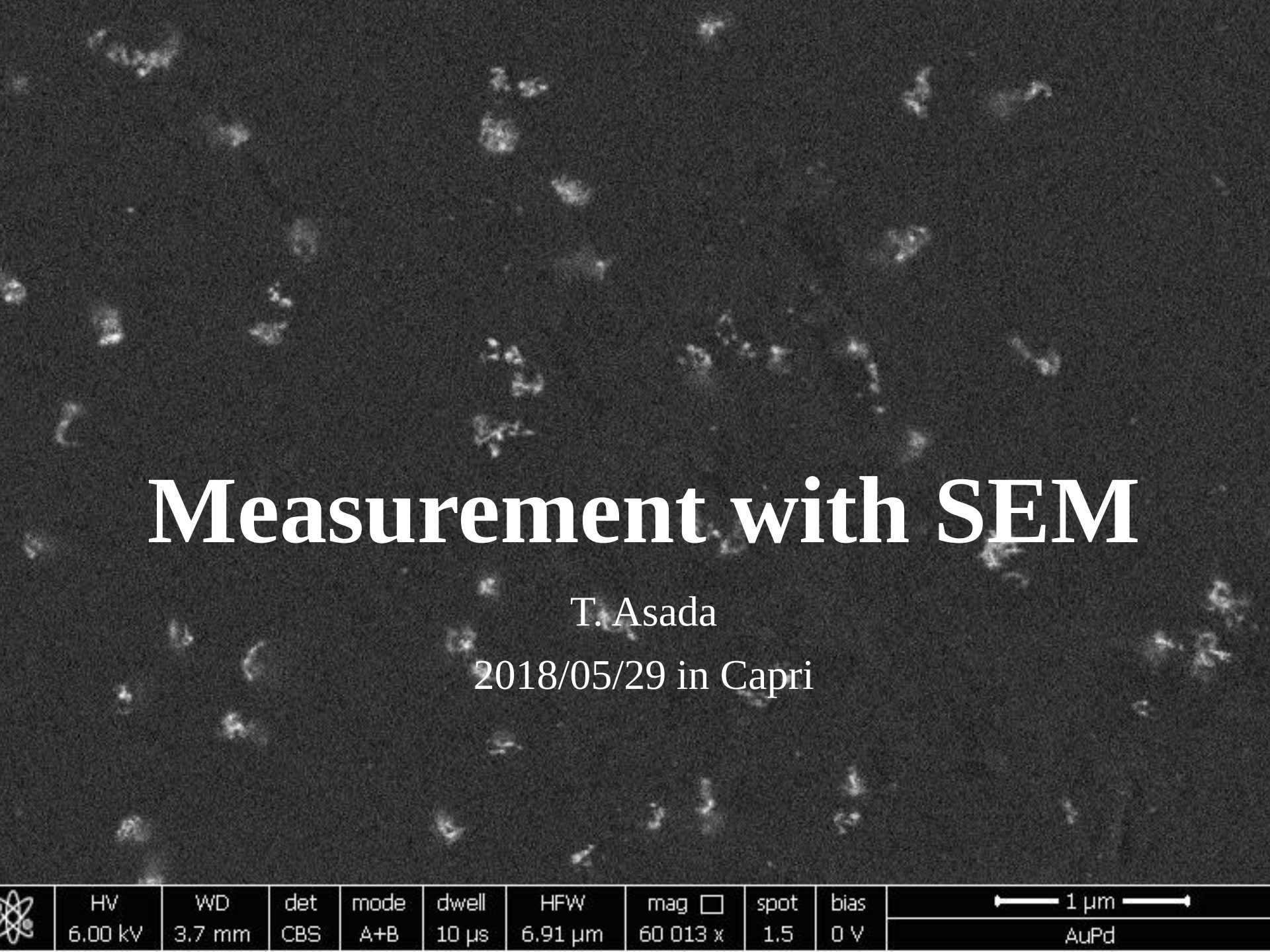
Measurement with SEM

T. Asada

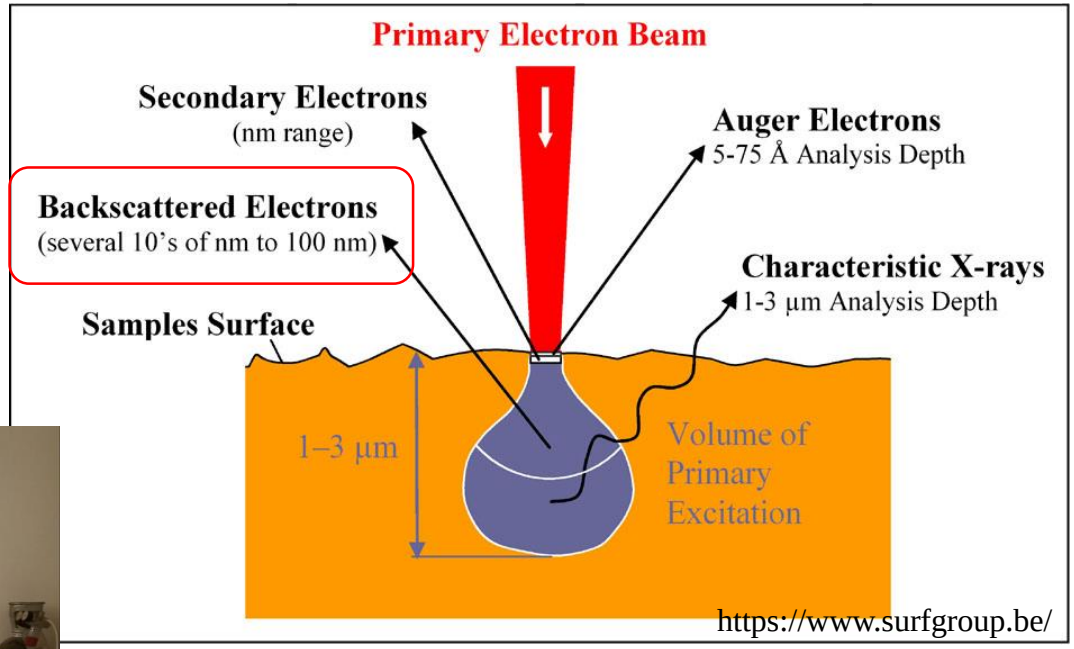
2018/05/29 in Capri



Purpose

- Comparison of actual filament structure and Plasmon resonance
- This report uses eye check of SEM images and scanned data of Naples system, aiming the check of validity of parameters
- Machine learning is expected as prospect

Scanning Electron Microscope



Chemical department
Help by Dr. Fabio Borbone

- depth of focus is very high, but penetration depth is several hundred nm
- the resolution depends on the width of electron beam and its diffusion

Requirement for SEM

- resolution < 10 nm/pix to see filament structure (width ~20 nm)
- V_{acc} > 5 kV for scattering of electron (see next page)
- conservative observation for structure destruction
  - at least 2 times sequential scanning gives same filament structure

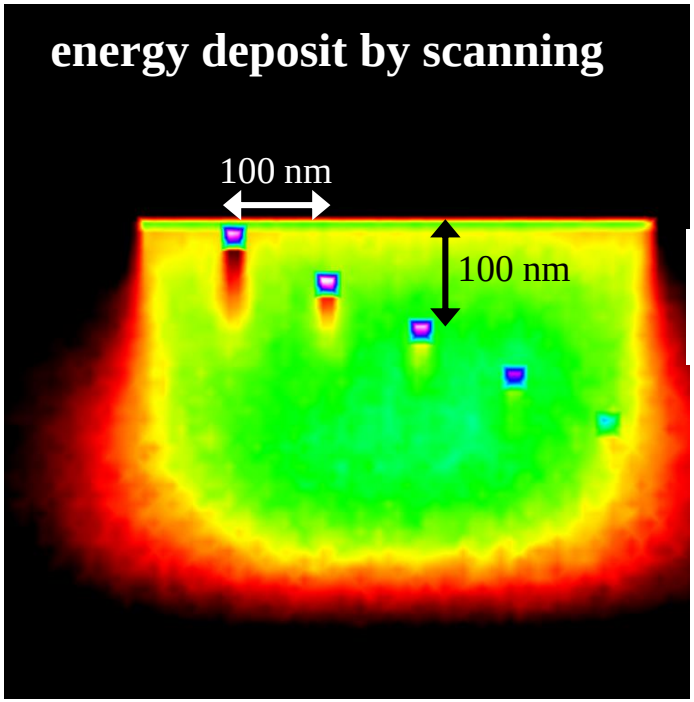
  → V_{acc} < 7 kV pre-check shows 7 kV or more

