Pits created in the lab on welded niobium coupons

10/8/08

Lance Cooley

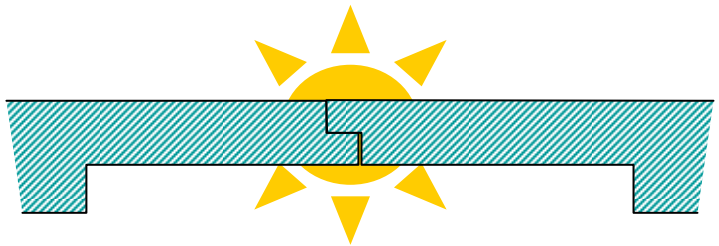
Acknowledgments

- Welding – Mike Foley, Sciaky
- EP – Charlie Cooper, Donna Hicks, Dave Burk, Rob Schuessler, Chad Thompson
  - 6 hours in sweaty chemistry suits…
- Photos – Dave Burk, Chad Thompson, Dmitri Sergatskov, Charlie Cooper, Rob Scheussler, Lance Cooley

Overview

- Corners of Nb sheets used in 9AES001 were used to simulate an equator weld
- They were machined to same contour as equator joint and given same weld prep, then welded
- After welding, inspection did not reveal defects
- EP was done in a lab beaker setup that attempted to use same parameters as was used on 9AES001
- Defects (pits) were observed on both coupons after EP
- The defects were found to have common features

Weld info



- Welds done by Mike Foley at Sciaky
- Weld parameters were as close as possible to actual parameters used on 9AES001
- 3 passes: tack, seal, full penetration

Equator Sample Weld Parameters

All four weld samples were prepared with two tack welds (one tack weld at each end of the joint), followed by a seal weld pass to get heat into the pieces to be joined. The tack and seal weld parameters for all four samples were:

Tack Weld

50 KVolts
20 mA
Focus 365 (~ 0.5 inches above joint)
30 inches per minute (ipm) material feed rate

Seal Weld

50 KVolts
25 mA (1.0 sec dwell) followed by 25 mA for weld
Focus 376 (~ 1.5 inches above joint)
18 inches per minute (ipm) material feed rate
E-beam oscillating on 0.040" diameter circular path at 100 Hz puddle frequency (PF)

The parameters for the final weld pass on each sample were:

SAMPLE	1	2	3	4
EB voltage	50 KV	50 KV	50 KV	50KV
EB current	45 mA (1.0 sec dwell) 44 mA for weld	43 mA (0.5 sec dwell) 43 mA for weld	43 mA (0.5 sec dwell) 41 mA for weld	43 mA (0.5 sec dwell) 43 mA for weld
Focus	378 ~ 1.8" above joint	378	378	378
Feed Rate	18 ipm	18 ipm	18 ipm	18 ipm
Oscillation	E-beam oscillating in 0.050" diameter circular path at 100 Hz PF			
Vacuum	3.1 x 10^{-5} Torr	3.5 x 10^{-5} Torr	2.0 x 10^{-5} Torr	4.2 x 10^{-5} Torr
Temperature	~ 123^{0} F	~ 106^{0} F	~ 112^{0} F	~ 98^{0} F

Each sample was allowed to cool down for 10 minutes under vacuum. The weld chamber was then backfilled with nitrogen to a level of ~ 25 Torr and the sample was allowed to cool for another 10 minutes. The temperature of the part was recorded immediately after the weld chamber was opened to atmosphere.

