

# FGT decapping summary

GeTe, thick Te, thin Te

Dante Prins QMI summer 2021

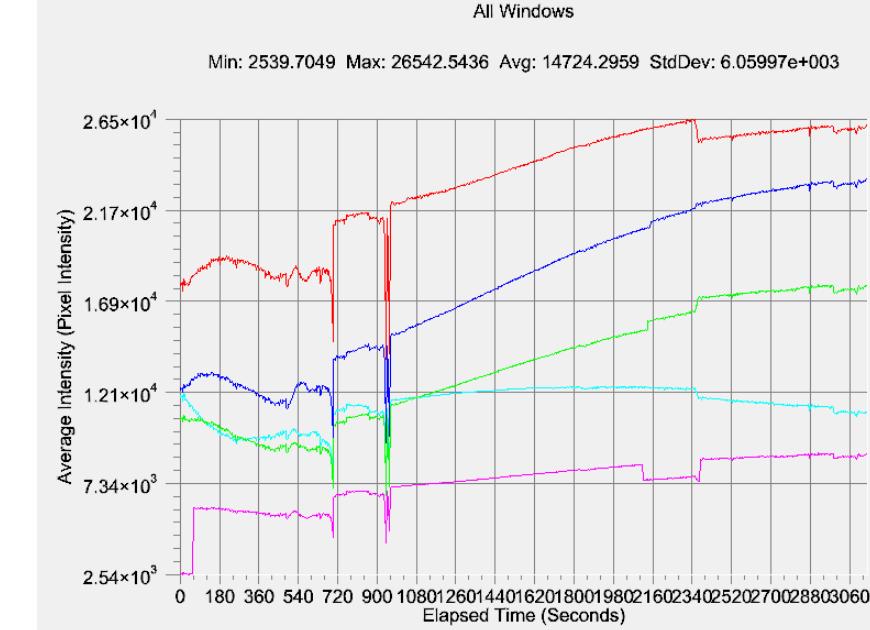
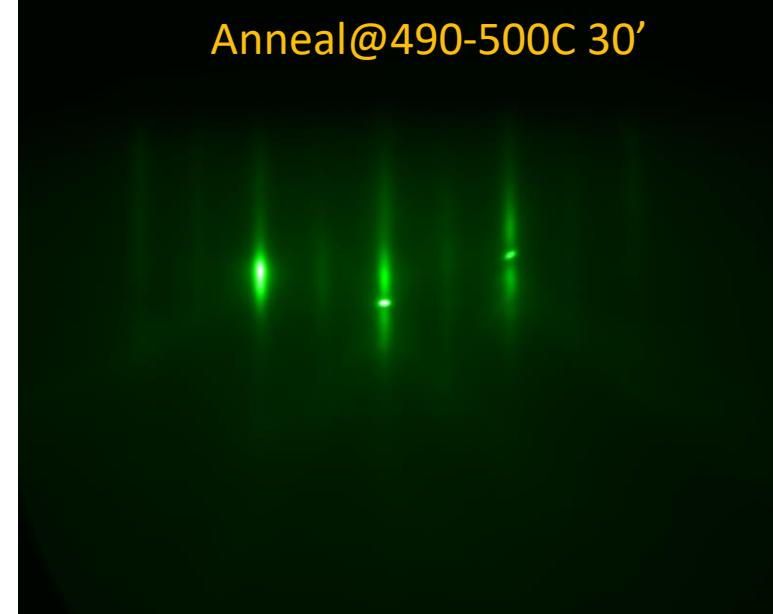
# Thick Te

507 and others later

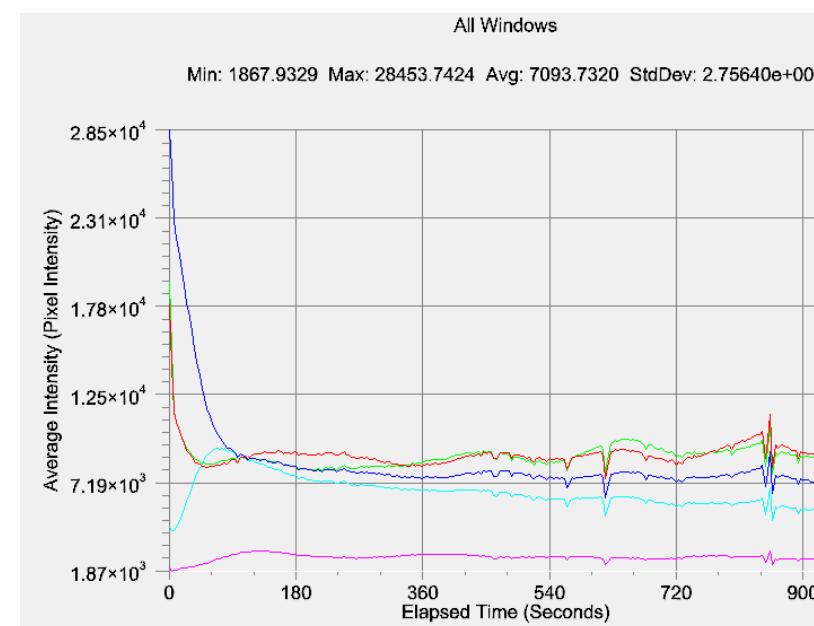
Conclusion: new phase is formed by reacting with FGT as FGT cannot exist above 360.

Ge-507 FGT + Te capping

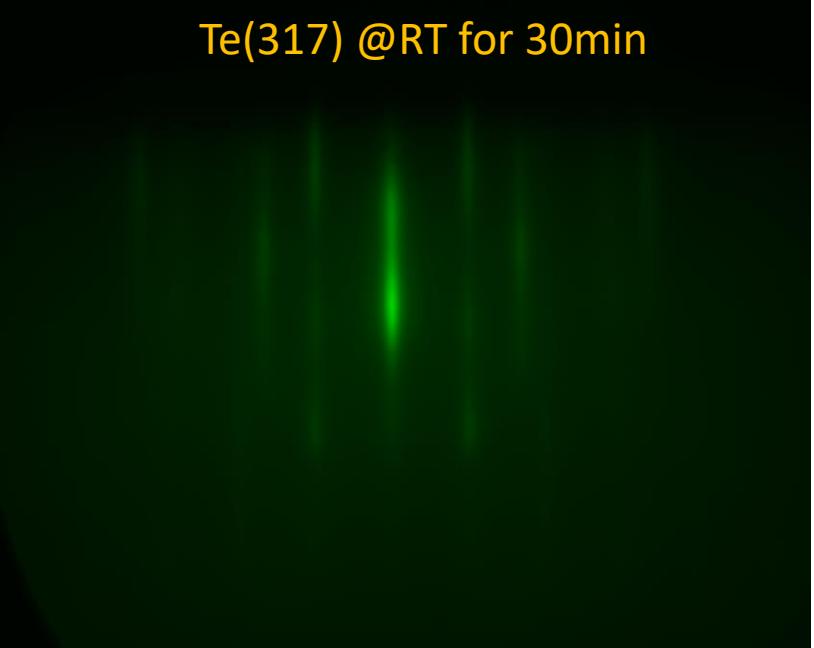
Ge-0507(NOVA) DI 10'+HCl 20'  
+Te@400



+ Ge(1090) @500 for 48'



Te(317) @RT for 30min



Decap @330



Decap @360 1hr



# GeTe

510 521a 521b 527b 527a 607b 607a

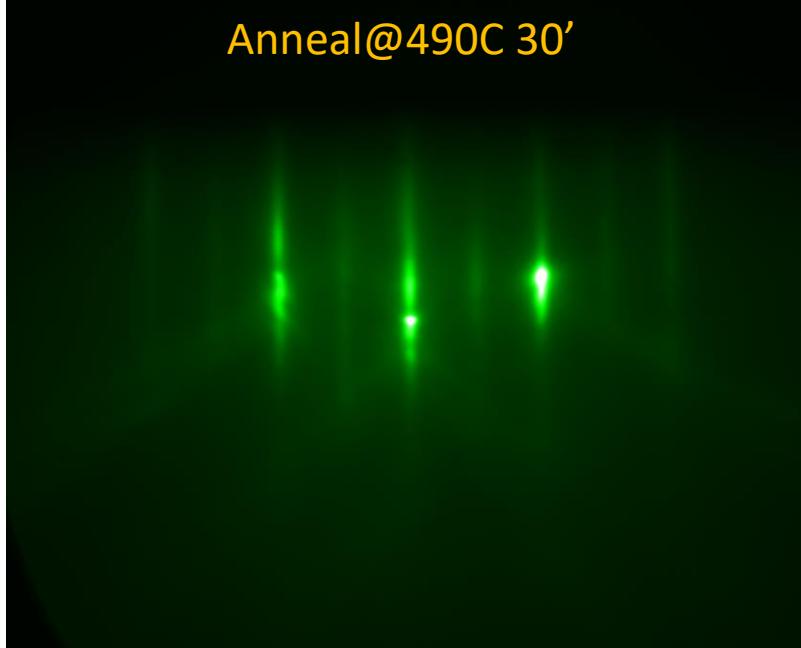
Conclusion: GeTe cannot be removed under 360C

Ge-510 FGT+GeTe cap decapped to 360

Ge-0510 (MTI old) DI 10'+HCl 20'  
@400



Anneal@490C 30'

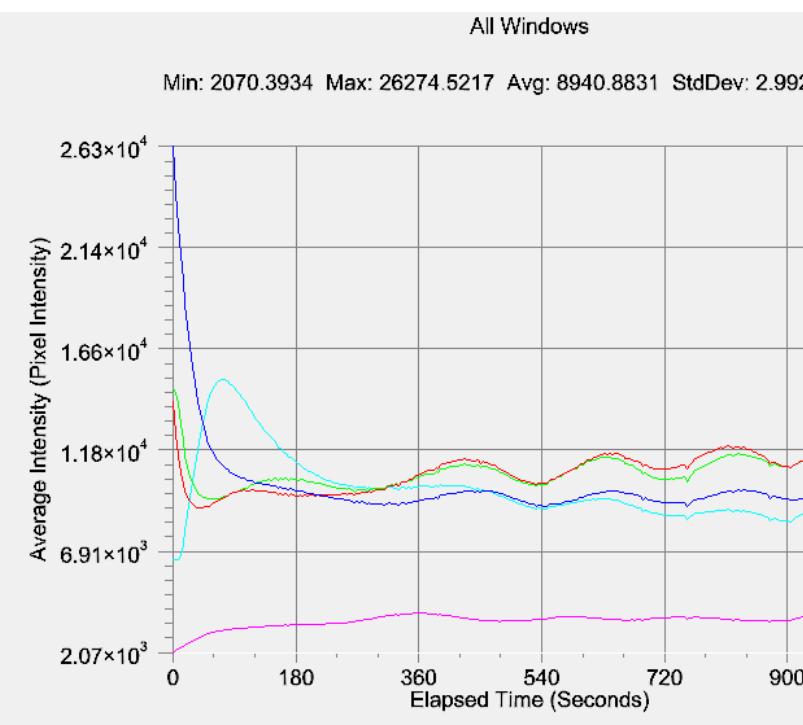


+ Ge(1090) @490 for 13.5'



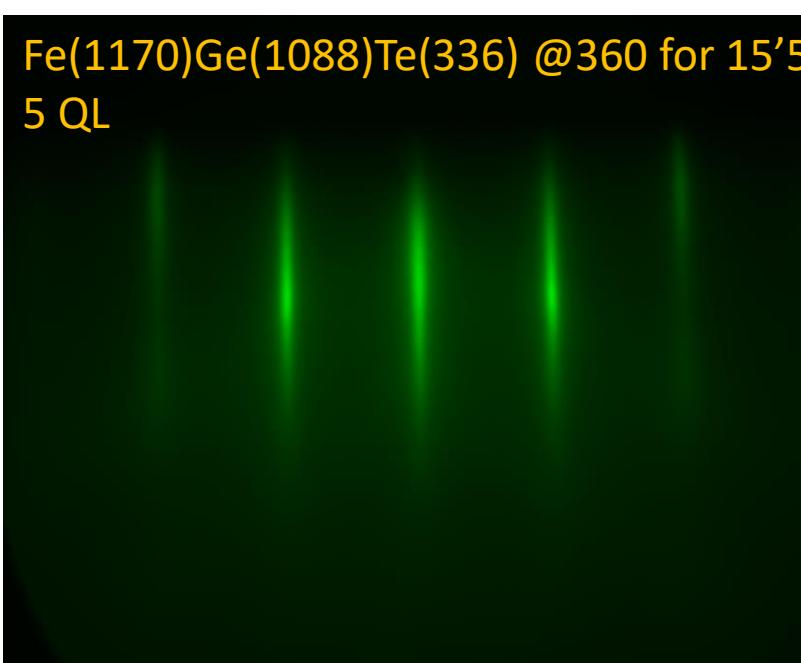
All Windows

Min: 2070.3934 Max: 26274.5217 Avg: 8940.8831 StdDev: 2.9921



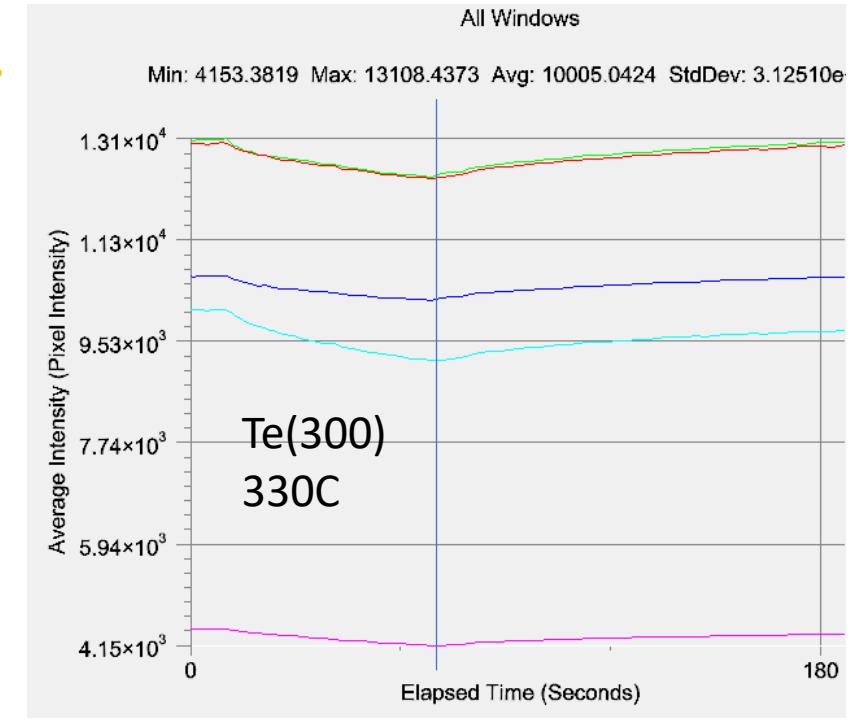
Fe(1170)Ge(1088)Te(336) @360 for 15'5'

5 QL



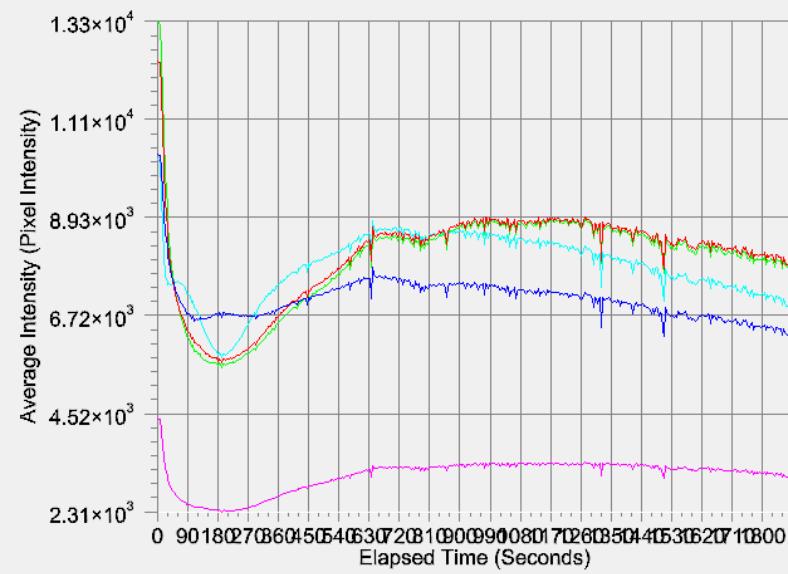
All Windows

Min: 4153.3819 Max: 13108.4373 Avg: 10005.0424 StdDev: 3.1251e

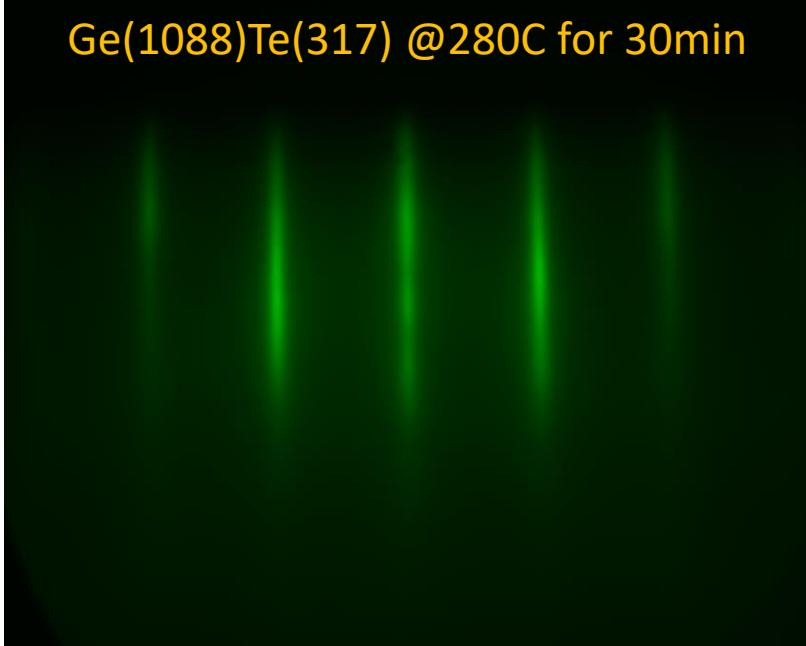


All Windows

Min: 2308.1511 Max: 13349.0102 Avg: 6804.5731 StdDev: 2.03695e+



Ge(1088)Te(317) @280C for 30min

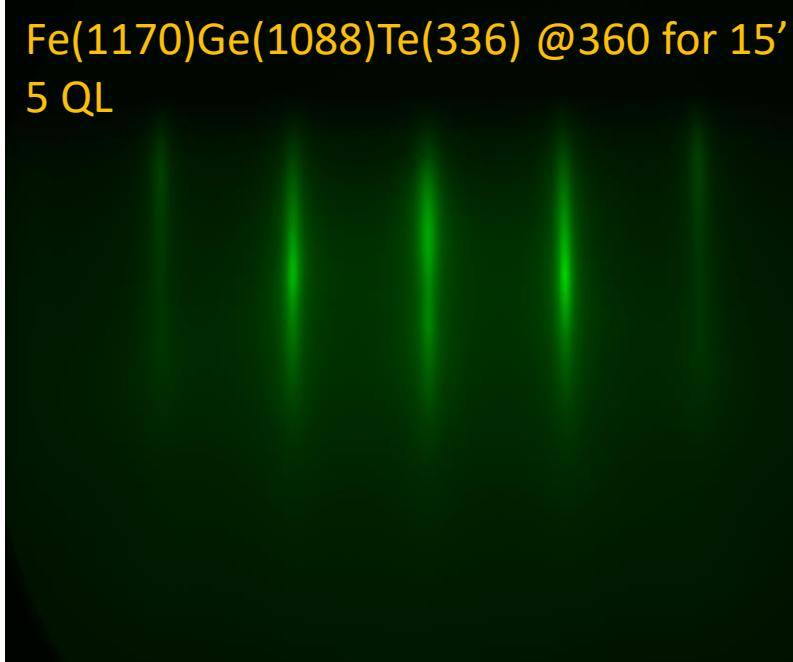
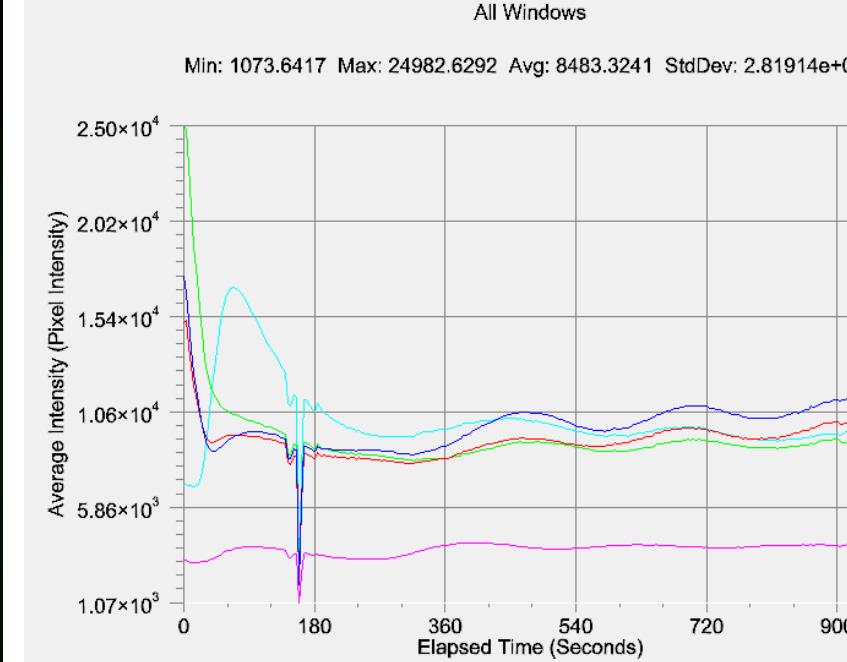


Decap @360 40'

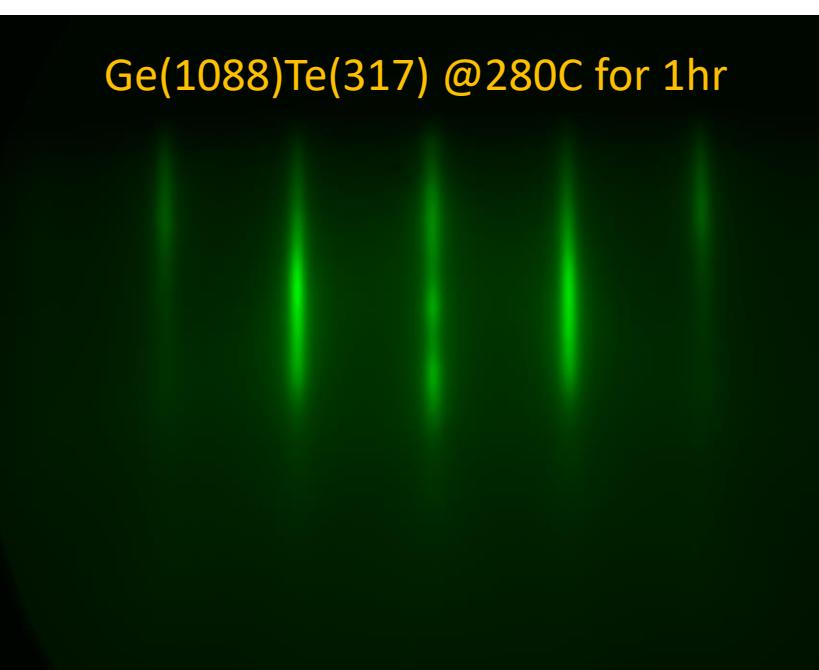


Ge-521a FGT + GeTe cap exposed to air  
decapped to 360

Ge-0521a (Nova) DI 10'+HCl 20'  
@490 40' + Ge

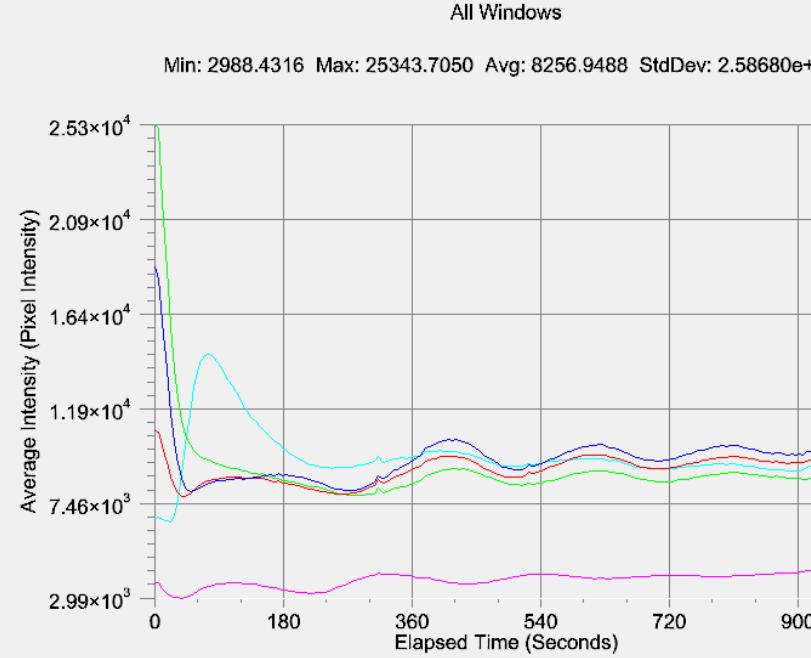


Ge(1088)Te(317) @280C for 1hr



Ge-521b FGT+GeTe + Se cap exposed to air  
decapped to 360

Ge-0521b (Nova) DI 10'+HCl 20'  
@490 40' + Ge



Fe(1170)Ge(1088)Te(336) @360 for 15'  
5 QL



Ge(1088)Te(317) @280C for 1hr



Ge-527a FGT+GeTe +Te + Se cap decapped to  
360

Ge-0527a (Nova) DI 10'+HCl 45'  
@490 30' + Ge

Fe(1170)Ge(1088)Te(336) @360 for 15'  
5 QL

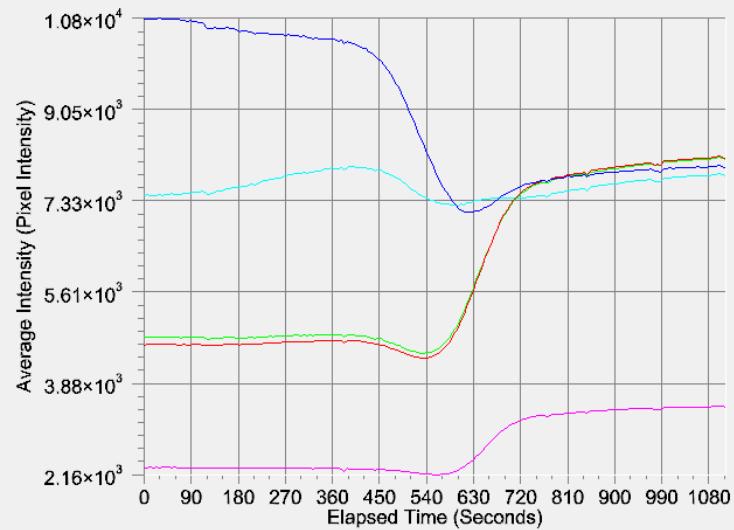
Ge(1089)Te(317) @280C for 1hr

+Te+Se, in air 20min

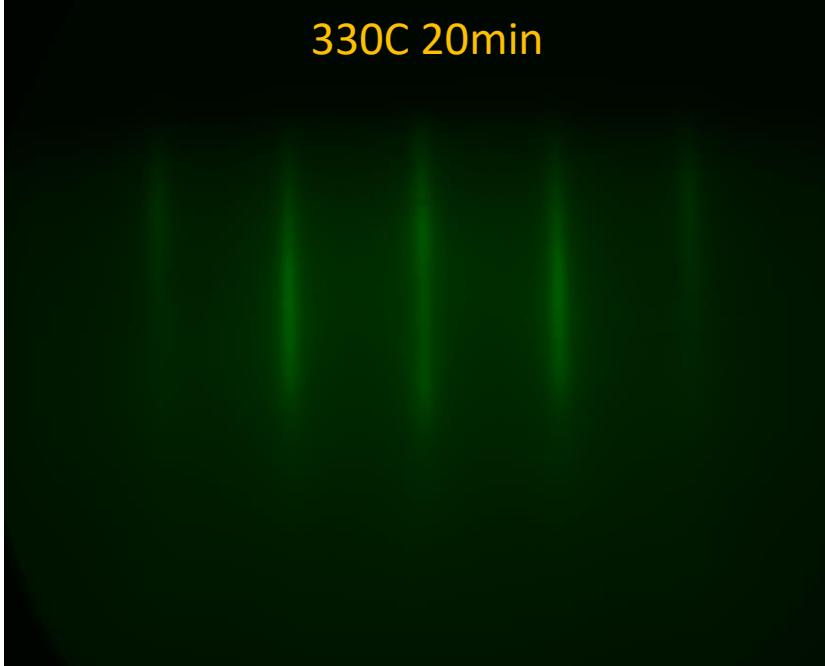
330C

All Windows

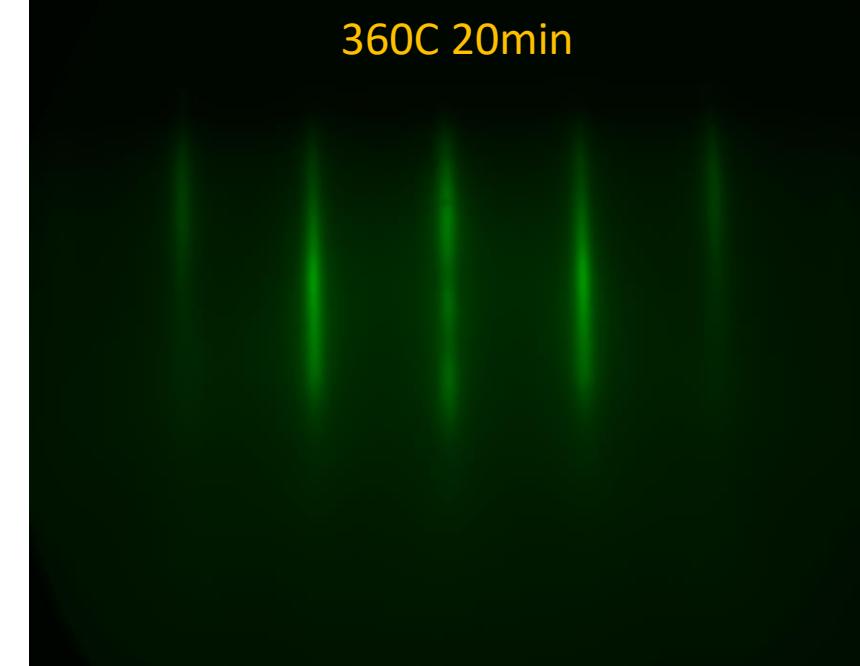
Min: 2162.4502 Max: 10773.1448 Avg: 6258.5938 StdDev: 2.40323e+0