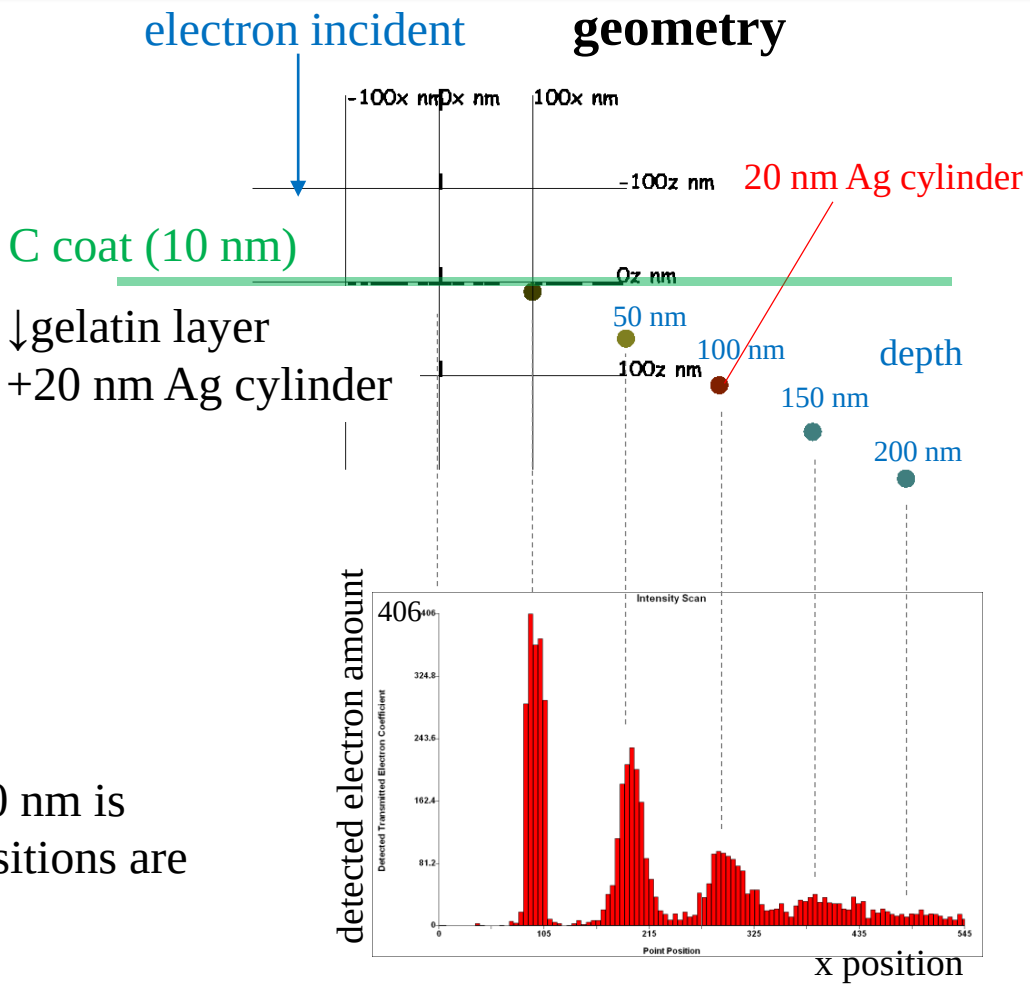
- position matching for comparison to optical scan

SEM simulation by CASINO v3

Reported in Oct 2016

SEM Vacc = 5 keV



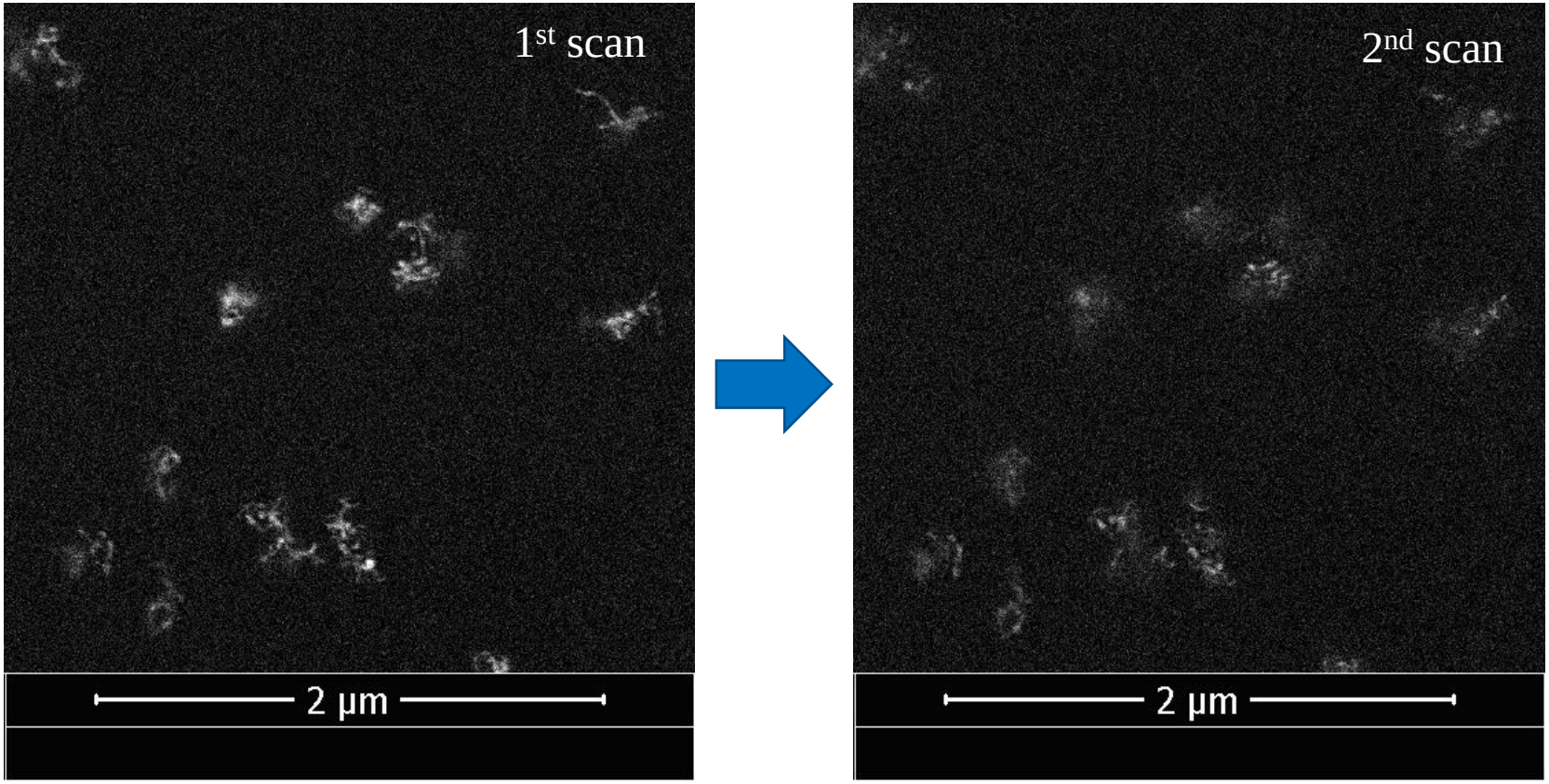


detected electrons / 1000 ev in sensor position

Ag filament which depth is up to 200 nm is detectable, however 100~200 nm positions are worse contrast and resolution.

The contrast effect may be not so strong in our target; Carbon, energy < 100 keV
(see nextpage)

problem of structure destruction



sequential scan show a destruction of filament structure in some condition
I afraid that even 1st image gives destructed structures, so I required 2 sequential scan giving same structure for conservative observation
→ $V_{acc} < 7$ kV ; $V_{acc} = 6$ kV considering requirement

condition check: surface coating

- Surface coating layer is need for electron conduction
- minimum coating is required to keep good contrast

Carbon sputter

- Carbon: transparent material, however thicker coating (20 nm) didn't contribute to film protection performance while resolution becomes worse

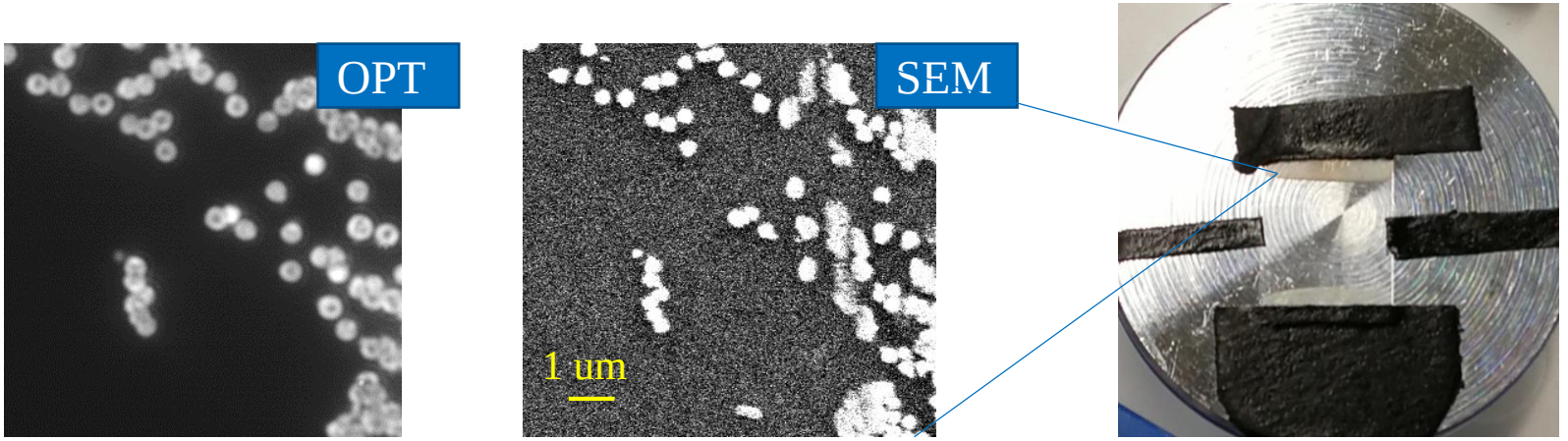
- Au/Pd: reflective material, however, very thin layer (4 nm) gives enough conductivity, and gives better contrast