EP details – caution! System not optimized

Both samples

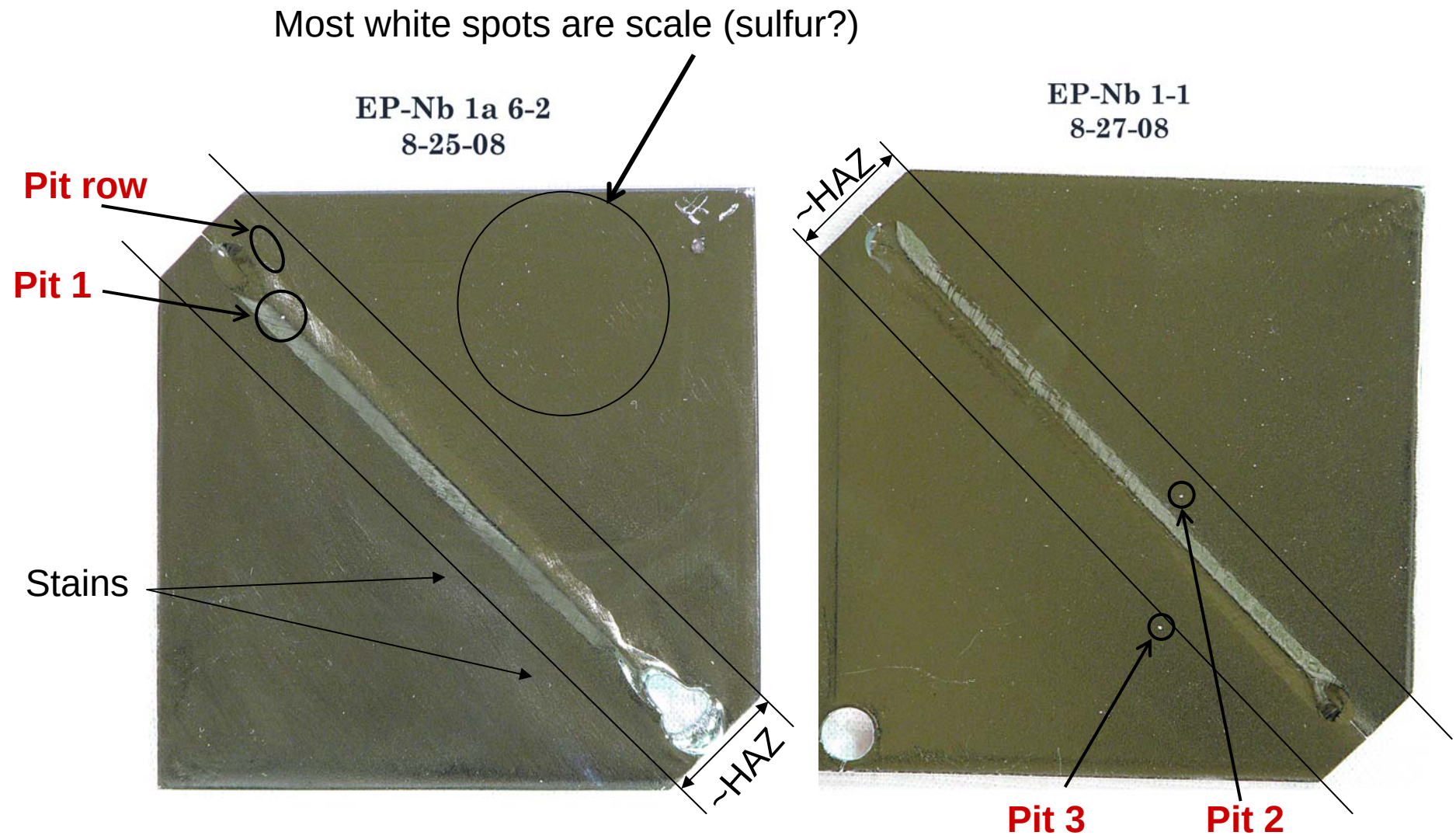
- Nb and Al parallel plates in a beaker, stir bar at the bottom. Beaker then sat in a chilled water bath.
- Niobium wire used to suspend niobium sample – wire etched through several times
- Weld side faced Al cathode; both sides were exposed
- Temperature not under control
- Current oscillations at fixed voltage once plateau reached

Sample 1A 6-2

- 210 µm total removal in 6 hrs (0.6 µm min^{-1})
- 14.5 V, not well controlled at first
- 2.3 to 3.2 A (60-80 A cm^{-2})
- Temp rose 18 to 32 °C