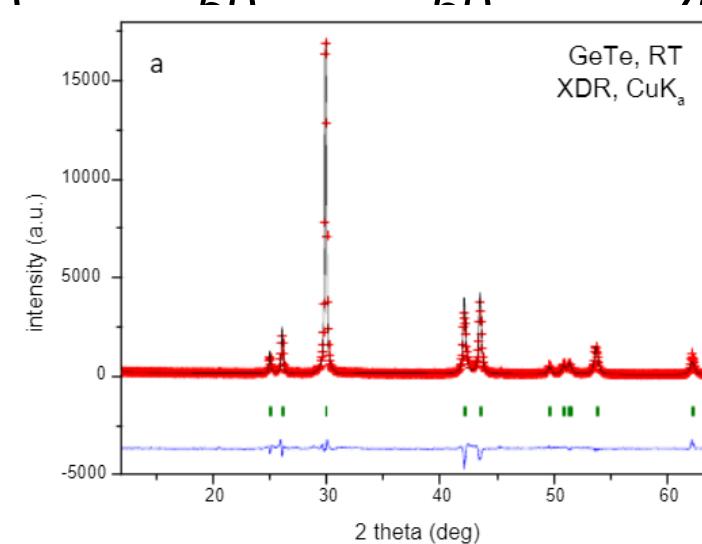
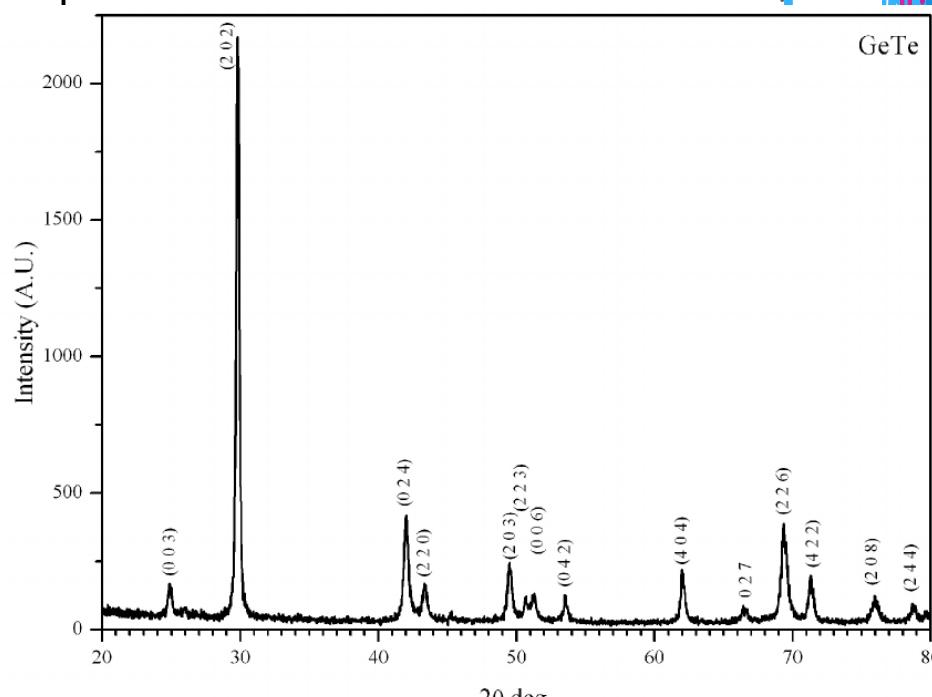
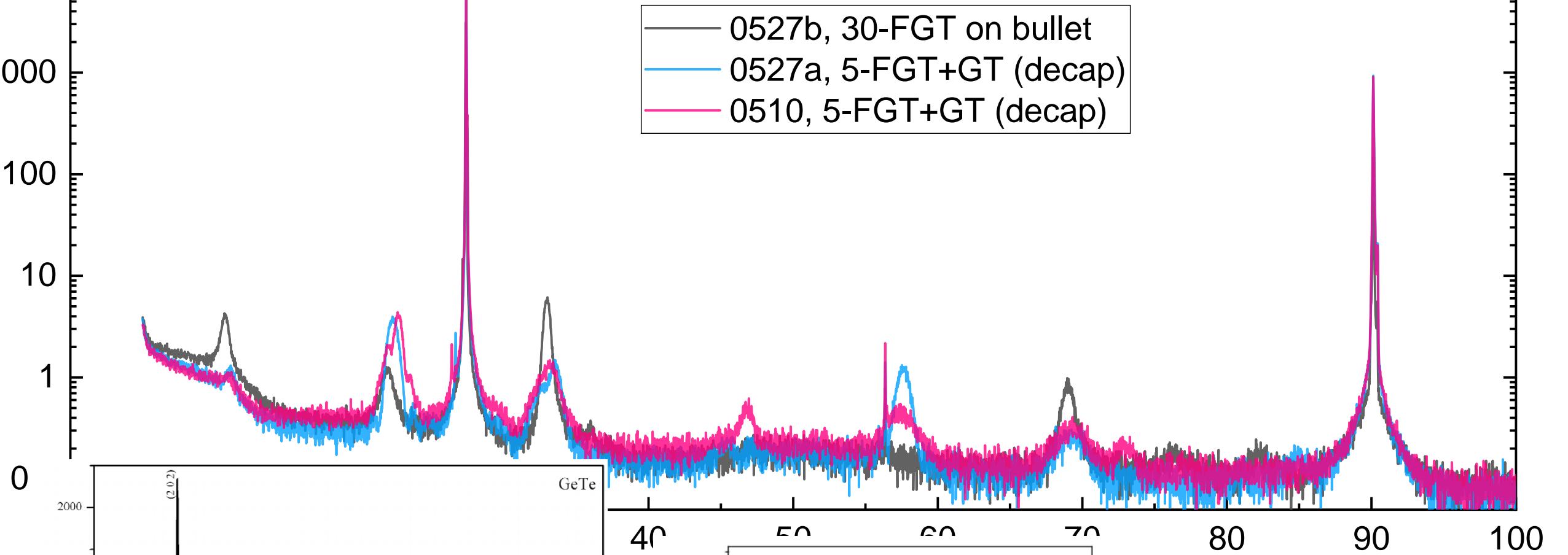


330C 20min



360C 20min





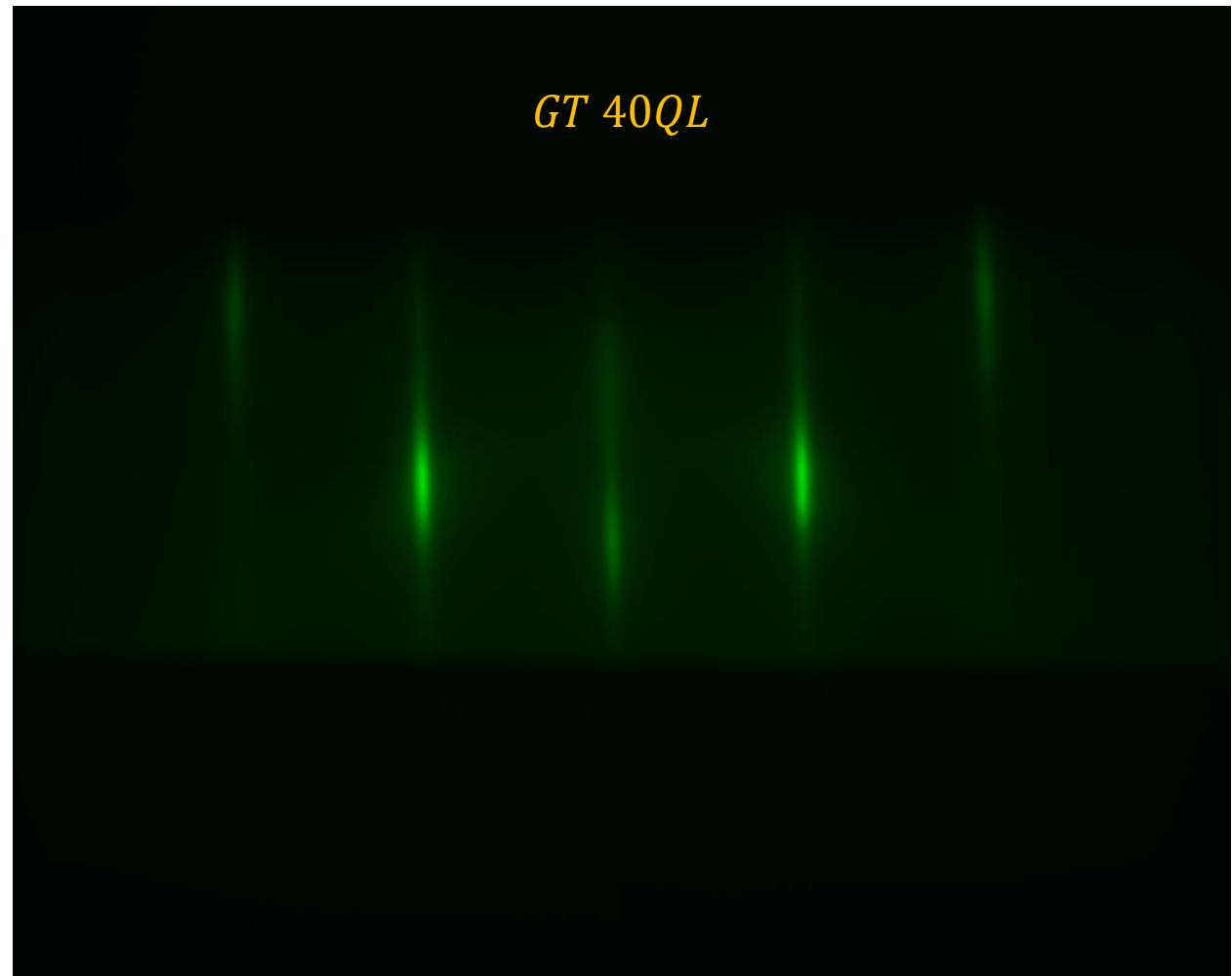
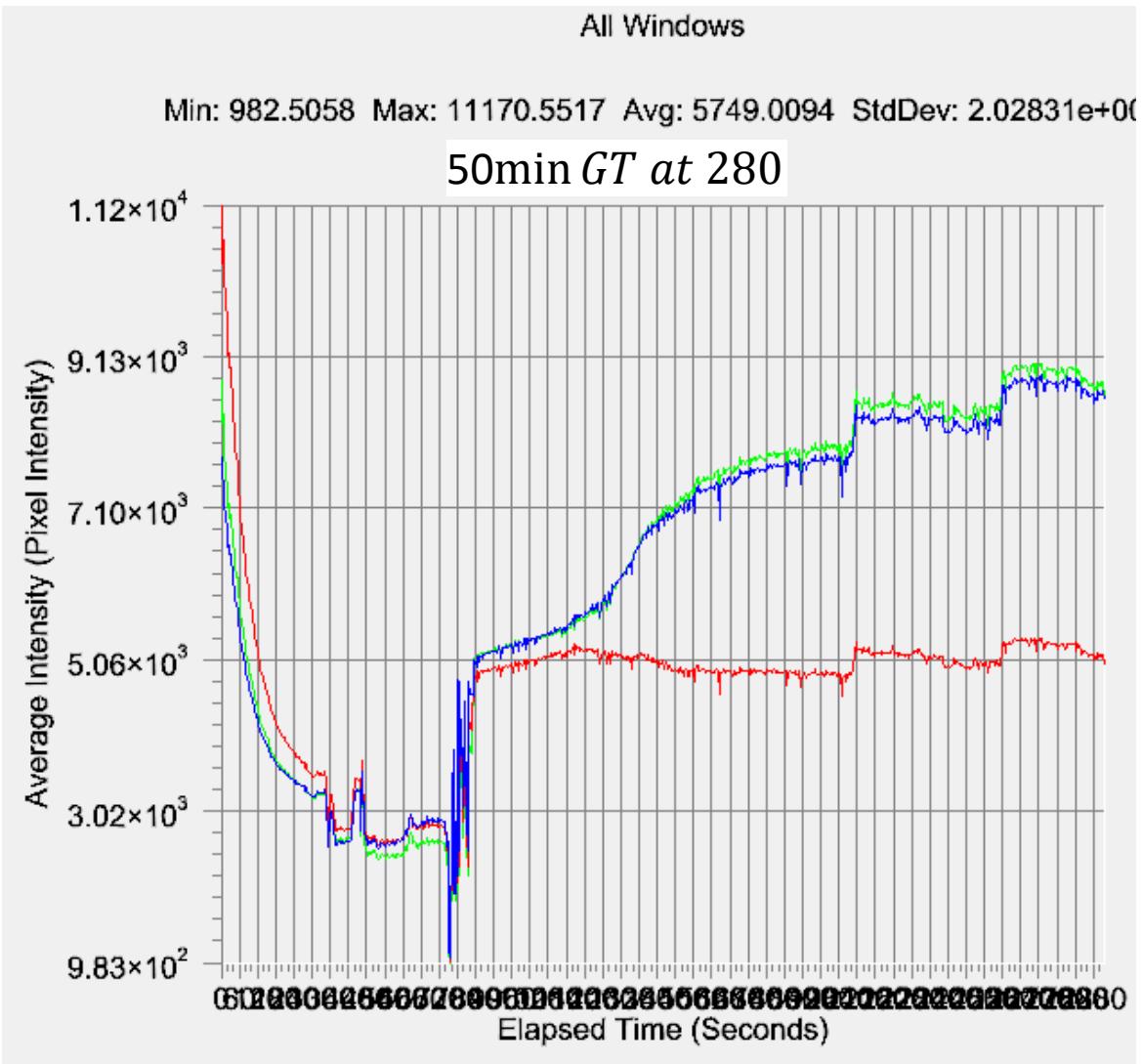
Ge-607b 40L GeTe (no fgt)

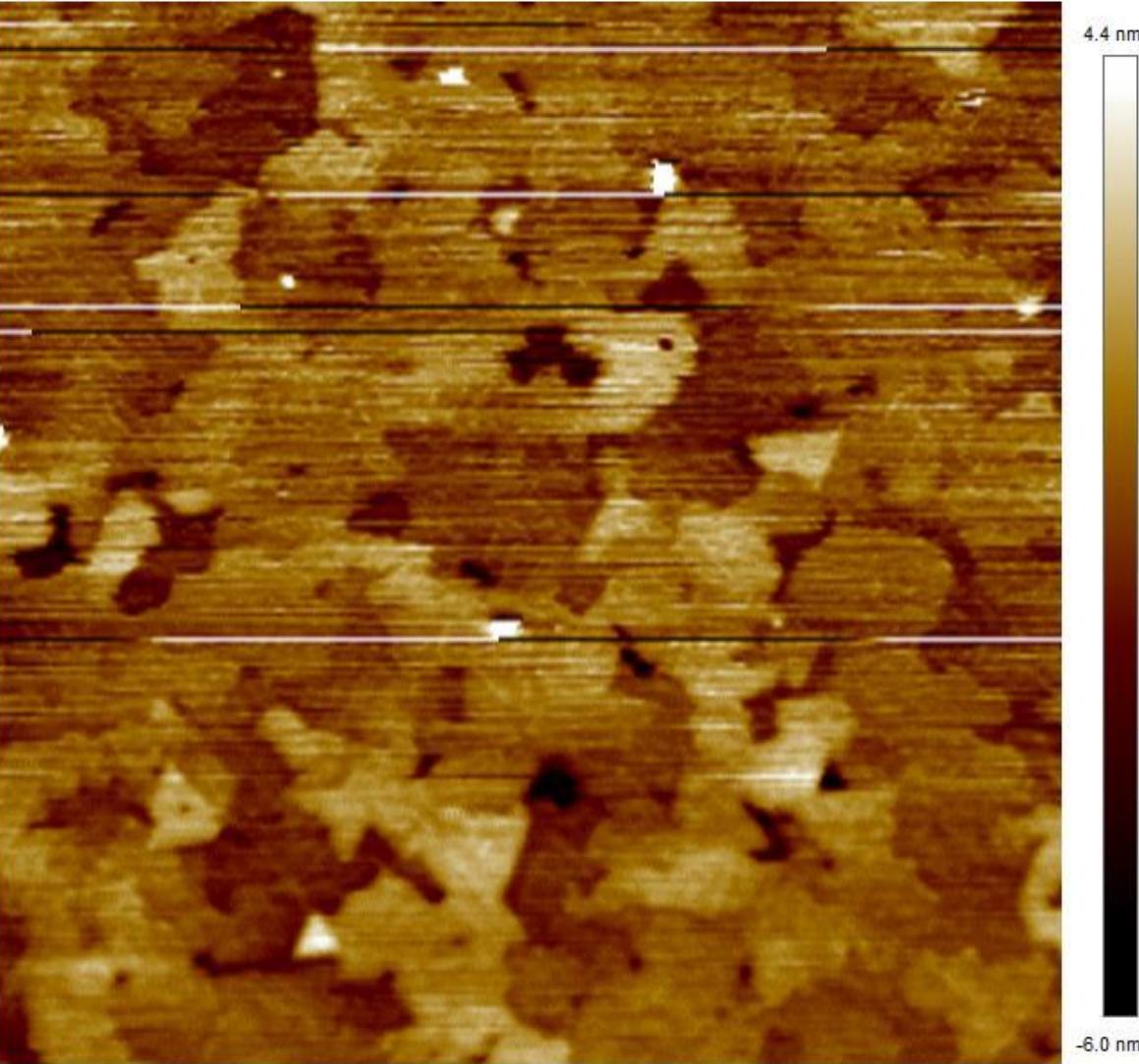
*at 490*

*anneal 30 min*

*+Ge [11 – 2]*

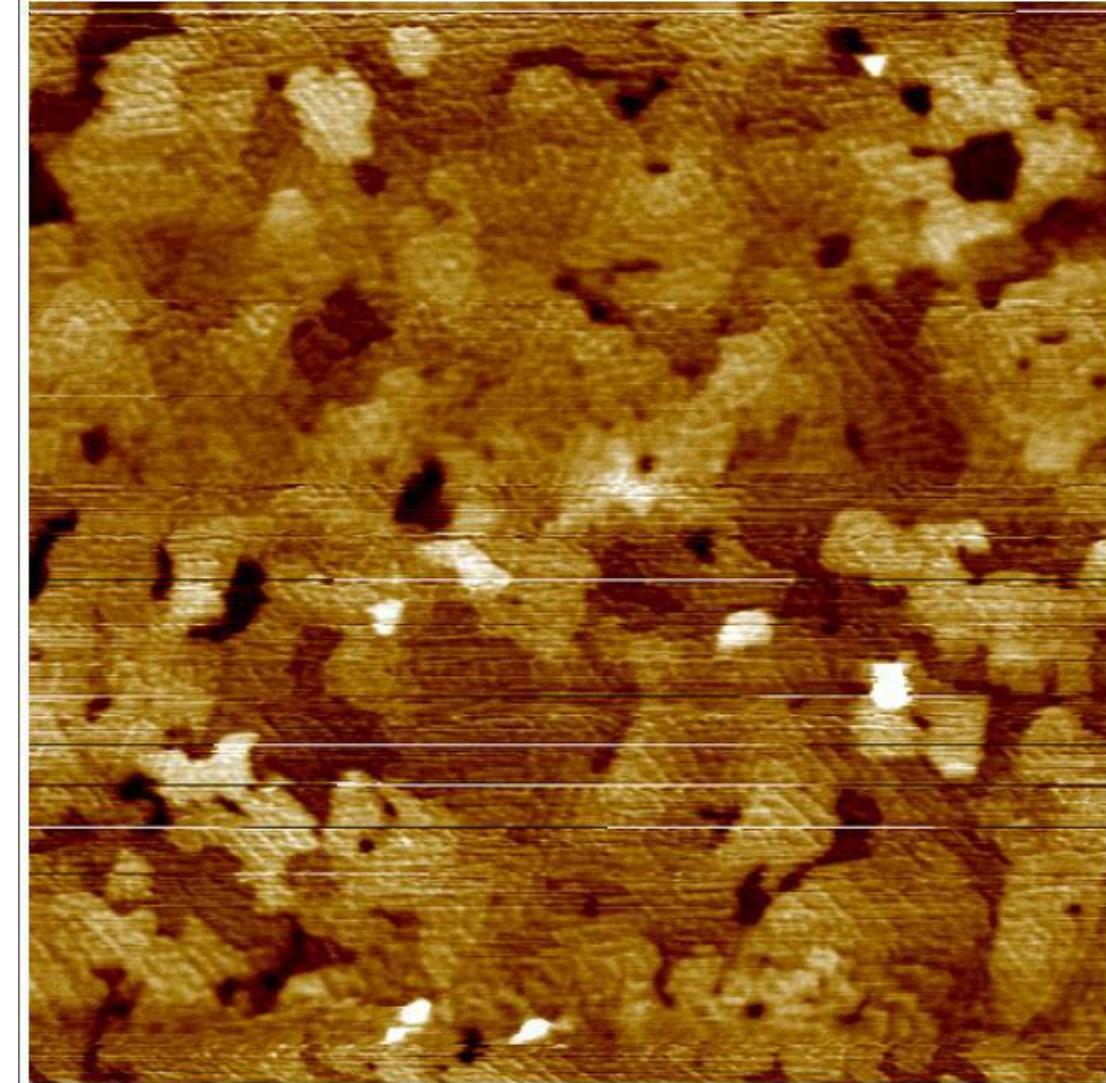
*+Ge [10 – 1]*





0.0

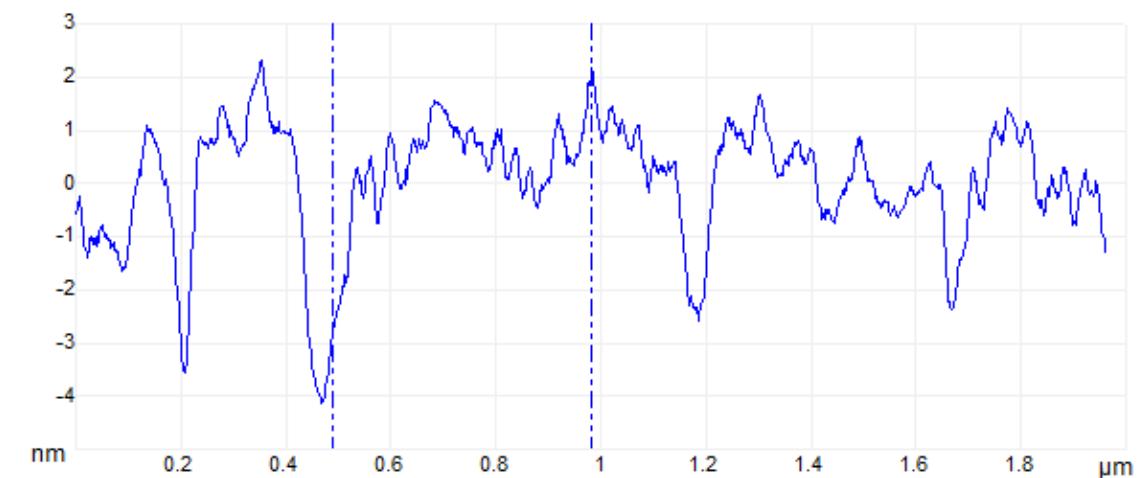
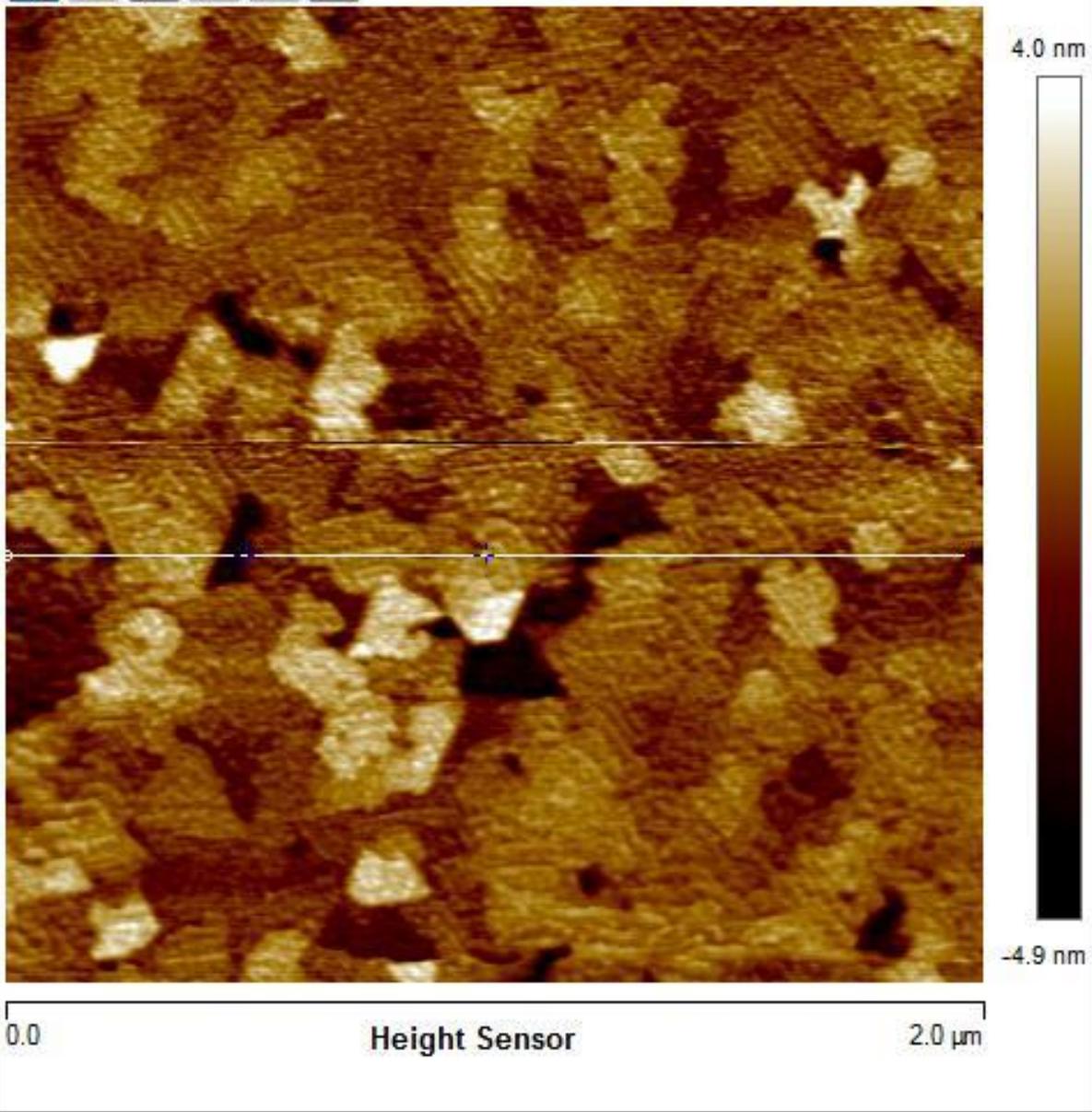
Height Sensor

2.0  $\mu\text{m}$ 

0.0

Height Sensor

2.0  $\mu\text{m}$ 



C=10.7A

Intensity (A.U.)

10000  
1000  
100  
10  
1  
0.1  
0.01

Intensity

Ge(111)

Ge(222)

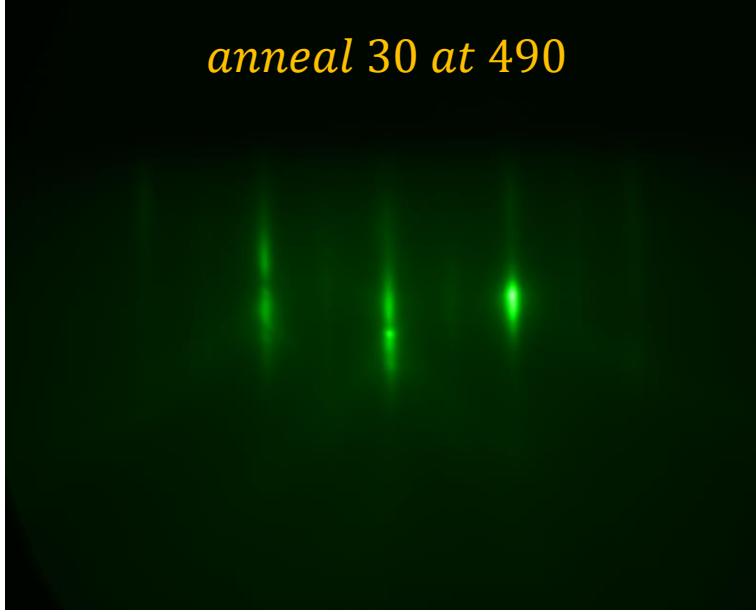
Ge(333)

0 20 40 60 80 100

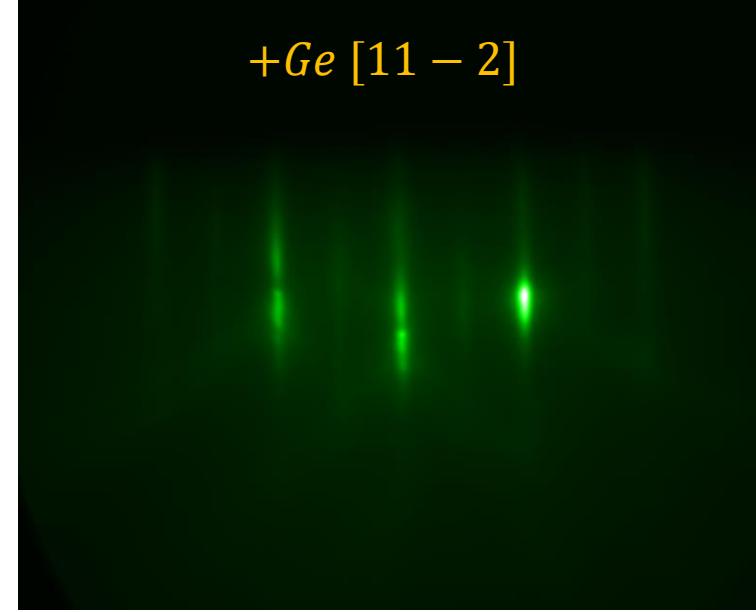
Angle  $2\theta$  (Theta)

Ge-607a GeTe decapped at 360 (no fgt)

