



FPI Cleaning Study Update

CASR

FAA Center for Aviation Systems Reliability

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Funding provided by the Federal
Aviation Administration as
Delivery Order IA052 as part of
Contract #DTFA03-98-D-00008

Objectives

- Determine the effect of chemical cleaning processes on the detectability of low cycle fatigue cracks in titanium alloys
- Update existing specifications to reflect the improved processes and provide best practices documents for use by the OEM's and airlines

Team Members

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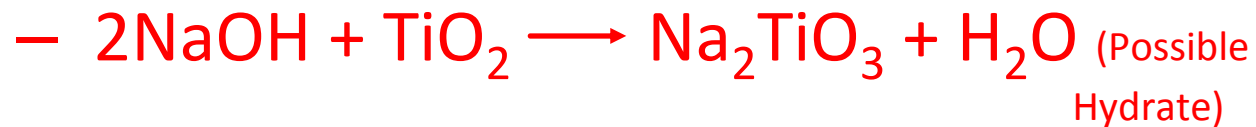
Rick Lopez

The problem

- ETC Phase II program looked at Ni and Ti cleaning using mechanical and chemical processes (Report at FAA and ISU websites)
- Samples of Titanium 6Al 4V, thru various chemical interactions during the cleaning process, became less responsive or unresponsive to fluorescent penetrant testing

Possible Cause?

Oxides forming when exposed to alkaline solutions:



- Reaction causes TiO_2 to EXPAND to Na_2TiO_3 and seal the cracks even tighter!
- Reaction might add water to the crack

- Can a final acid bath reverse the reaction?

The Process

Presently there have been three phases to the study

- Phase I using contaminated low cycle fatigue (lcf) samples from past work
- Phase II using pristine lcf samples with a second cleaning matrix and a heat treatment
- Phase III using pristine lcf samples and a third cleaning matrix and a heat treatment



Phase I

Phase I

Samples used for Cleaning Study

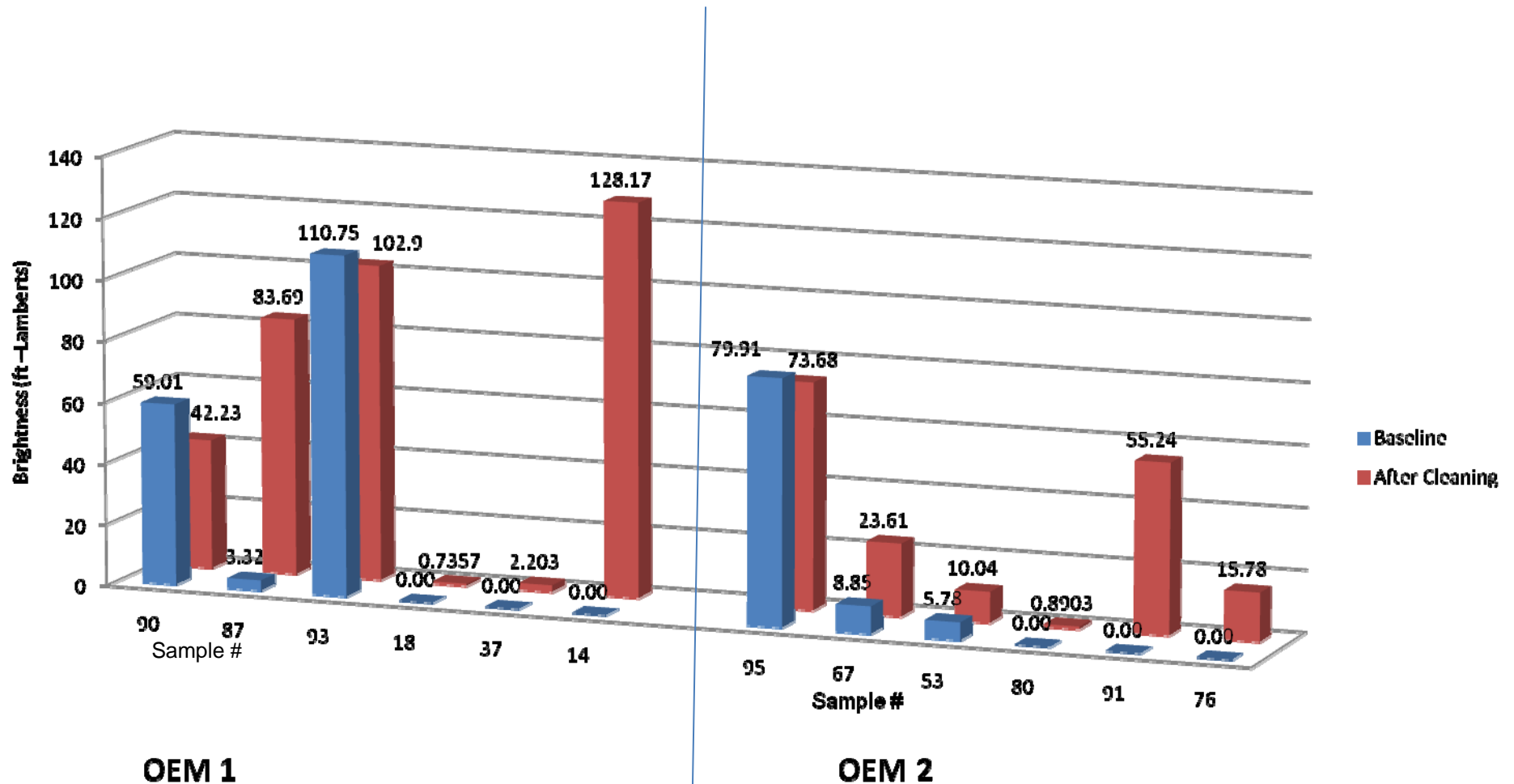
- Six samples that were not responsive and six samples that did not show adverse effects to the cleaning study were split into two groups
- Three non responsive and three responsive samples were sent to two OEMs for processing in their cleaning lines
- Contamination on samples consisted of soot and oxidation

OEM Cleaning Procedures

- OEM 1
 - Oil, grease, carbon cleaner – water rinse – scale conditioner – Nitric acid – water rinse - Oven dry
- OEM 2
 - Procedure 1
 - Aqueous alkaline cleaner – water rinse – liquid alkaline permanganate – water rinse – Sulfuric acid – water rinse - oven dry
 - Procedure 2
 - Aqueous alkaline cleaner – water rinse – liquid alkaline permanganate – water rinse – acid strippers – water rinse
 - Procedure 3
 - Molten salt bath – water rinse – Nitric acid – alkaline rust remover – water rinse – hot water dip/air dry

Final Results for Phase I

Baseline VS Post Cleaning



Samples w/o Baseline values were not detectable in the baseline runs but were recovered in the cleaning process

Phase I

Conclusions

- OEM 2 controlled etch process has clearly opened up the cracks - demonstrates effectiveness of etching as an aid to FPI but resulted in unacceptable changes to the surface despite a cautious approach.
- Six samples descaled by OEM 1 process although not as clean as would like.
- Neither alkaline deruster process cleaned the bars or aided FPI, in fact they may have contributed to loss of indication.

Phase I

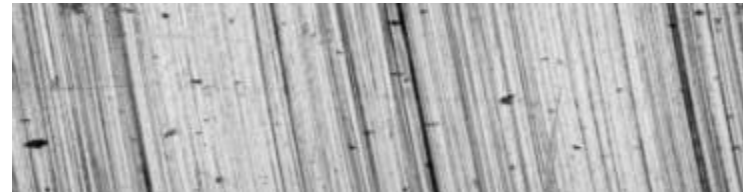
Conclusions

- Assuming results of FPI are same or better for OEM 1 processed specimens, then the use of a final HNO₃ acid step appears to be aiding the descaling process when compared to alkaline deruster process alone.
- If Titanates are in fact formed and are soluble in acid, then a final acid step should be beneficial.

Phase I

Conclusions

- In two of the samples that were recovered, it appears that the molten salt bath has removed a considerable amount of surface material, making the crack wider than the initial width.