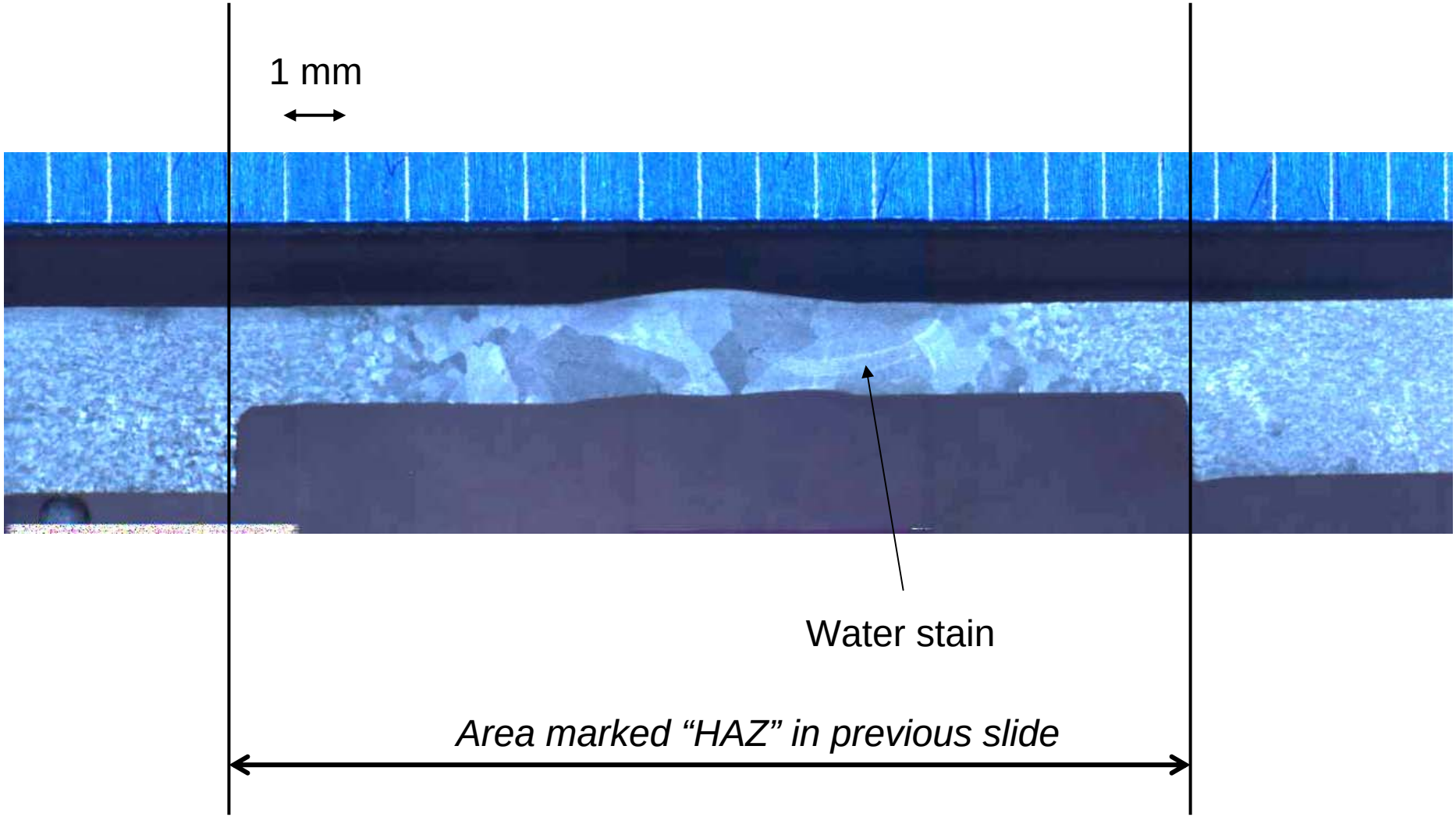
Sample 1-1

- 110 µm total in 3.5 hrs (0.5 µm min^{-1})
- 14.5 V, 2.3 to 3.2 A
- 17 to 37 °C



- *210 total µm removed*
- *110 total µm removed (total)*

Cross-section of the “HAZ”



Pit 1

Pit 1 is a whopper on the weld – 50 µm deep!!
Profile is across red line
Imaged using Keyence 3D microscopy

Height:
50
Reset
Display:
Higt/Clr
Higt
Clr

Profile measure

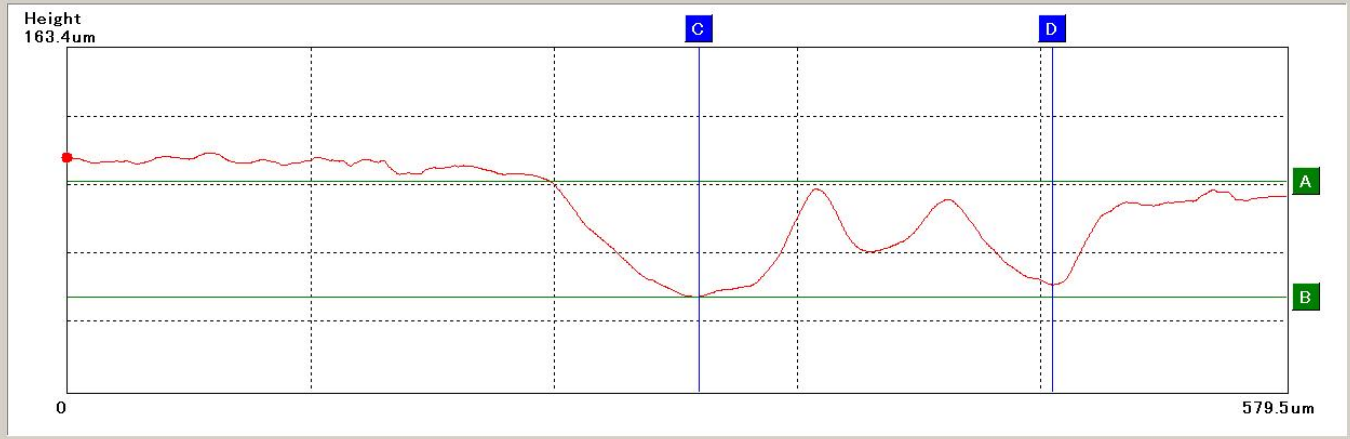
Record/Measure equipment: VHX
Save Date: 08/28/2008 02:37
Lens power: X 500
3D Record interval: 4.00 [um]
No. of record images: 40