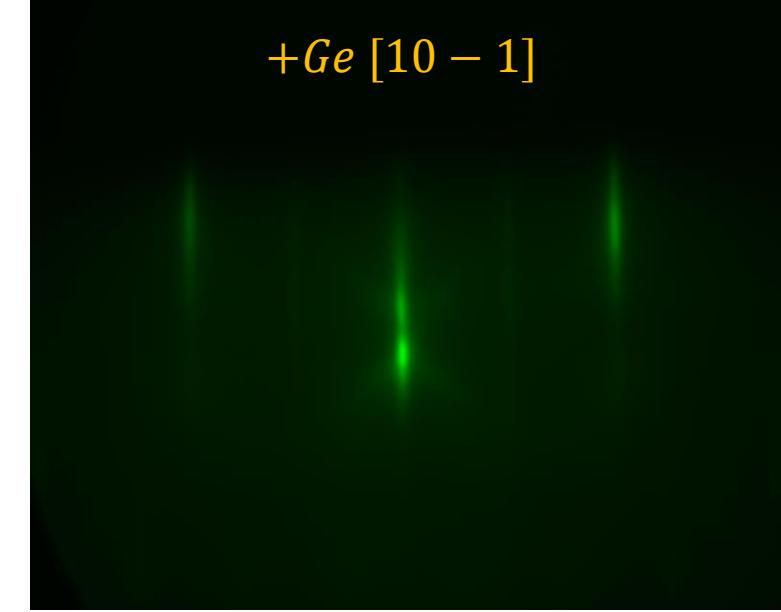
*anneal 30 at 490*



$+Ge\ [11 - 2]$



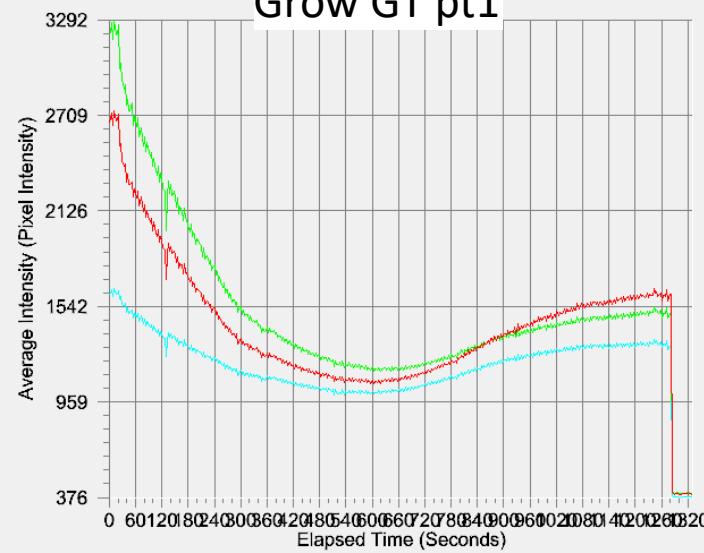
$+Ge\ [10 - 1]$



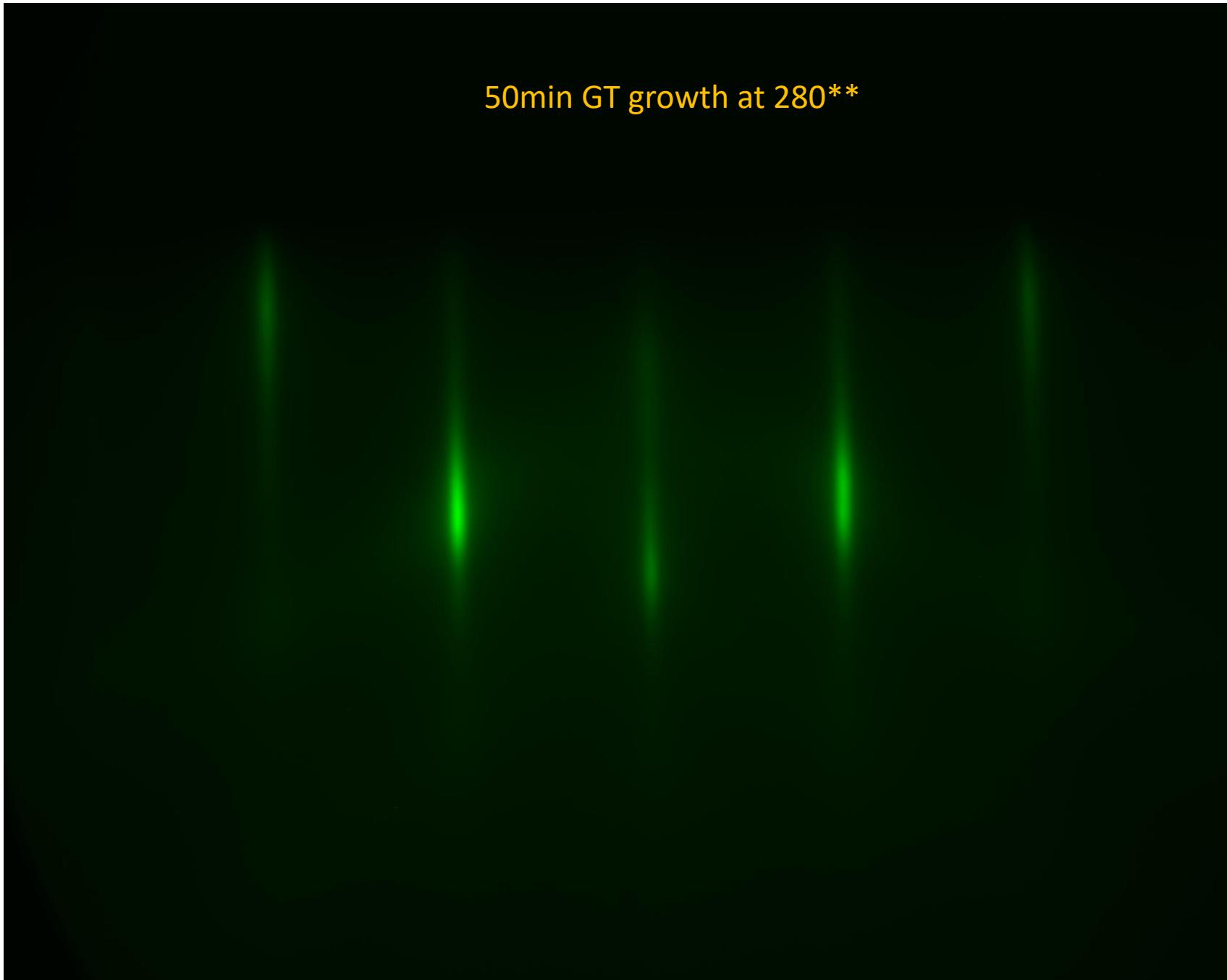
All Windows

Min: 376.0927 Max: 3291.7976 Avg: 1361.9409 StdDev: 4.14320e+002

### Grow GT pt1



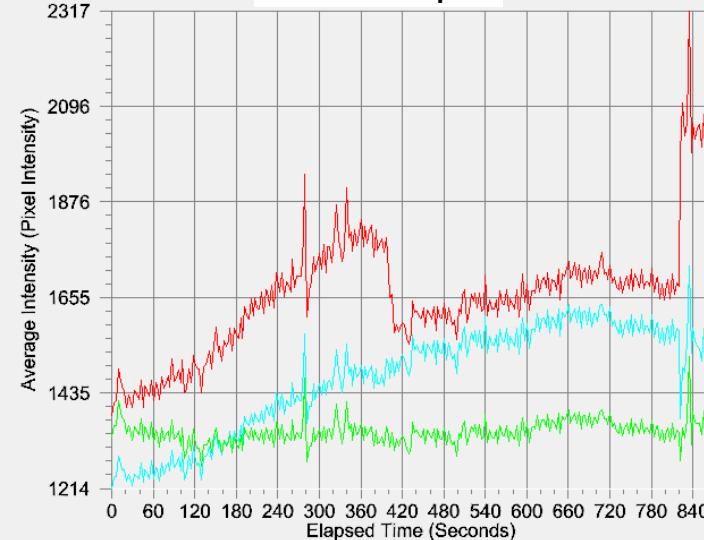
50min GT growth at 280\*\*



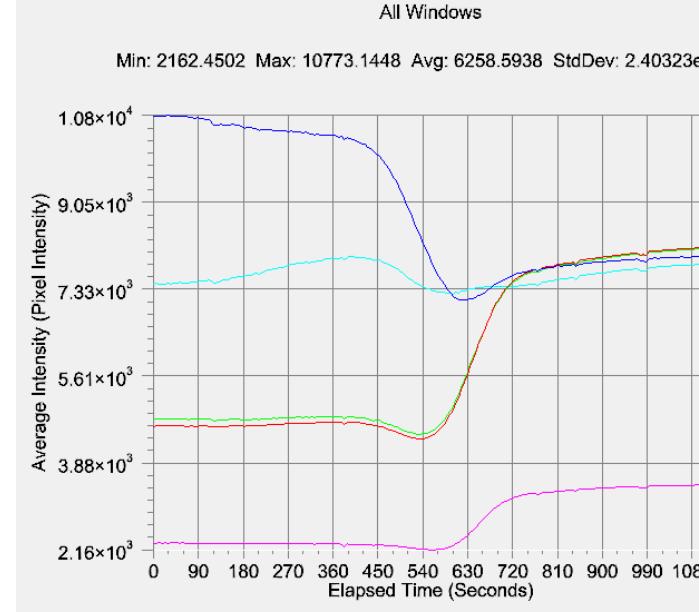
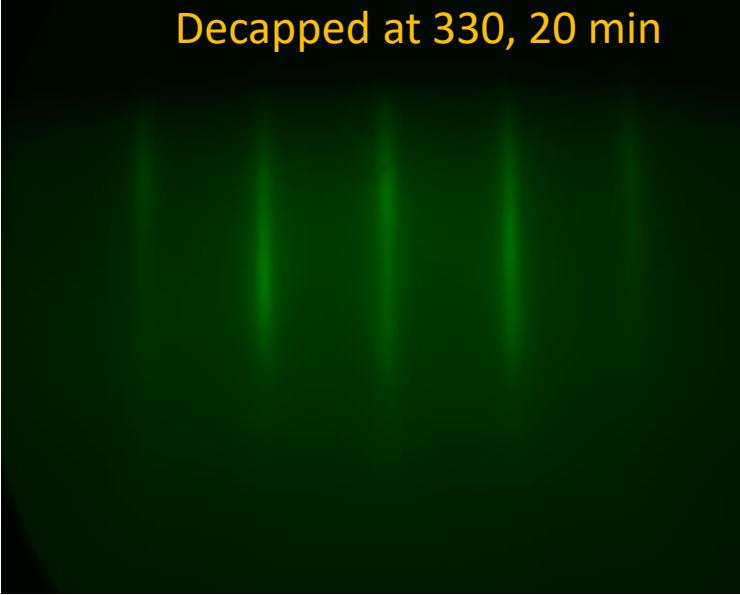
All Windows

Min: 1214.0491 Max: 2316.8479 Avg: 1424.8416 StdDev: 1.69408e+000

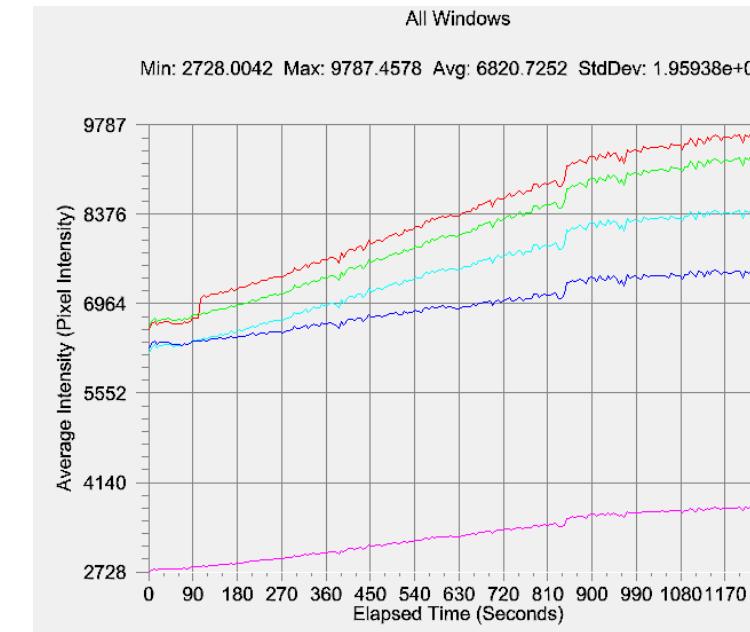
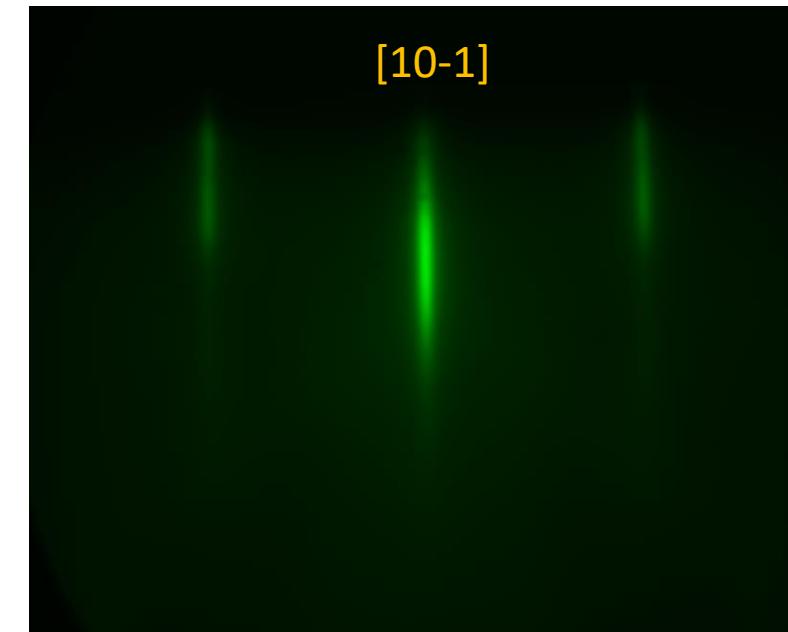
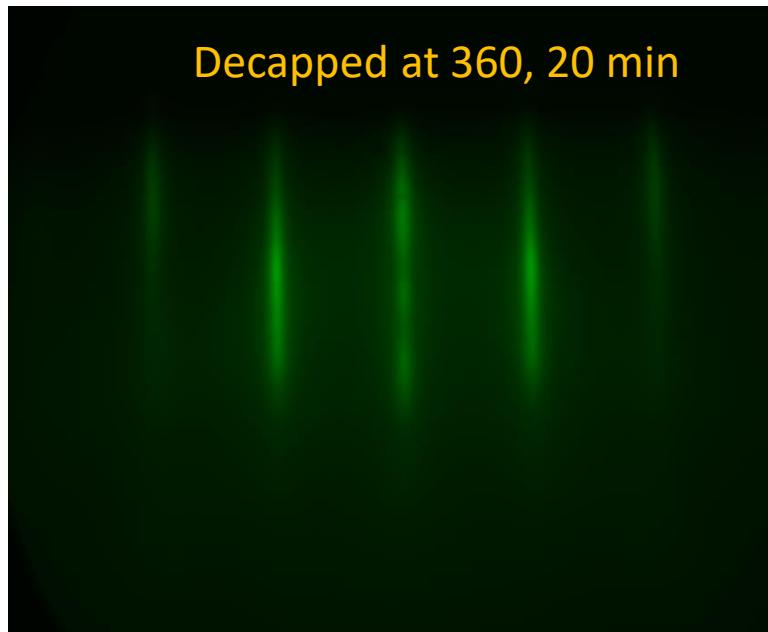
### Grow GT pt2

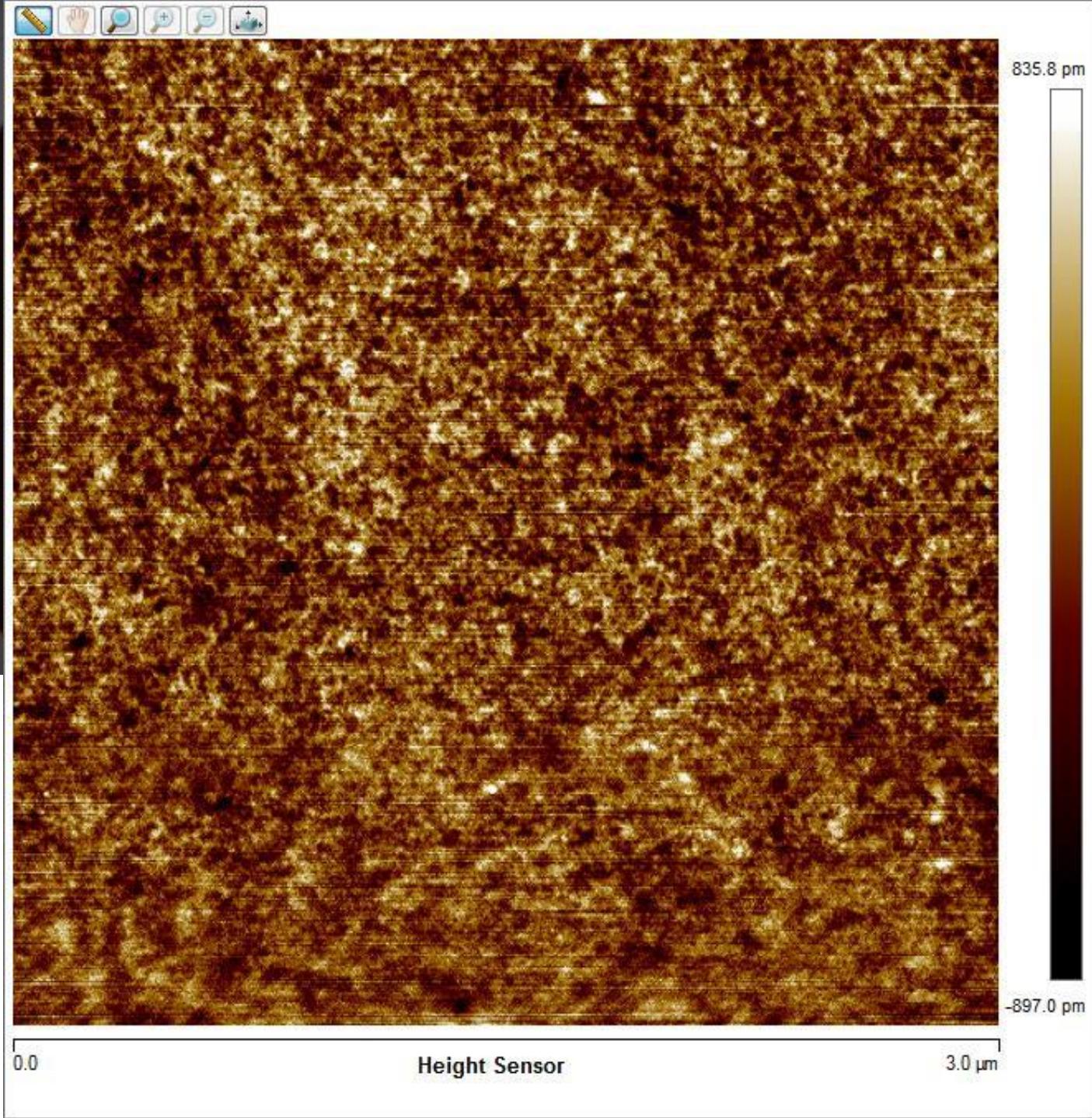
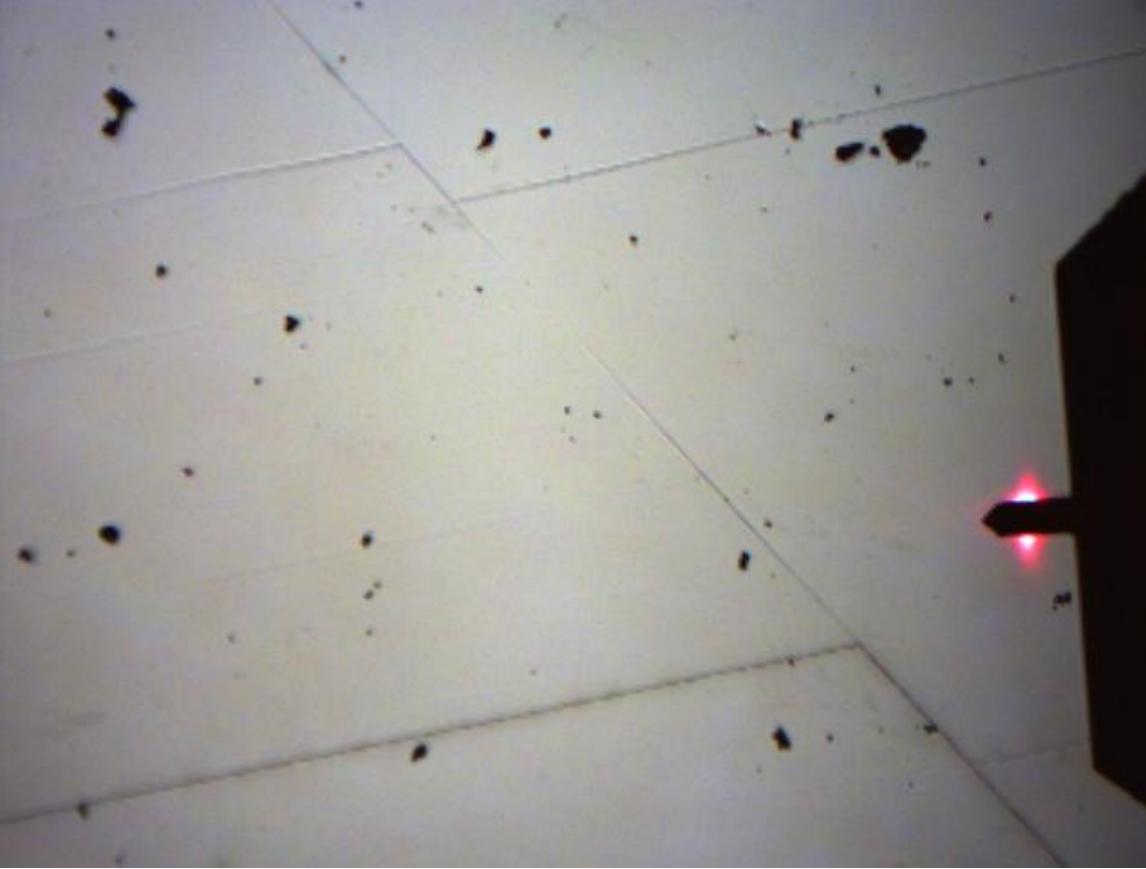


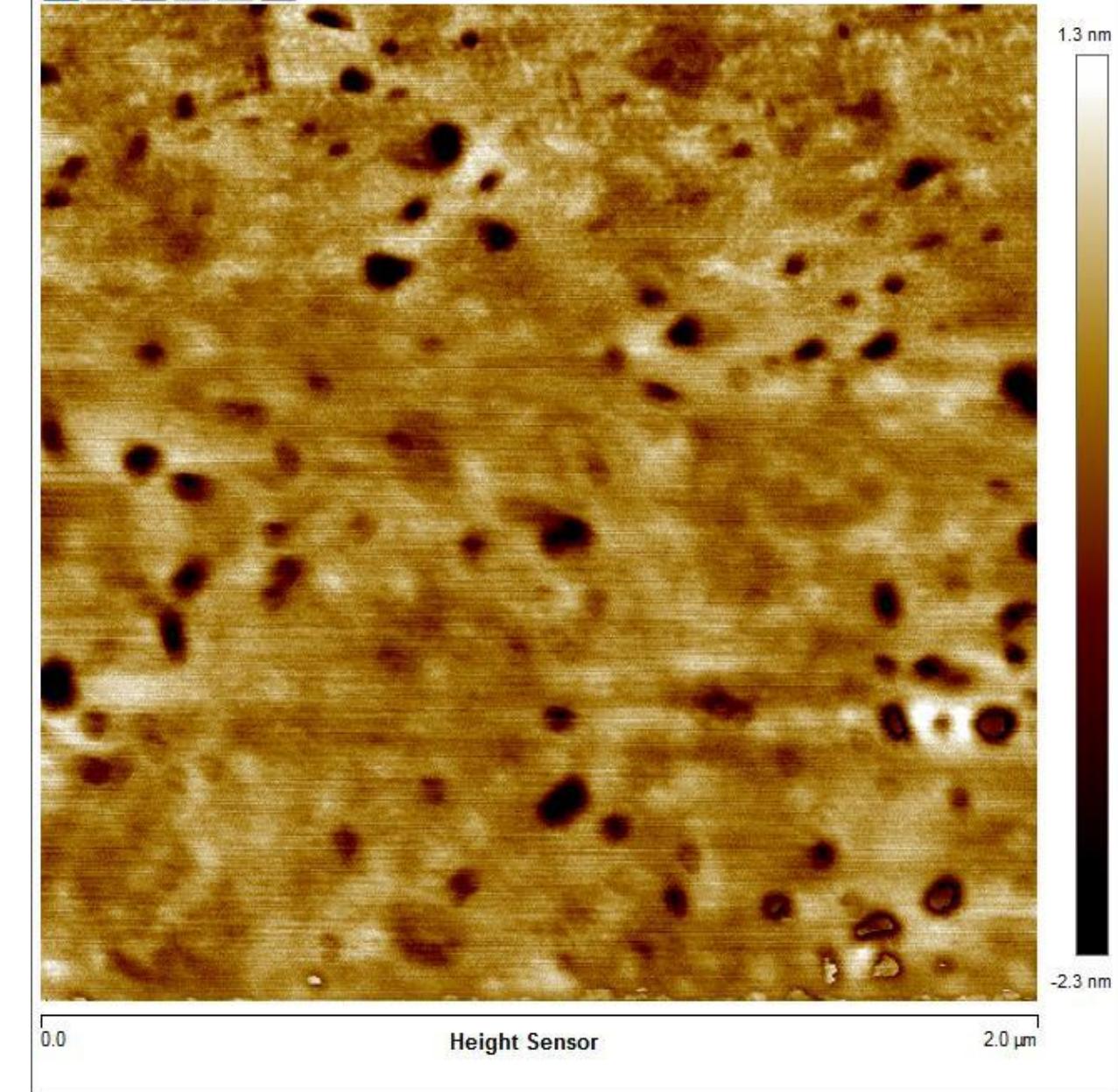
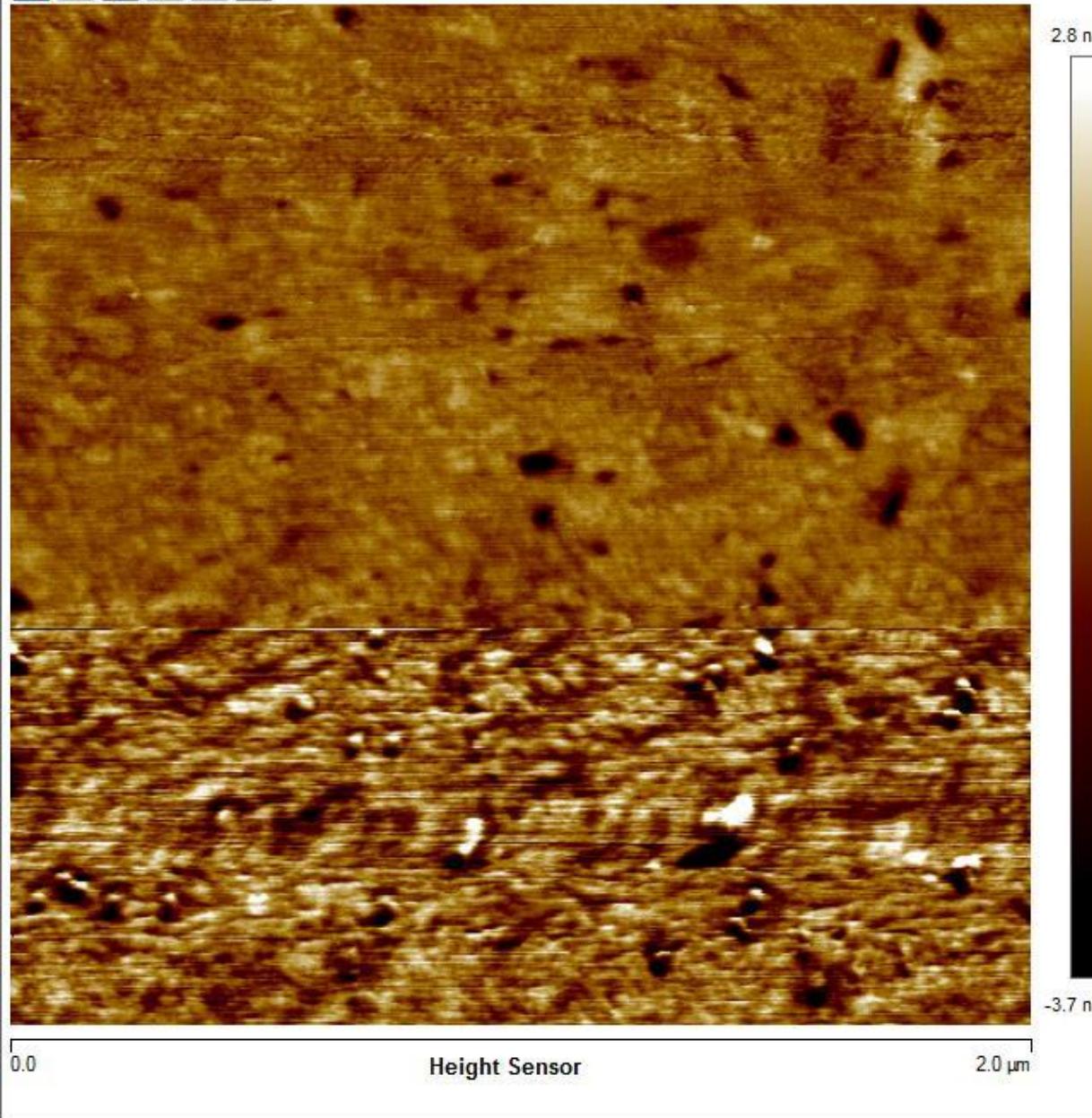
Decapped at 330, 20 min

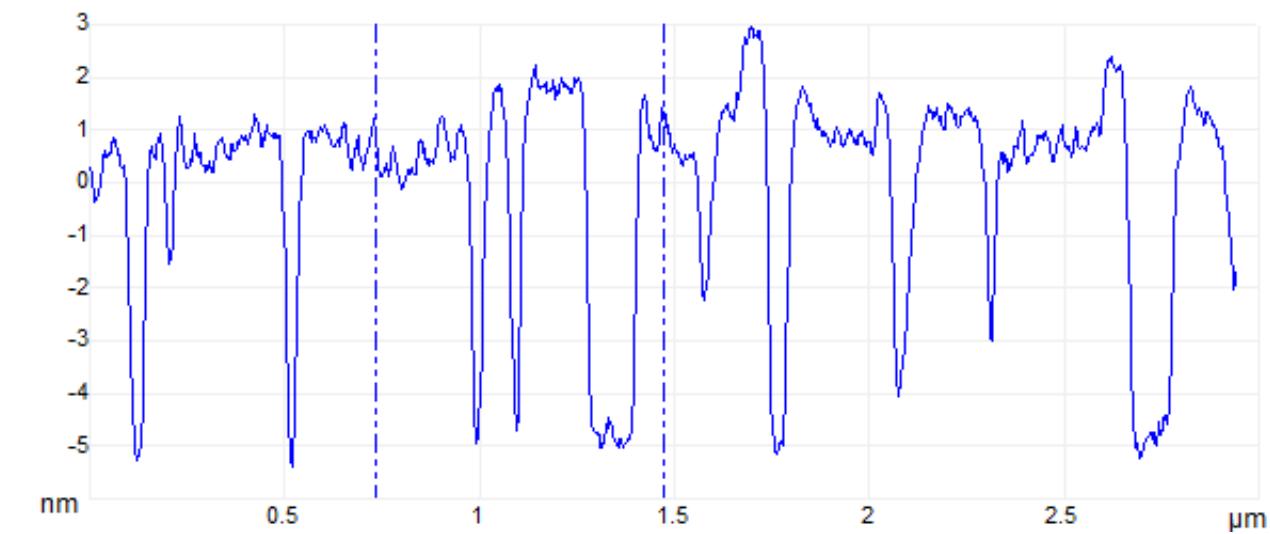
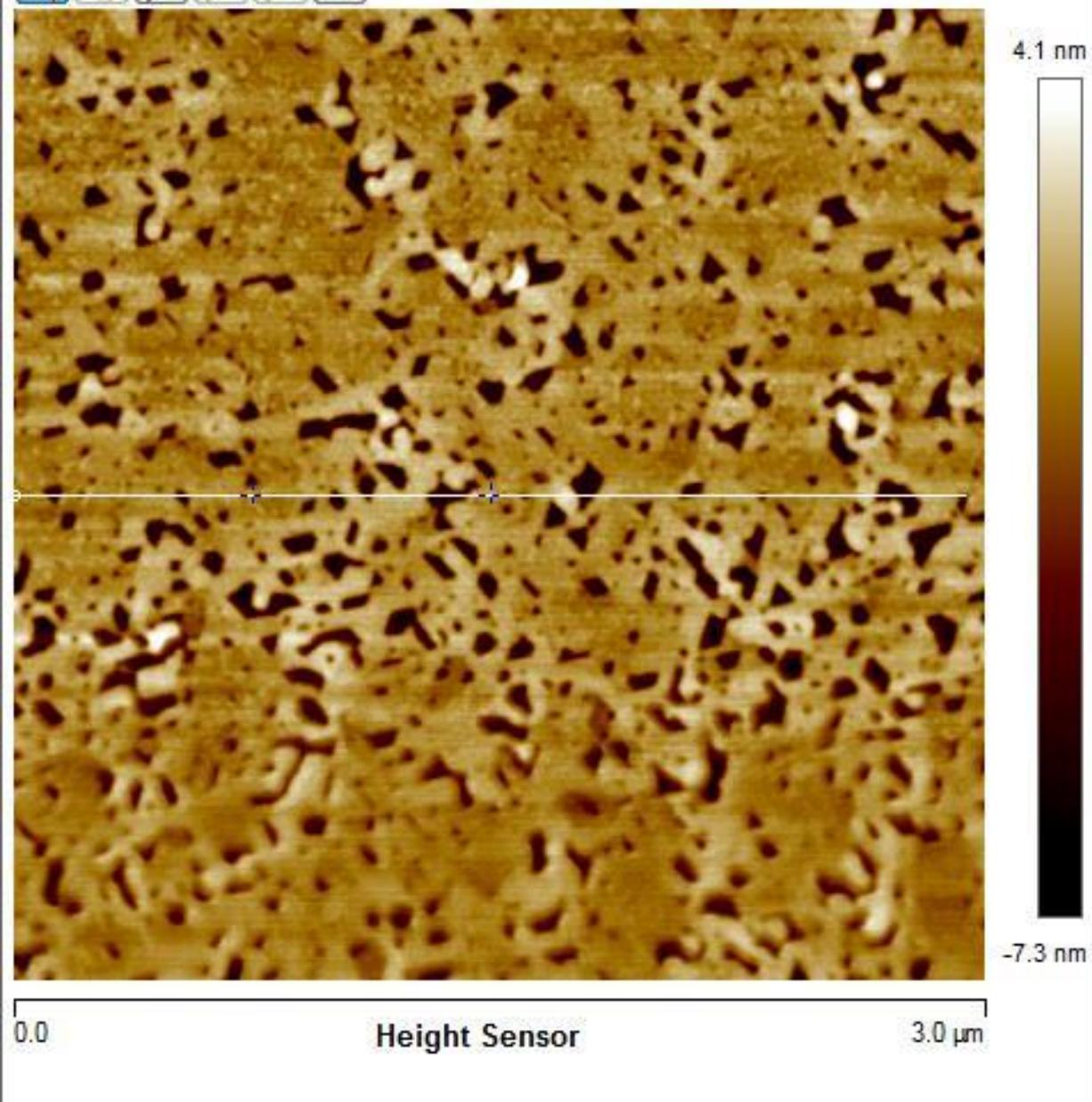