→ Au/Pd 4 nm is best

Au/Pd sputter

position matching

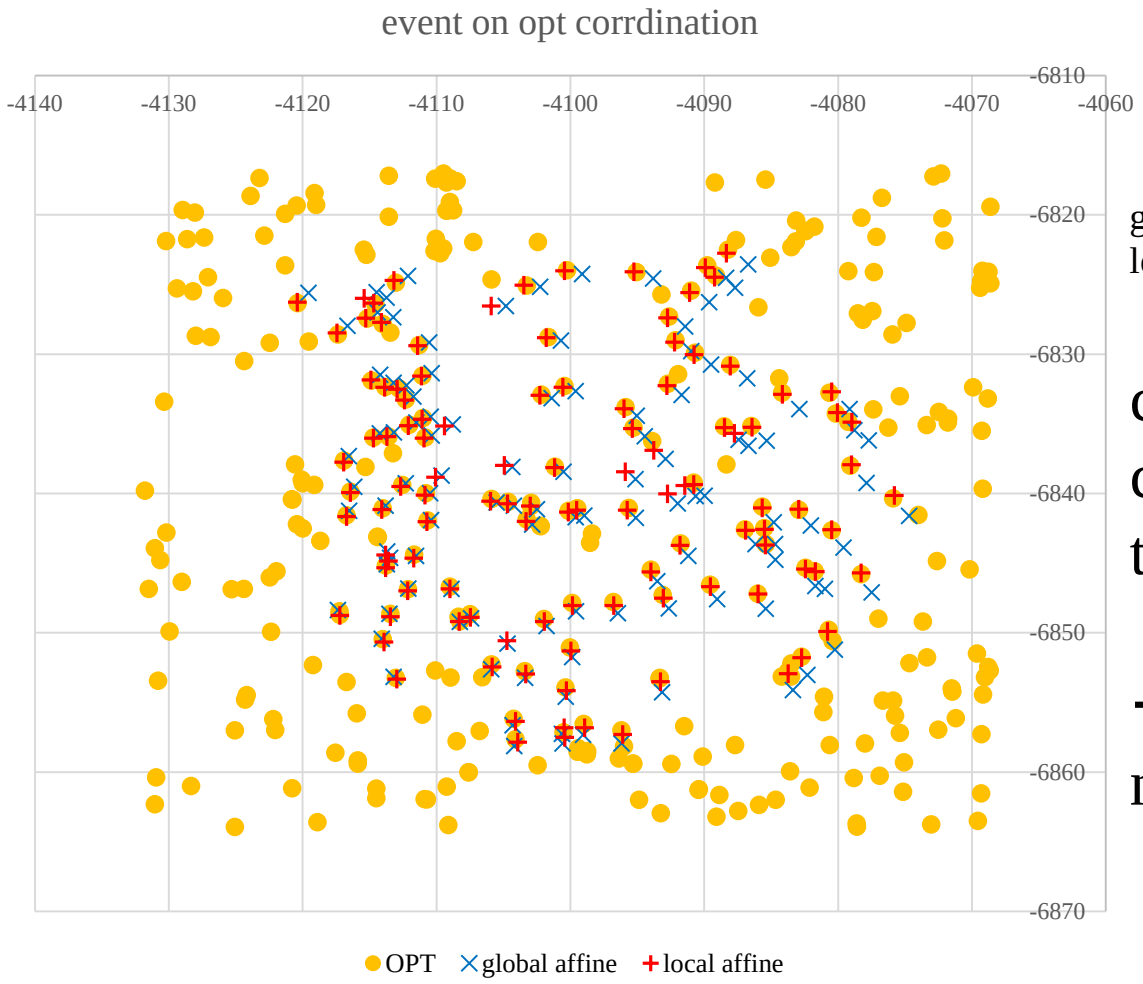
- global matching : optical marker by large crystal



- affine conversion using same corner markers give ~1um of RMS
- actual check of observing view position still keep few micron error

  → enough for prediction, but local affine conversion is needed for dense ion sample

position matching



global affine: corner optical marks
local affine: event matching inside view

currently local affine conversion needs manual treatment for view by view

→ pattern matching is needed for large statistics

Scan sequence

- maximum view size 55.1 x 36.8 um (fiducial area ~ circle of 40 um diameter)
- almost equivalent to optical view size (60 x 45 um)

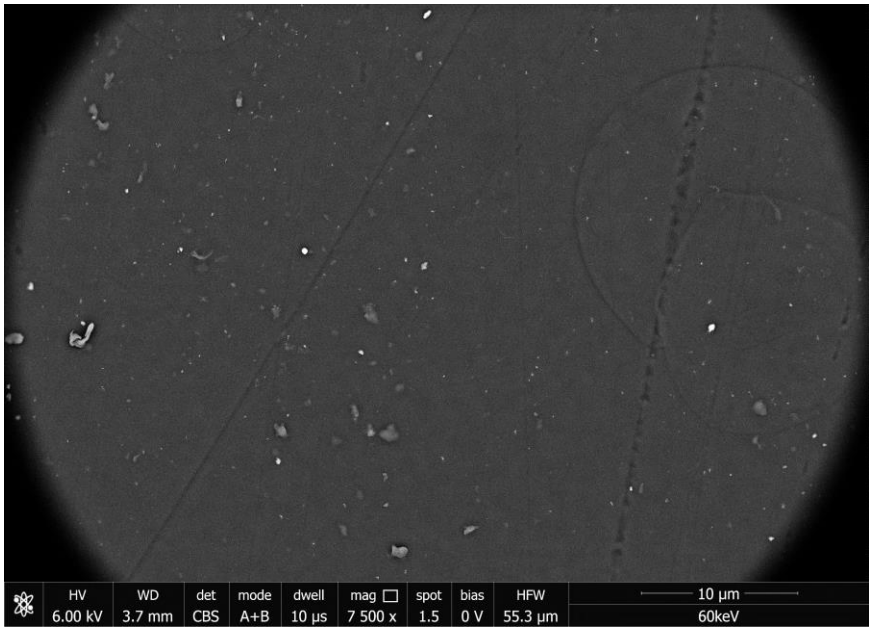
  →view by view scan
- scan speed ~9 min/view
- position matching takes ~2 hour

- we are planning to automatic scan during night time for machine learning data

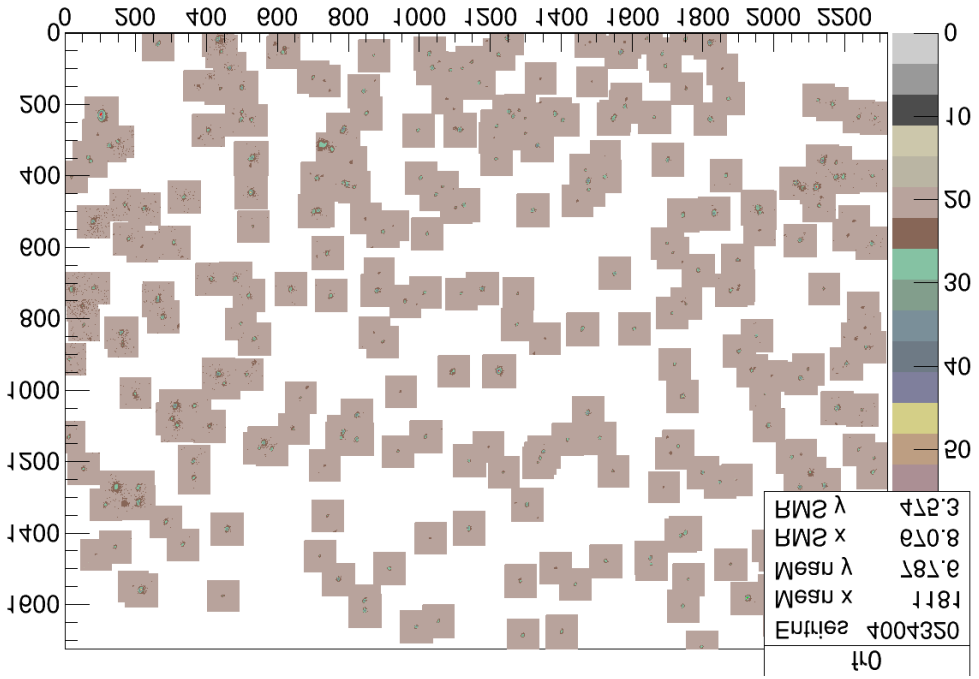
  (ex. 18:00–9:00; 15 hour → 100 images)

1 view matching

(C 60 keV)