Profile mode
Horizontal line
Vertical line
Specify 2 point

2 line compare mode
Range cursor[C-D] disp
Color Setting

Inclination correct
Auto
Manual
OFF

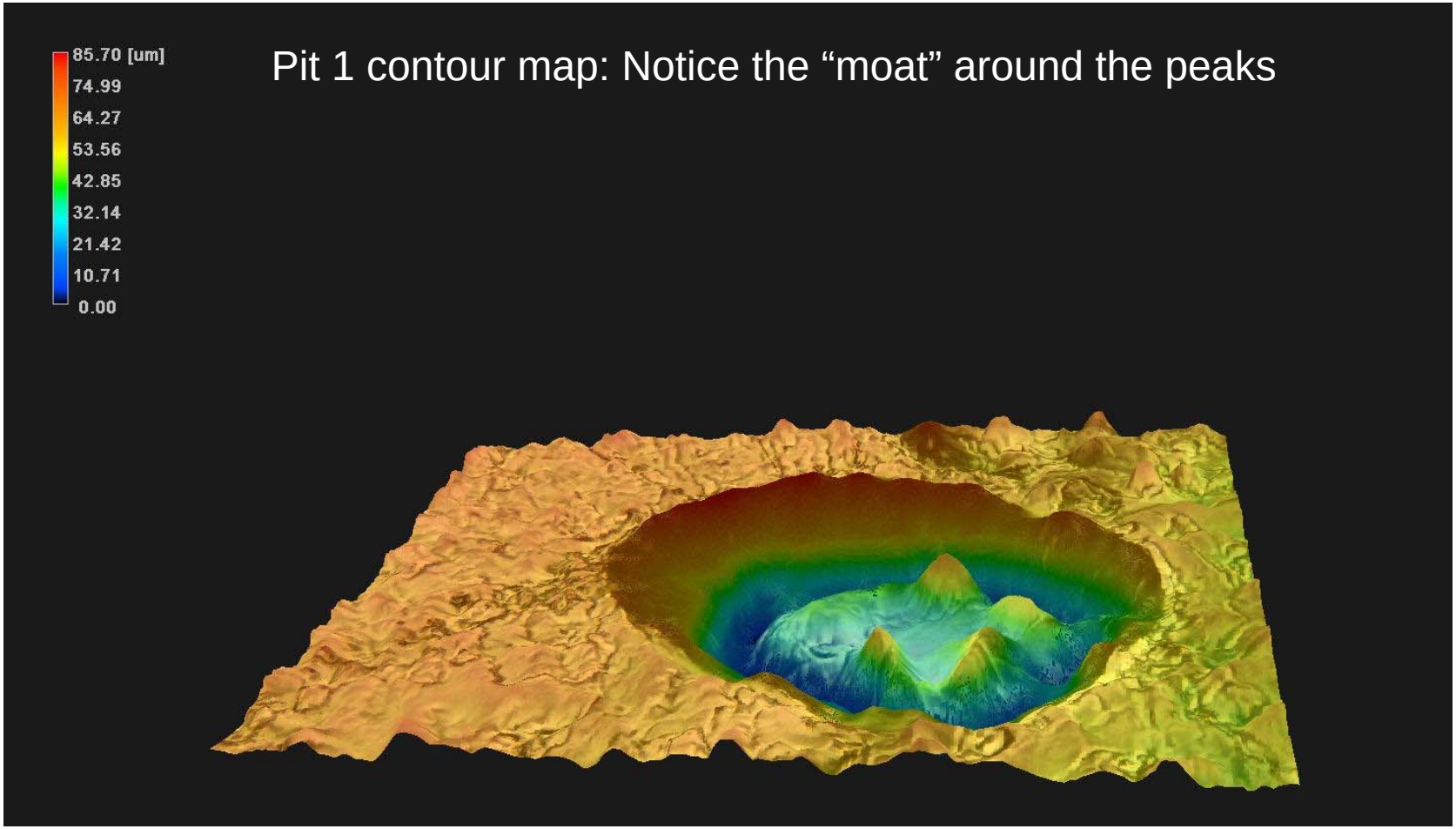
Height fit display



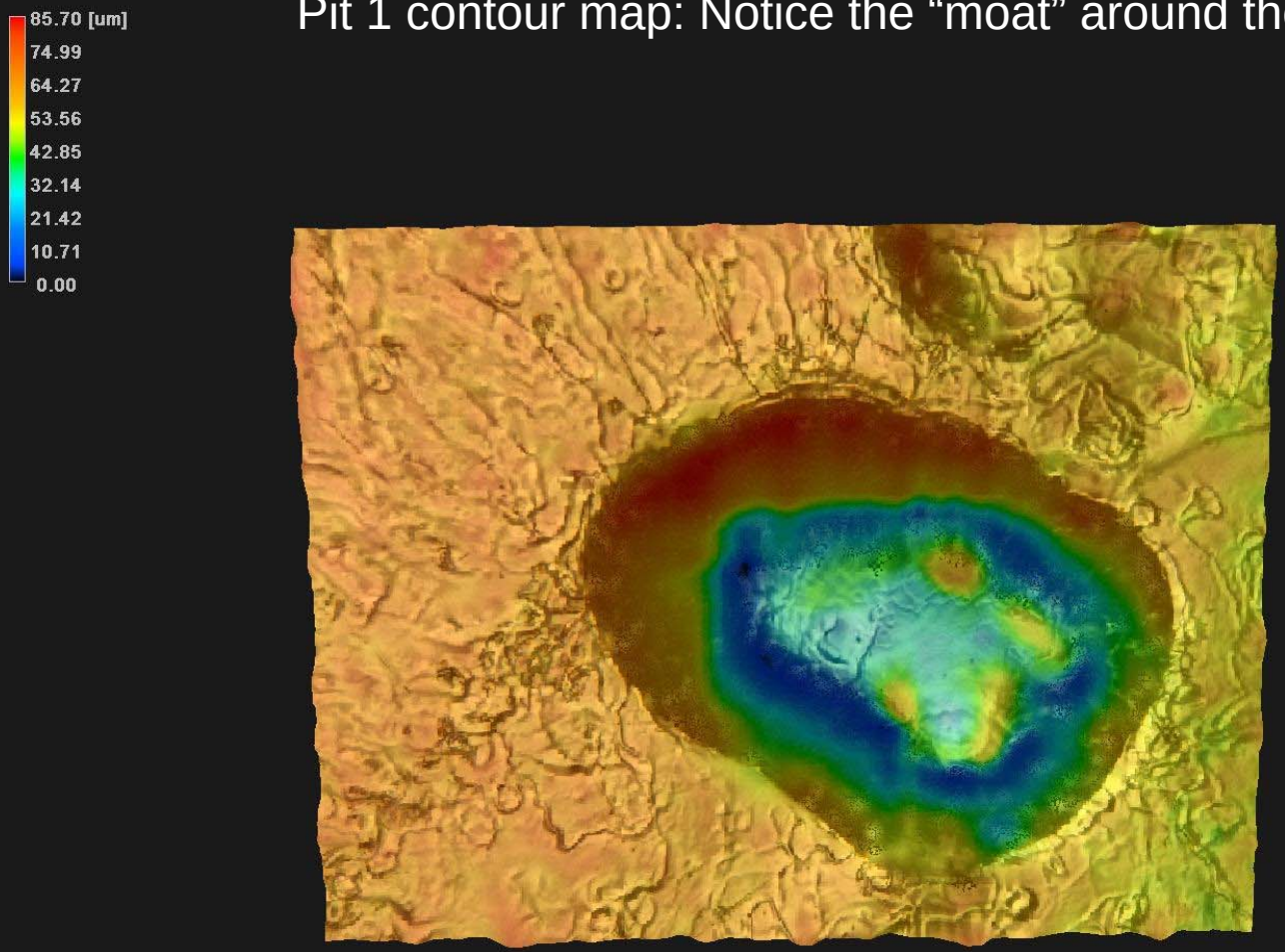
Height [A-B]: 55.0 µm
Width [C-D]: 168.0 µm
Cursor value: Add list