Decapped at 360, 20 min

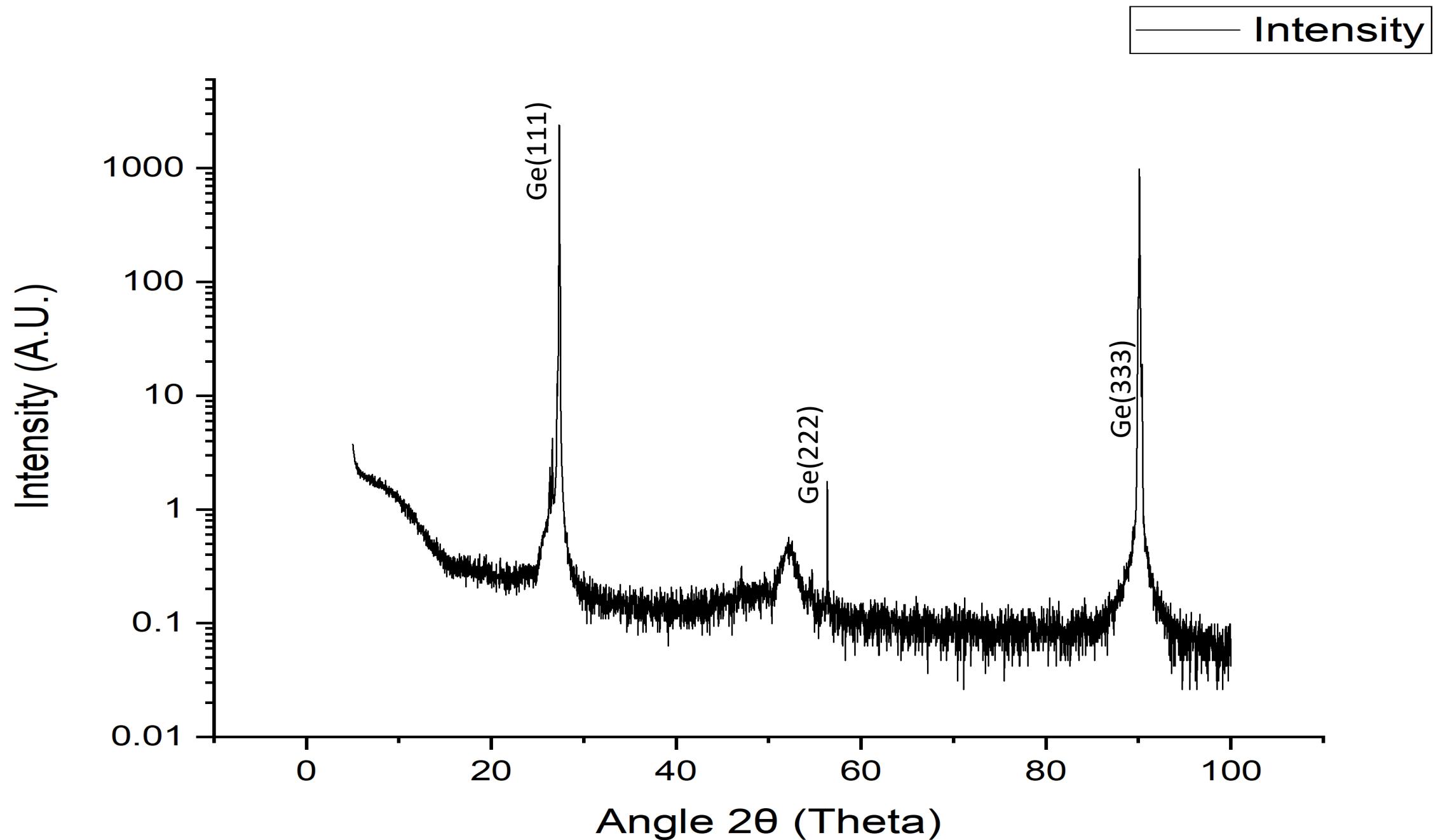




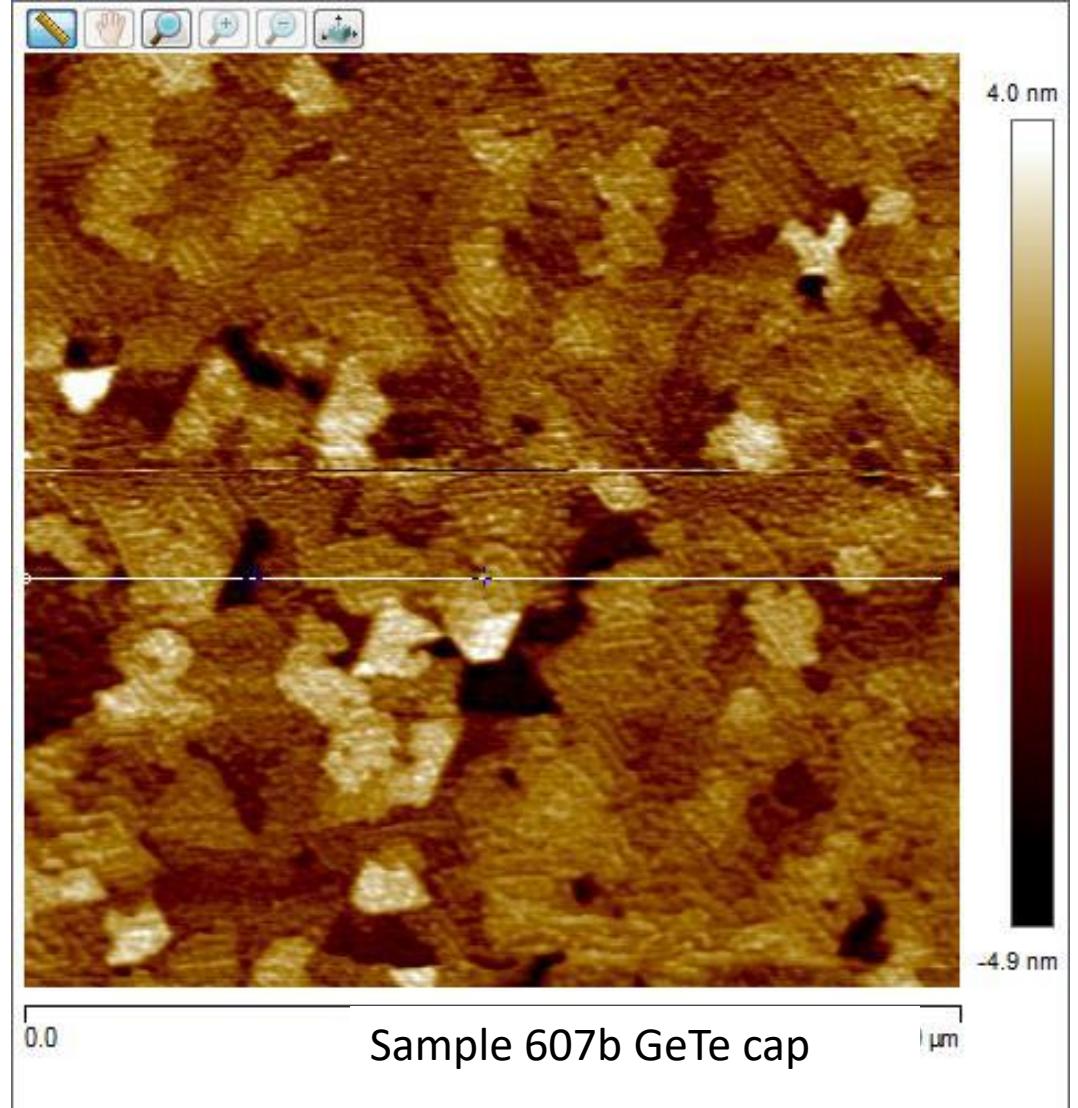
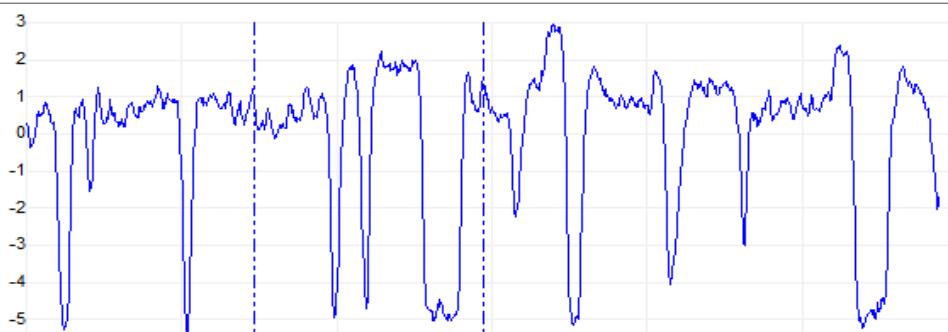
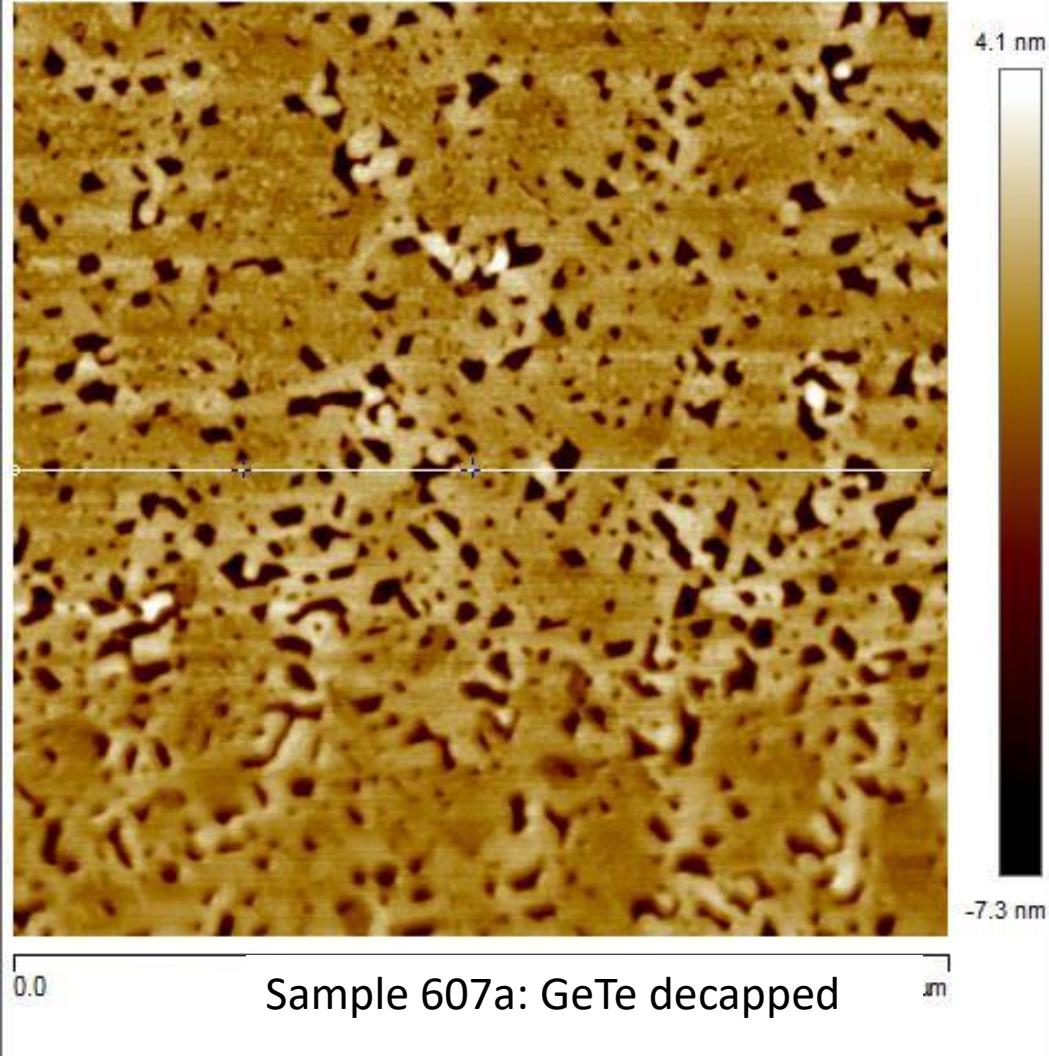


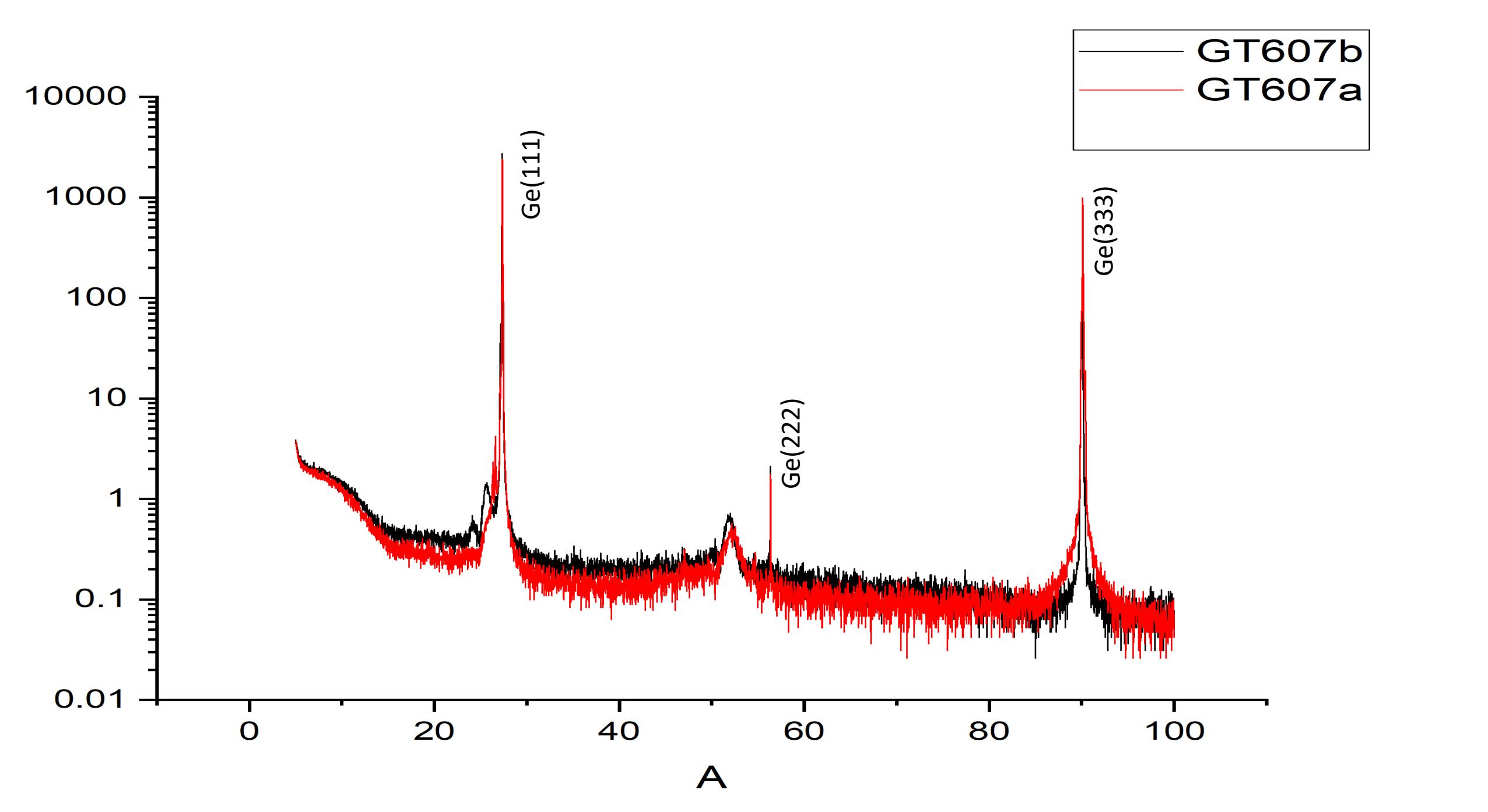


C=10.7A



GeTe decapping on Ge before/after





# Thin and mono Te

624a 624b 705a 708b 727a 729b 805b 805a 811b 817b 817a

Ge-624a 50L FGT + 6L Te at room temp.  
decapped at 260

Brought right to 260,  
no delay

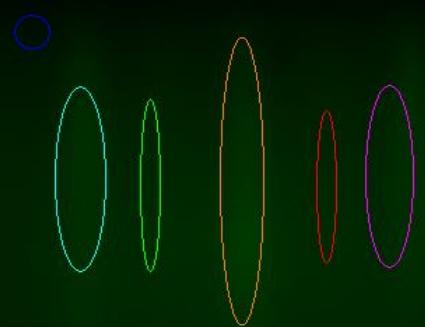
Ge at 490

30 min anneal +  
Ge@1090C 3min

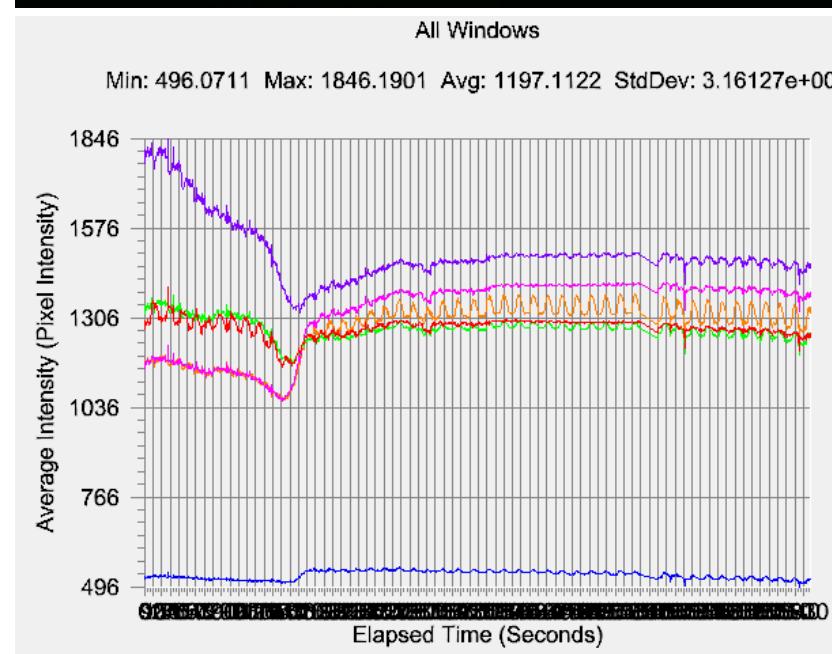
50L FGT

Capped at room temp, 6 layers

60 mins @260

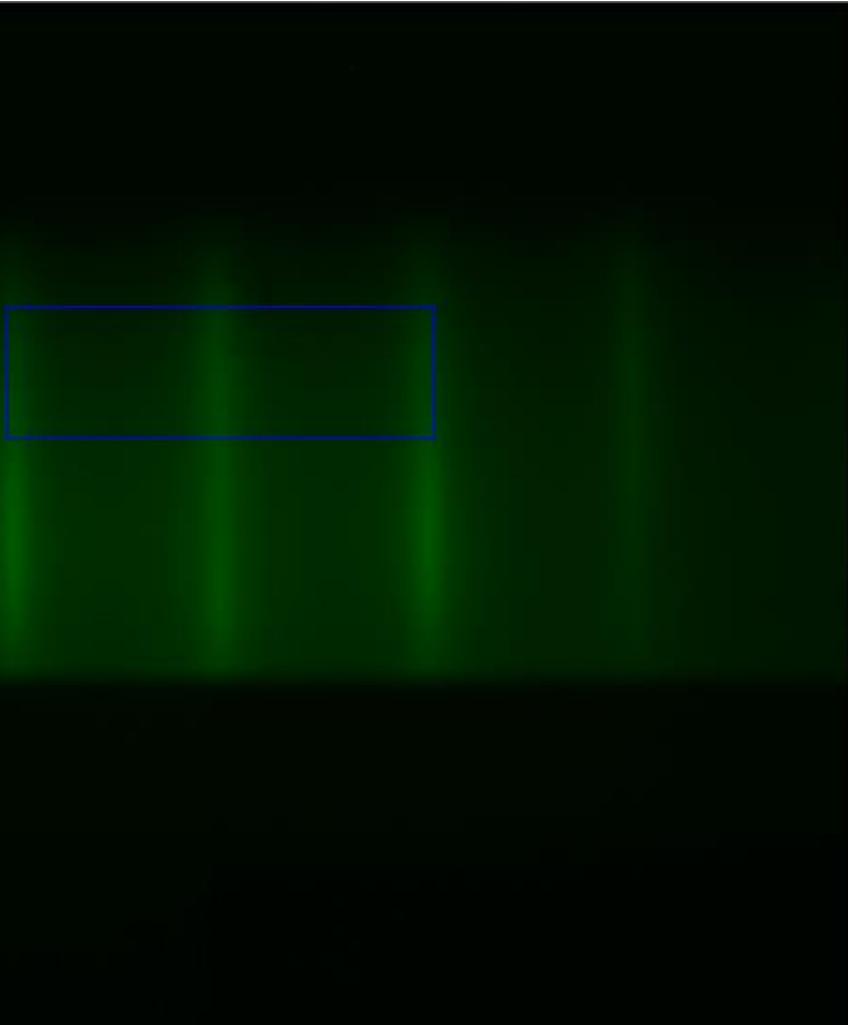


100min @260

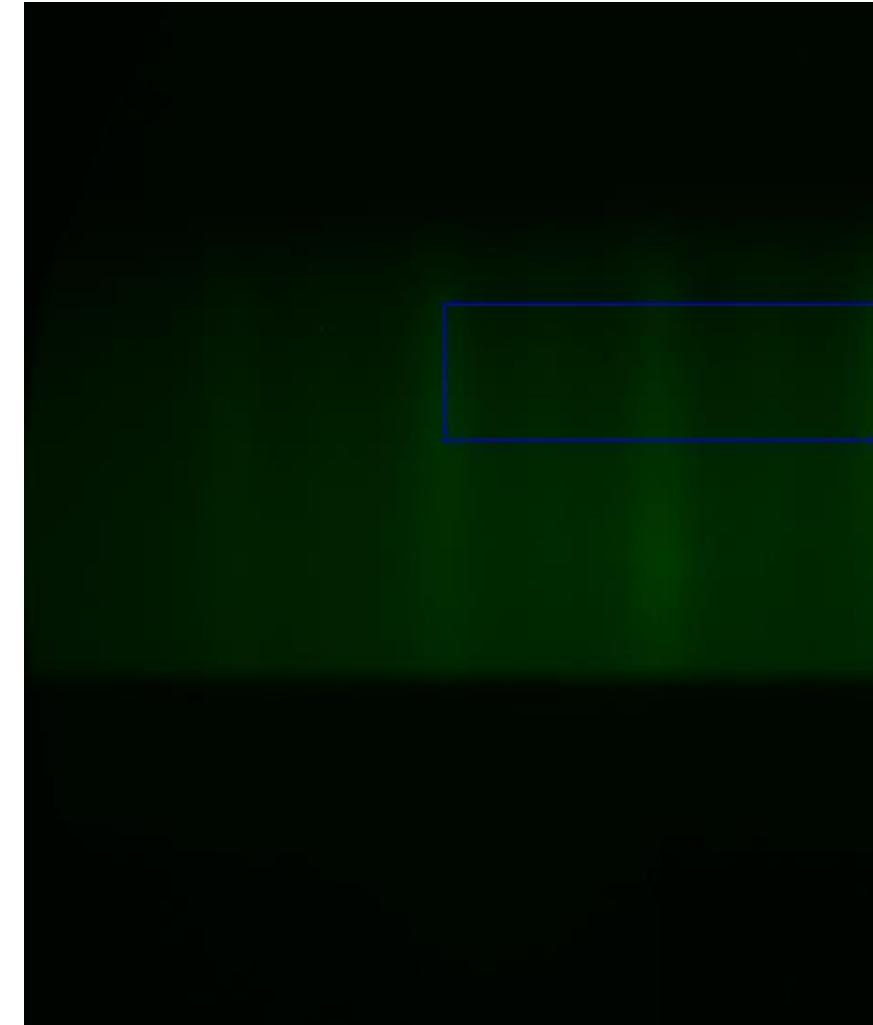


# Wrong spacing

FGT: 890 pix 3.99A



Decapped: 934 pix 3.84A

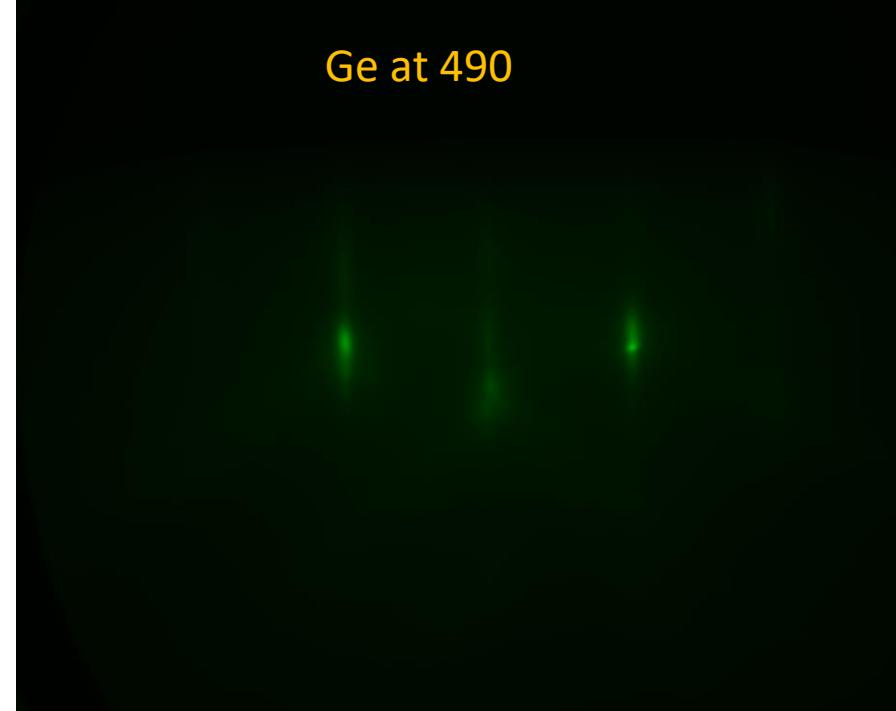


Ge: 888 pix 4.013A

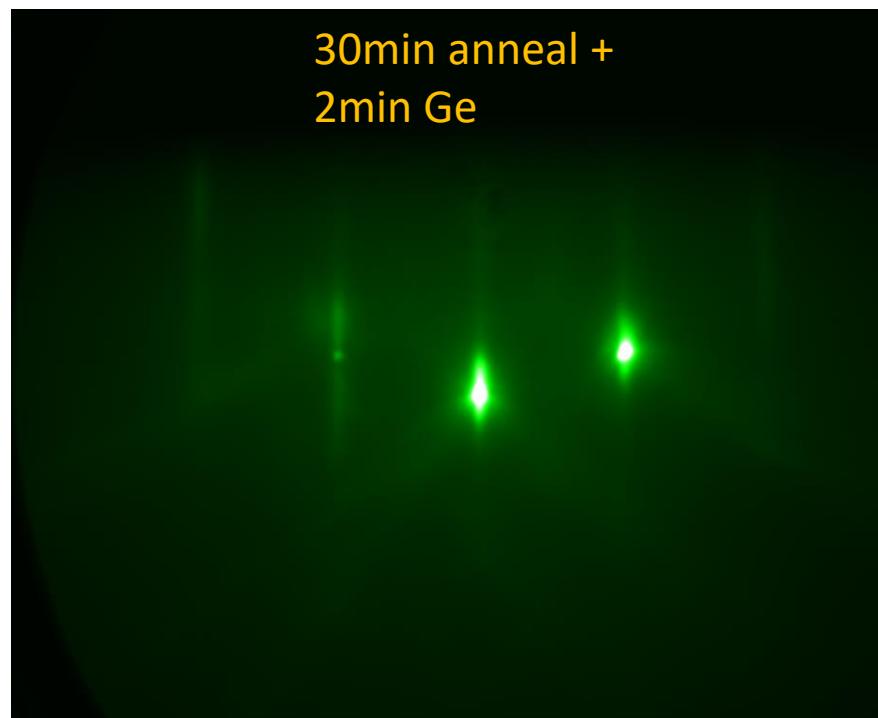
Ge-624b 50L FGT + 6L Te at 14C. decapped at  
260

Gradually brought to 260

Ge at 490



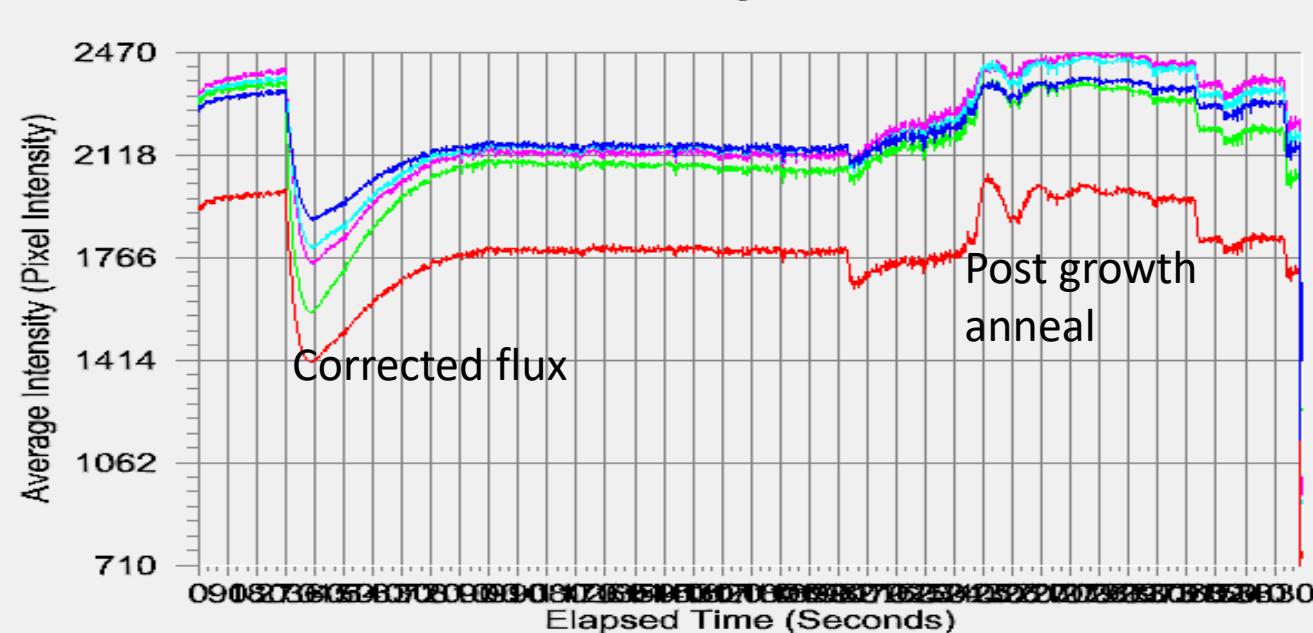
30min anneal +  
2min Ge



624b

All Windows

Min: 709.9439 Max: 2469.8472 Avg: 2110.3212 StdDev: 2.20317e+00



After 40 min Fe(1177) +  
30 min Fe(1170)



Capped 14C, 6 layers



capped

@150C (recrystallization)