Further Work – Phase II and Phase III

- Develop a new set of Icf samples for further studies
- Sample set to consist of Ti 6-4 bars
- Bar dimensions to be 6" L, 1" W, ½" H
- Crack length to be 0.060" +/- 0.010"
- Samples developed using Icf procedures with an EDM notch as crack initiator.
- Consider the effects of heat treatment on crack detectability

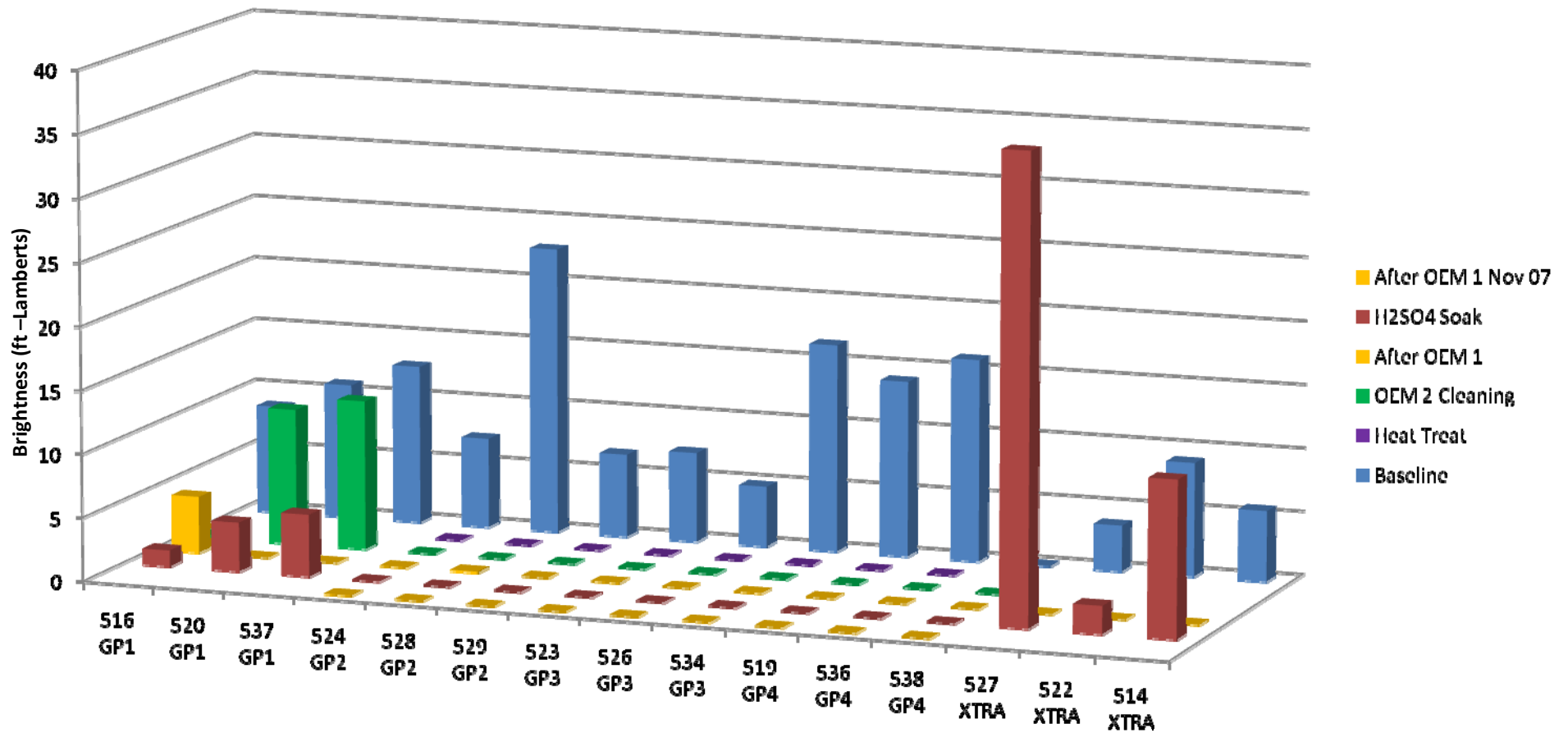


Phase II

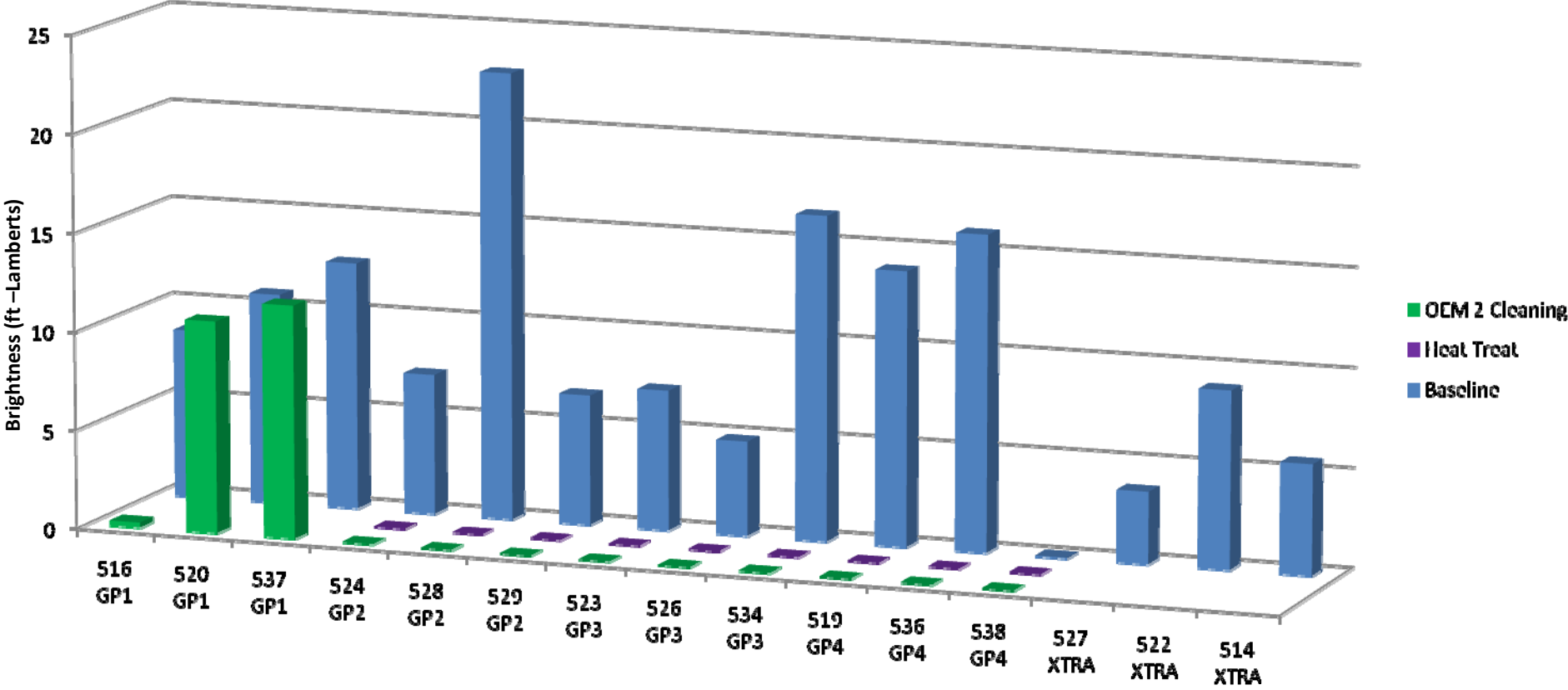
Cleaning Matrix – Phase II

- **Group 1 not heat treated**
 - Alkaline clean – water rinse – oven dry – FPI – OEM 1 process 2X – FPI – hot H₂SO₄ – FPI
- **Group 2 Heat treated @ 975 F**
 - Alkaline clean – water rinse – oven dry – FPI – OEM 1 process 2X – FPI – hot H₂SO₄ – FPI – OEM 1 – FPI
- **Group 3 Heat treated @ 975 F**
 - Alkaline clean – water rinse – HNO₃ – water rinse – oven dry – FPI – OEM 1 process 2X – FPI – hot H₂SO₄ – FPI – OEM 1 – FPI
- **Group 4 Heat treated @ 975 F**
 - Alkaline clean – water rinse – alkaline permanganate – water rinse - HNO₃ – water rinse – oven dry – FPI – OEM 1 process 2X – FPI – hot H₂SO₄ – FPI – OEM 1 – FPI
- **Group XTRA (not Heat Treated)**
 - OEM 1 process 2X – FPI – hot H₂SO₄ – FPI
- **OEM 1 Process**
 - Aqueous degreaser – water rinse – hot alkaline degreaser – water rinse – HNO₃ – cold water rinse – hot DI water dip – air dry