Height [A-B]	Width [C-D]
77.1 um	154.1 um

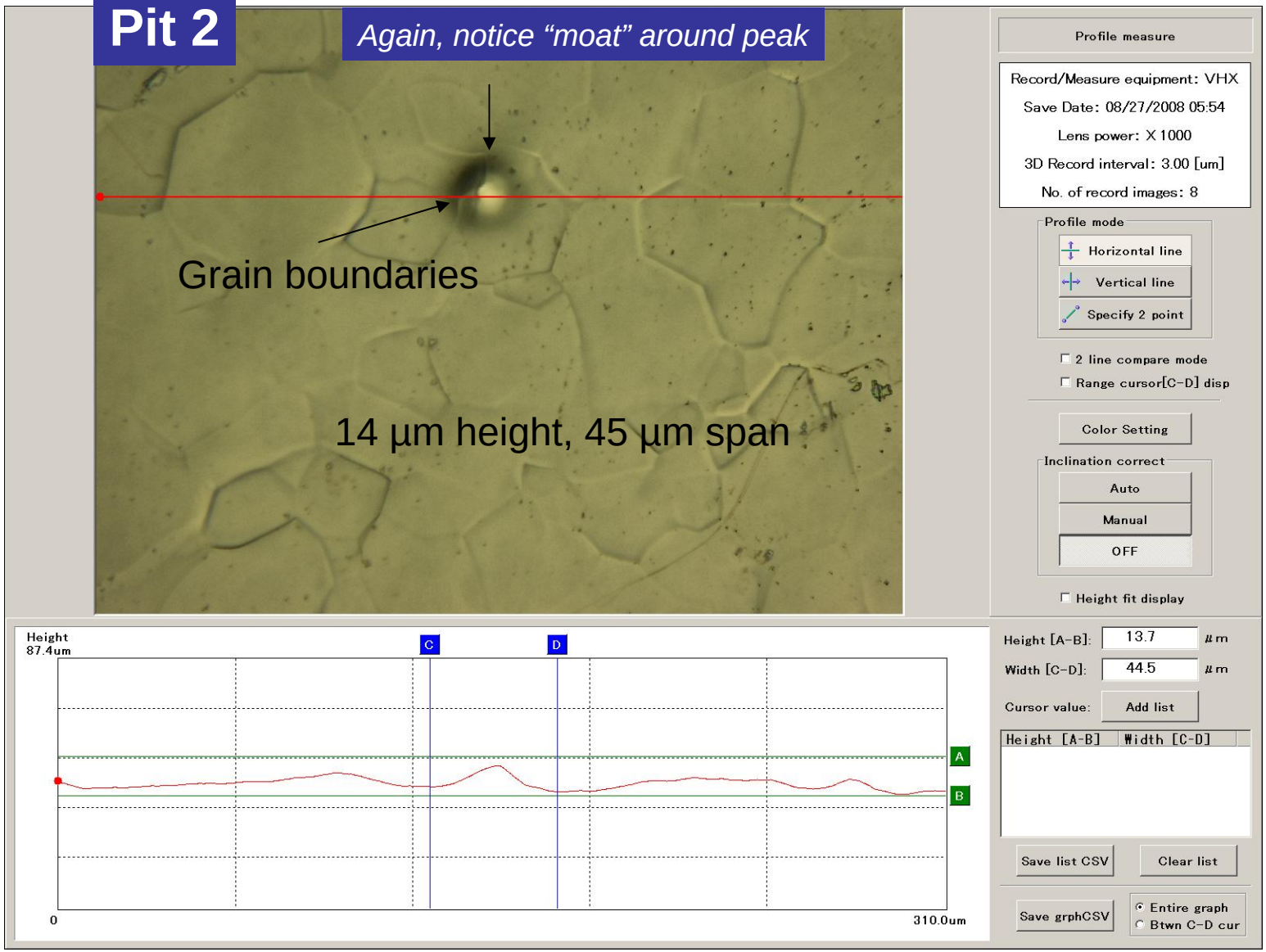
Save list CSV
Clear list
Save grphCSV
Entire graph
Btwn C-D cur