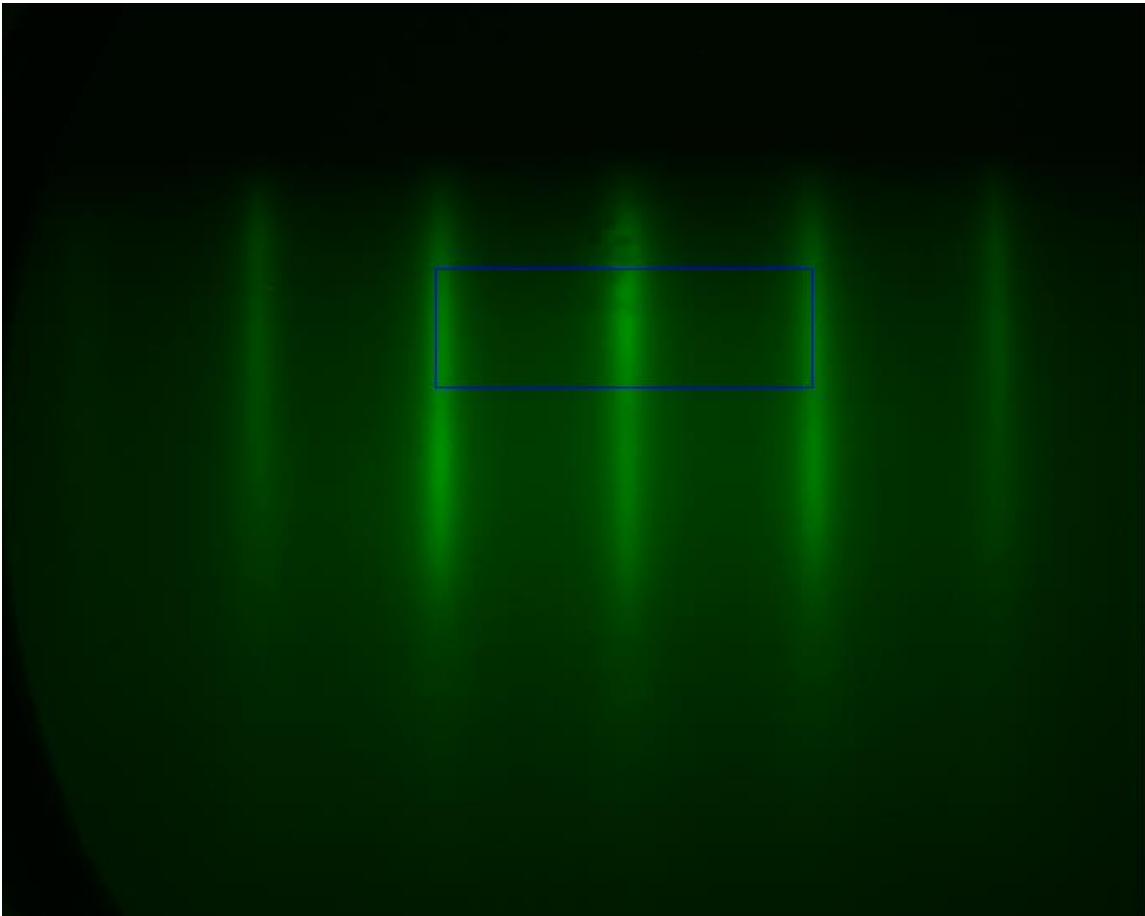
Decap @230

Decap @240

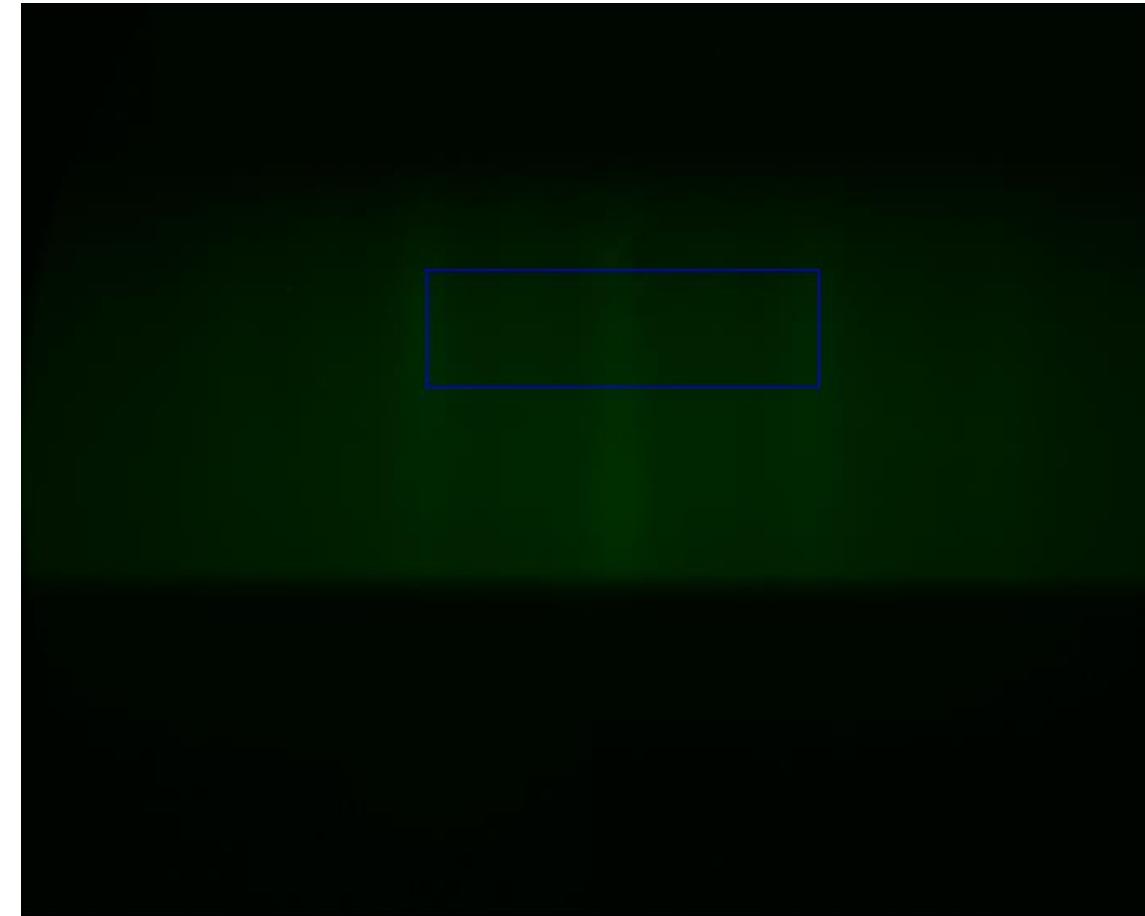
Decap @245

# Wrong spacing

FGT: 894pix 3.99A



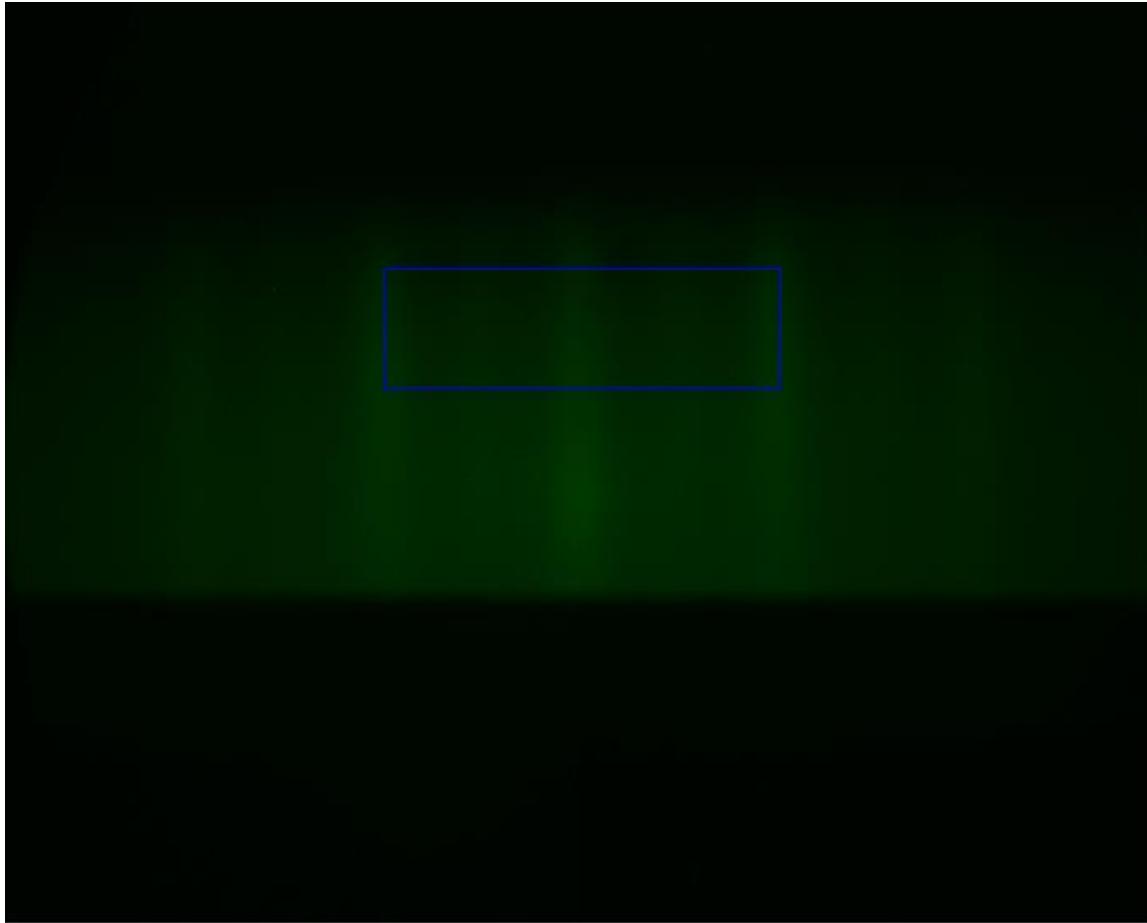
Decapped: 930pix 3.86A



Ge: 888 pix 4.013A

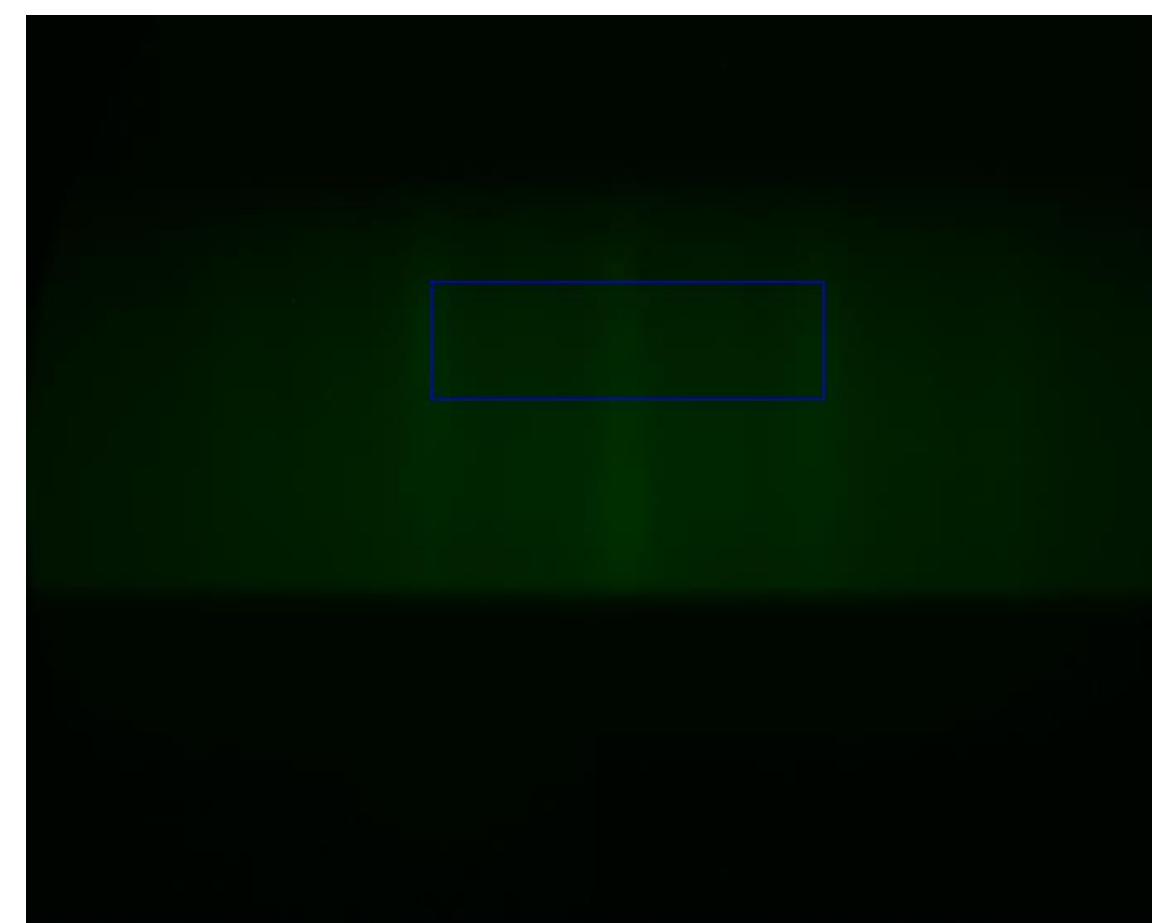
# Comparing decaps

624a decap



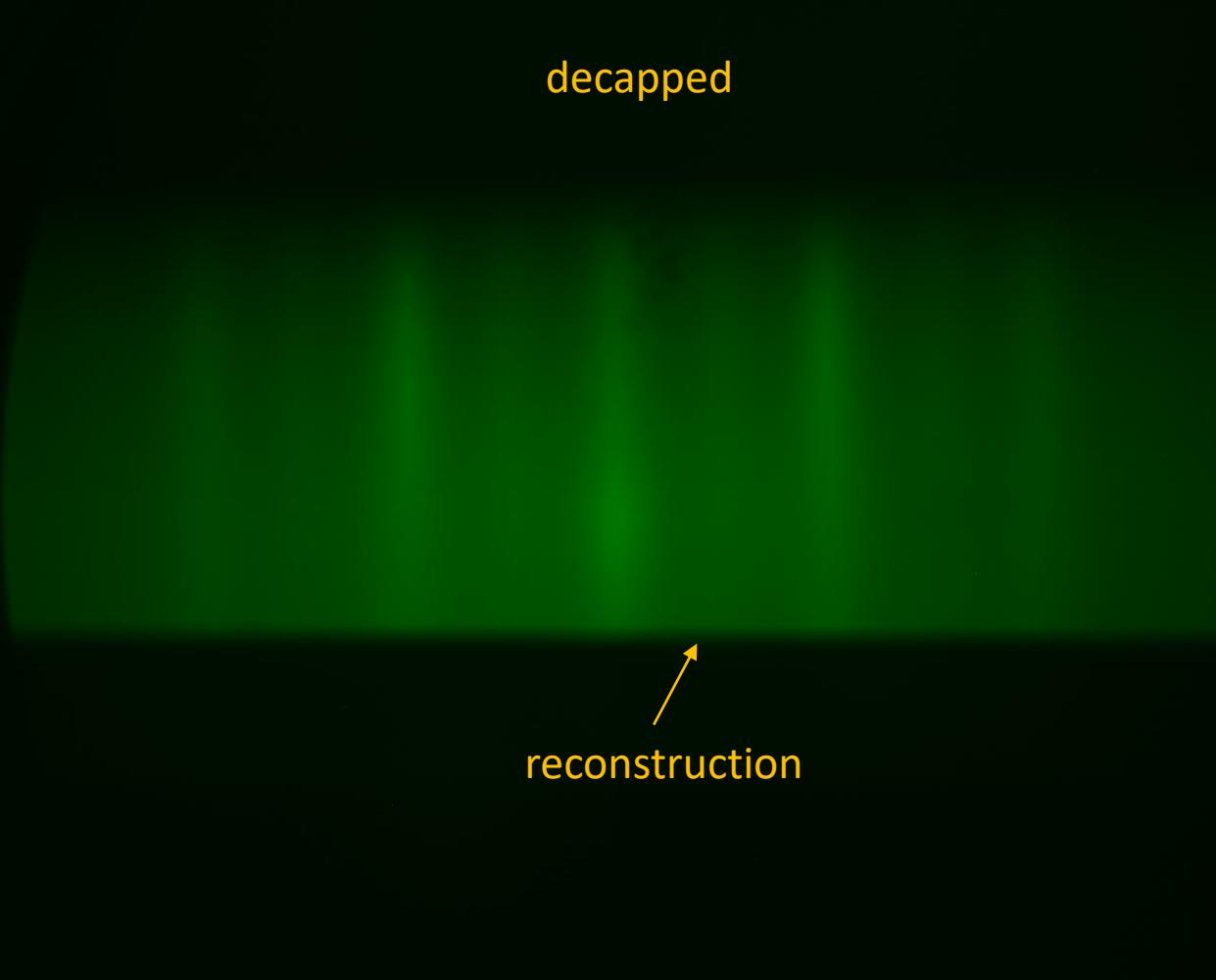
3.84A

624b decap



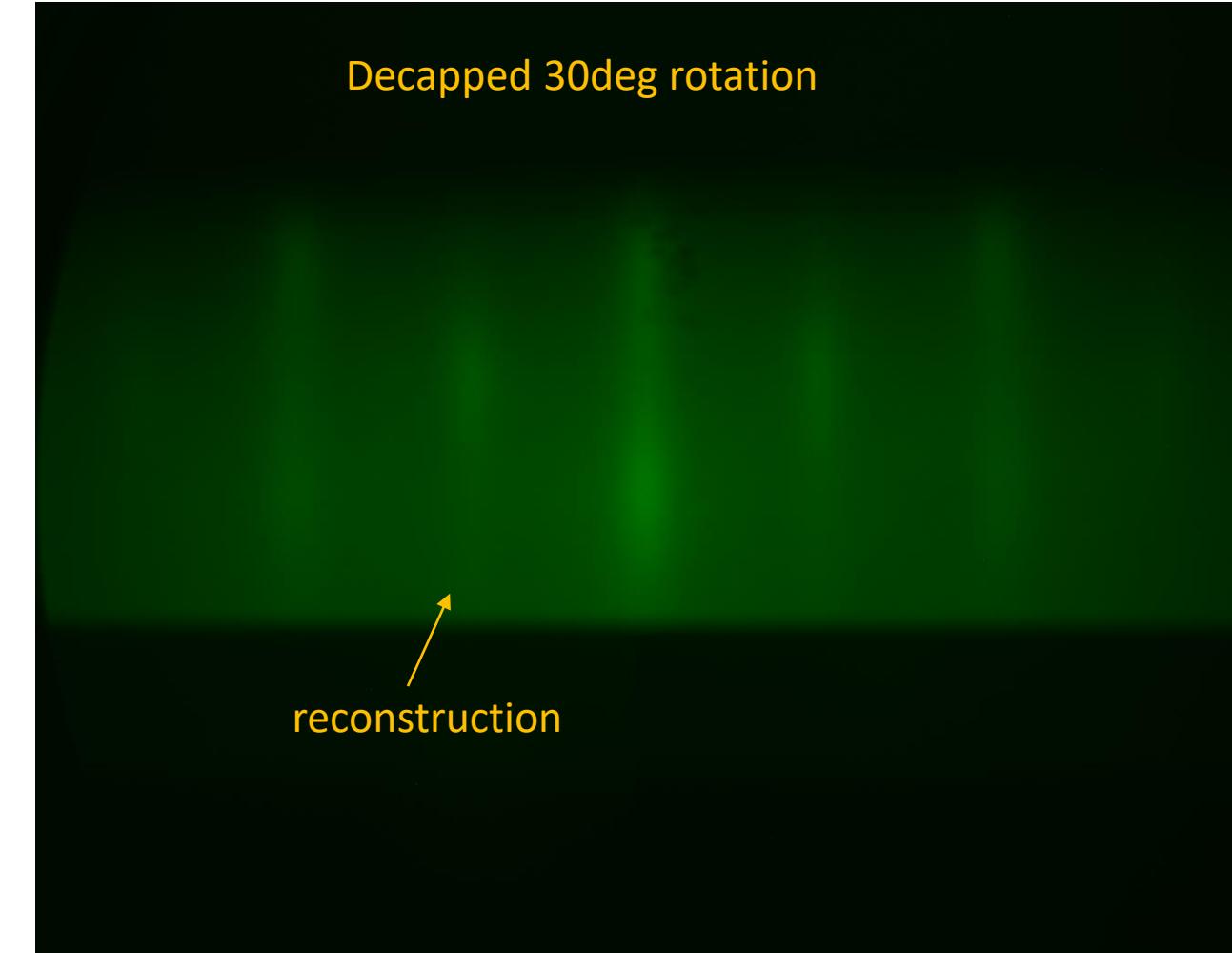
3.86A

New 240C 2x2 3.85A compound



decapped

reconstruction



Decapped 30deg rotation

reconstruction

Using Ge RHEED, new crystal has  $a=3.8\text{\AA}$ , 2x2 reconstruction

Clues:

New compound does not form during normal FGT growth (360C)

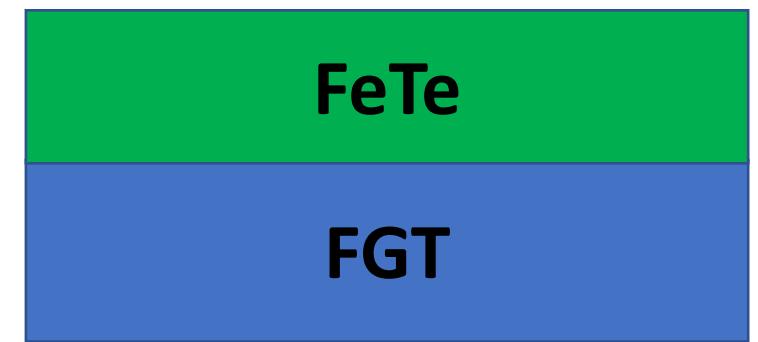
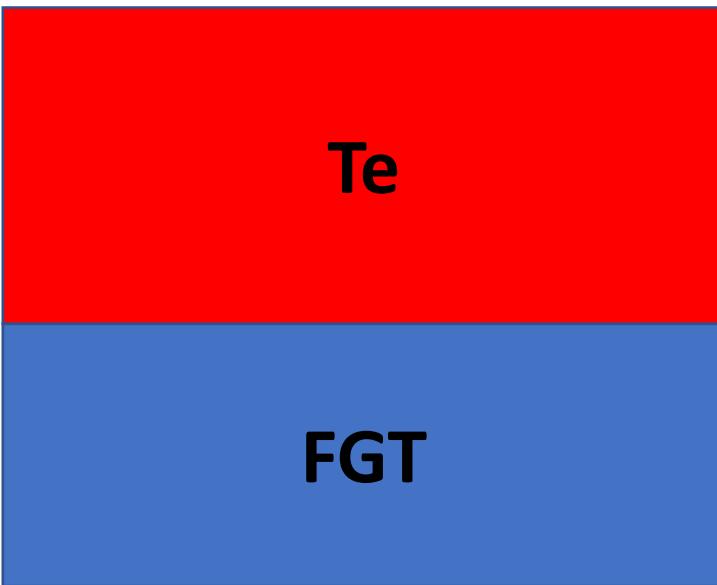
RHEED transition is smooth from Te pattern to new pattern

Idea:

Room temp

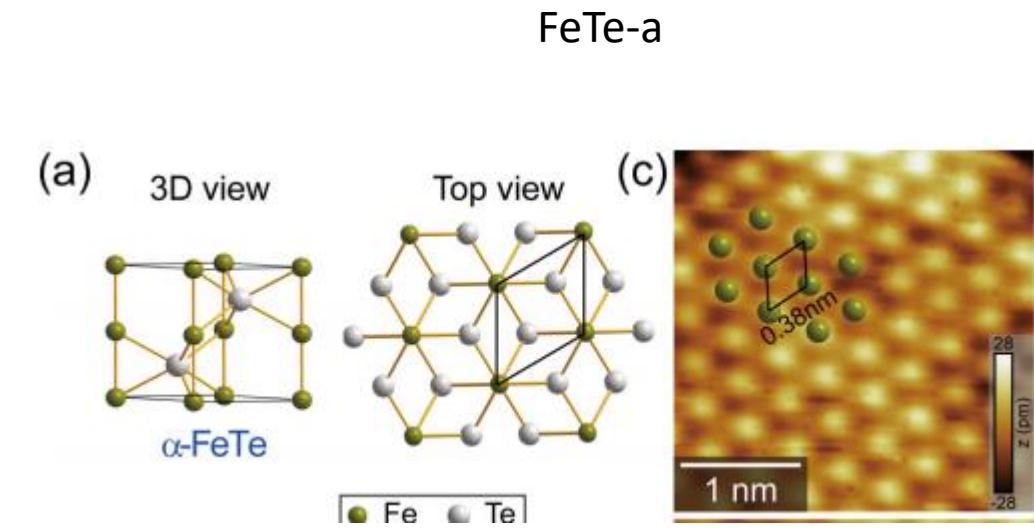
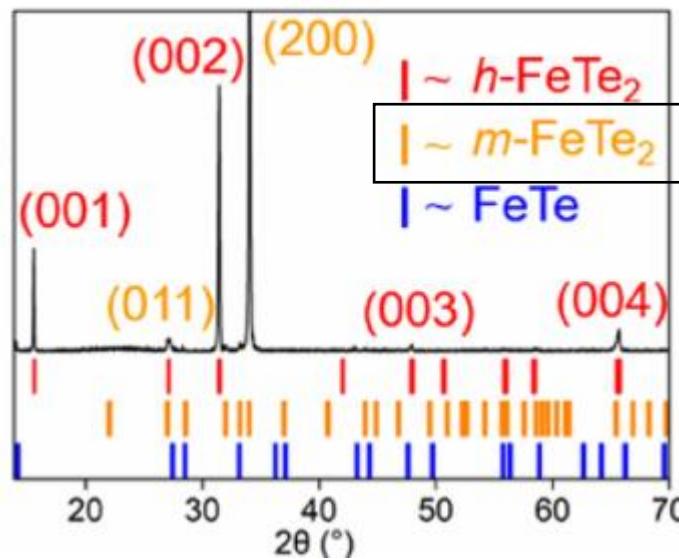
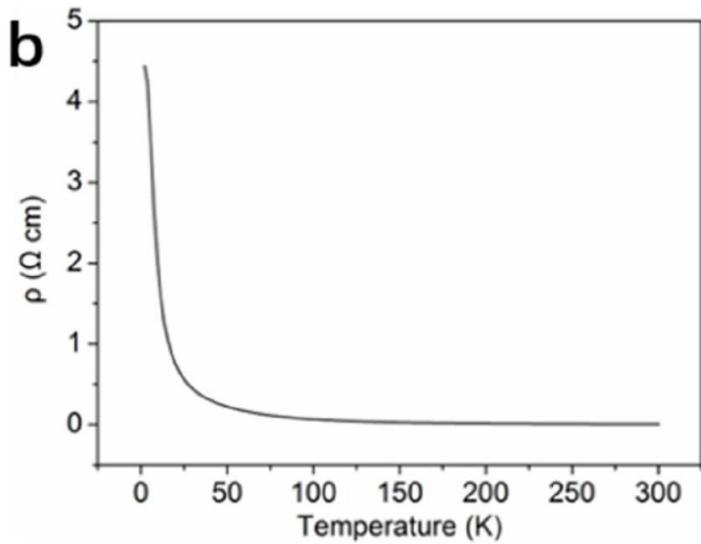
reaction temp

240C upward



# FeTe

a=3.8Å



<https://pubs.acs.org/doi/10.1021/acsnano.0c03863#>

3.8A

Ge-705a 10L FGT + 1.3L Te at r.t.. decapped to  
310

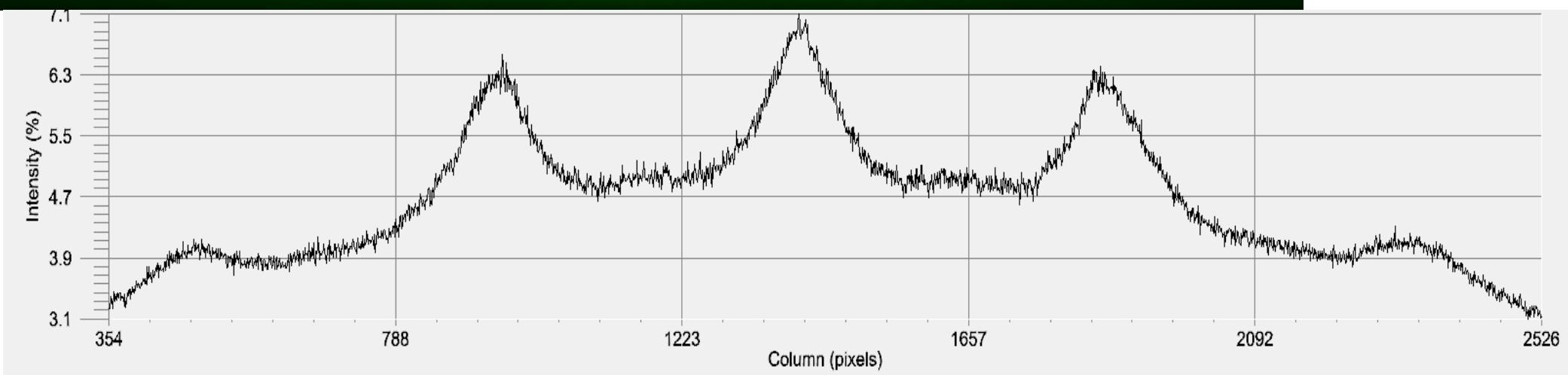
Ge 2mins + anneal

FGT 10L

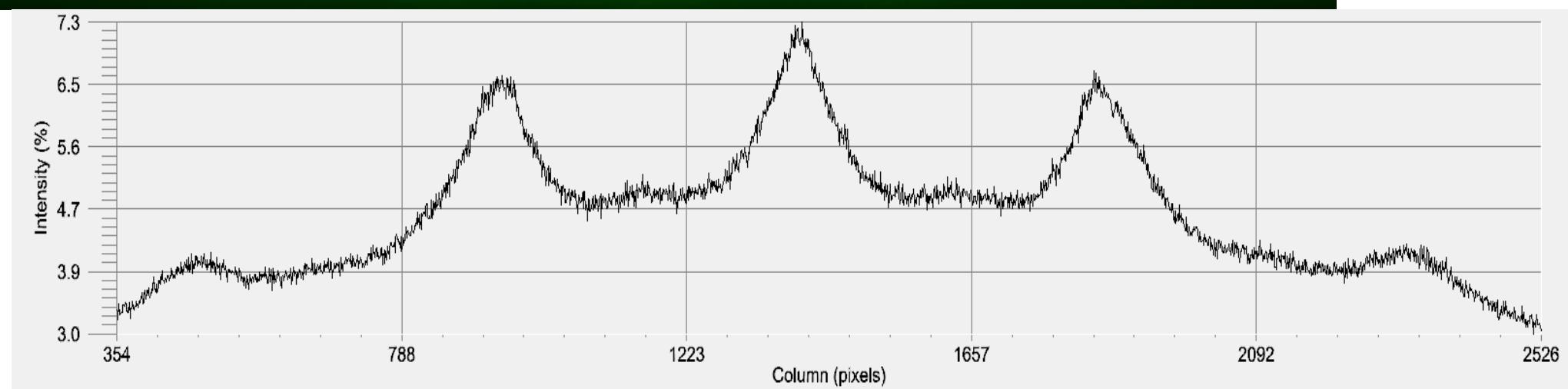
54sec 1.3 layer Te

1 hour at room temp  
Te recrystallizes

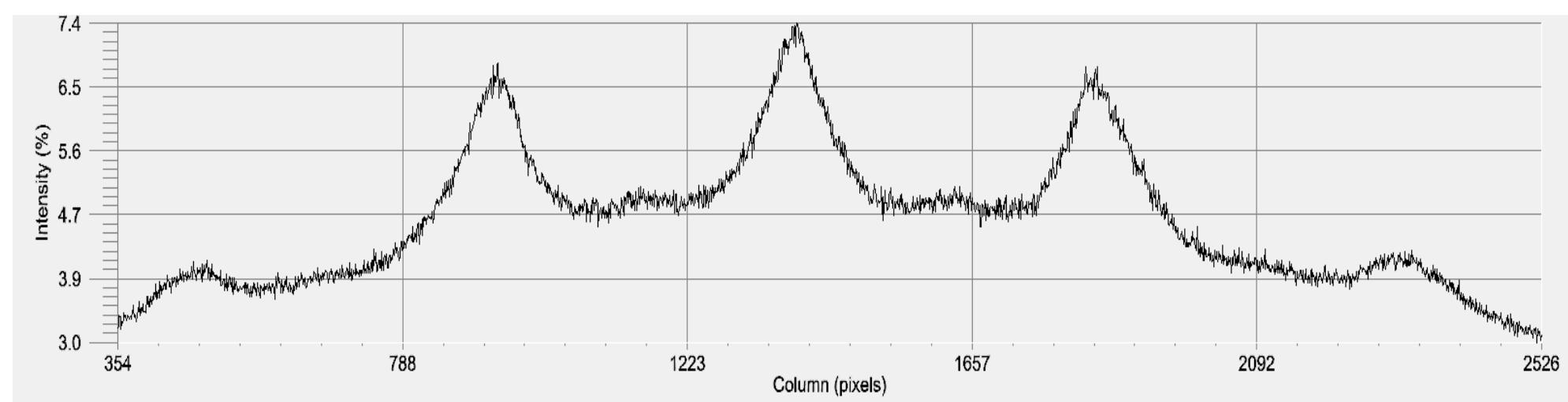
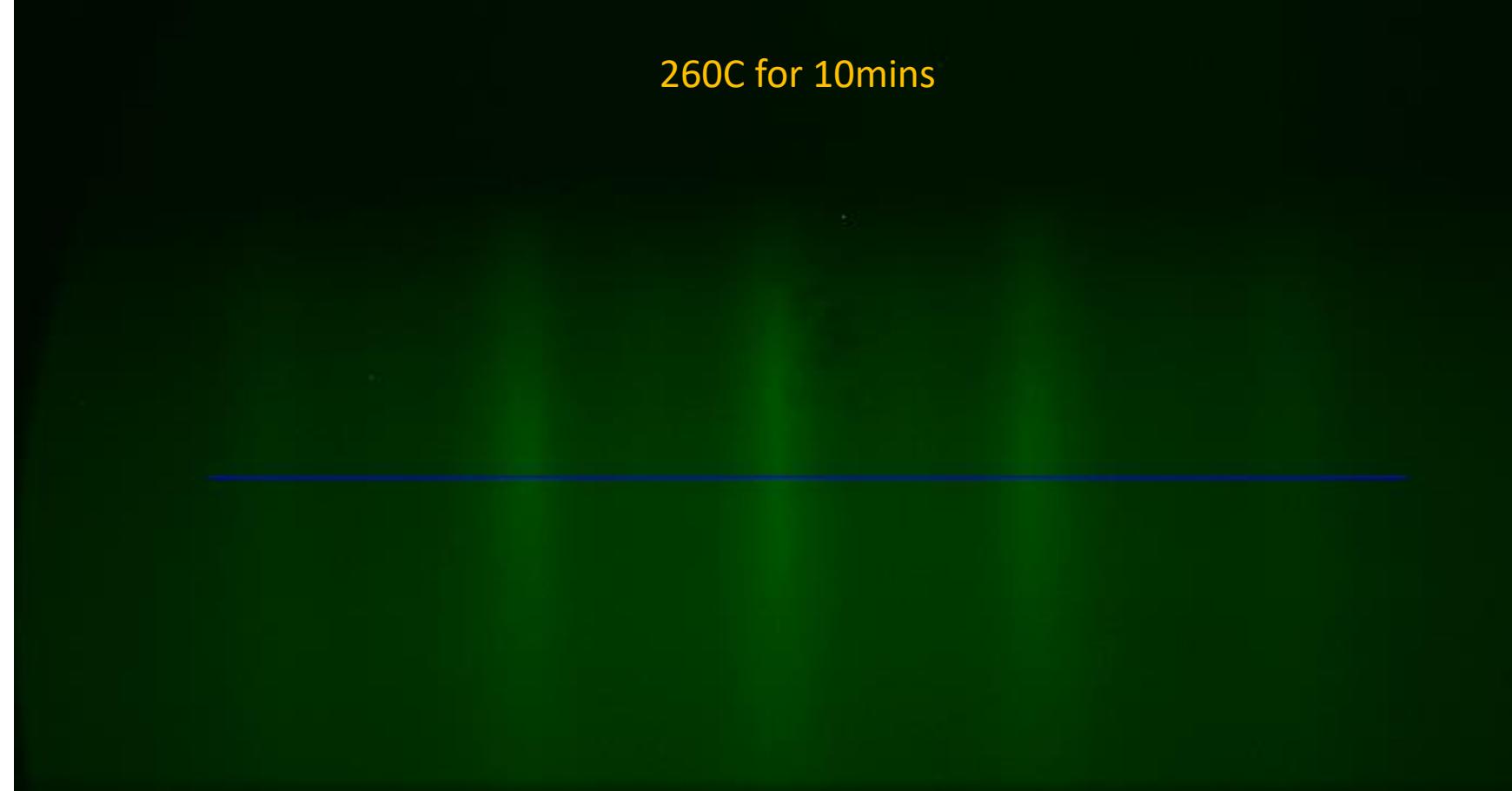
240C for 15mins