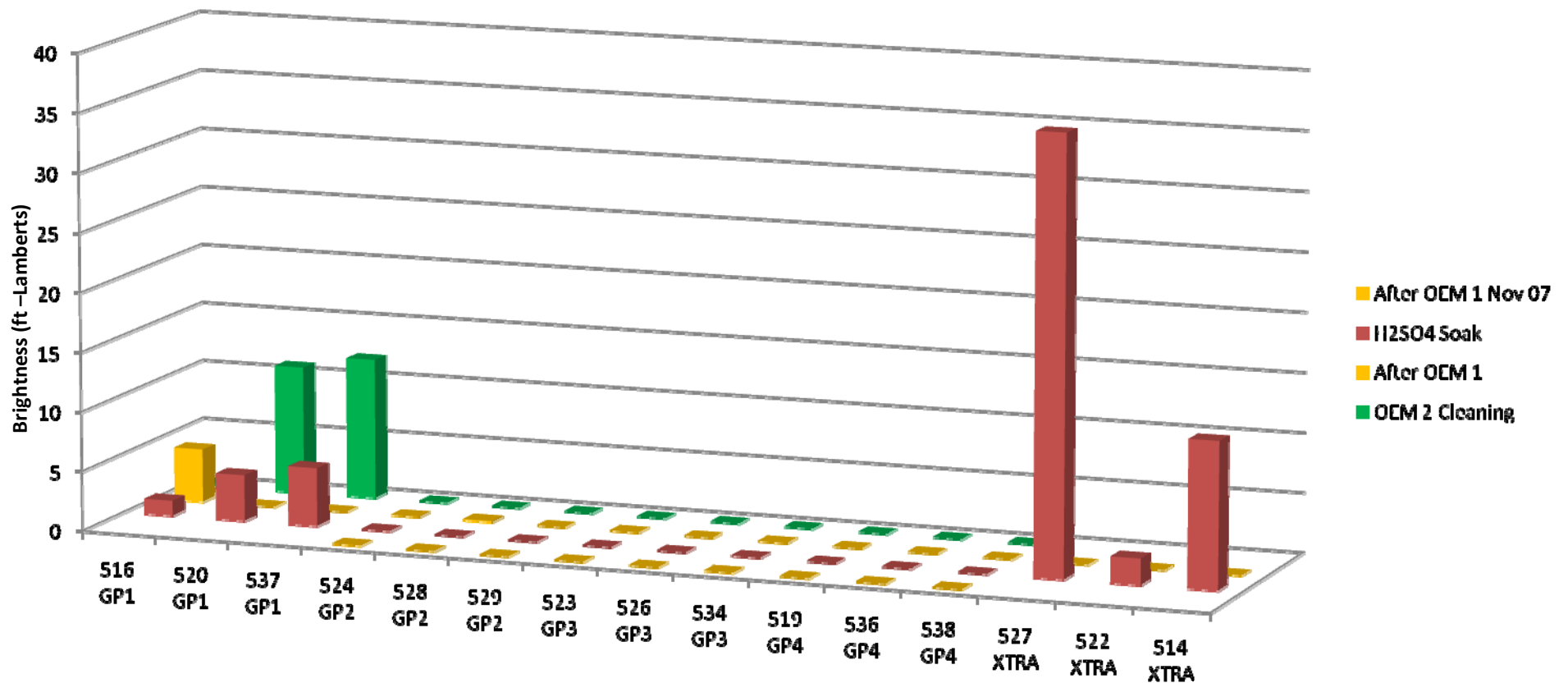
Phase II Results after Cleaning



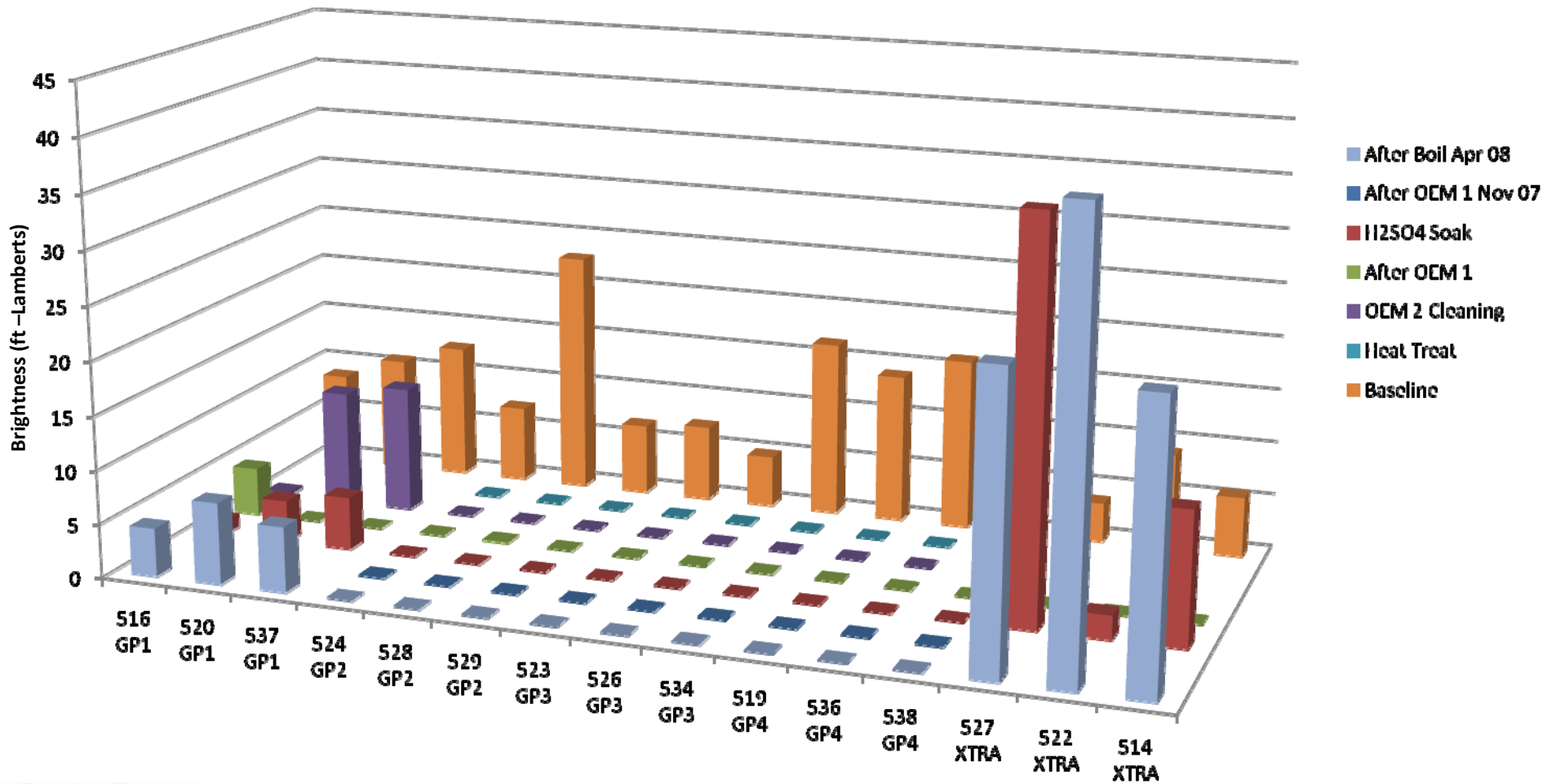
Phase II Results after Cleaning



Phase II Results after Cleaning and acid soak

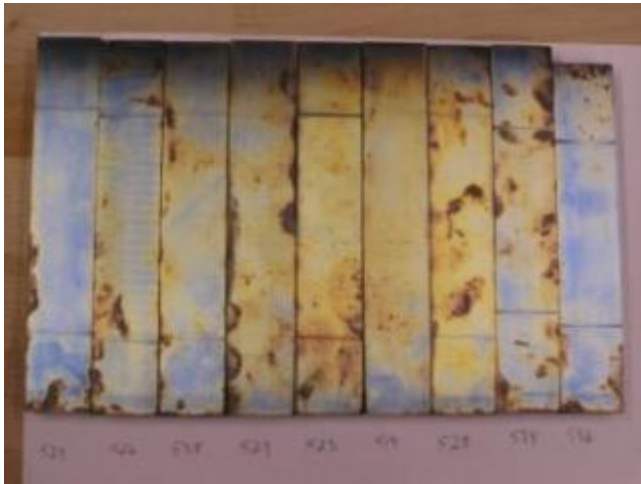


Phase II Results after Cleaning, acid soak and 30 min boil

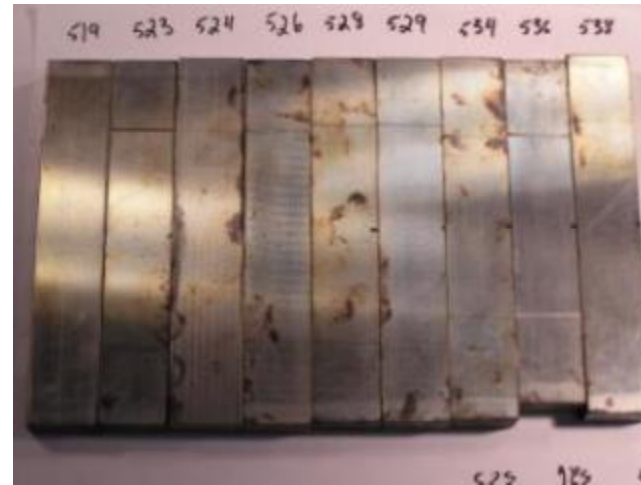


Sample	Group #	3 run Average From Baseline	Brightness after HT	Brightness after cleaning	After RR treatment	After H2SO4	After RR Nov 07	After Boil
516 GP1	1	8.5886		0.2576	4.4672	1.2717		4.4537
520 GP1	1	10.7204		10.7475	0.0011	3.8703		7.534
537 GP1	1	12.5786		11.8027	0	4.8915		6.0252
524 GP2	2	7.1290	0.0169	0.0182	0.0262	0.0359	0.0314	0
528 GP2	2	22.7076	0	0	0.0957	0	0.009199	0
529 GP2	2	6.6040	0	0	0	0	0	0
523 GP3	3	7.1113	0	0	0.017	0	0	0
526 GP3	3	4.7961	0	0.0083	0.0006	0	0	0
534 GP3	3	16.4417	0.0115	0.0114	0.0144	0.0172	0.011	0
519 GP4	4	13.9045	0	0	0	0	0	0.0059
536 GP4	4	15.9824	0	0	0	0	0	0
538 GP4	4	0.0514	0	0	0	0	0	0
527 XTRA	XTRA	3.6446			0	36.5717		26.6645
522 XTRA	XTRA	8.9735			0	2.1946		40.3948
514 XTRA	XTRA	5.5746			0.0225	12.2608		25.5913