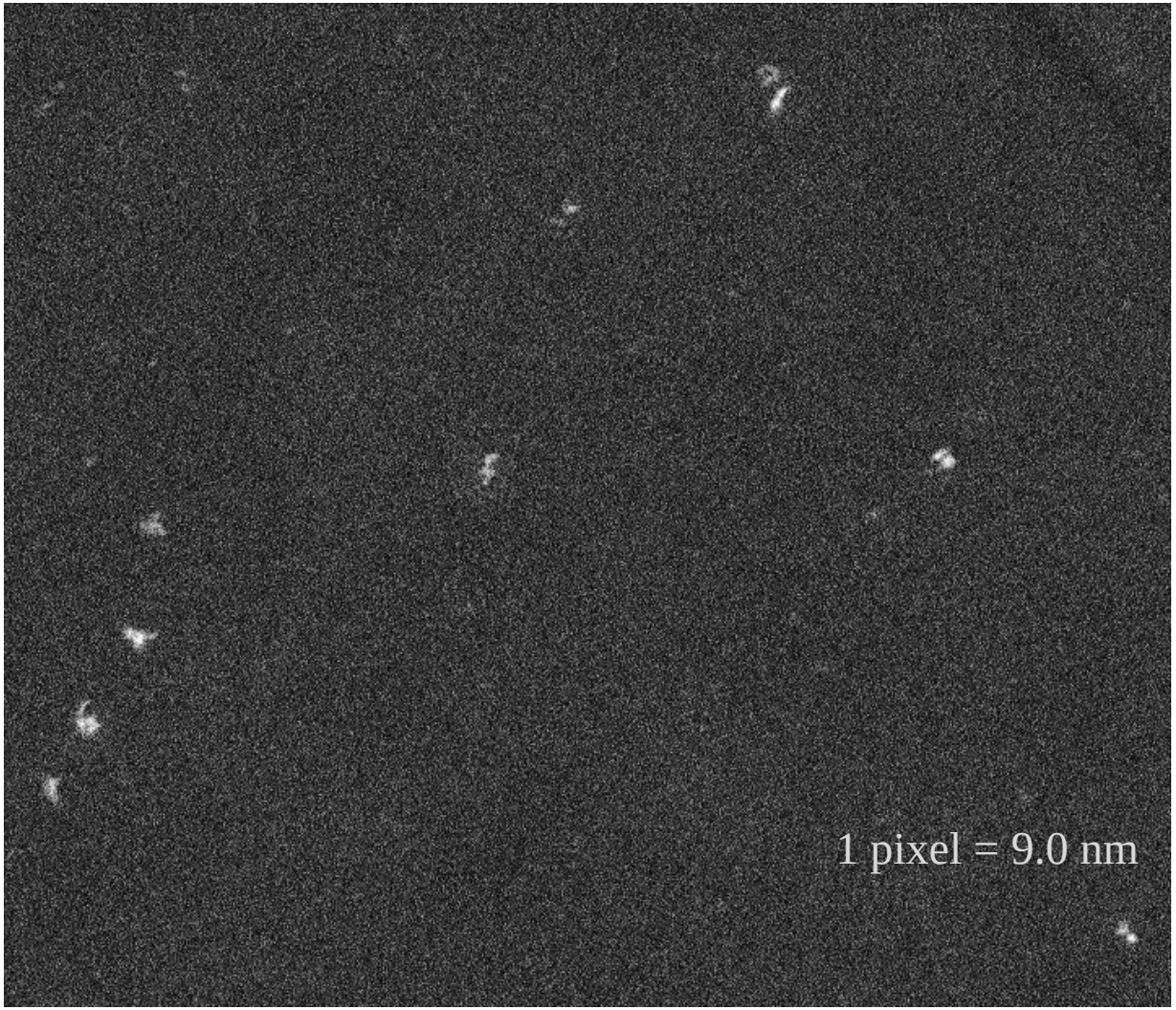
SEM view 55.3 x 36.8 um

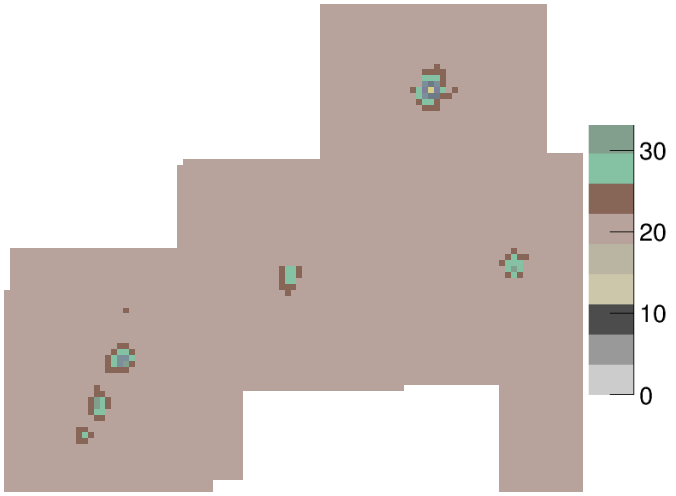


Napoli view 60 x 45 um

1 view matching / zoom

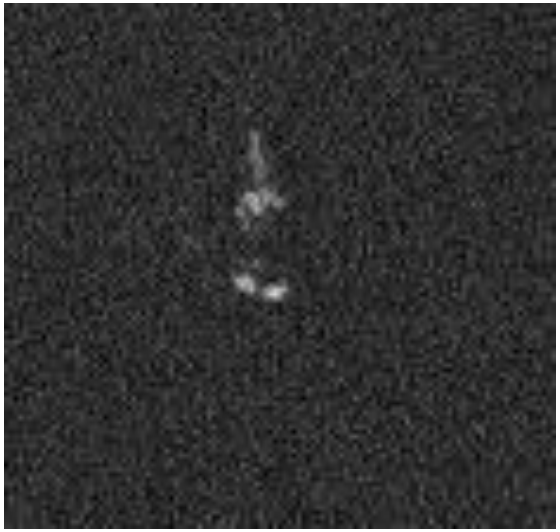
(C 60 keV)

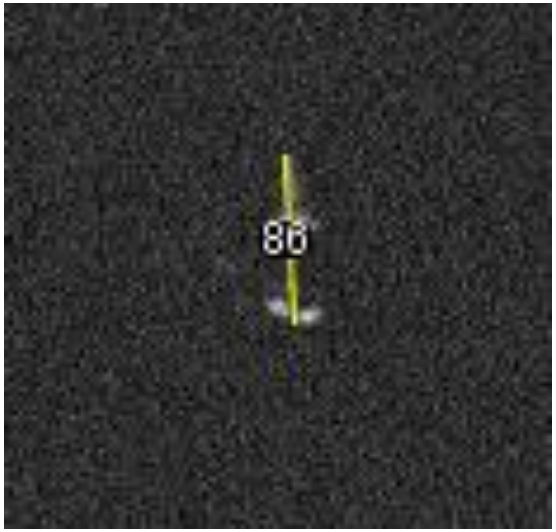




1 pixel = 27 nm

manual check

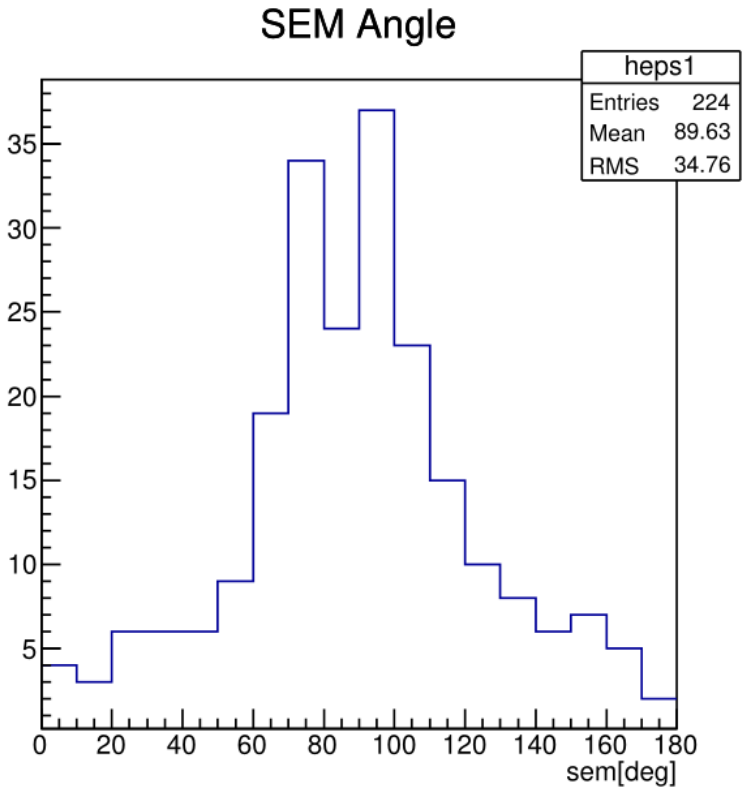


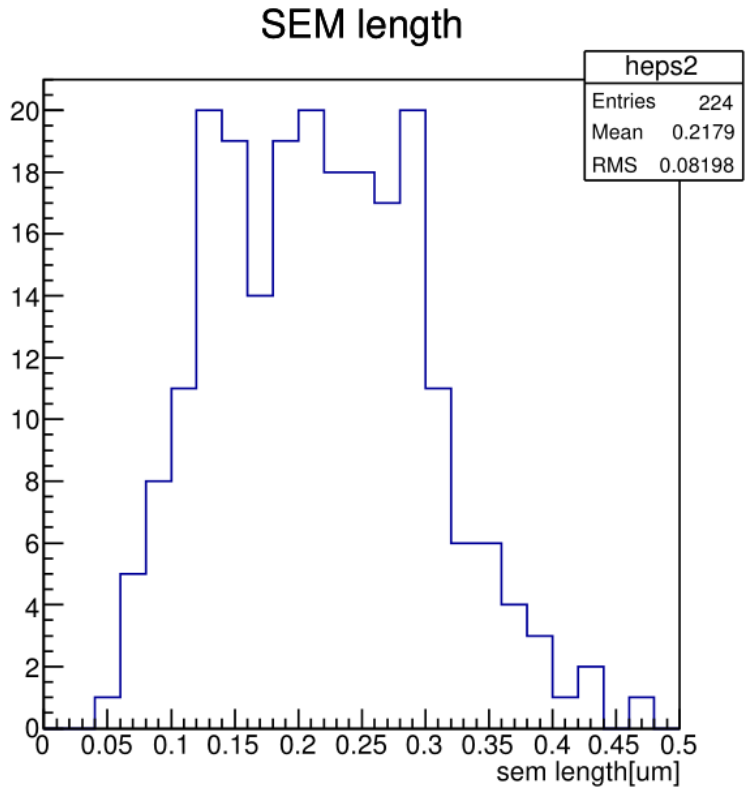


using a free software ImageJ

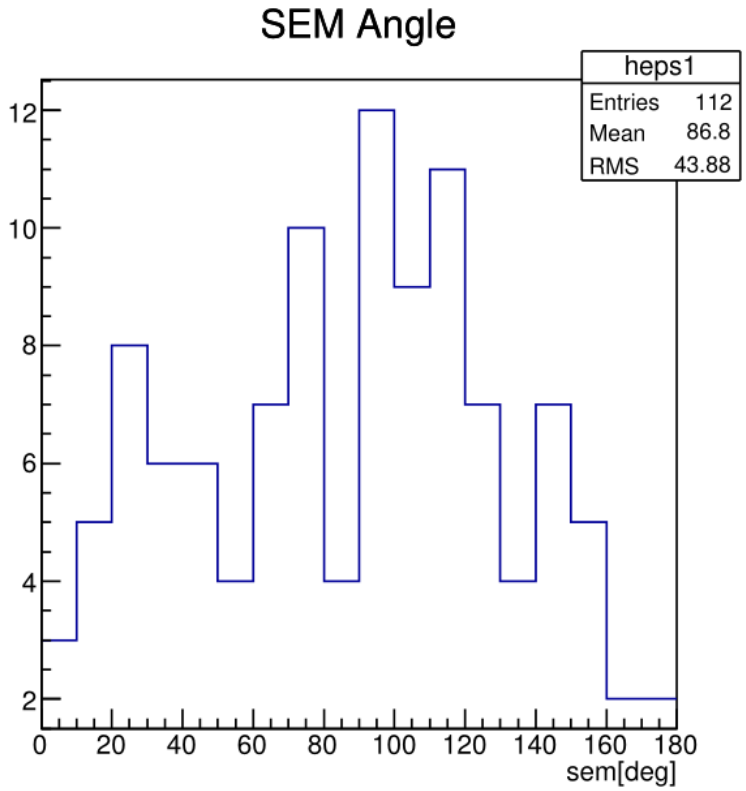
direct analysis from image is currently difficult due to dust, contrast, and structure.
As first step, I measured length and angle of tracks.
Measurement is based on the thought of Feret's Diameter(maximum length of structure)