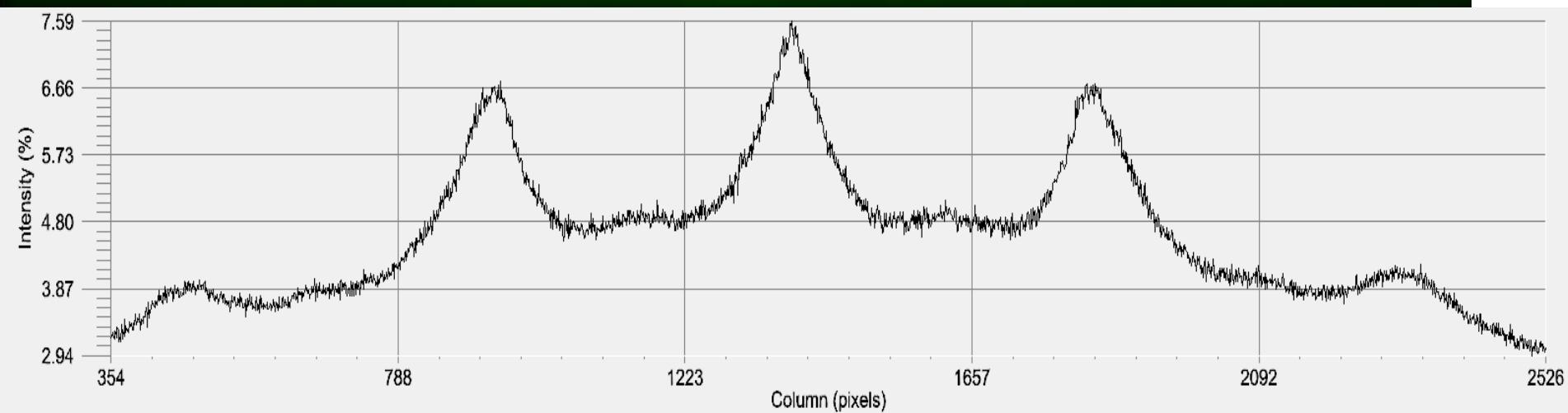
250C for 10mins



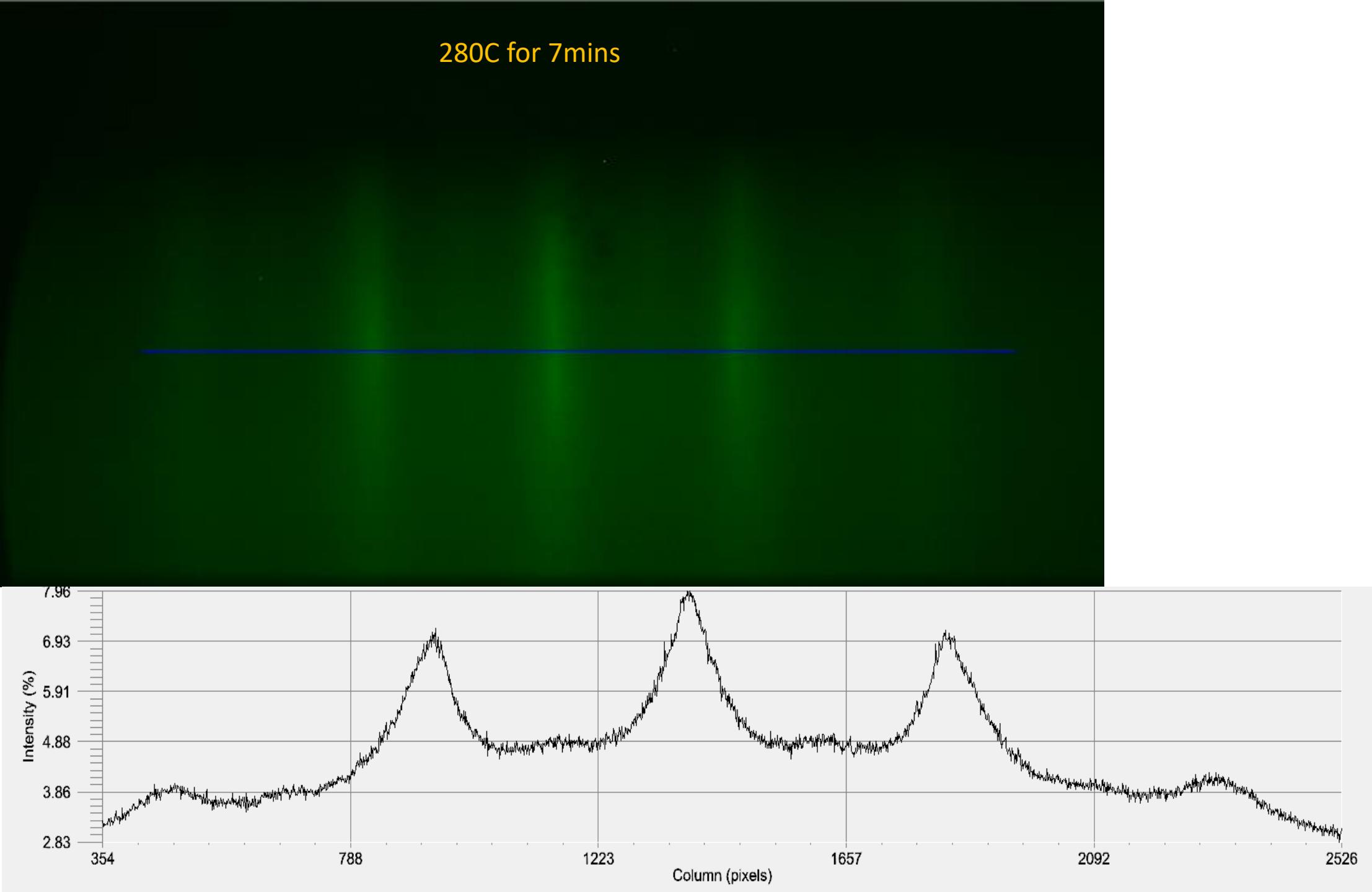
260C for 10mins



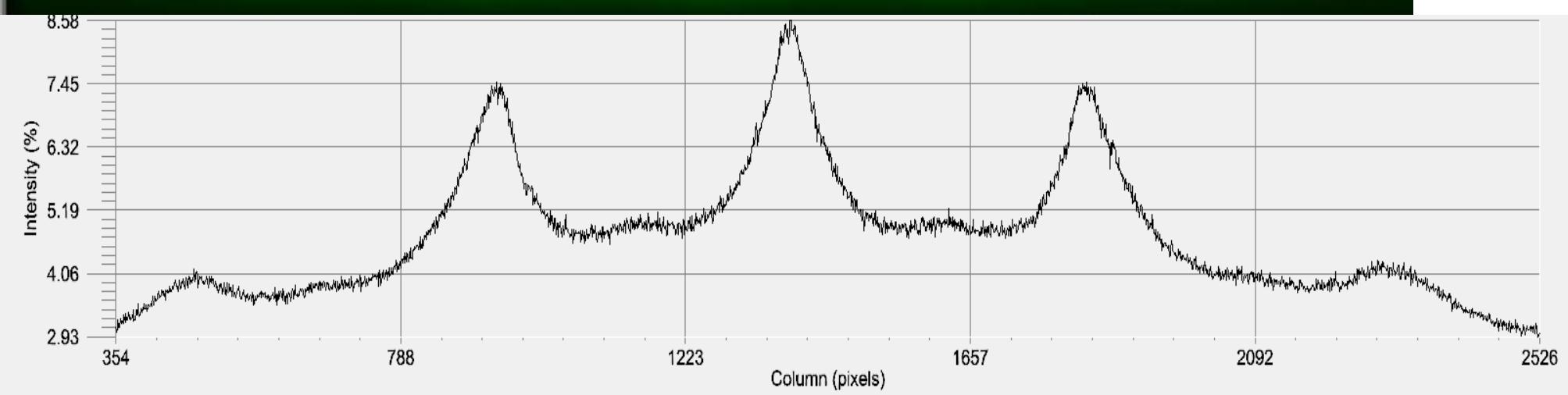
270C for 10mins



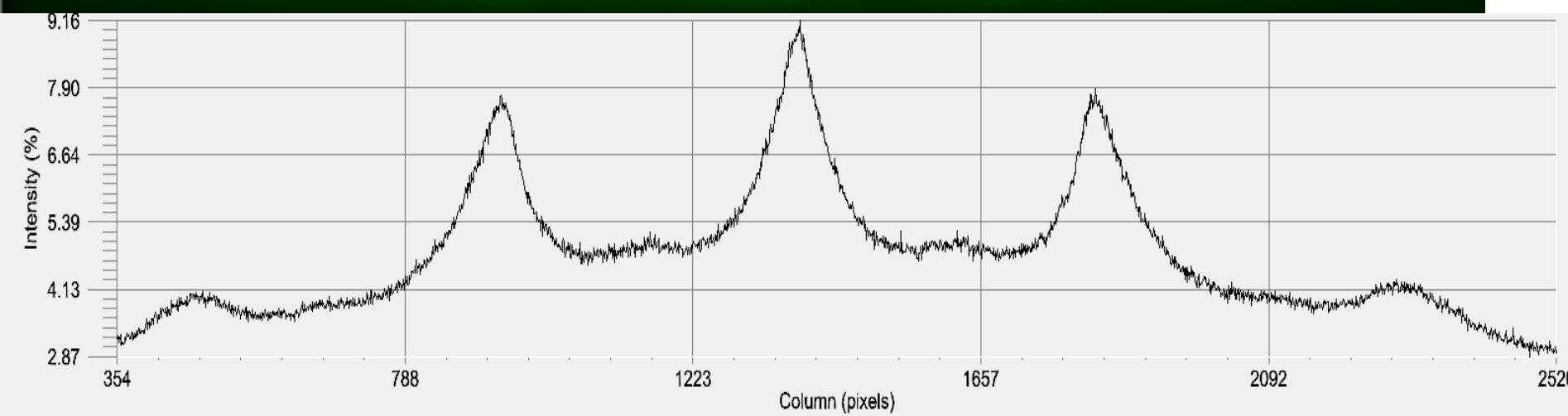
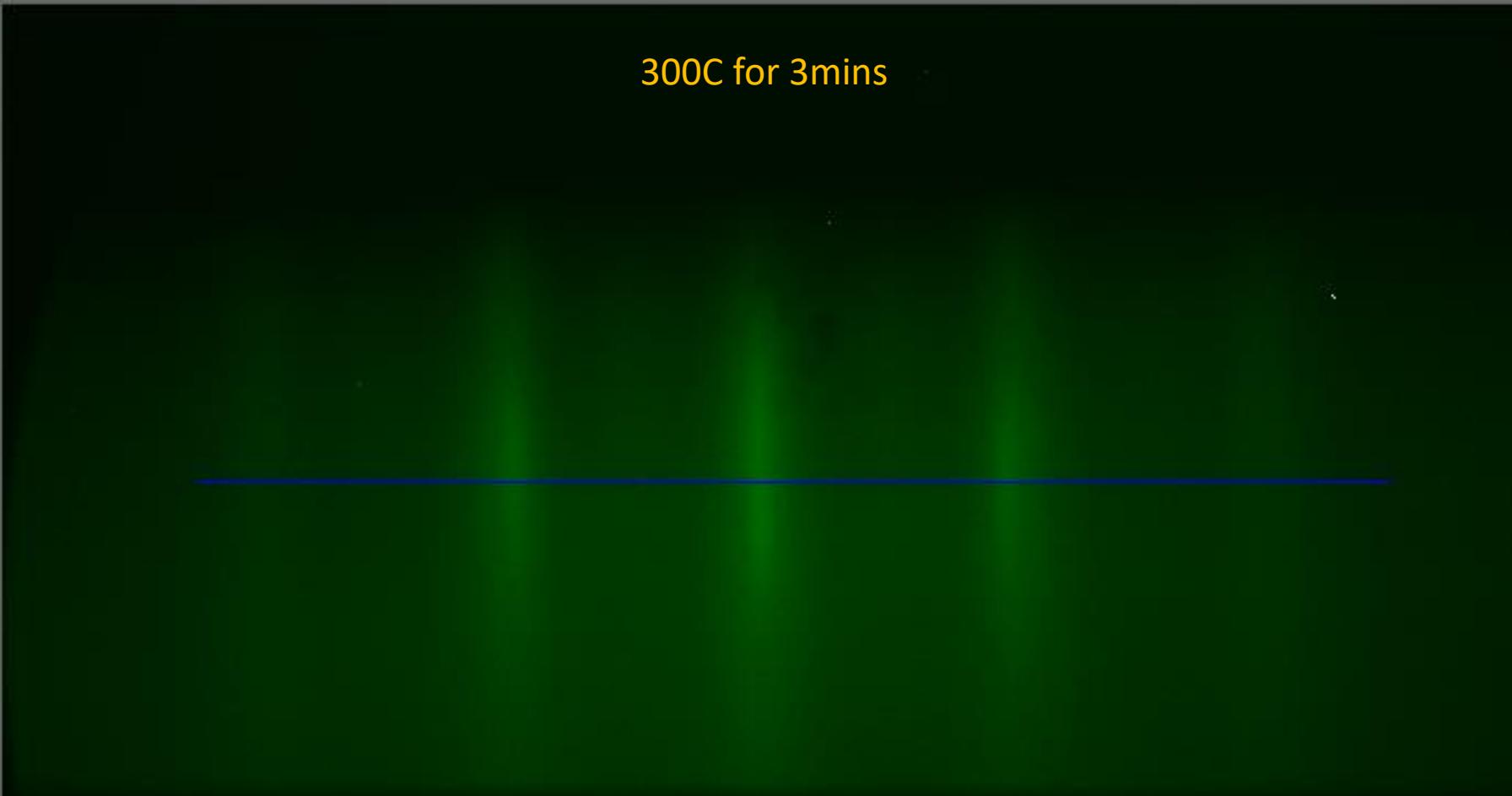
280C for 7mins



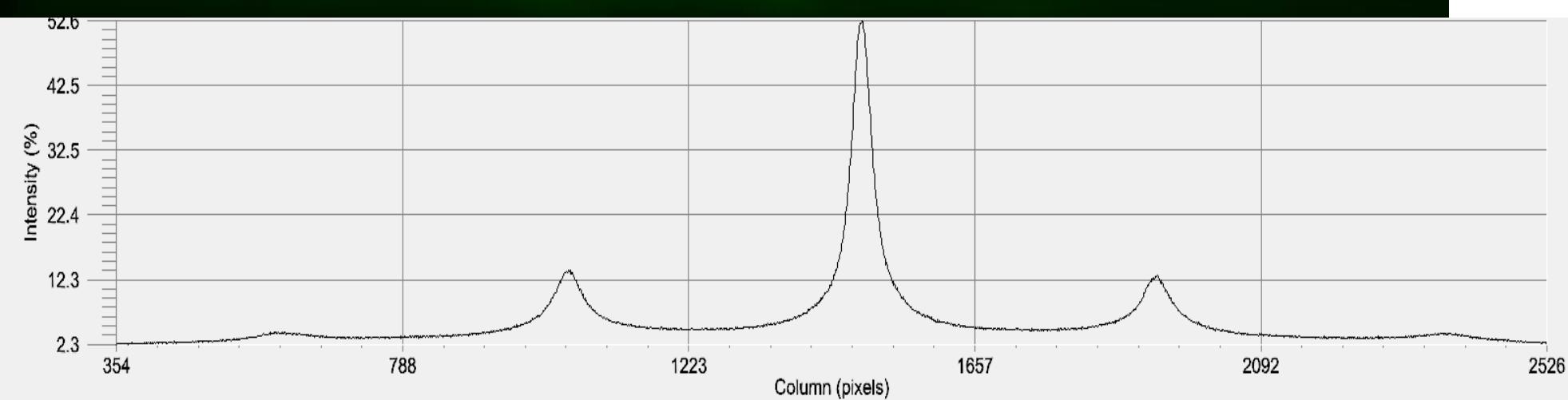
290C for 4mins

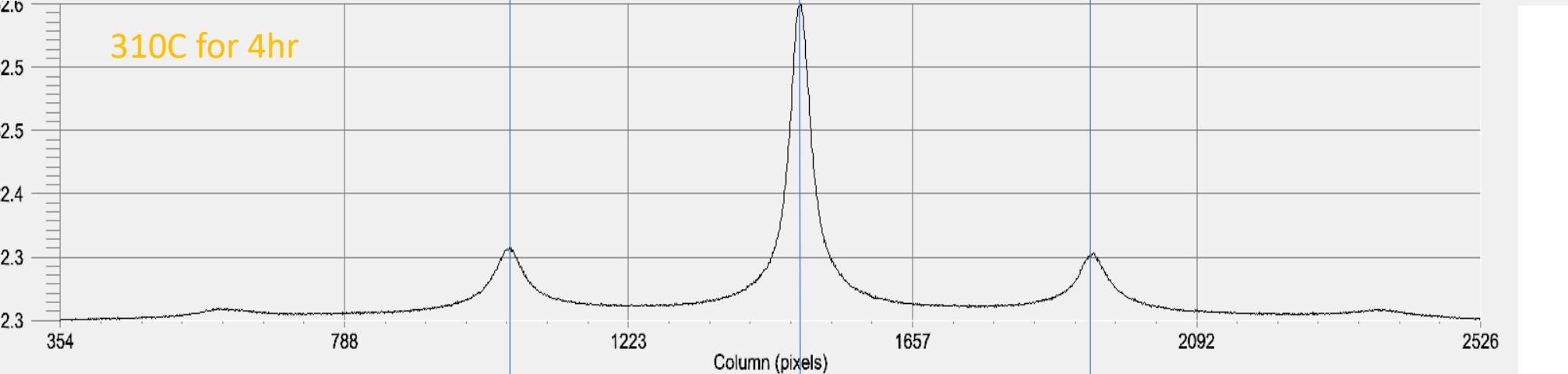
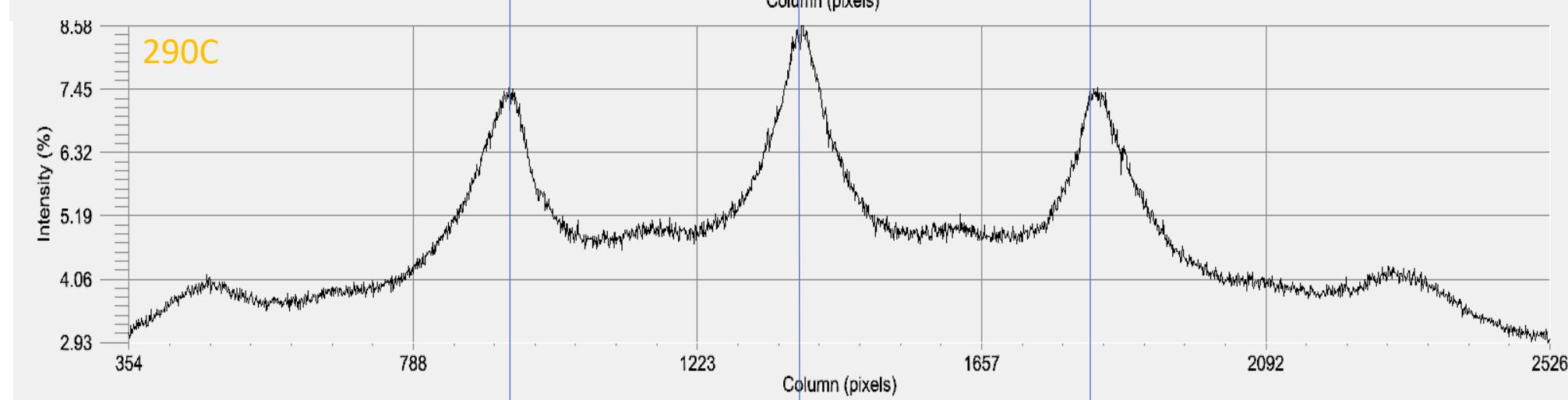
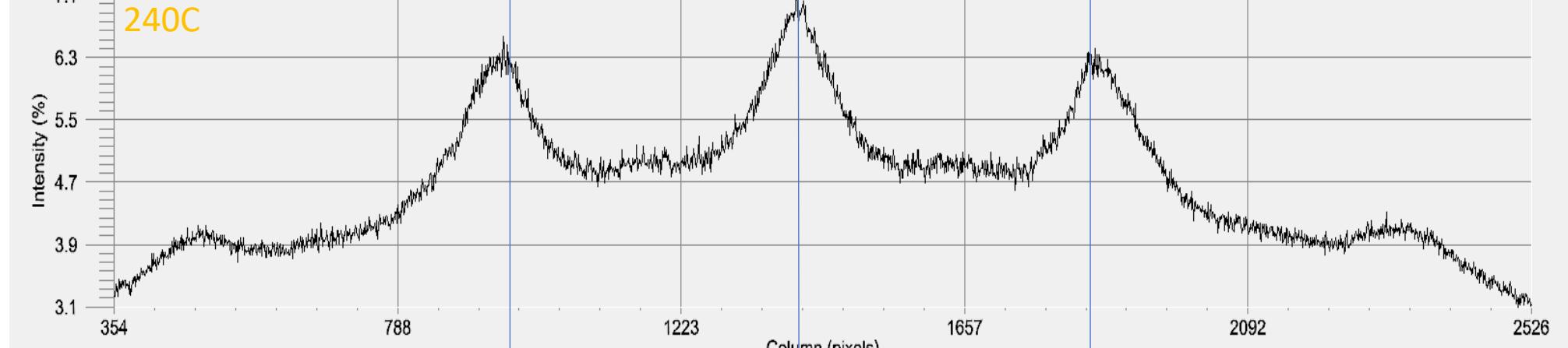