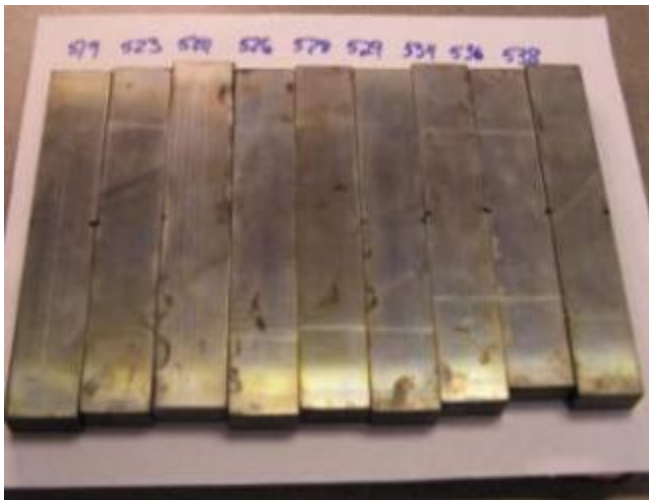
Heat treated @ 975° F



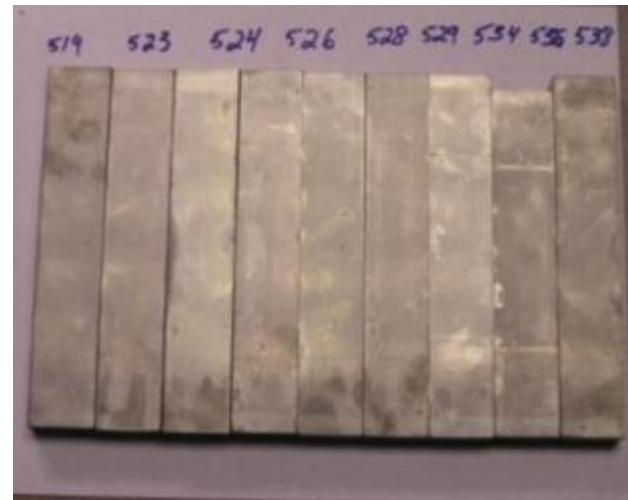
Samples prior to any cleaning efforts



After OEM 2 and OEM 1 Cleaning



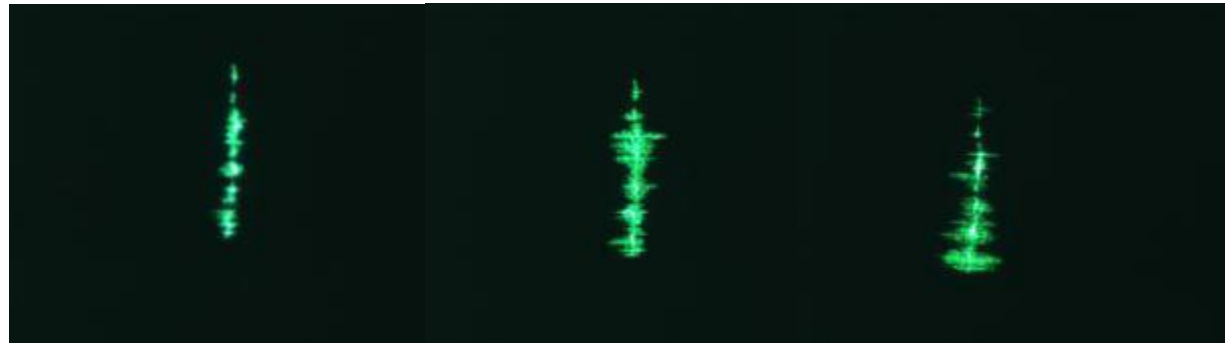
After OEM 2, OEM 1,
and ISU Cleaning



After OEM 1, Nov 07

Group 1(not-Heat Treated) Alk. Clean, Water rinse, Oven Dry/Flash Dry, OEM 1 (2X), Hot H₂SO₄, OEM 1

Baseline Runs

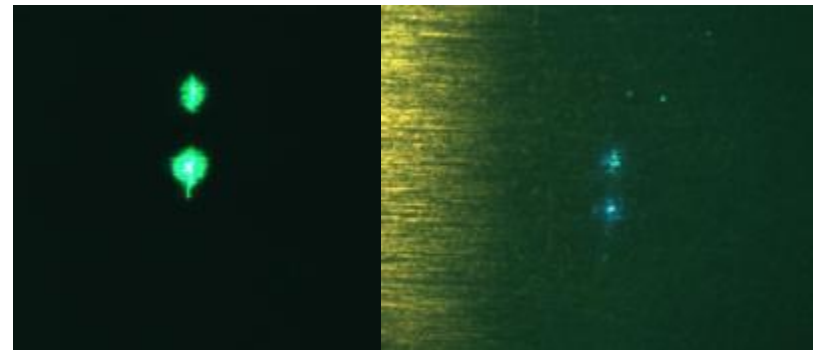


Run 1
B=6.689
E=822ms

Run 2
B=11.639
E=749ms

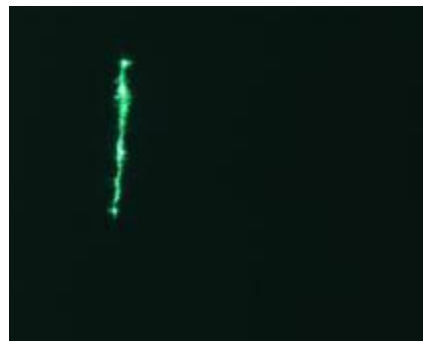
Run 3
B=13.832
E=464ms

No Image
Not Heat Treated



After Cleaning
B=10.747
E=522ms

After OEM 1
B=0.0011
E=2.4min



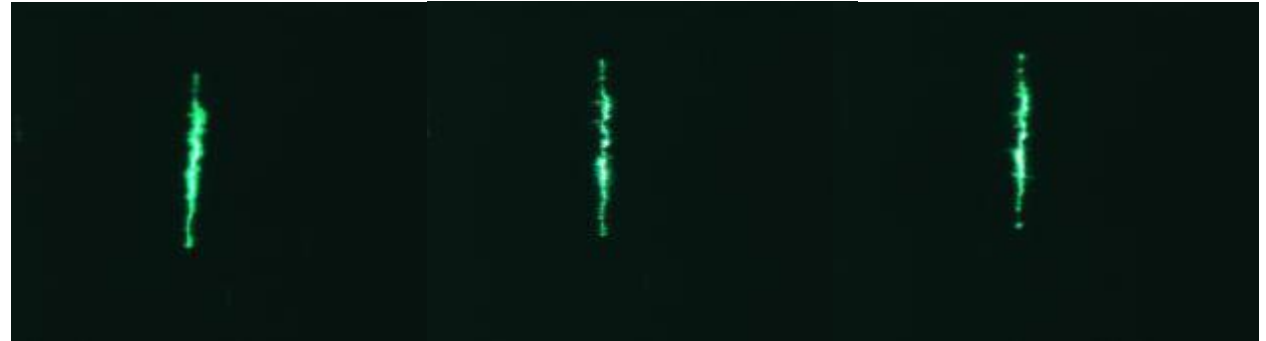
After ISU
B= 3.8703
E=1.88sec

After OEM 1, Nov 07
Not Tested

02-524 0.055"

Group 2(Heat Treated) Alk. Clean, Water rinse, Oven Dry/Flash Dry, OEM 1 (2X), Hot H₂SO₄, OEM 1