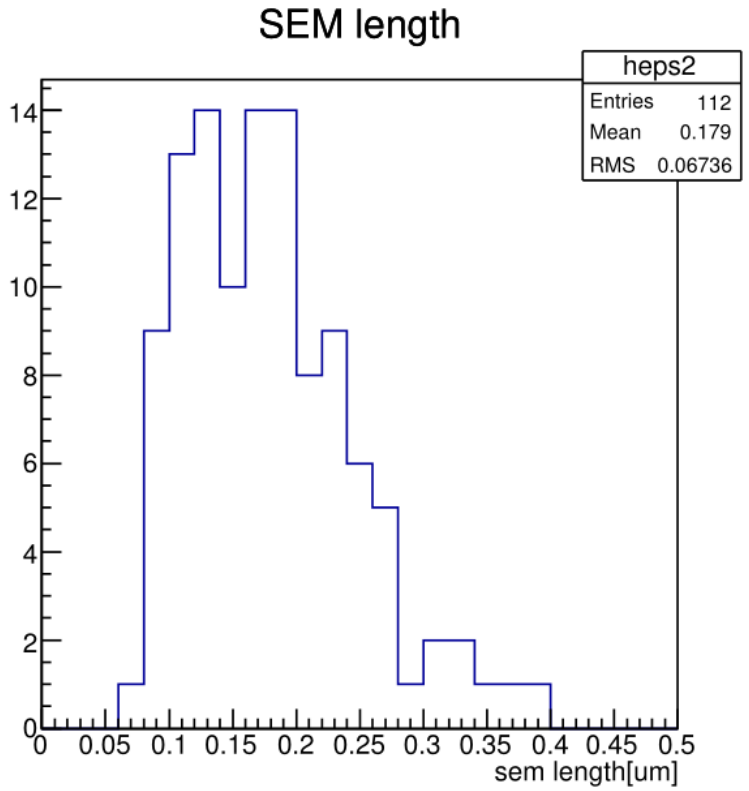
manual check result (C 100 keV)



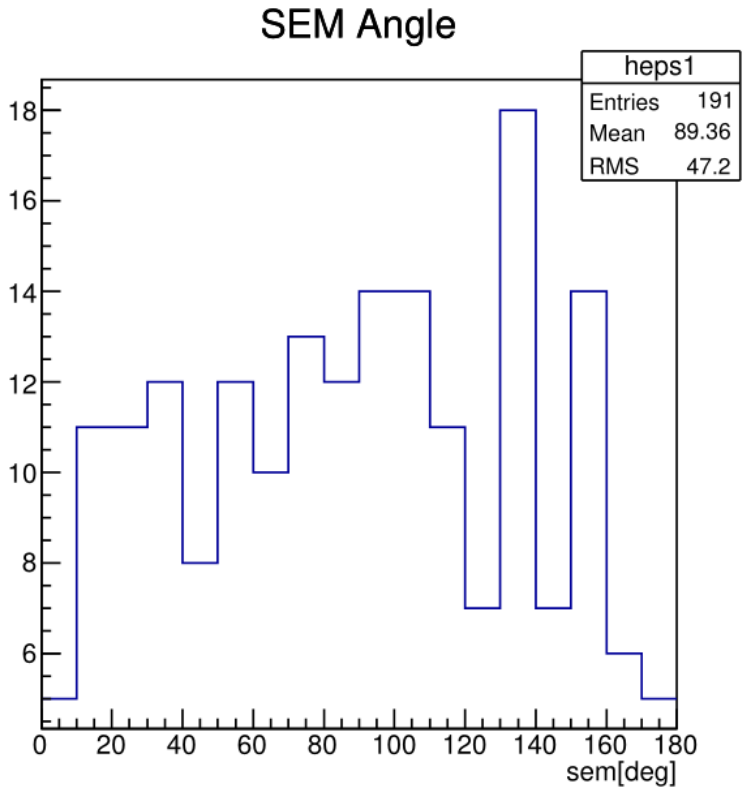


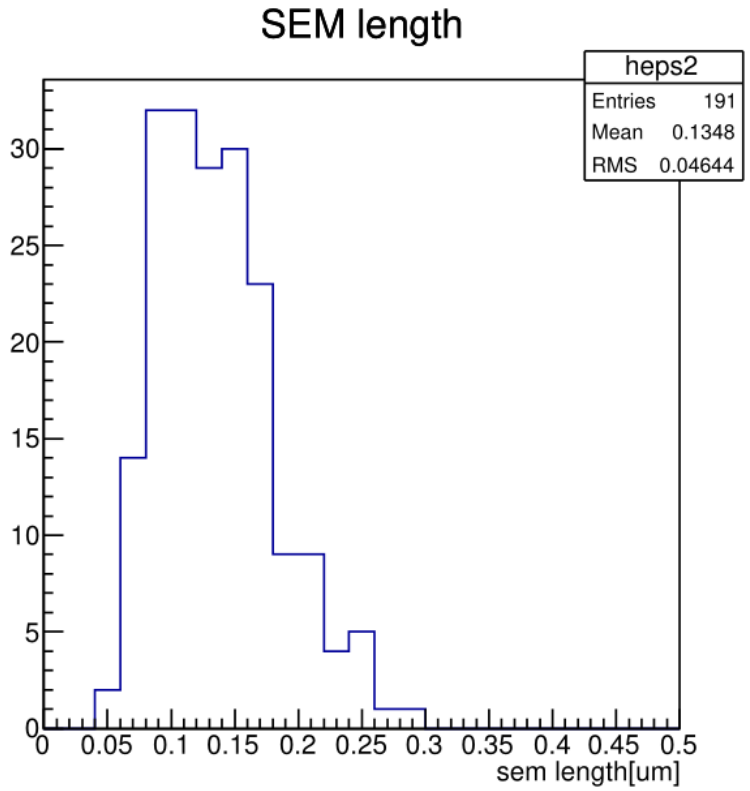
manual check result (C 60 keV)



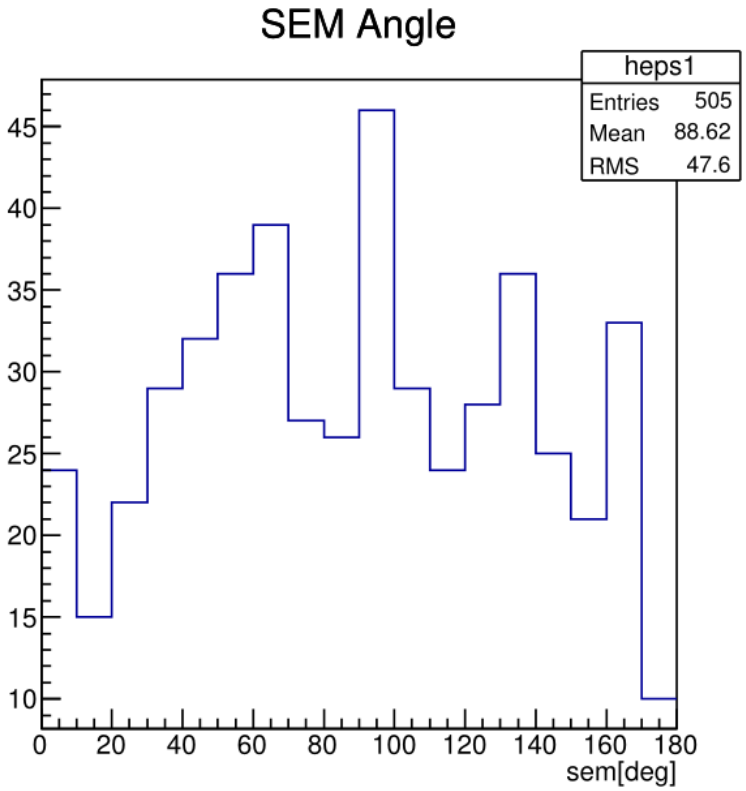


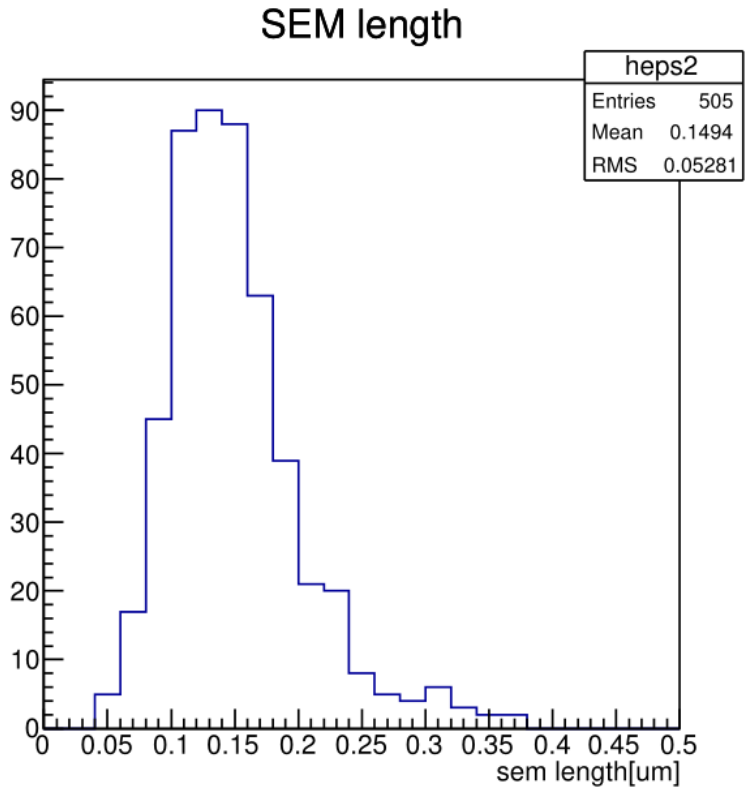
manual check result (C 30 keV)