85.70 [um]
74.99
64.27
53.56
42.85
32.14
21.42
10.71
0.00
Pit 1 contour map: Notice the “moat” around the peaks

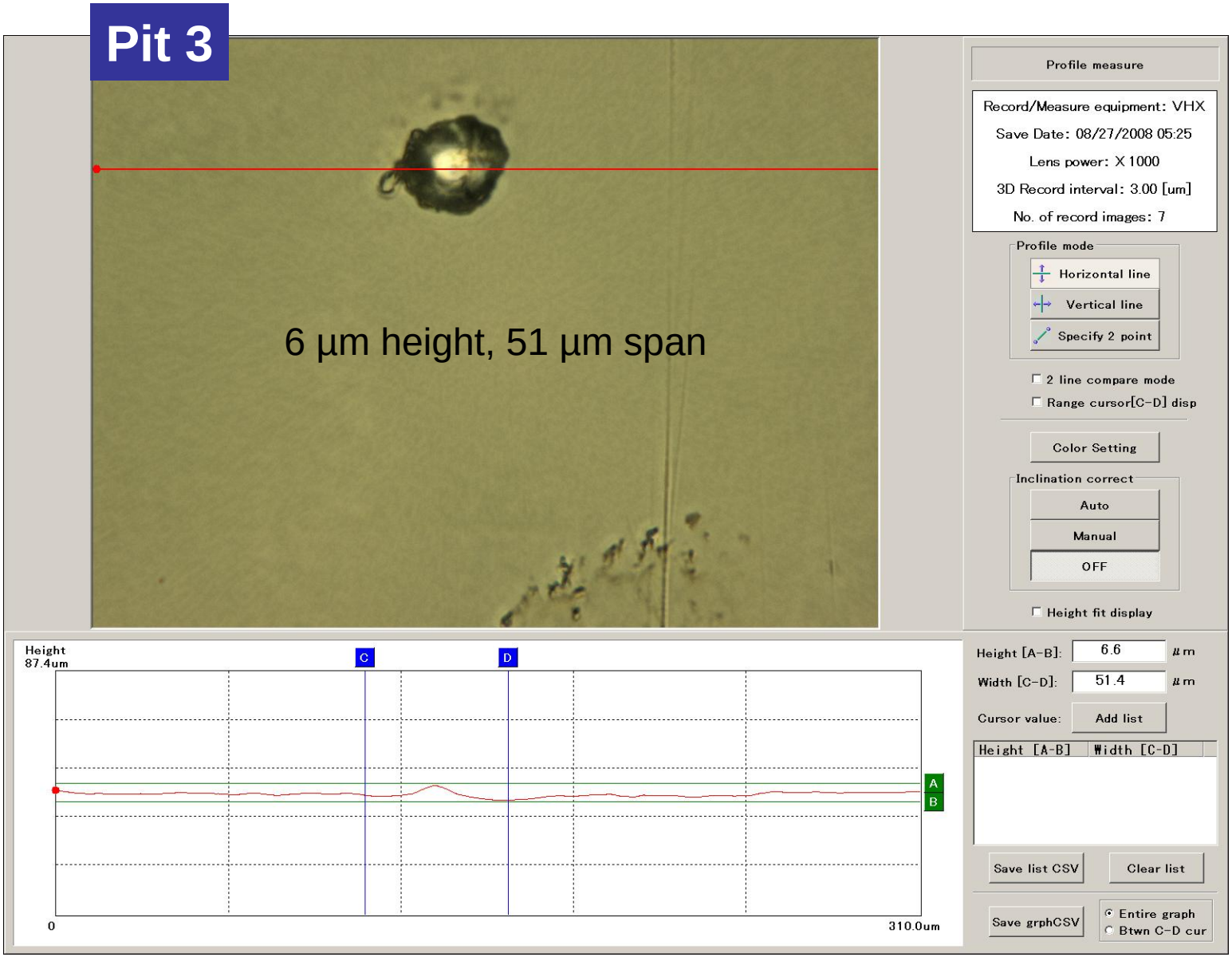
Pit 1 contour map: Notice the “moat” around the peaks