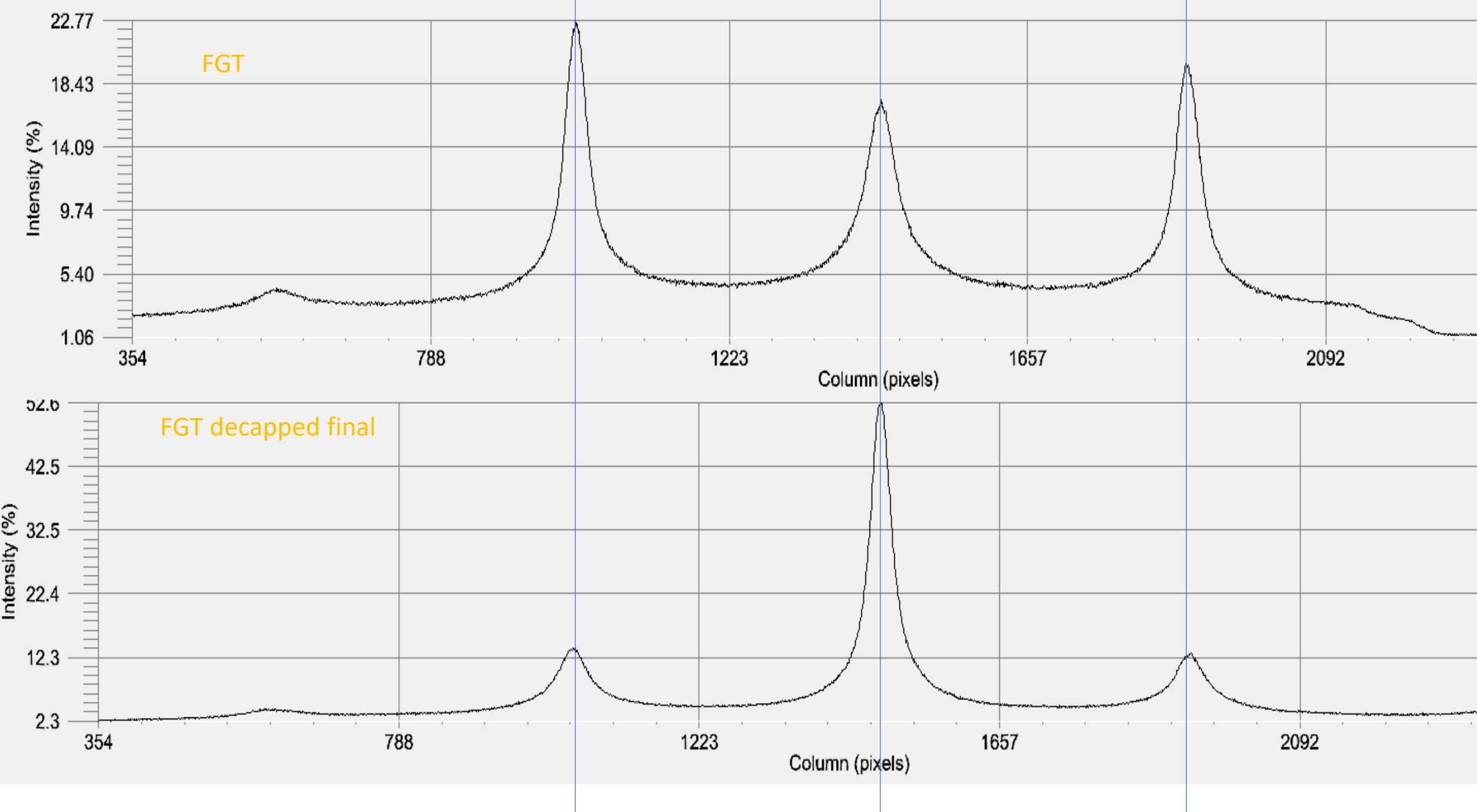
300C for 3mins



310C for 4hr



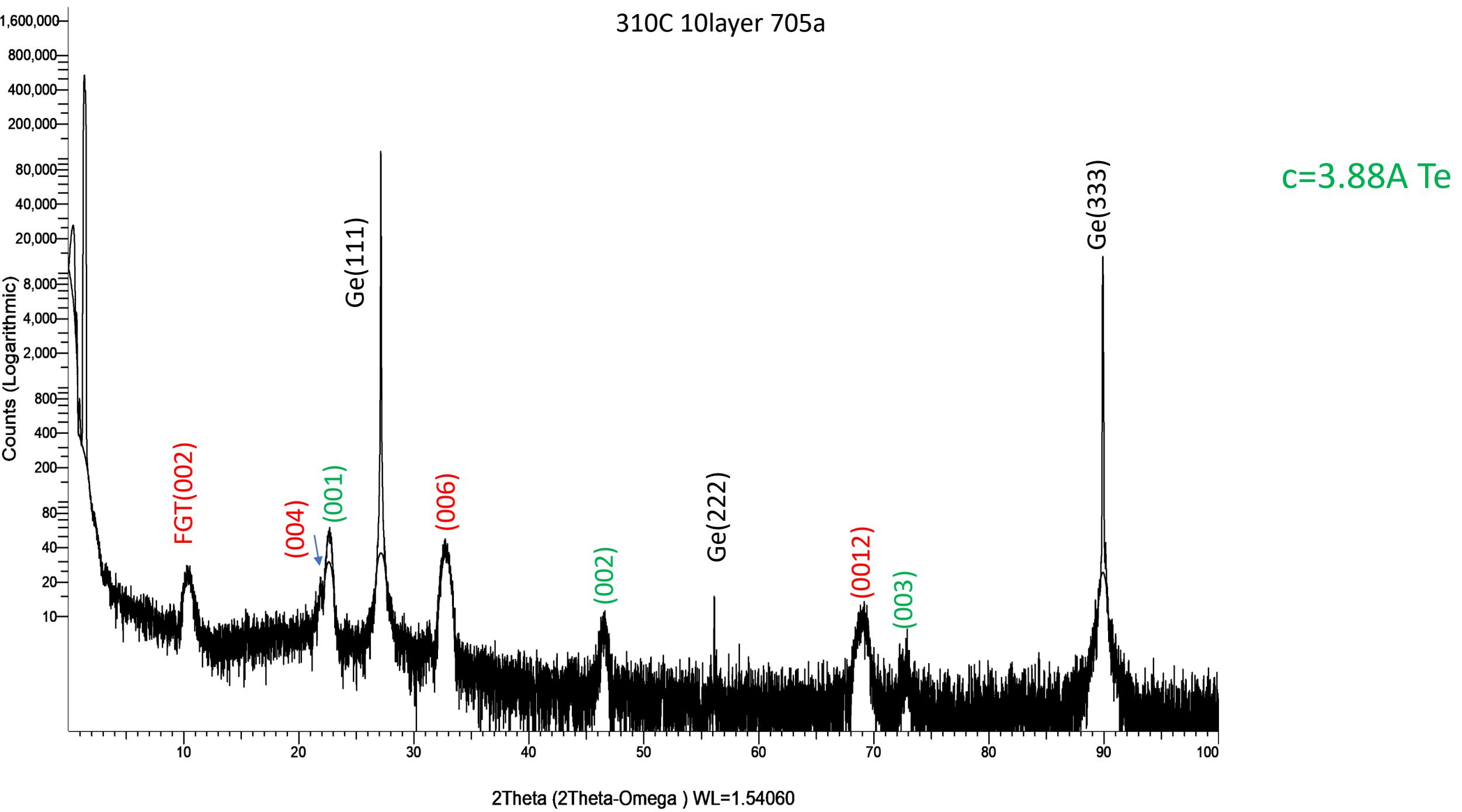




FGT 4.0A

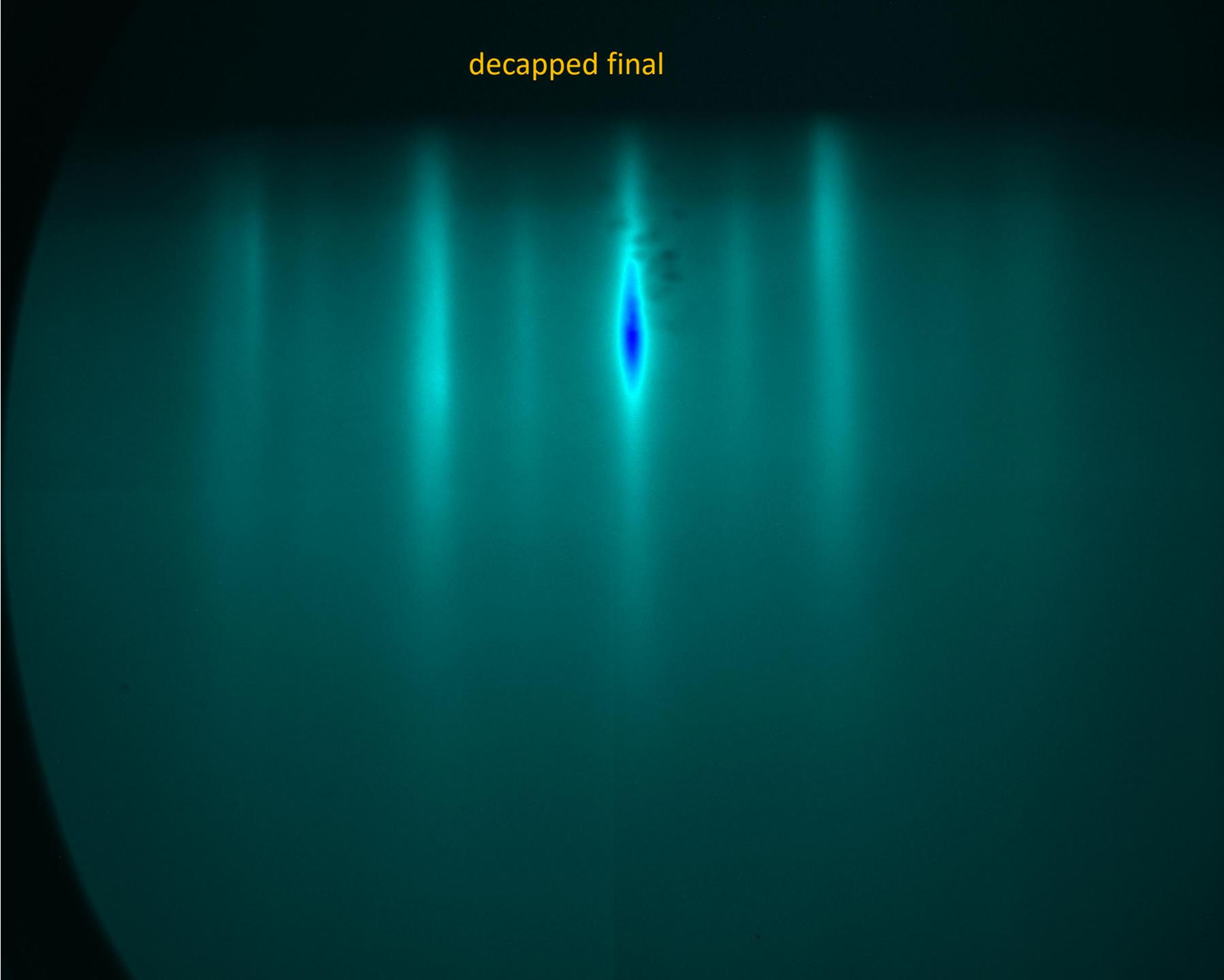
FGT decapped final 4.0A

310C 10layer 705a



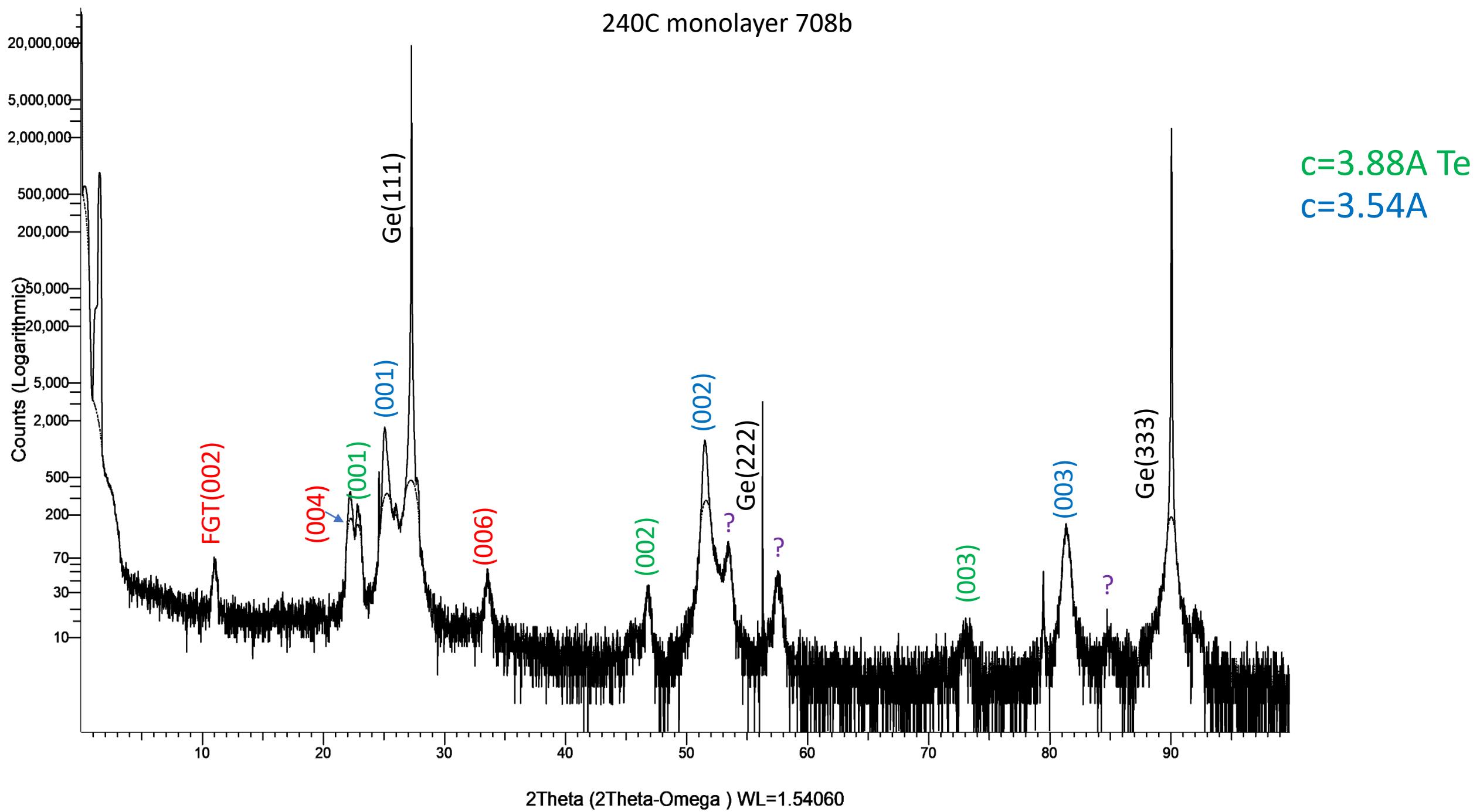
Ge-708b 1L FGT + 1.3L Te at r.t.. decapped to  
240