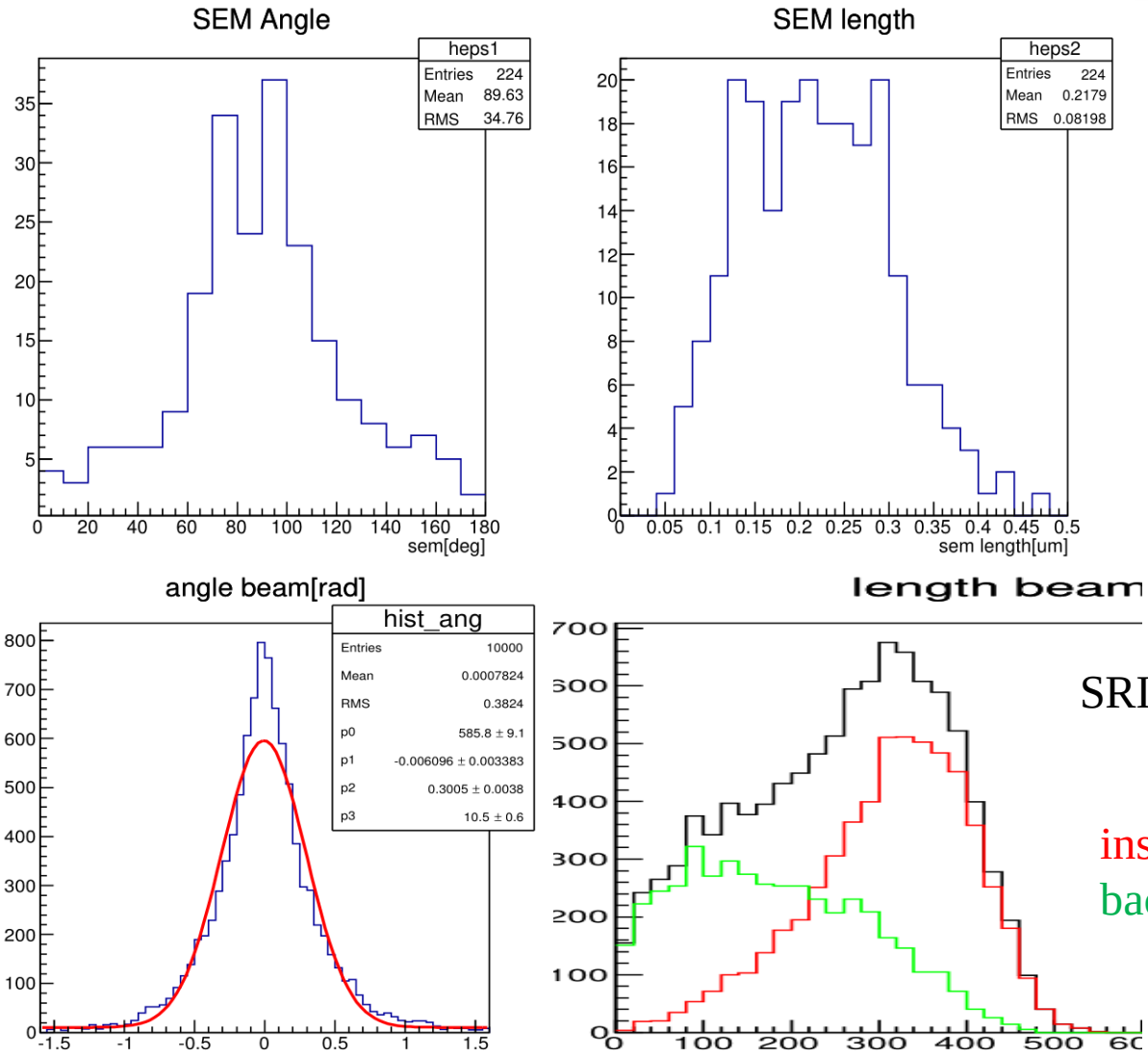


manual check result (C 30 keV Vertical)





comparison to SRIM simulation (100 keV)

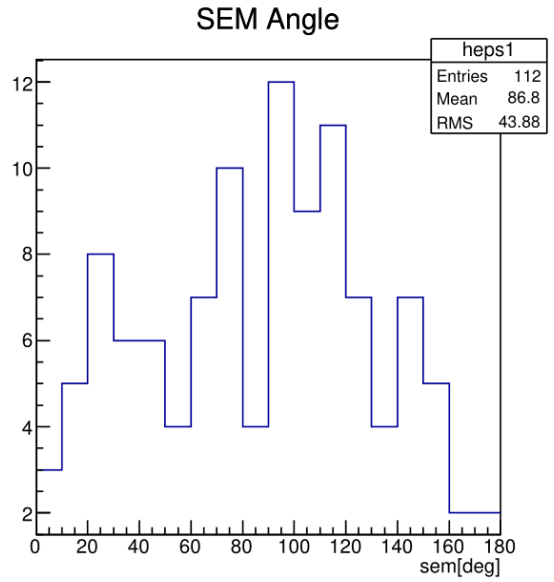


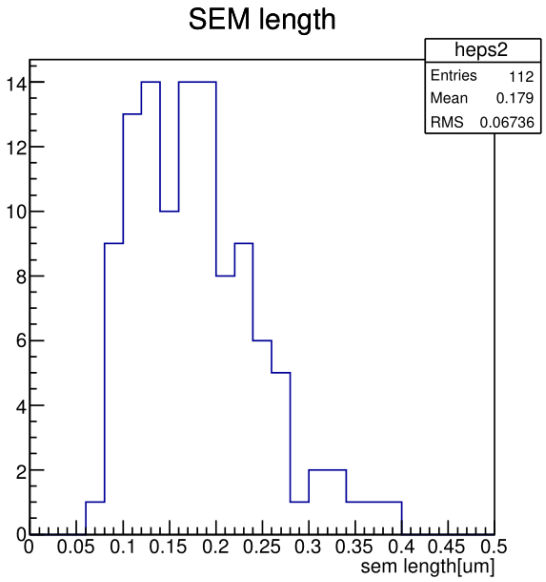
SRIM simulation

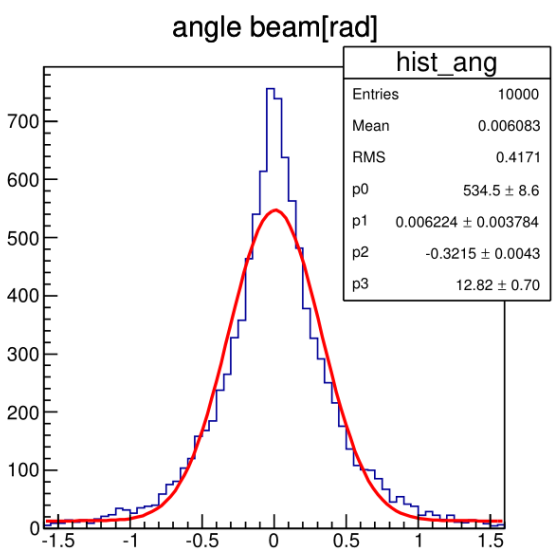
inside event

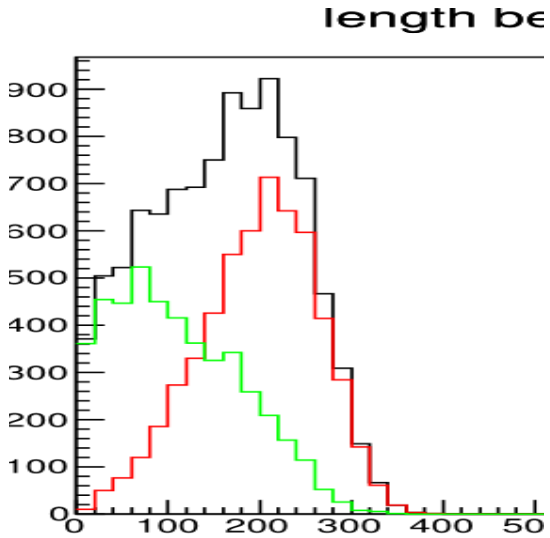
backscatter event

comparison to SRIM simulation (C 60 keV)