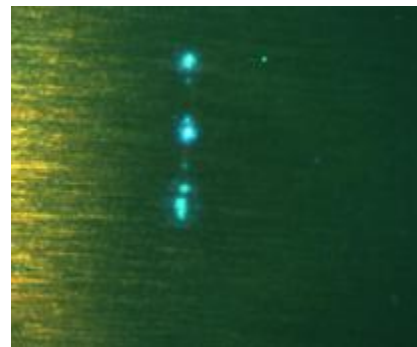
Baseline Runs



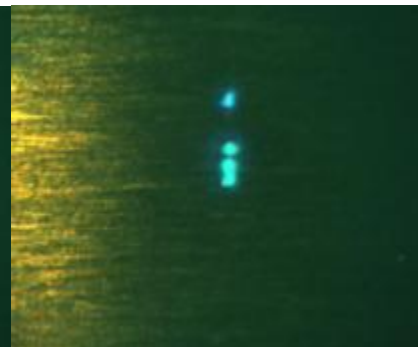
Run 1
B=8.969
E=510ms

Run 2
B=6.450
E=752ms

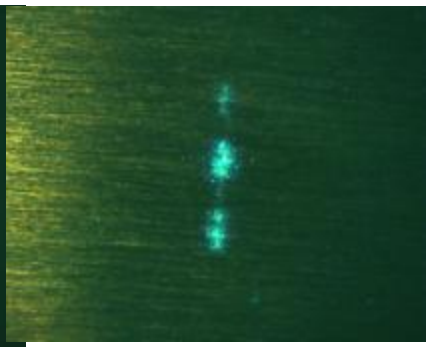
Run 3
B=9.66
E=728ms



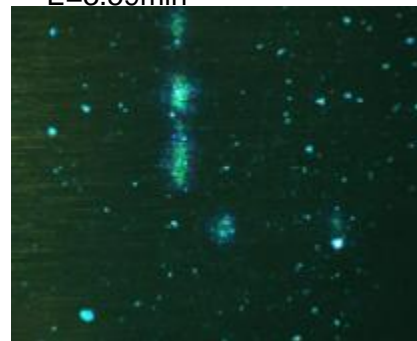
After Heat Treat
B=0.0169
E=3.59min



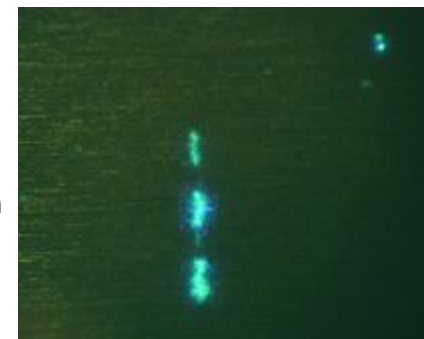
After Cleaning
B=0.0182
E=3.26min



After OEM 1
B=0.0262
E=2.86min



After ISU
B= 0.0359
E=1.49min



After OEM 1,
Nov 07
B=0.0314
E=2.34min

02-523 0.054"

Group 3(Heat Treated) Alk. Clean, Water rinse,HNO₃,
Water rinse, Oven Dry/Flash Dry, OEM 1 (2X), Hot H₂SO₄,OEM 1

Baseline Runs



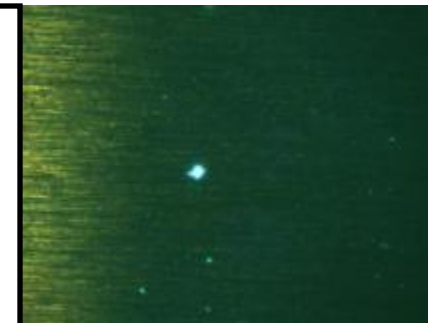
Run 1
B=0.570
E=4sec

Run 2
B=12.354
E=728ms

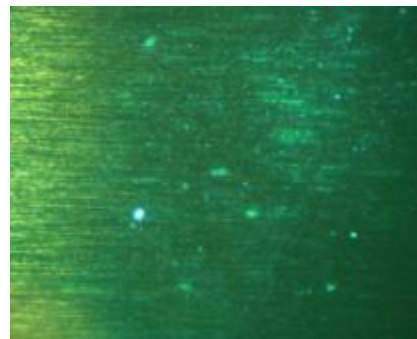
Run 3
B=8.409
E=759ms

No Indication
After Heat Treating

No Indication
After Cleaning



After OEM 1
B=0.017
E=2.61min

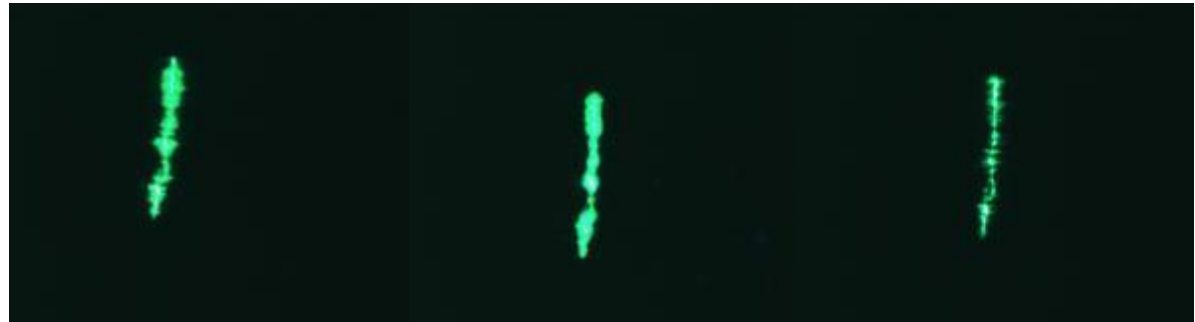


After ISU
B= -0.0065
E=5.29min
No real
indication

No Indication
After Nov 07
OEM 1 Cleaning

Group 4 (Heat Treated) Alk. Clean, Water rinse, Alk. Perm,
Water rinse, HNO₃, Water rinse, Oven Dry/Flash Dry, OEM 1 (2x),
Hot H₂SO₄, OEM 1

Baseline Runs



Run 1
B=16.135
E=547ms

Run 2
B=16.268
E=424ms

Run 3
B=9.309
E=509ms

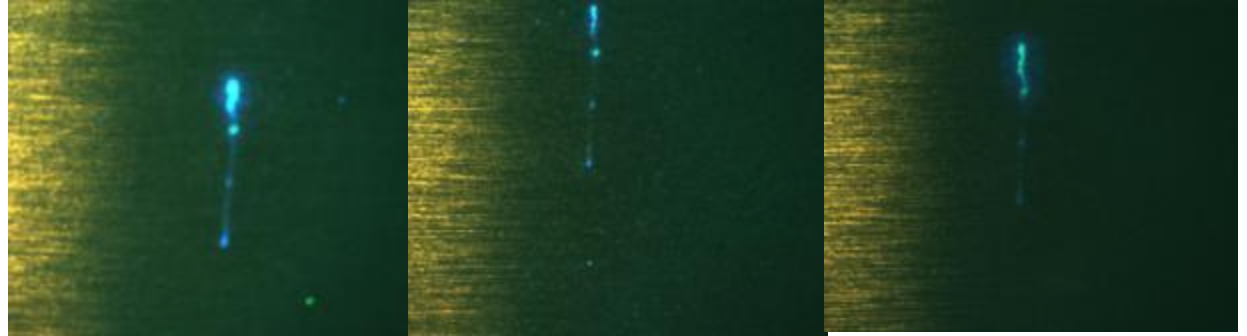
No Indication After Heat Treating	No Indication After Cleaning	No Indication After OEM 1 Cleaning
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After ISU
No Indication
After Acid

No Indication
After Nov 07
OEM 1 Cleaning



Baseline Runs



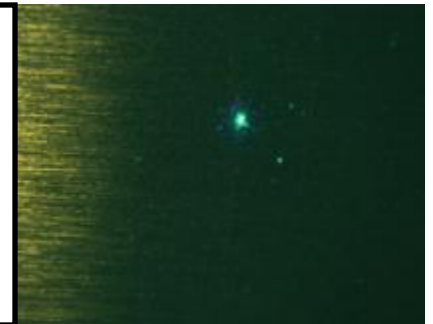
Run 1
B=0.0083
E=2.7min

Run 2
B=0.0131
E=2.36min

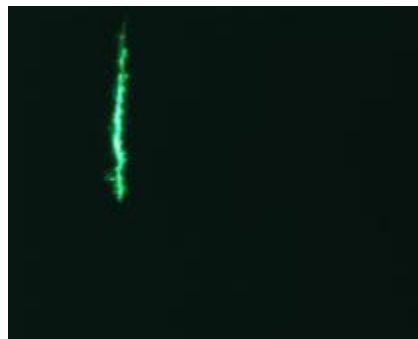
Run 3
B=0.008
E=1.74min

No Image taken
Not Heat Treated

No Image taken
Not Cleaned



After OEM 1
B=0.0225
E=1.73min



After ISU
B= 12.2608
E=443ms

Phase II Conclusions

- Heat treated samples lost indications after the heat treatment
- Neither OEM cleaning process, or hot H₂SO₄ soak recovered crack indications after the heat treatment
- Samples that were not heat treated responded well to both the OEM's cleaning processes and the hot sulfuric acid soak (ISU)

Phase II Conclusions

- From the work in phase two, a more in-depth look at acid treatments was pursued for the third phase of the work
- Questions were raised regarding the effect of temperature on responsiveness to cleaning (975°F verses 800°F)