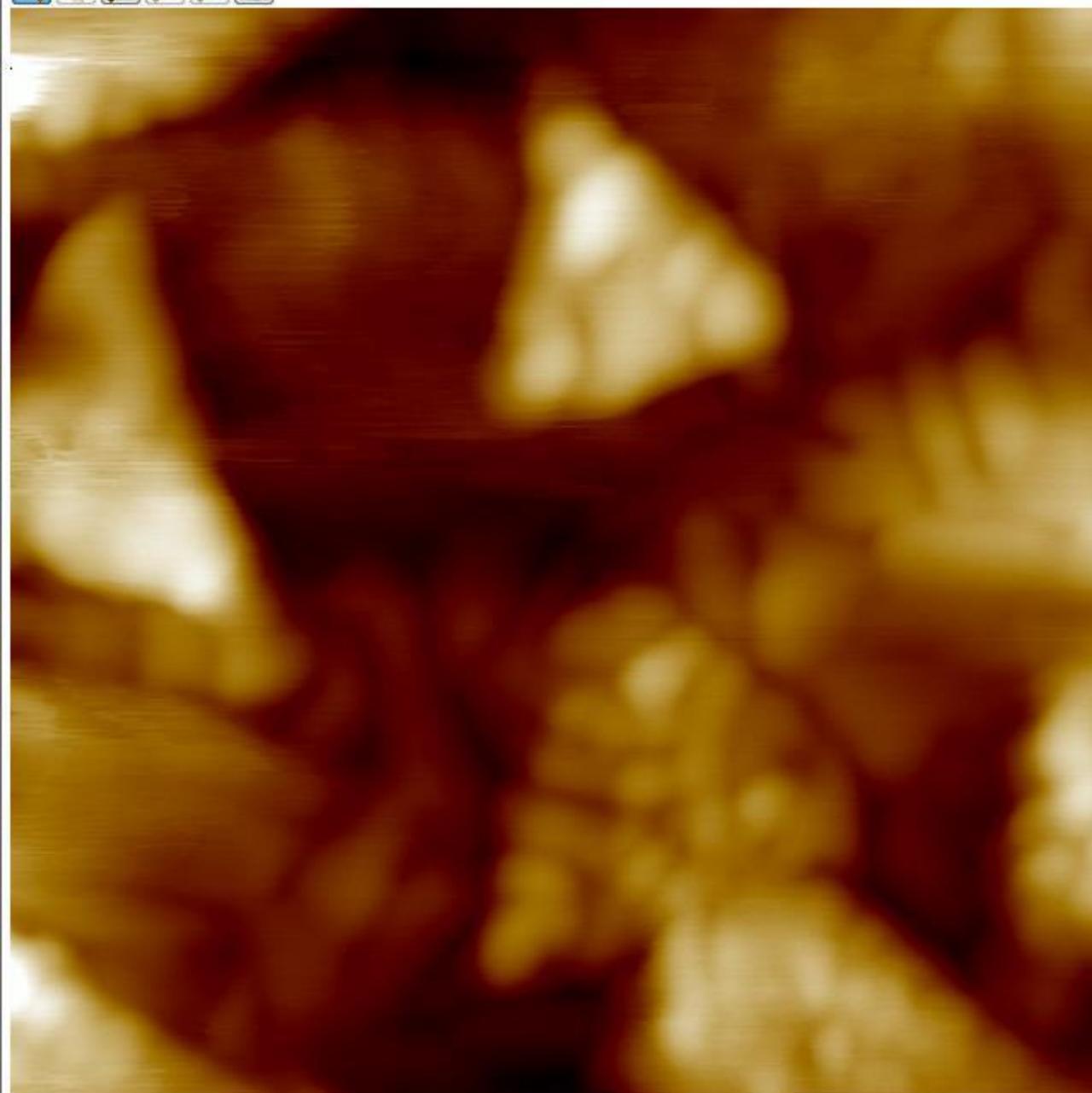
decapped final



## 240C monolayer 708b

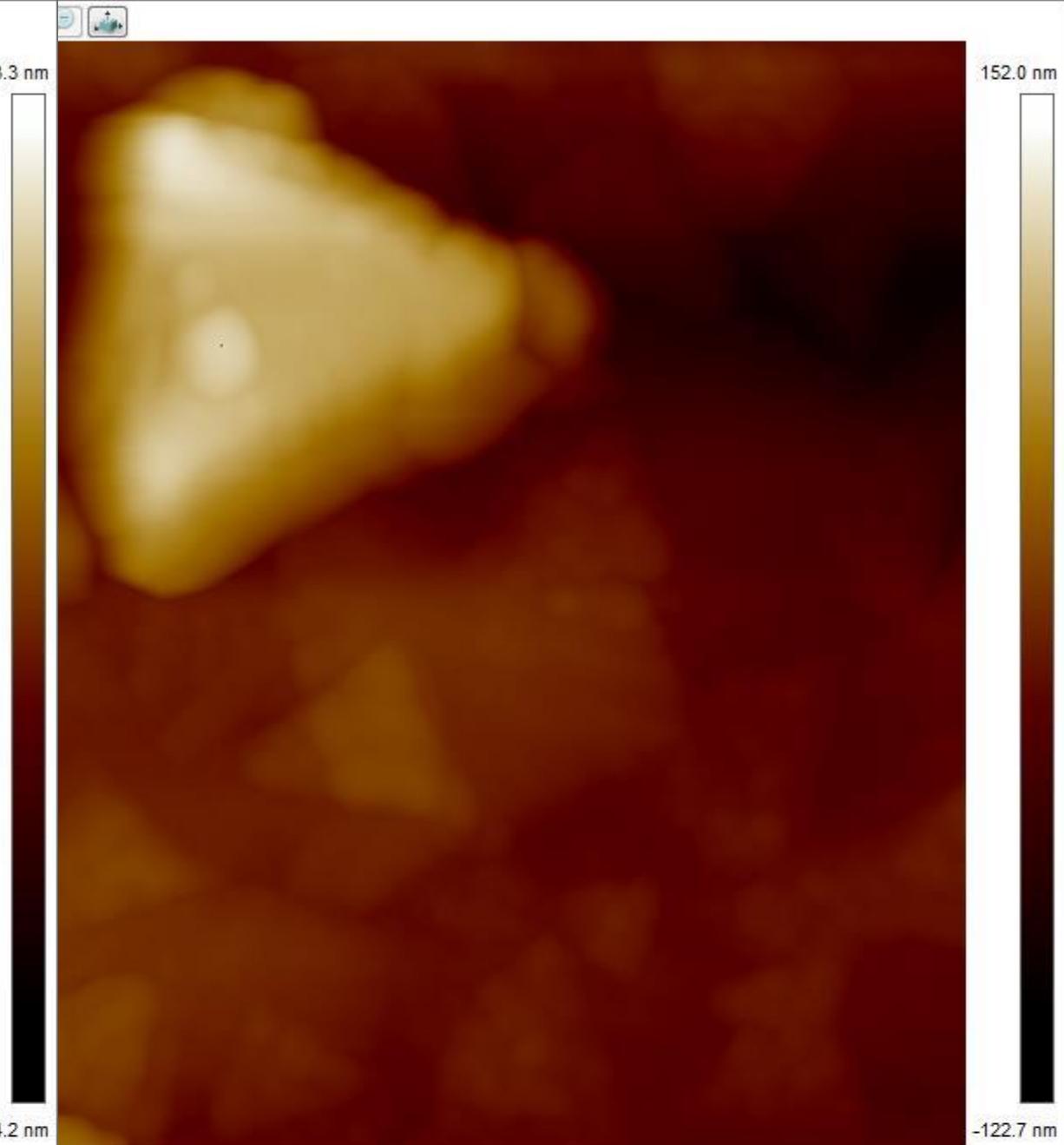
c=3.88A Te  
c=3.54A

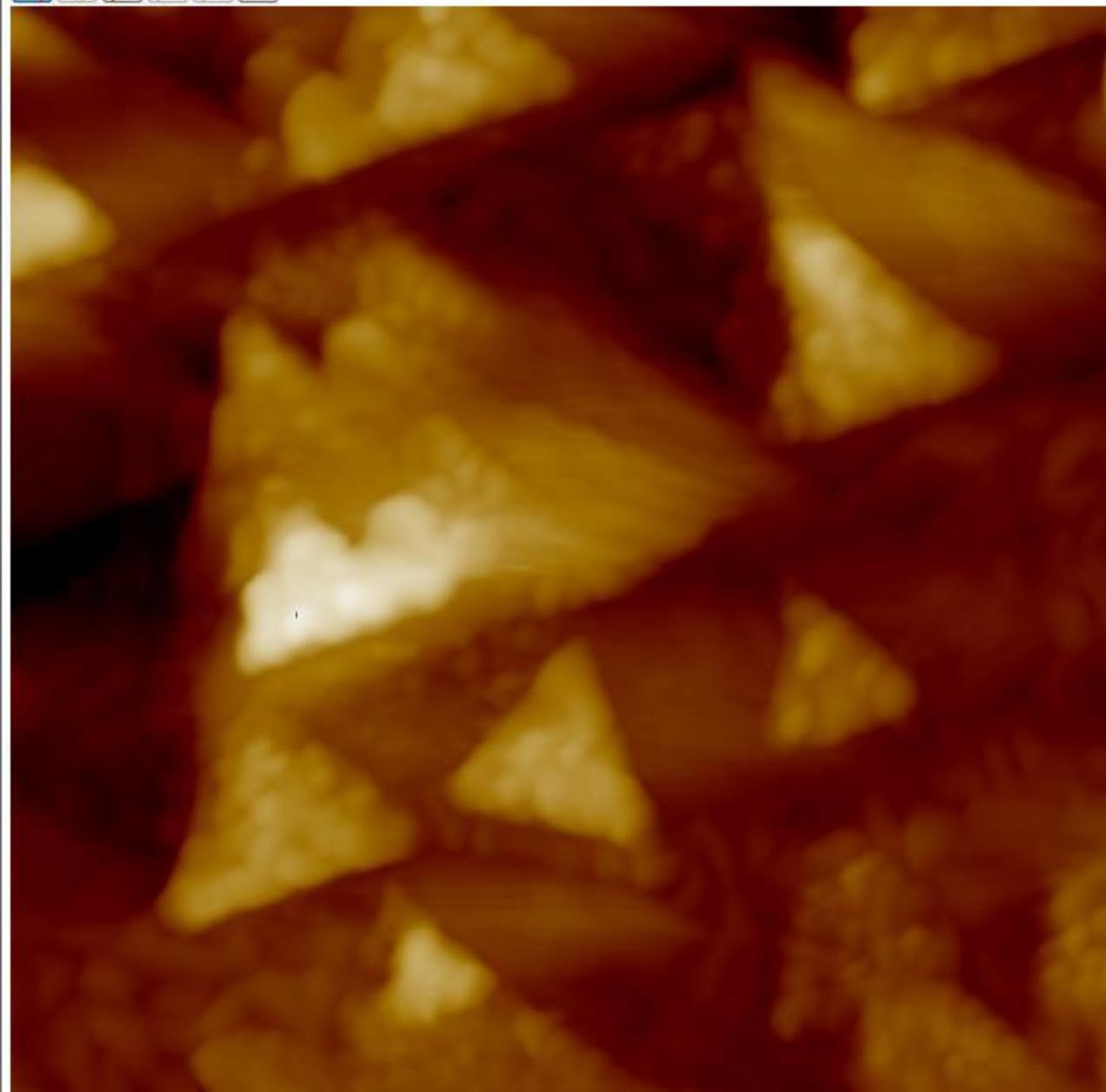




0.0

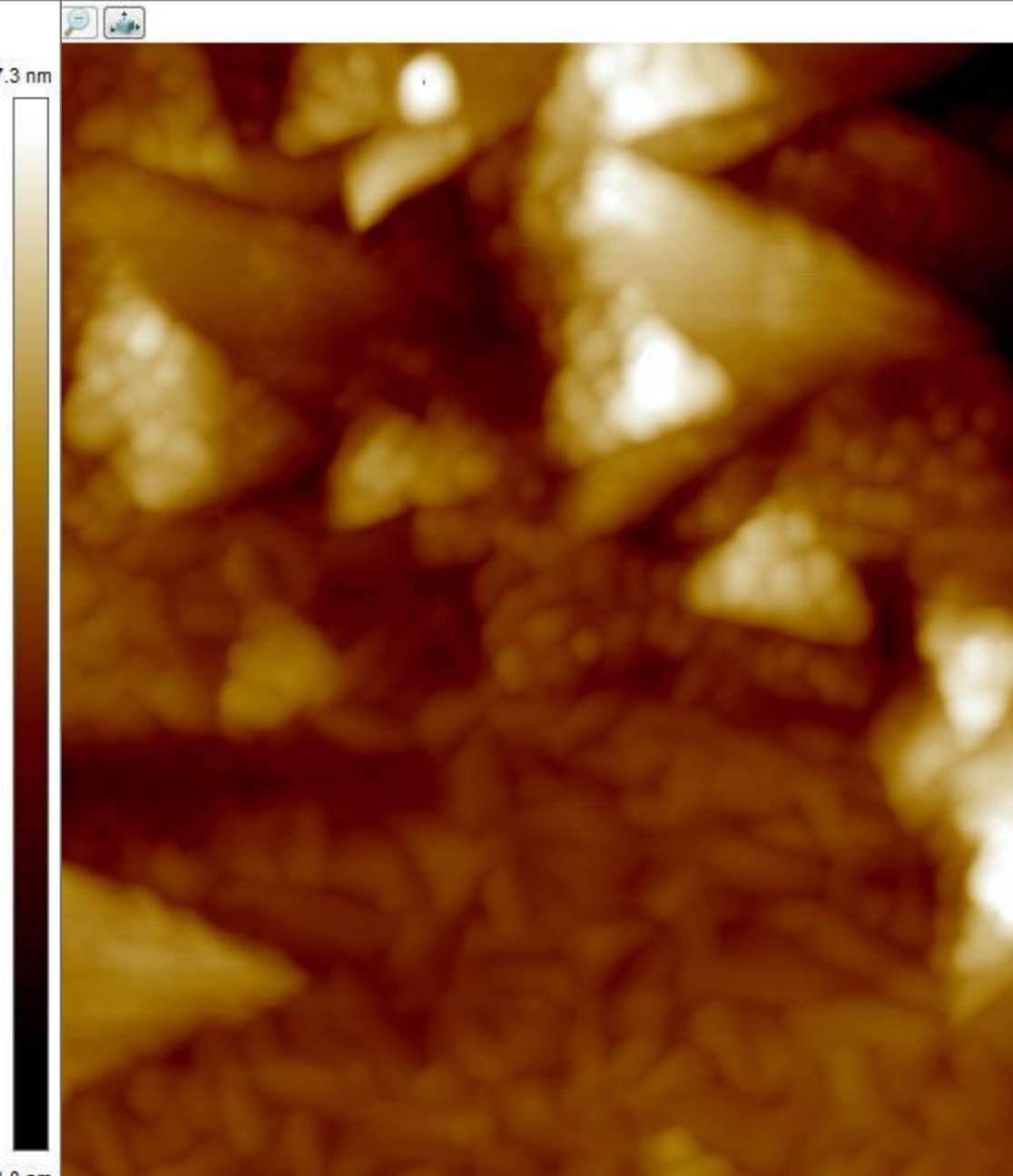
Height Sensor

1.0  $\mu\text{m}$ 2.0  $\mu\text{m}$



0.0

Height Sensor

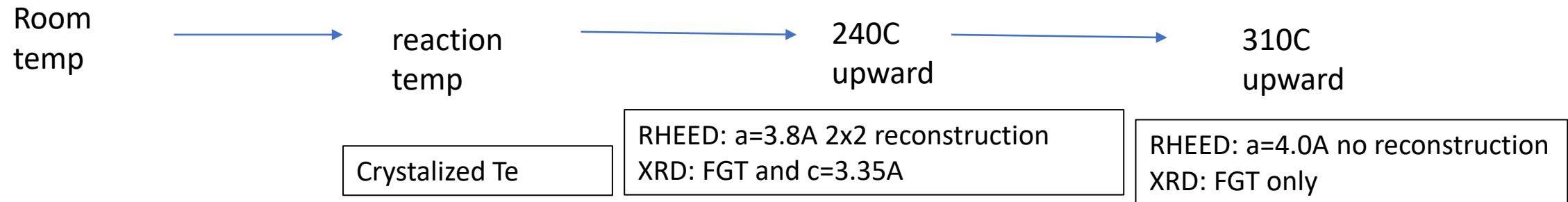
2.0  $\mu\text{m}$ 

Height Sensor

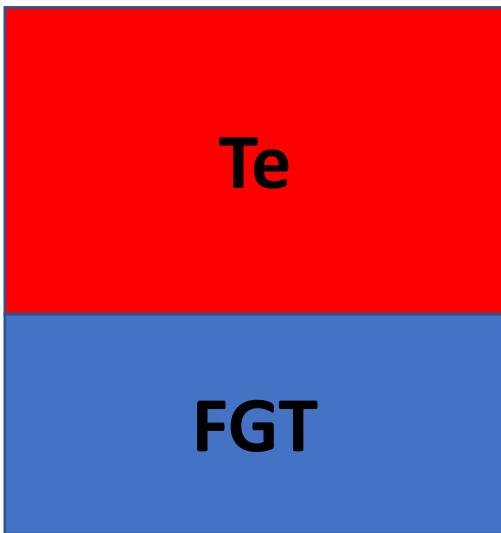
2.0  $\mu\text{m}$ 

27.1

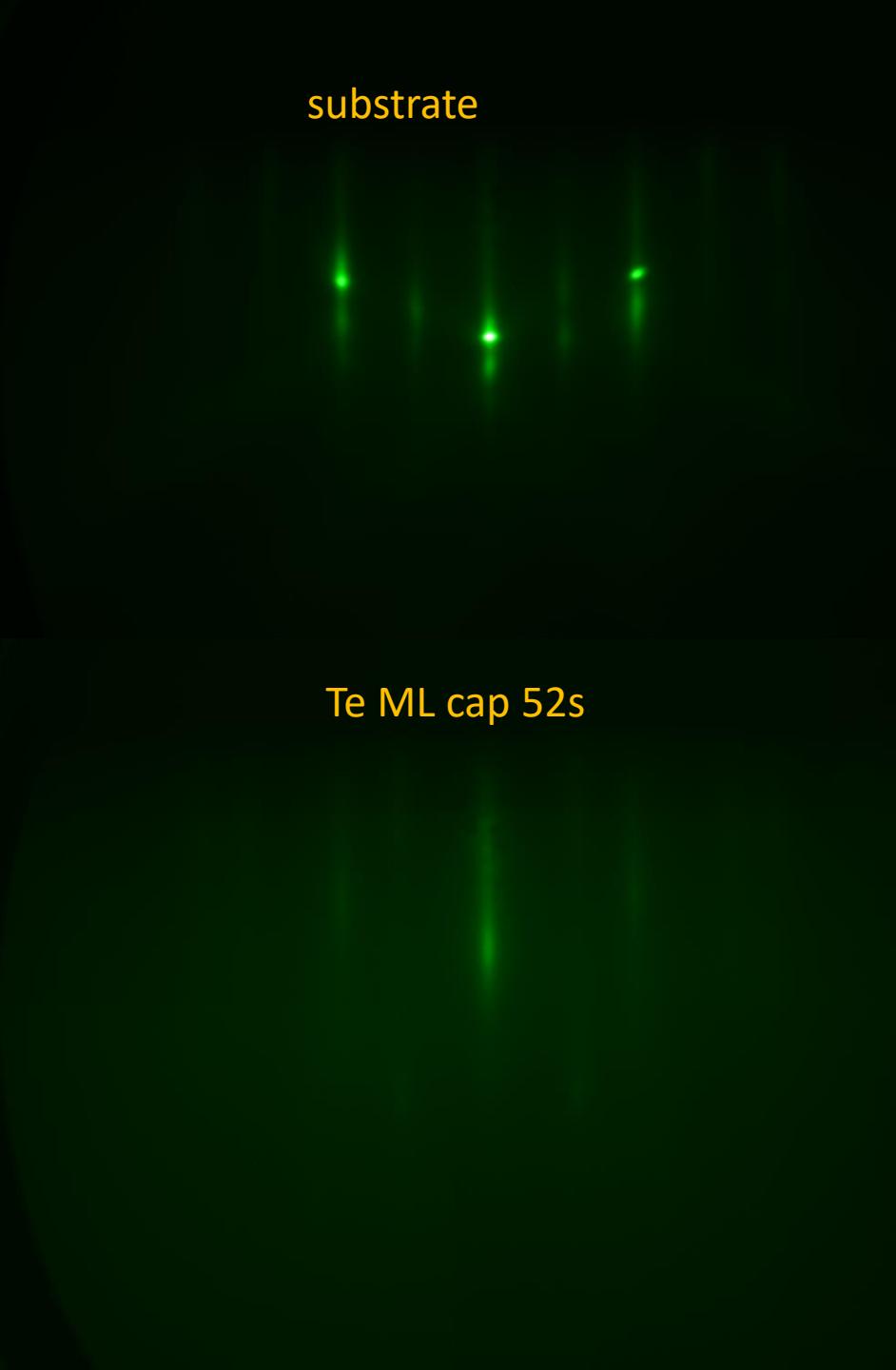
-29.8



## Speculation:

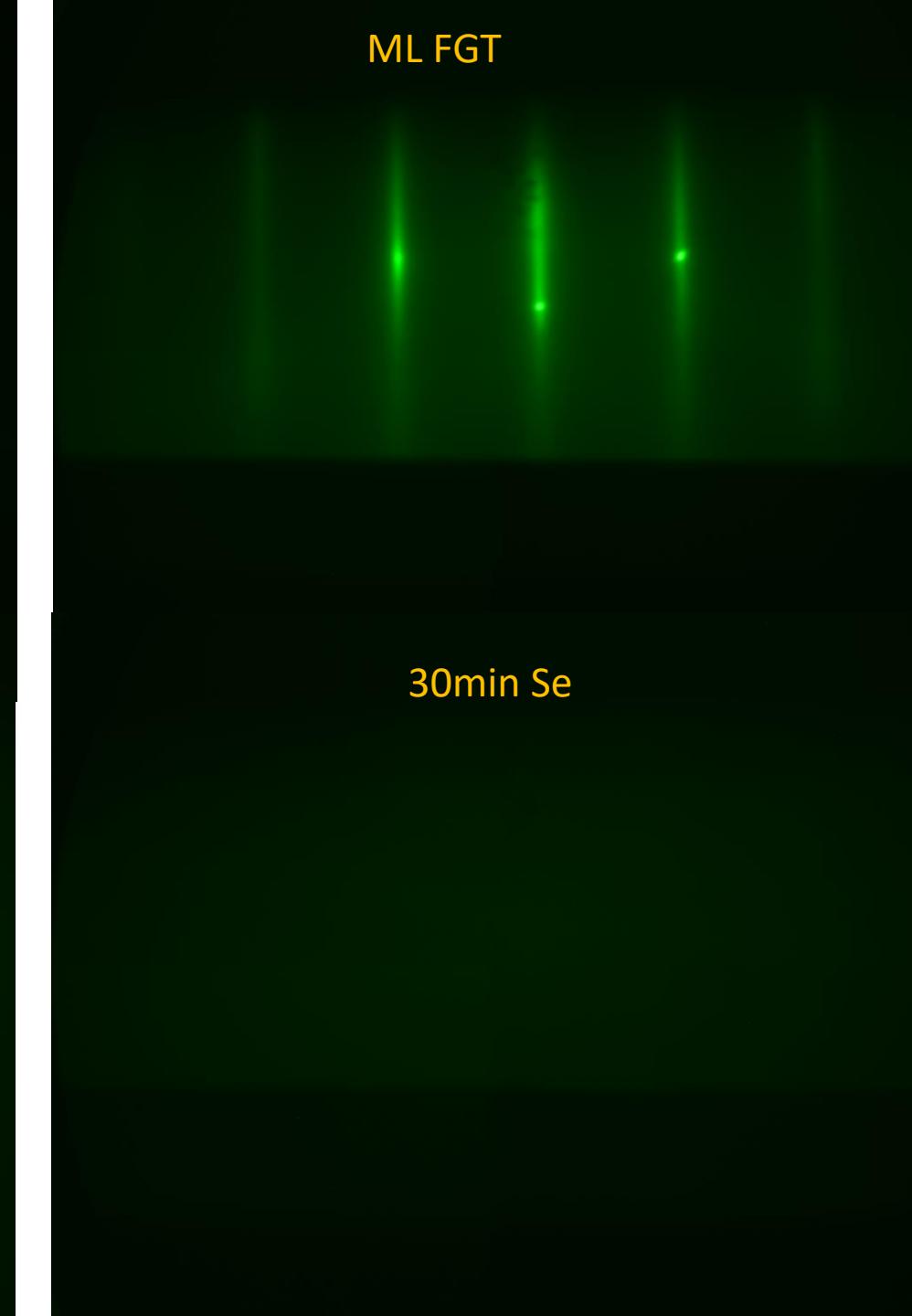


Ge-727a 1L FGT + 1.3L Te at r.t. 30 min Se  
@200. exposed to air. decapped



ML FGT

substrate



Te ML cap 52s



30min Se

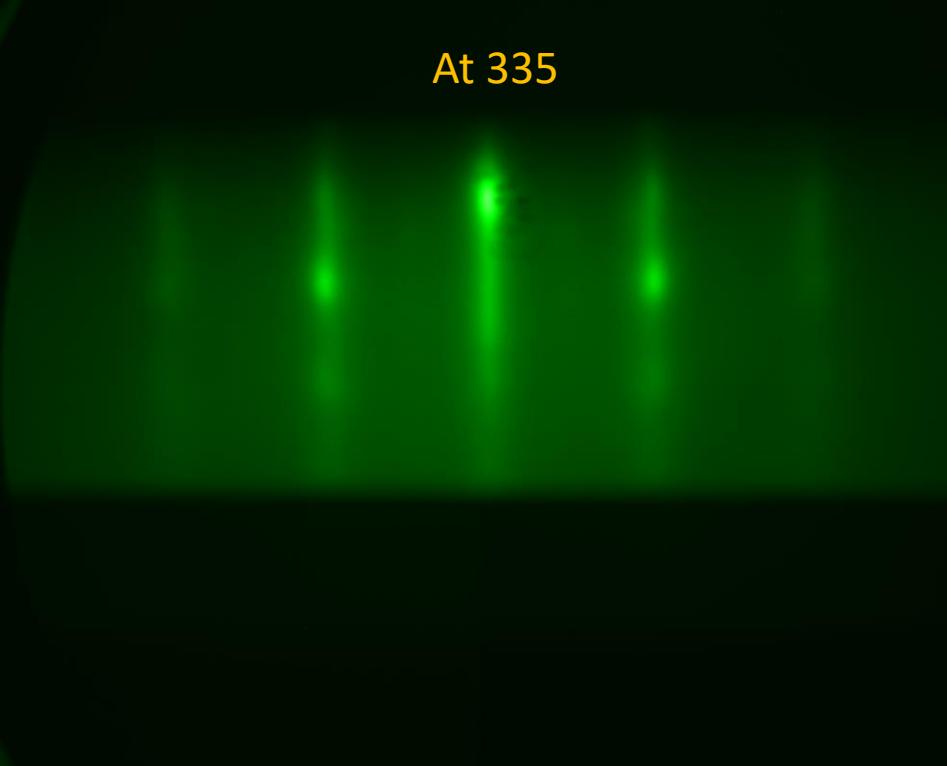
50 MIN AT 200

At 310

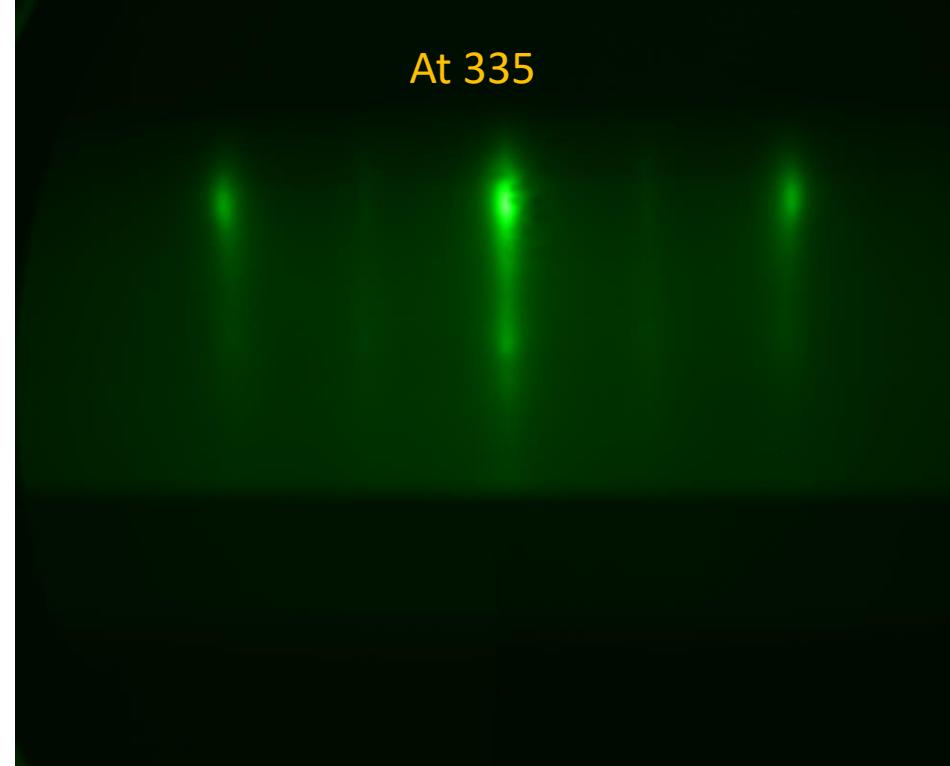
Another combined 80 mins to 230

At 335

At 335



At 335



Ge-729b 1L FGT + 1.3L Te at r.t. 24 min Se  
@200. decapped

after this we conclude ML FGT cannot be decapped

substrate

Te ML cap 48s

ML FGT

30min Se

30 MIN AT 240

4hr at 340

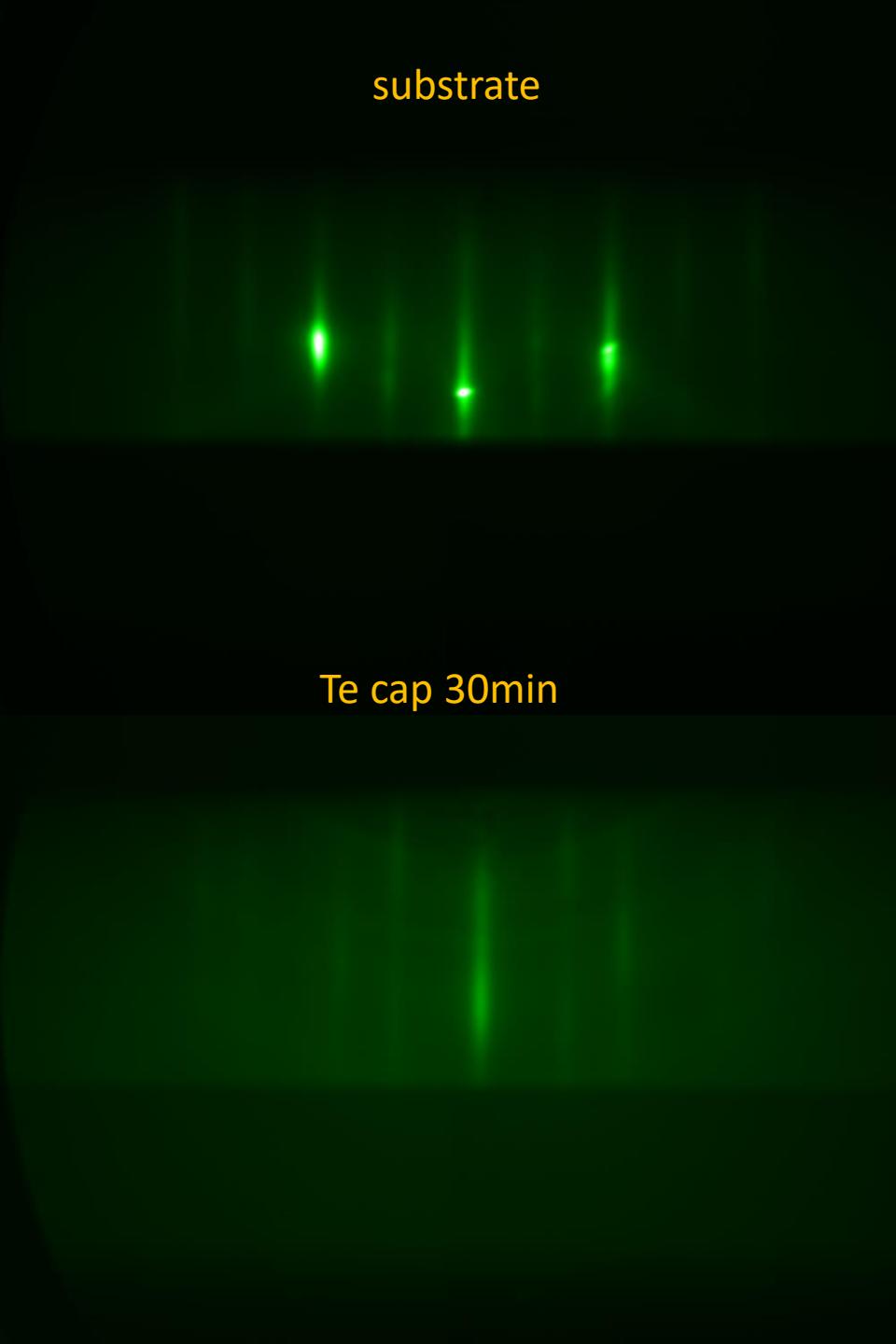
4hr at 310

Ge-805b 11L FGT + 30min Te at r.t. .  
decapped

substrate

Te cap 30min

10L FGT poor growth, LN2 empty midway through

A scanning electron micrograph (SEM) showing a substrate with three bright, sharp features. Below the substrate, there is a dark, textured region labeled "Te cap 30min". The overall image is dark with these specific features highlighted.

At 240

310

335

360

410

# Ge-805A 11L FGT 48s Te. Decapped and recapped

AFM suggests decapping was successful. Next steps is to investigate how many layers with transport and to try another sample with Se ontop

substrate

11L FGT

Te 48s at RT

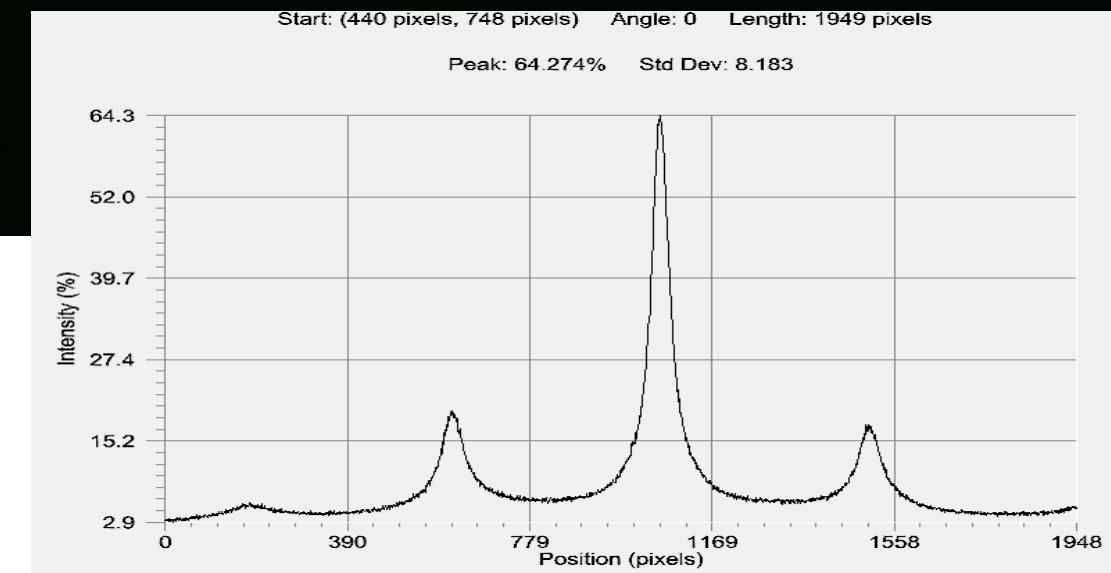
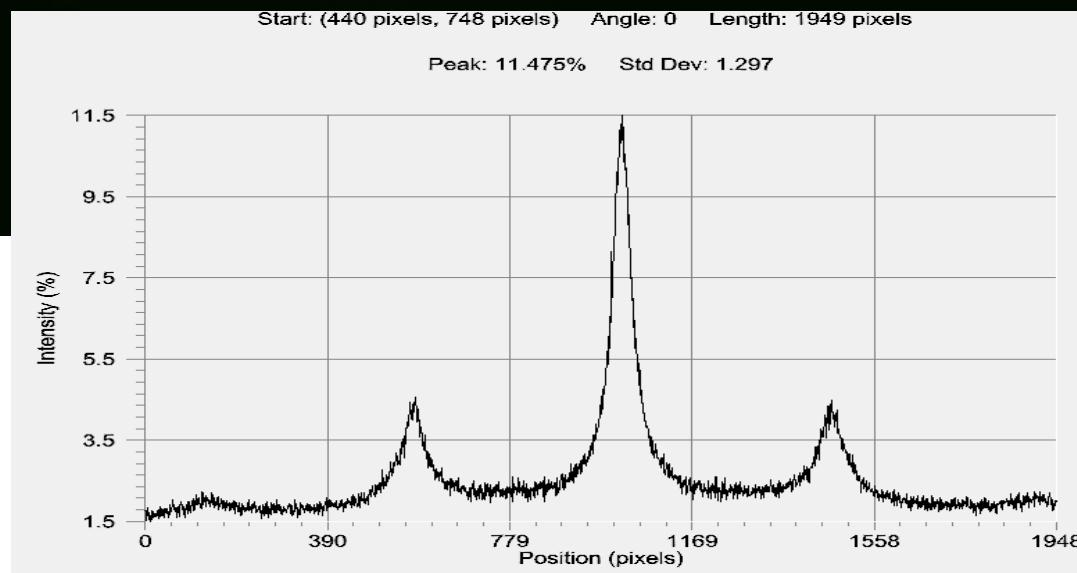
At 100

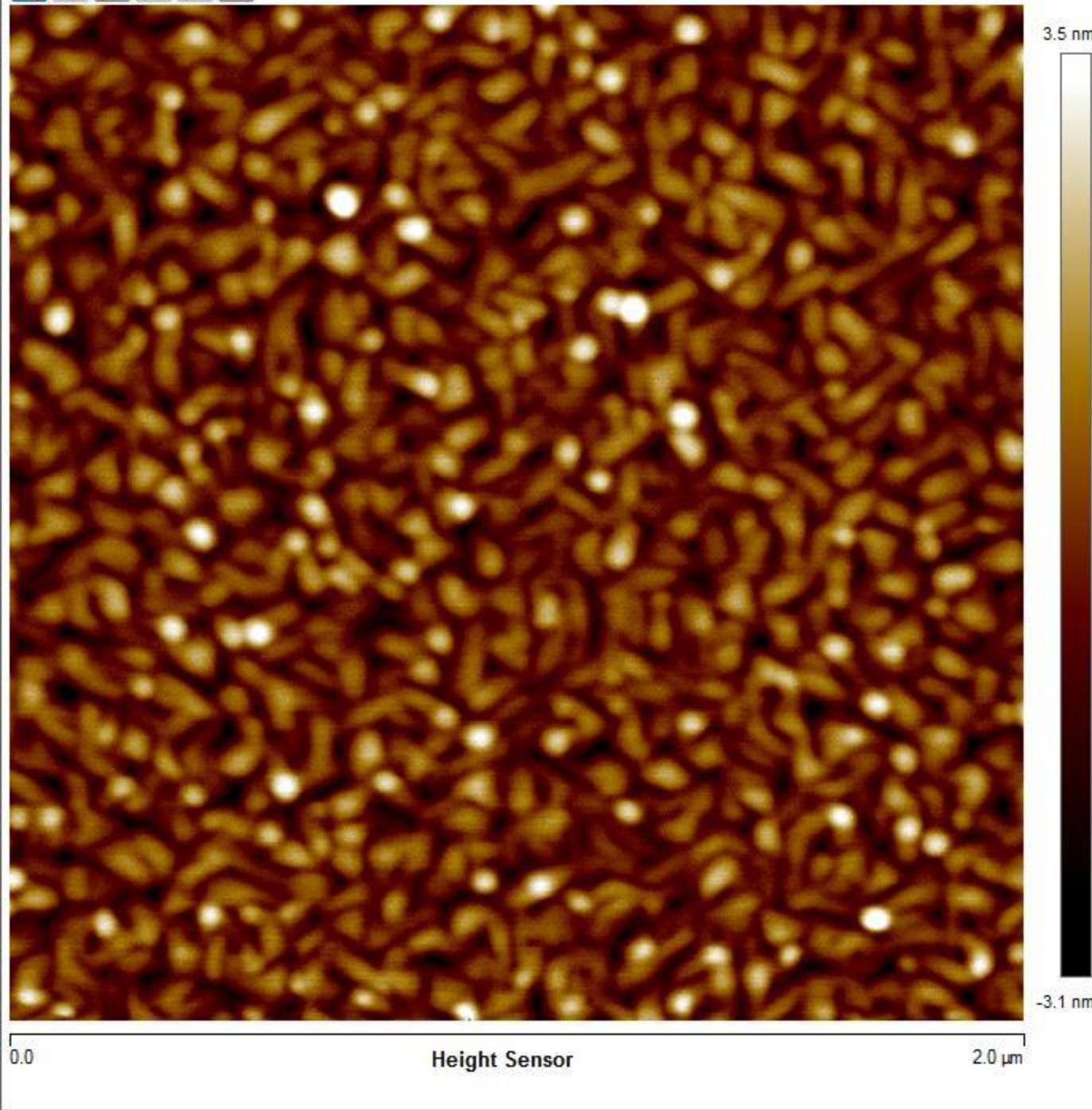
At 240 for 15min

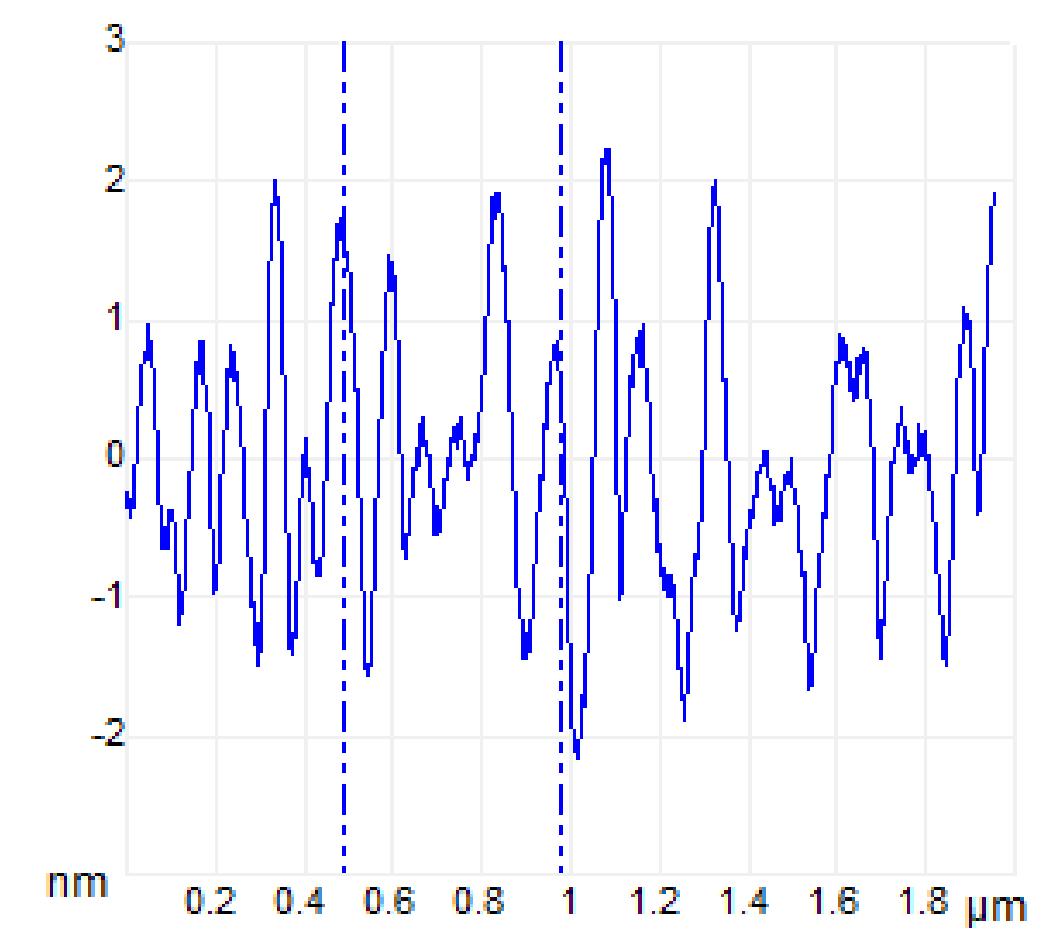
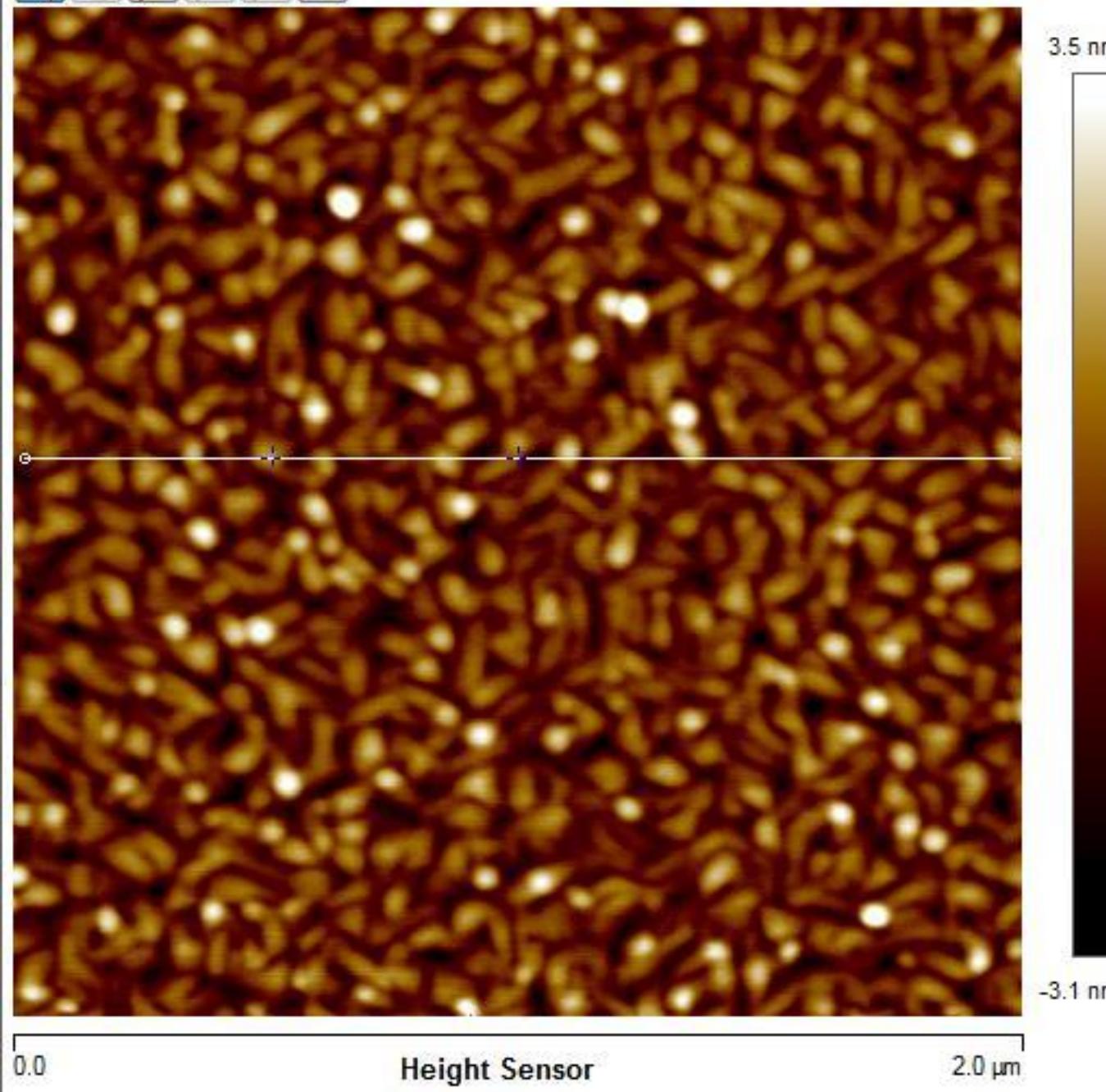
2hr at 310

decapped

Original FGT







Ge-811B 9L FGT 48s Te. 30 min Se. Exposed  
to air. Decapped.

Final attempt for 1L Te capping

substrate

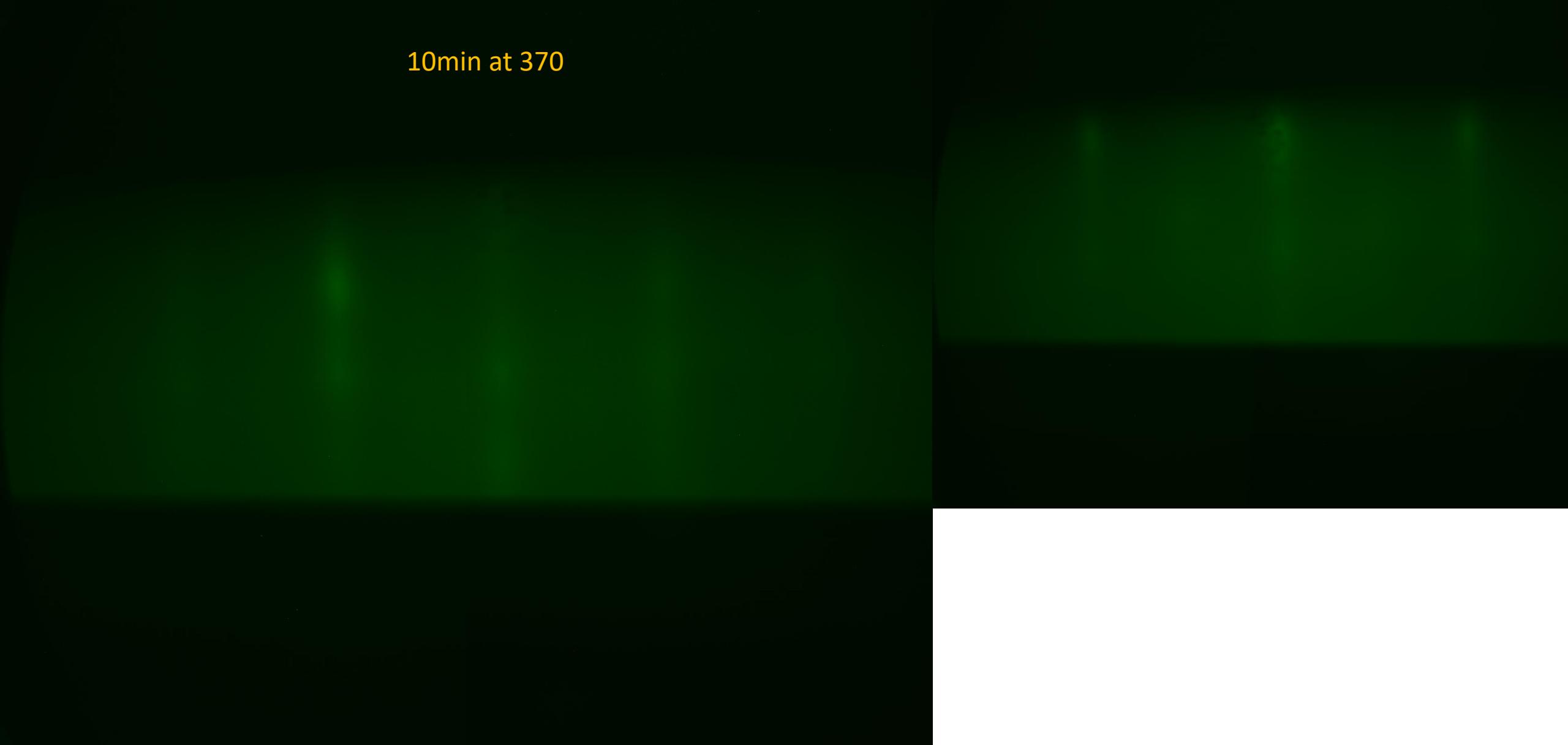
Te 48s at RT

9L FGT

Se 30min



10min at 370



Ge-817B 9L FGT 243s Te. 30 min Se. Exposed  
to air. Decapped.

substrate

9L FGT

Te 6L at RT

Se 30min

In air 25 min

20min at 210

20min at 230

10min at 245

15min at 310

20min at 340

Ge-817a 9L FGT 243s Te. 30 min Se.  
Decapped.

substrate



Te 6L at RT

9L FGT

Missing sorry, I automated it, so no rheed.  
Look at 817b for identical treatment, only difference was air exposure

Se 30min and overnight



Se cap

40min at 200

30min at 245

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20min at 275

---

10min at 310

---

20min at 345

# The conclusions

11L FGT + 30 minutes of Te  
805b

- Decapped to 4A at 410C with 3.8A 2x2 phase at 310C.
- We suspect the 310C phase is FT but did not have conclusive data.
- Because FGT decomposes at 360C we suspect the 410C phase is FG.
- This data shows thick Te cannot be decapped, and it is also interesting that the Te->930pix 2x2 -> 890pix Heating sequence also happens for the thinner Te caps. This does not necessarily mean the 890pix phase cannot be FGT for thin layer Te, but it does suggest it may not be, instead this same phase we get on thick Te.

Selenium Monolayer FGT + NoTe decapping		yes	Yes+air exposure
708b 240C: 890pix 'FGT'  AFM shows triangles up to 30nm above the surface  <b>Decapping a monolayer FGT to 240C creates a 3D structure.</b>	729b  Blurry at 240C  340C: 935pix 3D and 2x2	727A  Blurry at 240C  335C: 933pix 3D and 2x2	  <b>Se leakage affects FGT and creates new crystal. The loss of coherence in rheed at 240C is also present here as 6L Te. The trials seems identical.</b>

-Clearly the Se does not work and reacts with FGT.

-708b show that either the reconstruction pattern creates triangle islands, or decapping 1L FGT creates a 3D phase and we can't decap monolayers.

-It is possible all the 2x2 930pix phases look like triangle islands in AFM.

-If you have some other breakthrough related to decapping, try heating 1L FGT + 1L Te to 310C and see if the AFM still has islands. OR do AFM on the 2x2 930pix reconstruction phase on thick FGT + 1L Te and see if it looks the same. I theorise the triangles are actually caused by us looking at the 930pix 2x2 phase, not the FGT being monolayer.

~9L FGT + 1L Te decapping

Selenium?	No	yes	Yes+air exposure
	<p>805a 705a 310C: 890pix 'FGT' AFM suggests decapping was successful</p> <p>In comparison to the same trial with 1L FGT it seems thicker FGT preserves the surface under these conditions</p>	untried	<p>811b 370C: <u>*check the rheed spacing on the computer, I am guessing it is the 2x2 960pix phase but didn't check before I left.</u></p> <p>Se reacted with the FGT and formed the same phase as the thicker Te trials.</p>

-again on closer inspection it seems the AFM done on 708b was of the 240C phase, not the 310 one. This might mean that although it appears thicker FGT preserves the surface, it may be a property of the 240C V.S. 310C phase.

-we did not try the two other experiments because we expect them to go the same as the symmetrical trials on 1L FGT so now the cap needs to be thicker.

~9L FGT + 6L Te decapping

Selenium?	No	yes	Yes+air exposure
	<p>624a+b 245C: 930 '2x2 flat' No 310C data!</p> <p>This could preserve FGT, we moved onto Se trials because 6L will not protect surface alone so the results are irrelevant and we expect it to decap in the same pattern the 1L Te 805a did.</p>	<p>817a</p> <p>At 200C: Te signature with some amorphic noise At 245: Te signature is weaker, more amorphous At 310C and 345C: 3D 960pix 2x2 phase</p> <p>Se is still getting through 6L, in FG trial with Se and thick Te cap we did not have the return to amorphousness at 245C which suggests Se penetration through 6L</p>	<p>817b</p> <p>At 200C: Te signature with some amorphic noise At 245: Te signature is weaker, more amorphous at 310C and 340C: 3D 960pix 2x2 phase</p> <p>Se is still getting through 6L, in FG trial with Se and thick Te cap we did not have the return to amorphousness at 245C which suggests Se penetration through 6L</p>

-it looks like 6L Te is still too thin to keep out Se. Given that thick Te didn't work, we could continue to increase the Te thickness to see if we can find an integer low enough to decap, but doesn't admit Se.  
-Still, we don't know if the 310C phase is even FGT, but if you were able to decap to the phase with Se ontop by increasing layers, we could do ARPES to determine if it is FG, FGT or others.

If someone is going to pick up this project we can know this for sure:

- GeTe cannot be decapped under 360C.
- Thick Te (30mins at 336C) cannot be decapped under 360C.
- Thin Te caps under 7L do not keep Se off FGT.
- I do not think we can say for certain that monolayer FGT behaved differently than thicker layers for Te decapping because again that AFM was of the 245C 2x2 3.8A phase, not the final 310C 4A phase we looked at for thicker FGT. This could be determined easily with AFM of either the 245C phase on thick FGT or the 310C phase of monolayer FGT.