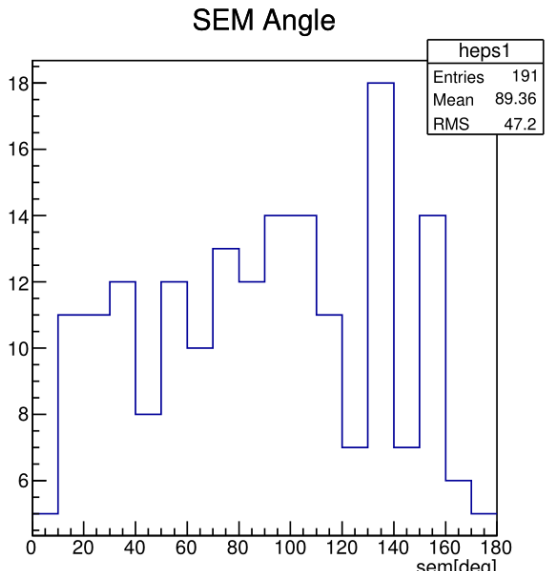


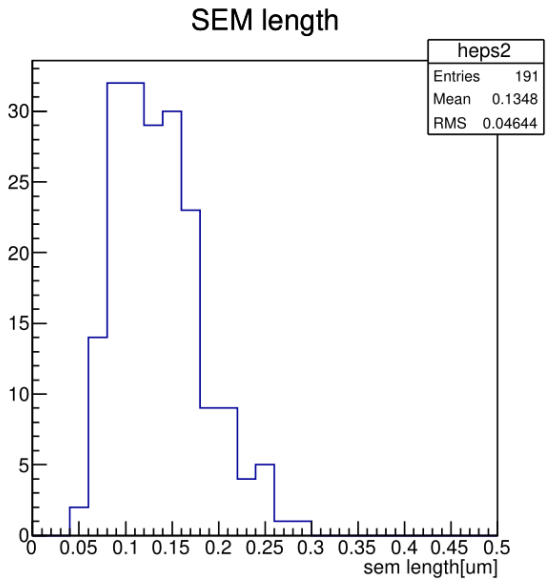
SRIM simulation

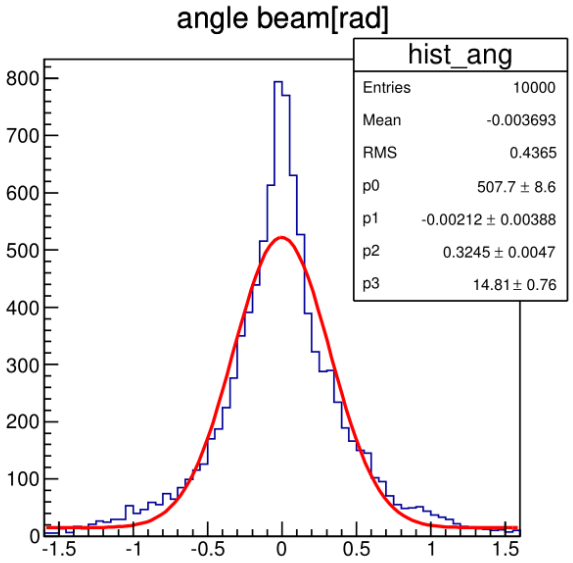
inside event

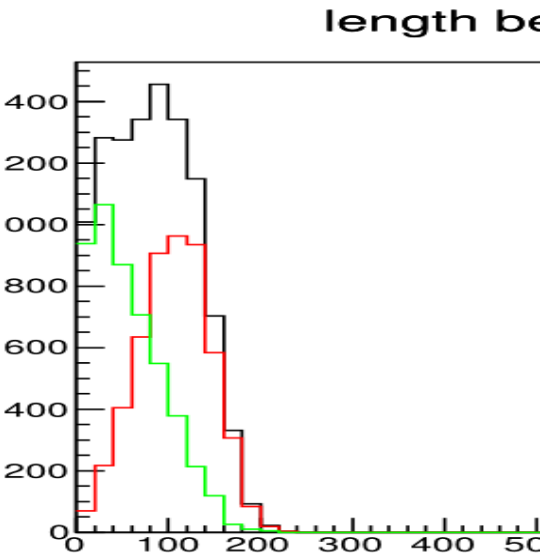
backscatter event

comparison to SRIM simulation (C 30 keV)







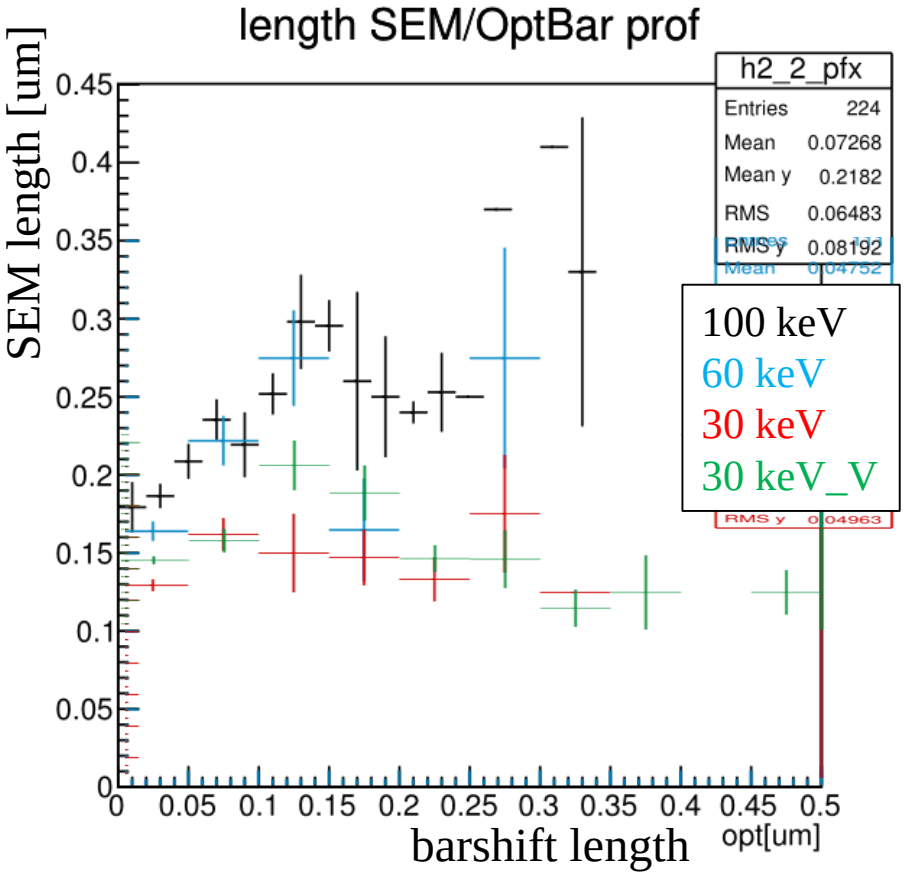


SRIM simulation

inside event

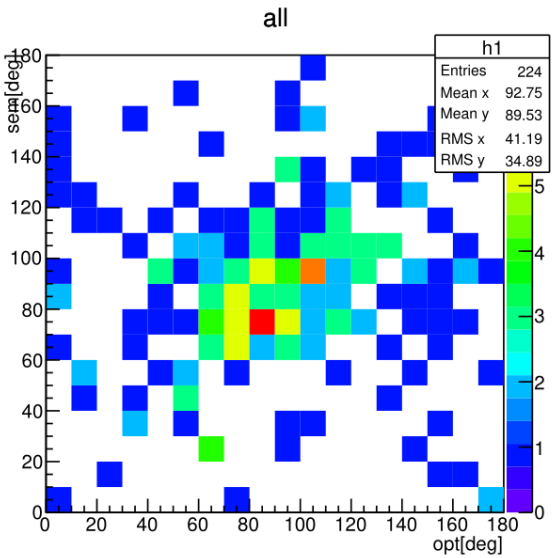
backscatter event

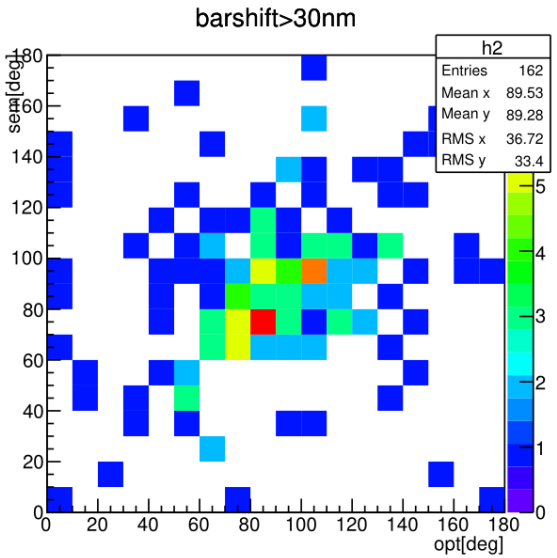
correlation between bar-shift and SEM measurement

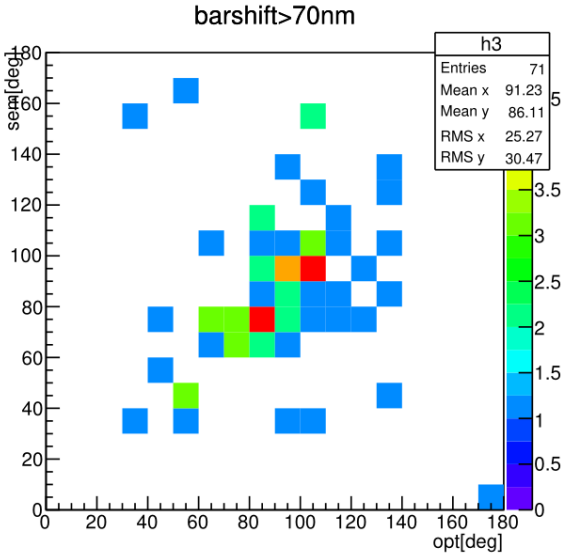


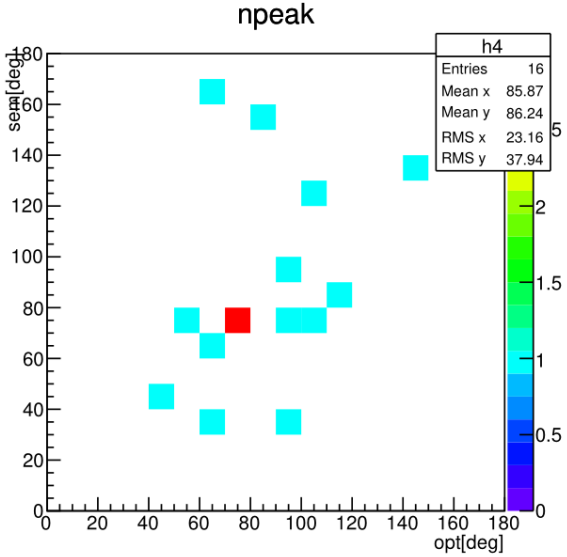
- longer tracks length than 150 nm is less correlated to bar-shift
- 30 keV (expected 1.6 grains) samples seem to have another behavior of bar-shift?