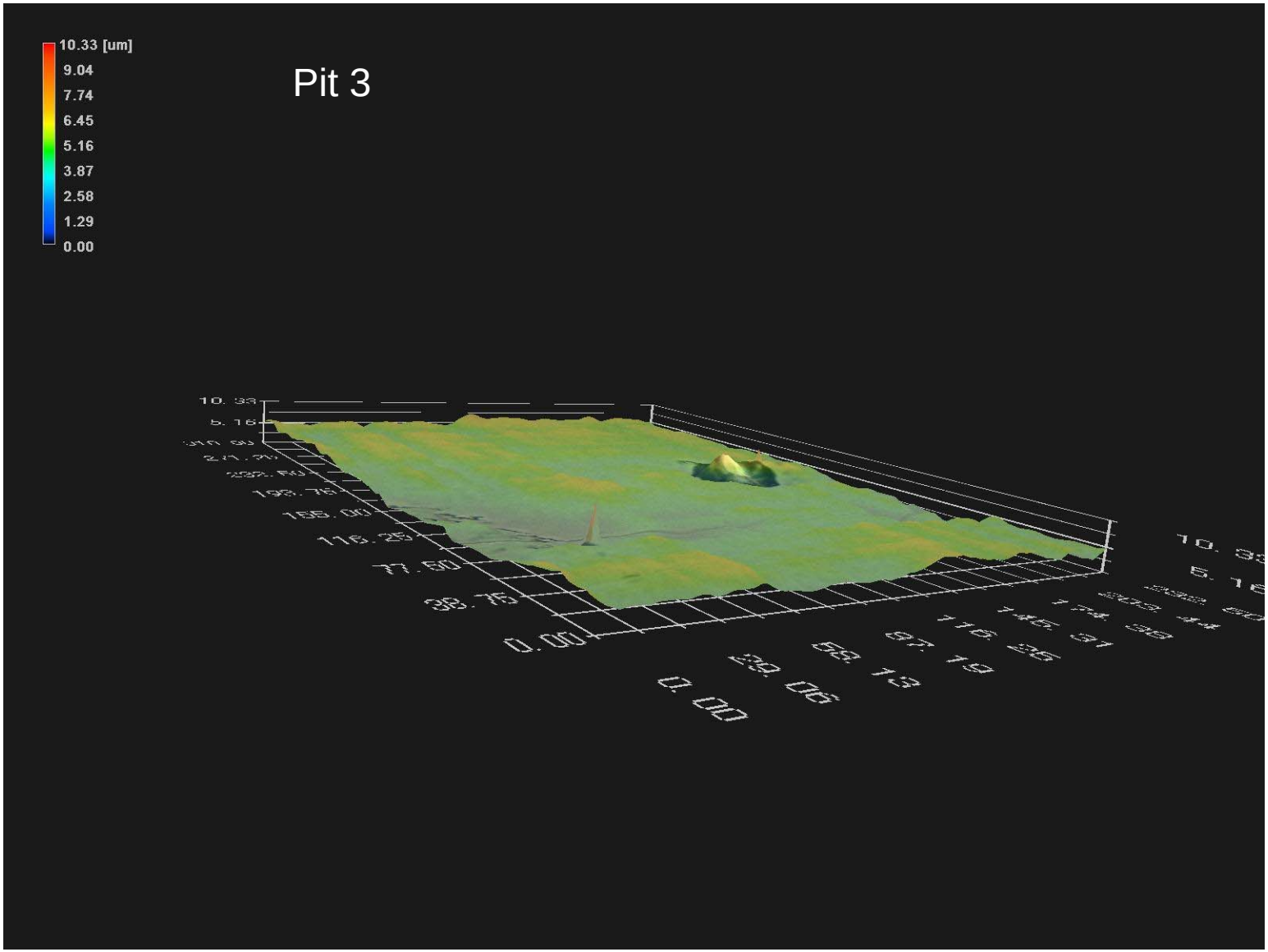
Pit 2


Again, notice “moat” around peak
Grain boundaries
14 µm height, 45 µm span
Profile measure
Record/Measure equipment: VHX
Save Date: 08/27/2008 05:54
Lens power: X 1000
3D Record interval: 3.00 [um]
No. of record images: 8
Profile mode
Horizontal line
Vertical line
Specify 2 point
2 line compare mode
Range cursor[C-D] disp
Color Setting
Inclination correct
Auto
Manual
OFF
Height fit display
Height
87.4um
C
D
A
B
0
310.0um
Height [A-B]: 13.7 µm
Width [C-D]: 44.5 µm
Cursor value:
Add list
Height [A-B]
Width [C-D]
Save list CSV
Clear list
Save grphCSV
Entire graph
Btwn C-D cur