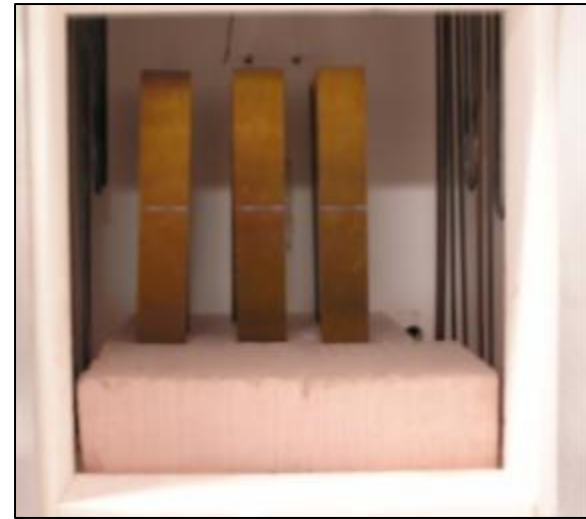
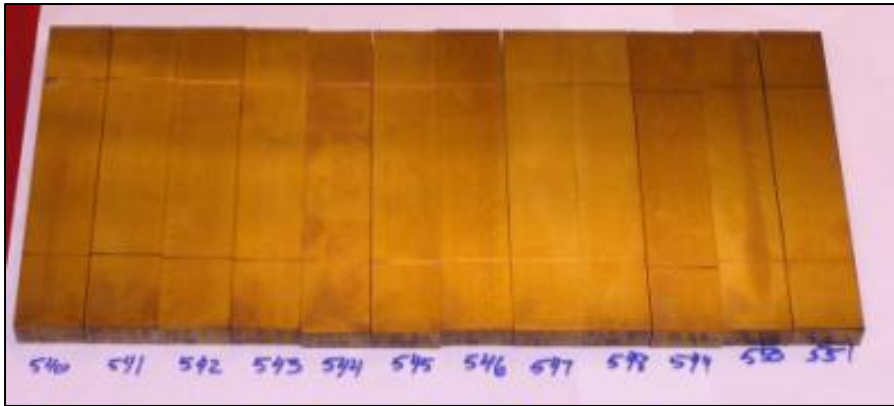


Phase III

Cleaning Matrix – Phase III

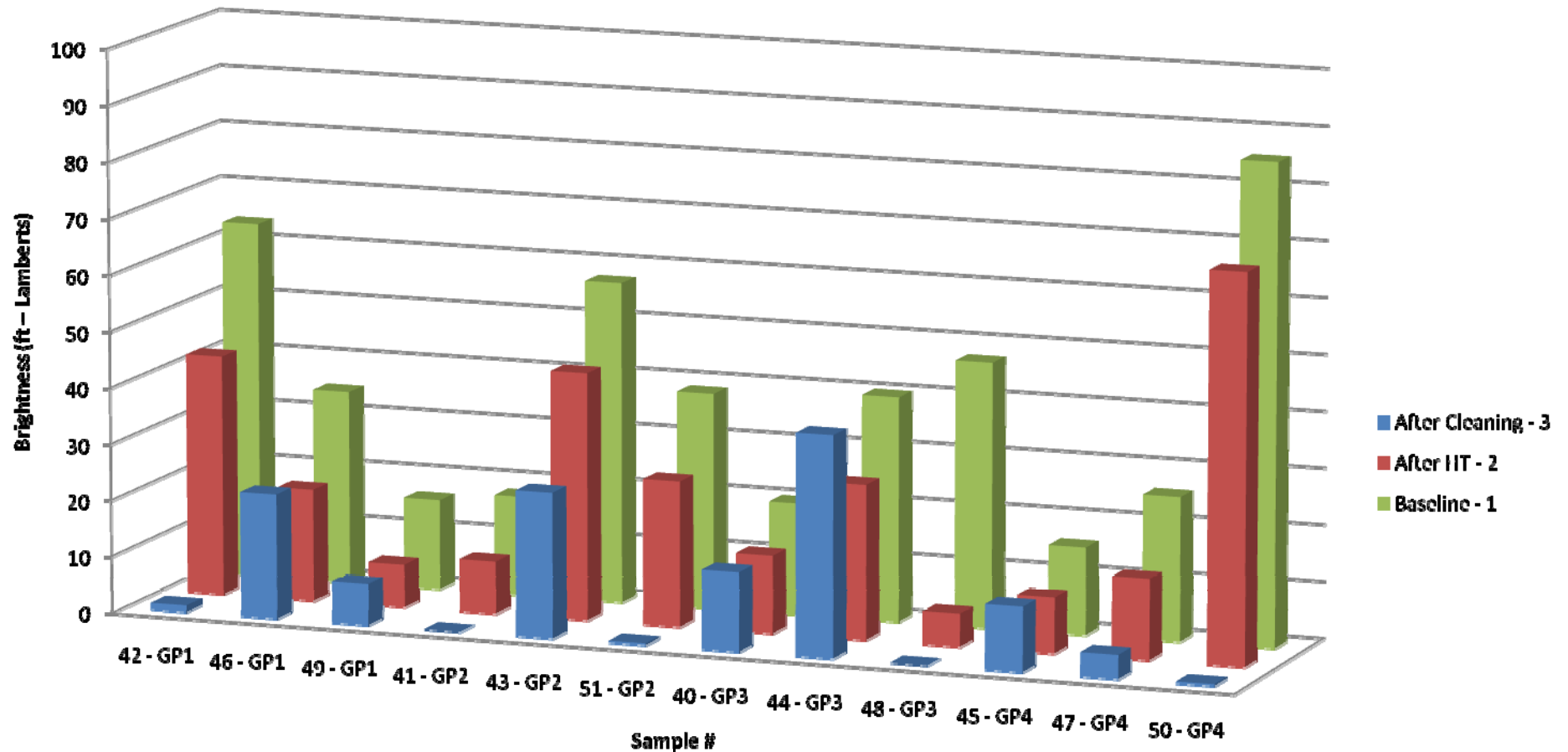
- **Group 1 Heat treated @ 800F**
 - Alkaline clean – water rinse – oven dry – FPI – hot H₂O Boil – FPI – 1 Hour Acetone UT - FPI
- **Group 2 Heat treated @ 800 F**
 - Alkaline clean – water rinse – H₂SO₄ – water rinse – oven dry - FPI – hot H₂O Boil – FPI – 1 Hour Acetone UT - FPI
- **Group 3 Heat treated @ 800 F**
 - Alkaline clean – water rinse – HNO₃ – water rinse – oven dry – FPI – hot H₂O Boil – FPI – 1 Hour Acetone UT - FPI
- **Group 4 Heat treated @ 800 F**
 - Alkaline clean – water rinse – Acid Descaler – water rinse – oven dry – FPI – hot H₂O Boil – FPI – 1 Hour Acetone UT – FPI