angle dependency of SEM / optical bar-phi









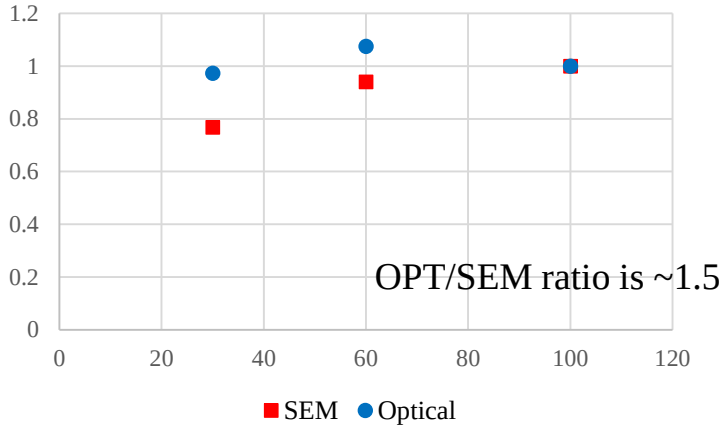
angles both have peak around 90 deg, but not linearly
Stronger bar-shift still not have good correlation to SEM

One possible reason is definition of SEM manual measurement (effect of filament tail)

contrast comparison

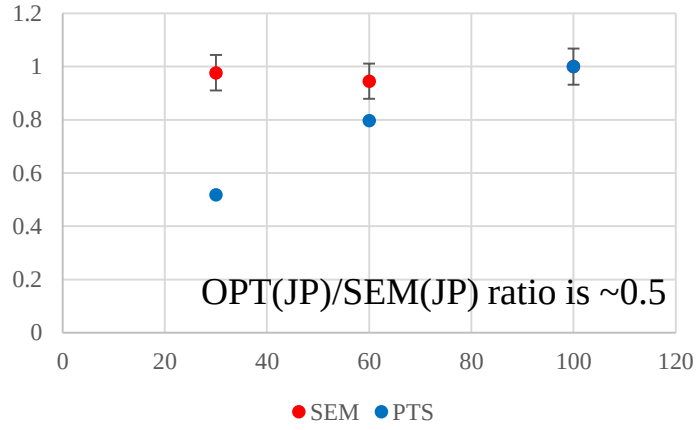
measurement in Naples

detected event density ratio
respect to 100 keV sample

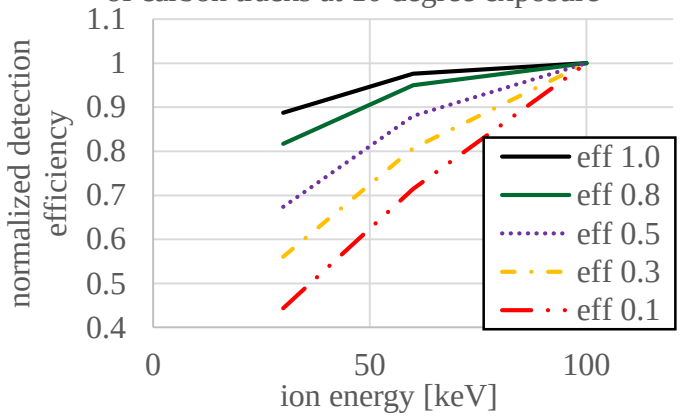


Old measurement in Japan-PTS

detected event density ratio
respect to 100 keV sample



expected detection efficiency
of carbon tracks at 10 degree exposure



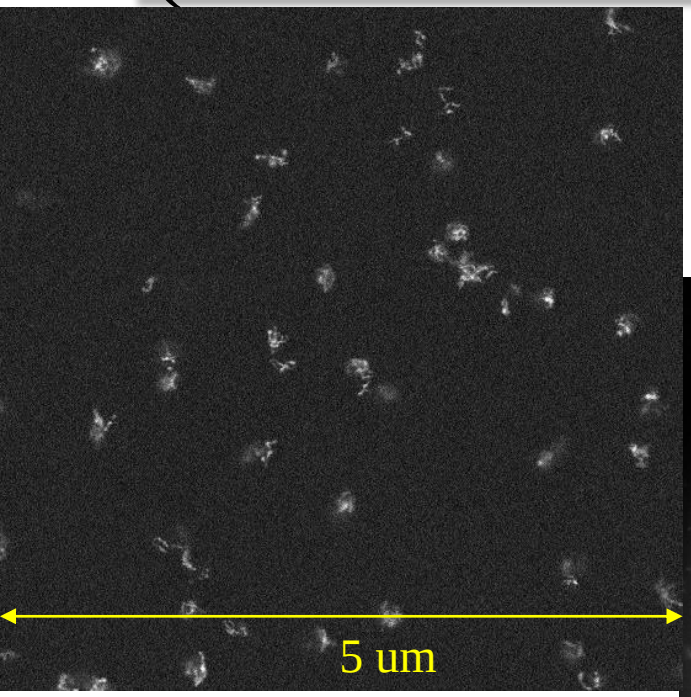
Absolute value is not good due to dust, cluster event, etc.
Japan has low contrast at lower energy tracks

P: detection efficiency of 1 crystal
$1-\Sigma(P^n)$: possibility of no crystal detected

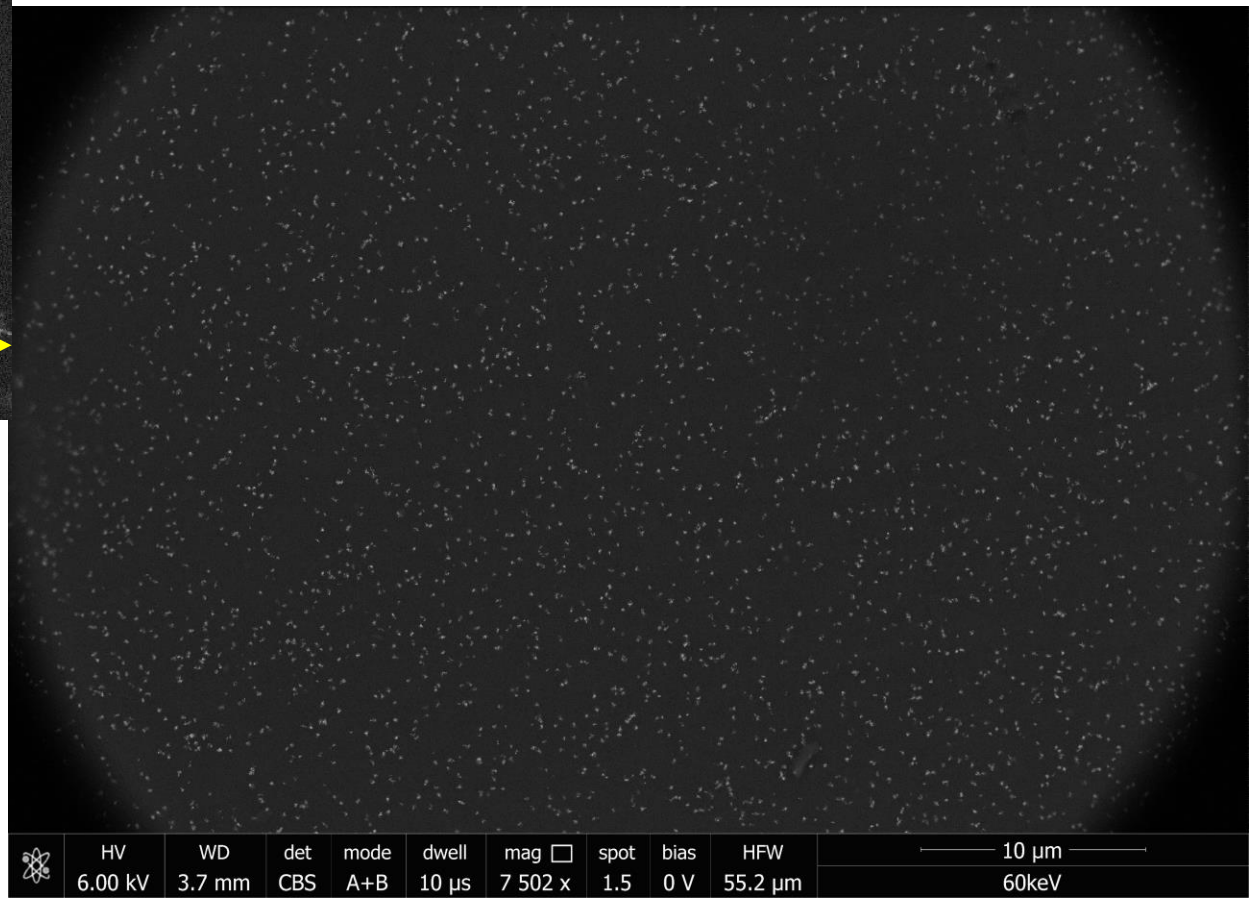
to be improved

- to measure and see the correspondence to optical image, Feret's Diameter is not good (filament tail looks too effective?)
- we should also measure other information (grain number, filament volume, etc.)
- measured samples has bad surface condition (it probably due to repeated scans) and cause problems on the kind of automatic analysis in the future
- comparison and connection to machine leaning
  - automatic image selection → image processing, pattern matching
  - new clean samples

Au/Pd Sputtering sample (C100 vertical, after soaking to oil)



very clean view
dusts of measuring samples should be depend on repeated scanning…



summary

- we performed 1 by 1 matching comparison between optical and SEM images
- Current analysis (manual check of length and angle) shows some behavior but not best method
- several update is needed to perform machine learning