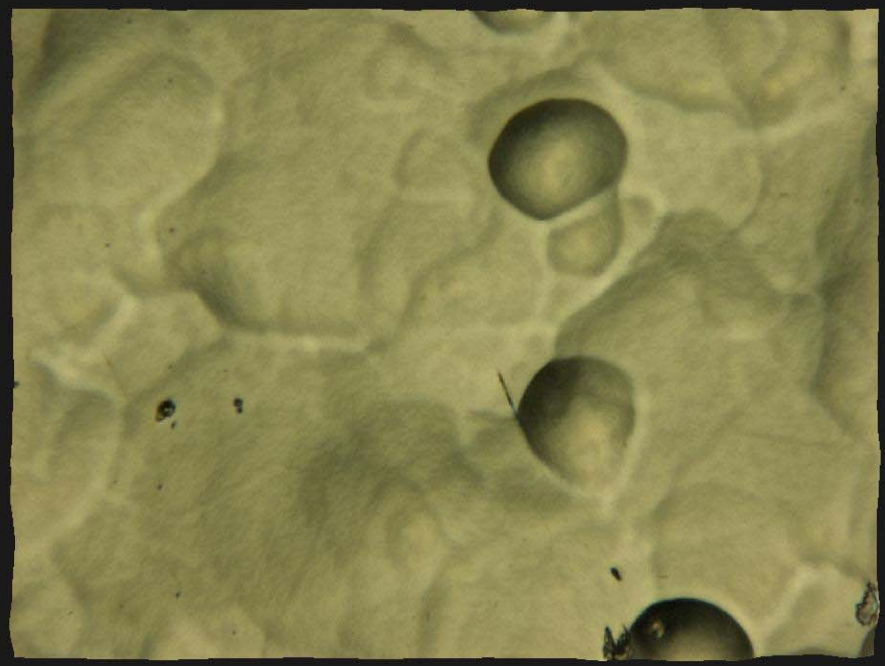
Pit 3


6 µm height, 51 µm span
Profile measure
Record/Measure equipment: VHX
Save Date: 08/27/2008 05:25
Lens power: X 1000
3D Record interval: 3.00 [um]
No. of record images: 7
Profile mode
Horizontal line
Vertical line
Specify 2 point
2 line compare mode
Range cursor[C-D] disp
Color Setting
Inclination correct
Auto
Manual
OFF
Height fit display
Height
87.4um
C
D
A
B
0
310.0um
Height [A-B]: 6.6 µm
Width [C-D]: 51.4 µm
Cursor value:
Add list
Height [A-B]
Width [C-D]
Save list CSV
Clear list
Save grphCSV
Entire graph
Btwn C-D cur

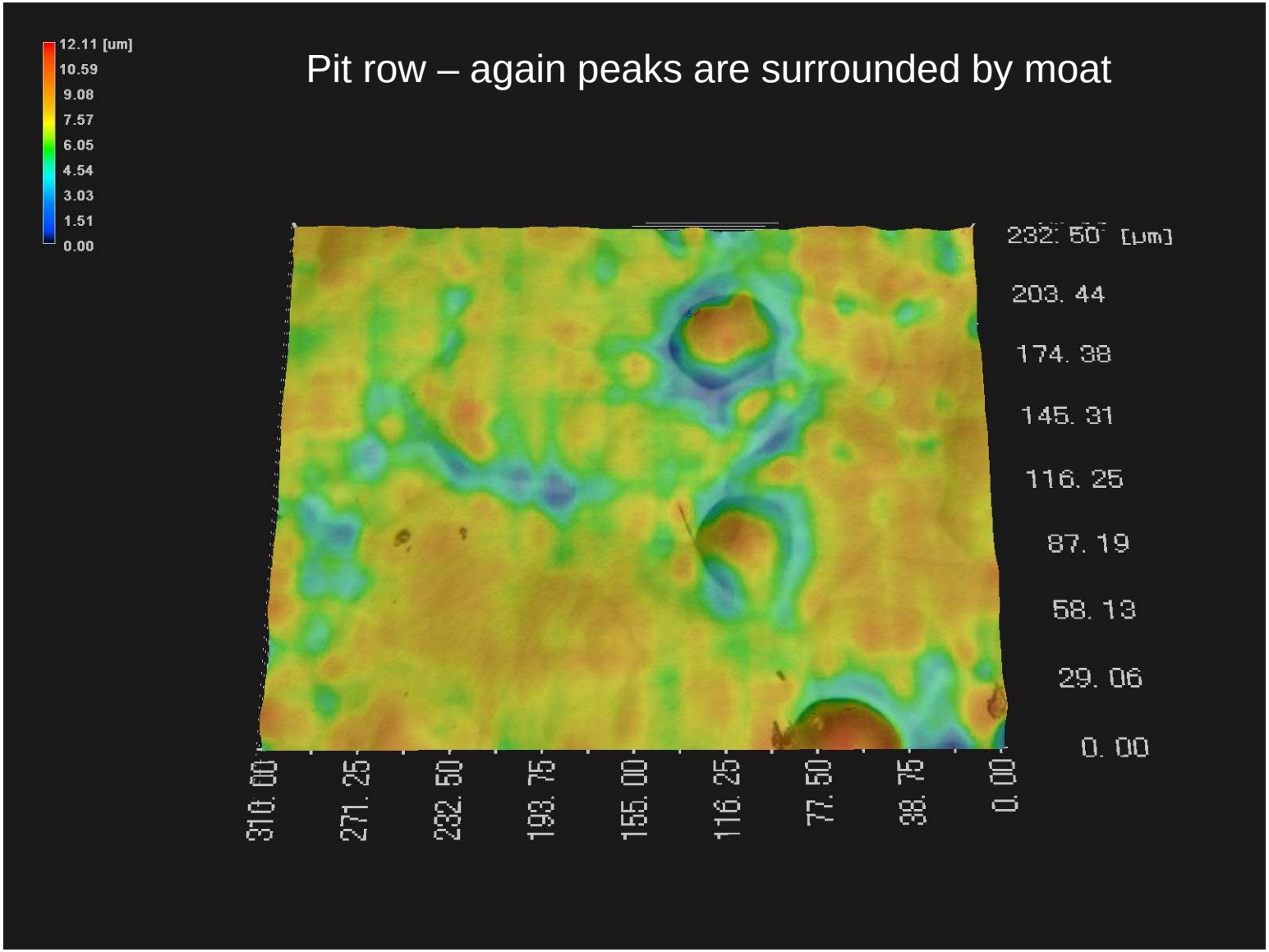
Pit 3



Pit rows that occur at the edge of the HAZ



Pit row – again peaks are surrounded by moat



Common themes and summary observations

- Pits are really peaks surrounded by a circular trough.
- ~10 um tall and ~50 um diameter pits are already formed at 110 µm removal
- Grain boundaries lead in to one pit – others too? This was seen in 9AES001.
- We're fairly confident that welding and weld prep was done very carefully and is not a source of flaws.
- EP was in no way optimized or under control.
- Florida State U. is dissecting pits and doing chemical analysis now