Photo of samples after heat treatment

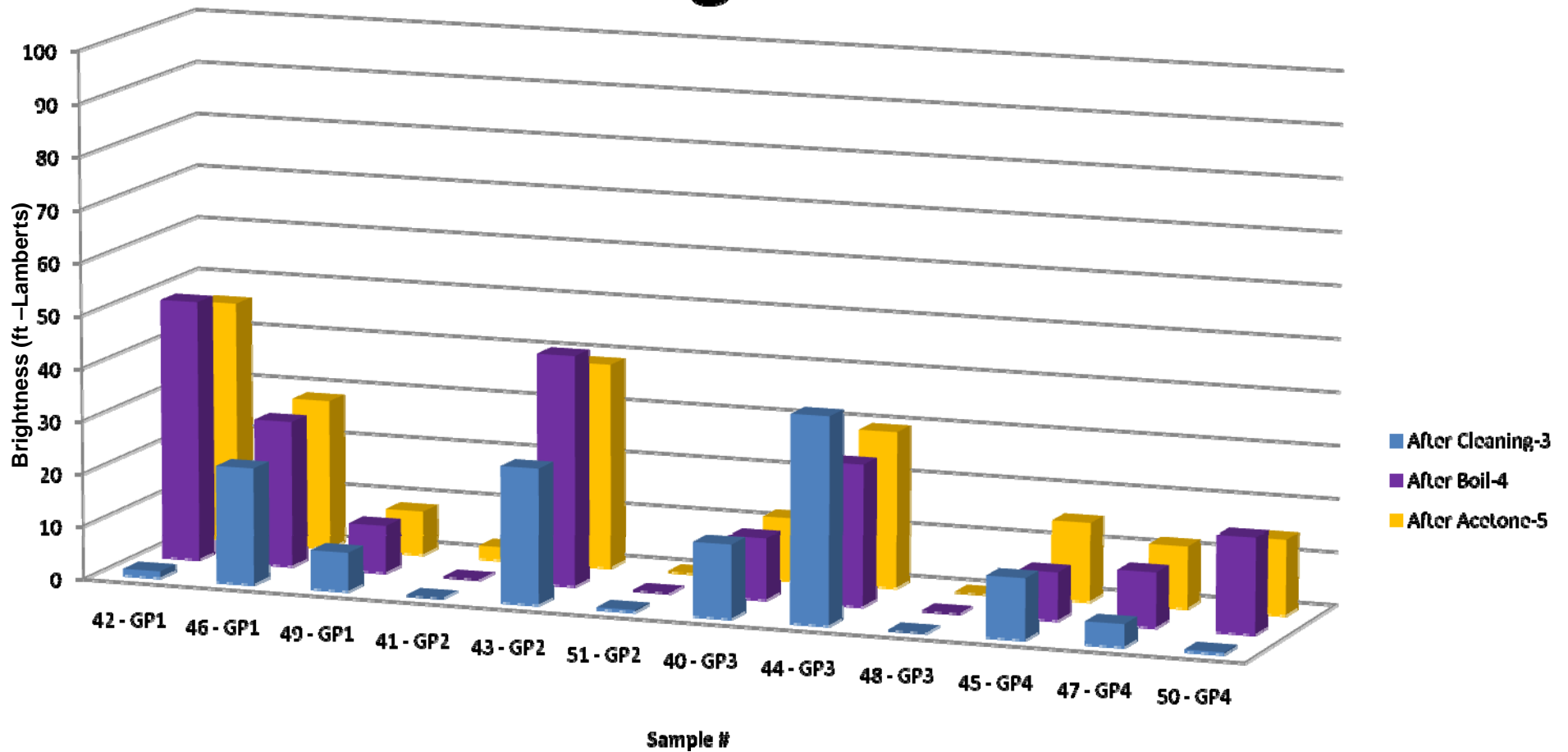


- Samples were cleaned in acetone in an ultrasonic cleaner for 30 minutes and kept in acetone until placed directly into the furnace.
- Heat treatment was done at 800°F for 96 hours.

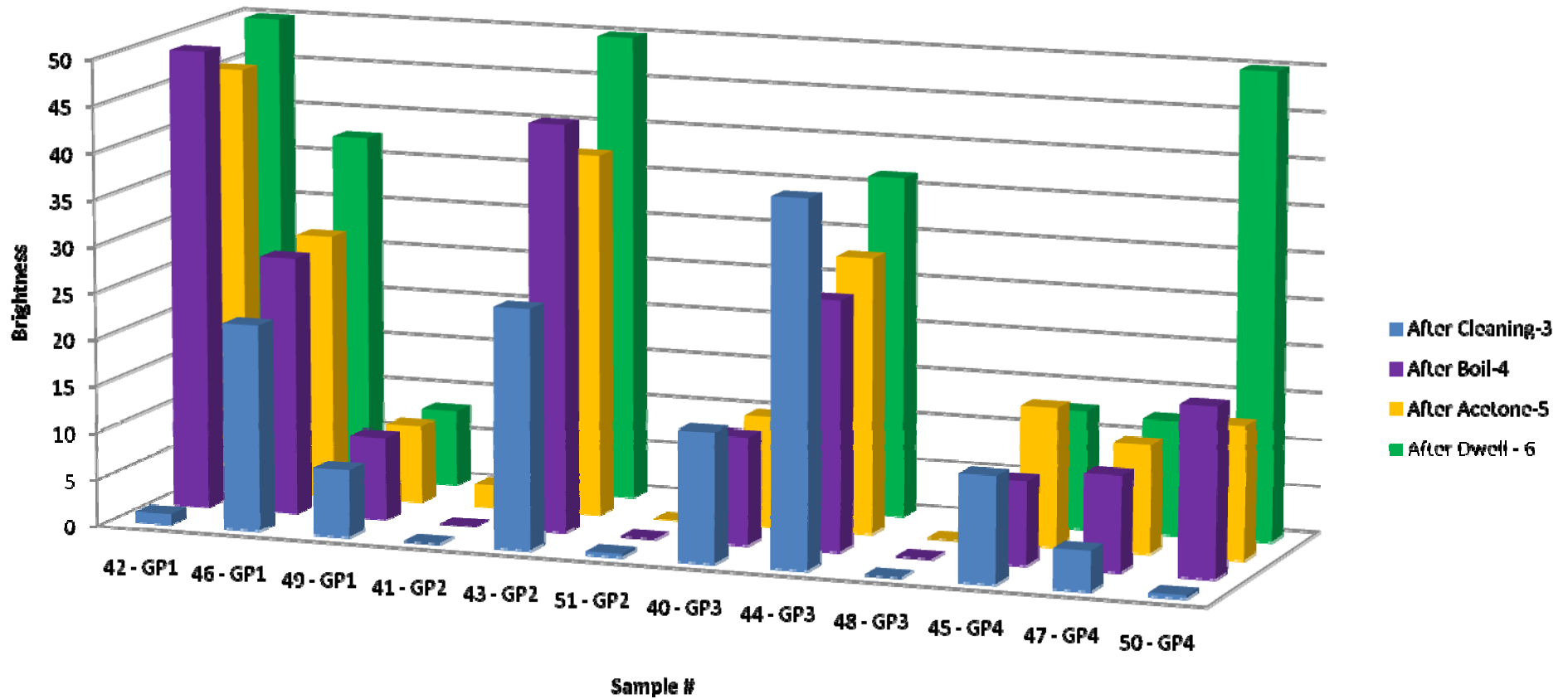
Baseline, Heat Treatment and Cleaning Brightness



Cleaning, Boil and Acetone Brightness

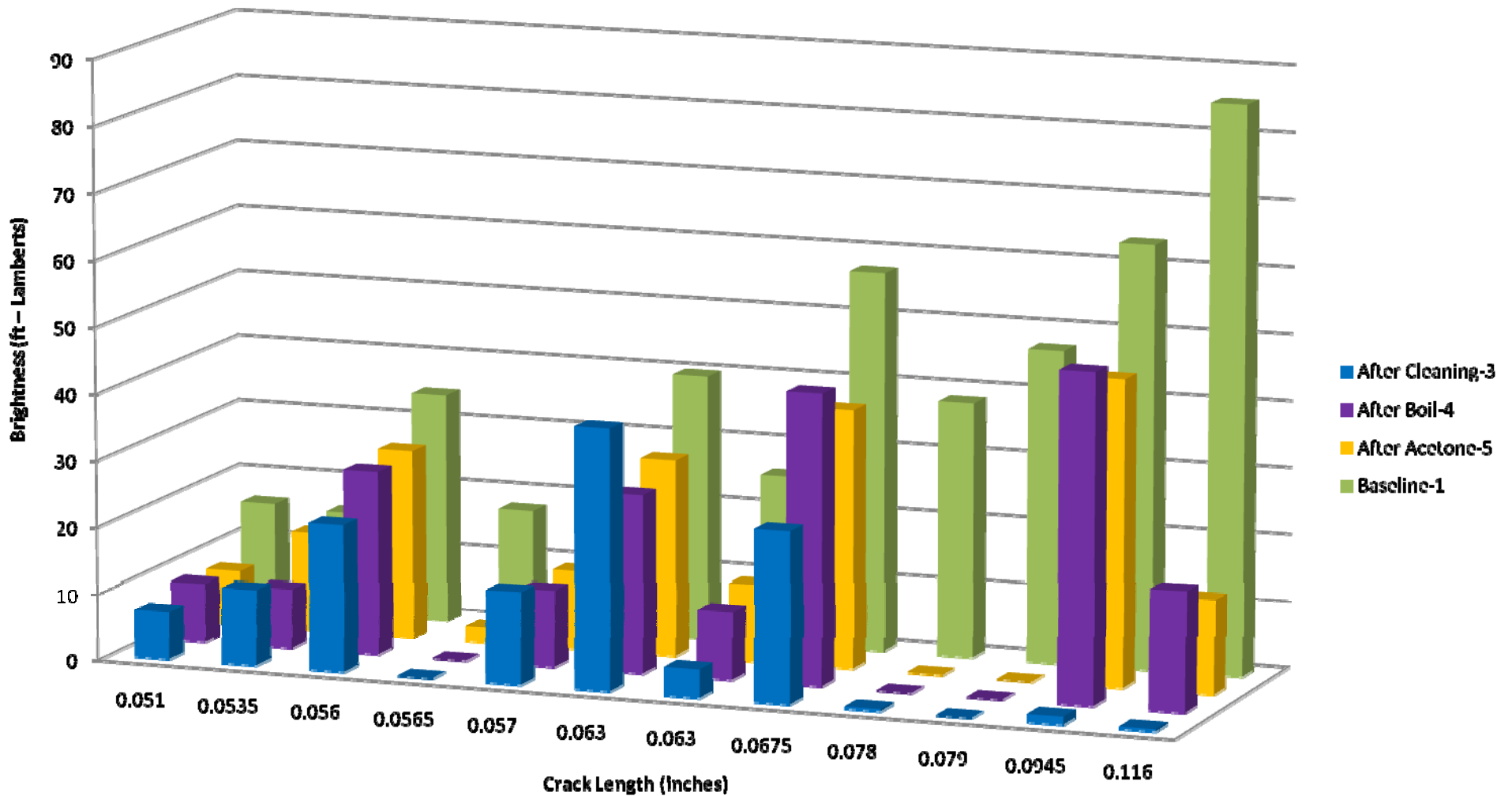


Cleaning, Boil and Acetone Dwell Brightness



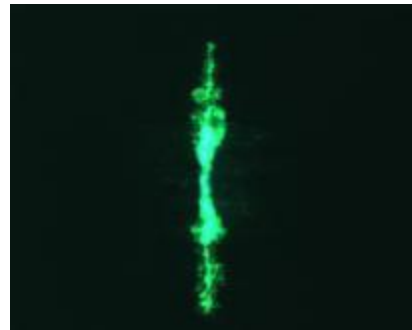
sample	B	AHT	AGE	Length	A Boil	A 1 Hr act	A 2 hr dwell
42 - GP1	63.6	42.6	1.2	0.0945	49.27	45.892	56.3746
46 - GP1	34.4	19.8	22	0.056	27.52	28.268	37.5106
49 - GP1	16	7.5	7.3	0.051	8.89	8.203	8.0347
41 - GP2	17.6	9.2	0.02	0.0565	0.00	2.463	6.954
43 - GP2	57	43.8	25.63	0.0675	43.57	38.753	56.0193
51 - GP2	38.3	25.7	0.34	0.078	0.00	0.070	0
40 - GP3	19.76	13.68	13.94	0.057	11.63	11.797	13.6487
44 - GP3	39.8	27.3	39.04	0.063	26.69	29.484	36.4919
48 - GP3	46.96	5.87	0.009	0.079	0.01	0.002	0.0466
45 - GP4	15.41	9.87	11.48	0.0535	9.03	14.796	12.6219
47 - GP4	25.44	14.21	4.25	0.063	10.31	11.666	12.2624
50 - GP4	85.1	68.68	0.35	0.116	18.07	14.202	62.7389

Brightness verses Length

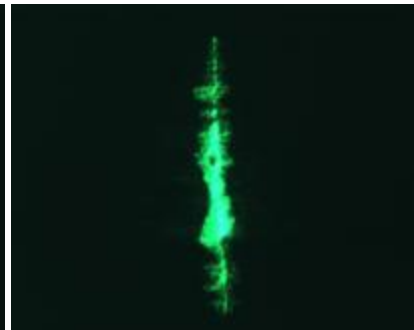


02-542 0.0945"

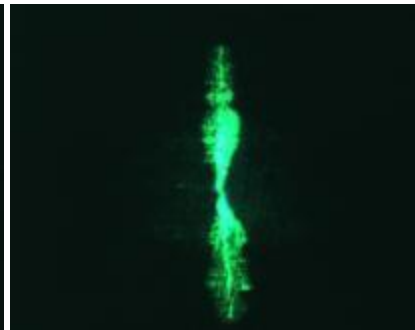
Group 1 Alkaline clean – water rinse – oven dry – FPI – hot H₂O Boil – FPI – 1 Hour
Acetone UT - FPI



Run 1
B=63.607
E=170 ms



Run 2
B=73.382
E=149 ms



Run 3
B=74.860
E=170 ms



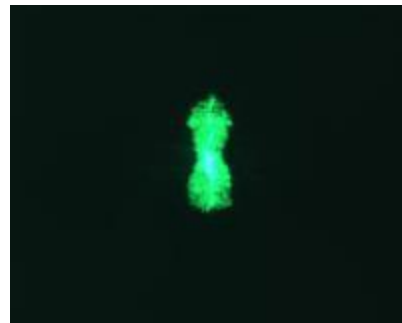
After Heat Treat
B=42.636
E=204 ms



After Clean
B=1.189
E= 4.44 sec



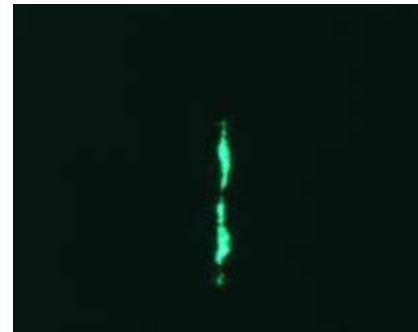
After ISU boil
B=49.27
E=225ms



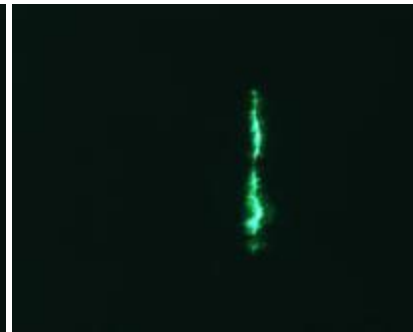
After ISU Acetone
B=45.89
E=189ms

02-541 0.0565"

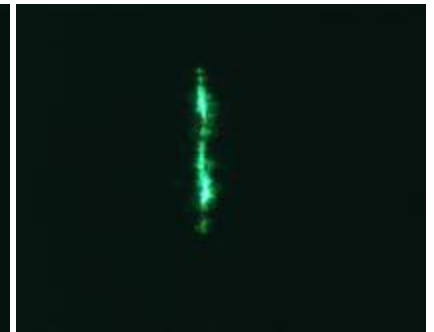
Group 2 Alkaline clean – water rinse – H₂SO₄ – water rinse – oven dry - FPI – hot H₂O
Boil – FPI – 1 Hour Acetone UT - FPI



Run 1
B=17.635
E=228 ms



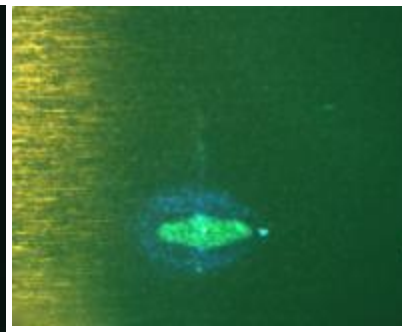
Run 2
B=17.990
E=245 ms



Run 3
B=19.003
E=257 ms



After Heat Treat
B=9.239
E=484 ms



After Clean
B= 0.0206
E= 6.44 min

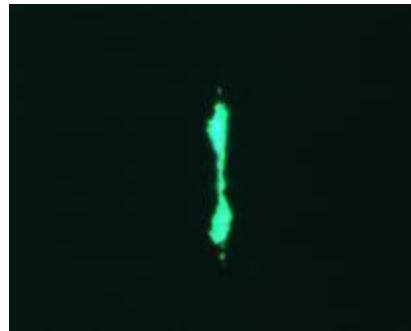
After ISU boil
B= No Indication
E= No Time



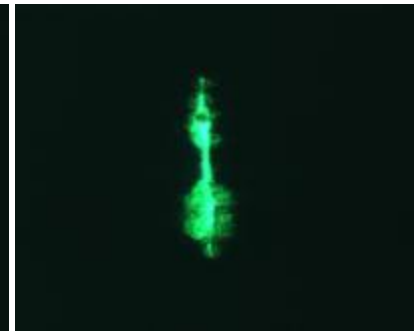
After ISU Acetone
B=2.463
E=1.2sec

02-544 0.063"

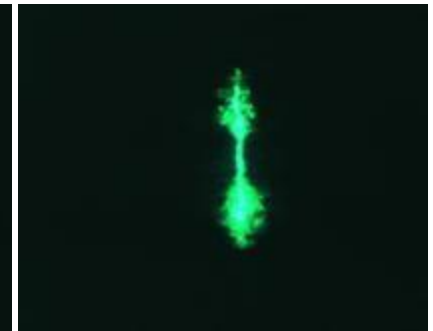
Group 3 Alkaline clean – water rinse – HNO₃ – water rinse – oven dry – FPI – hot H₂O Boil – FPI – 1 Hour Acetone UT - FPI



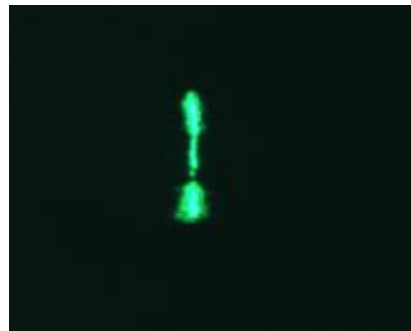
Run 1
B=39.805
E=147 ms



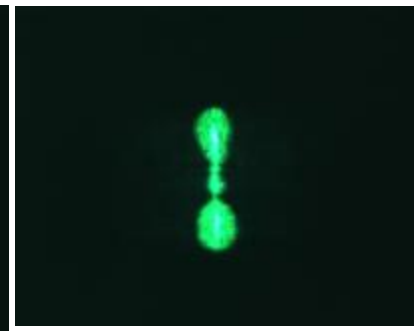
Run 2
B=51.952
E=166 ms



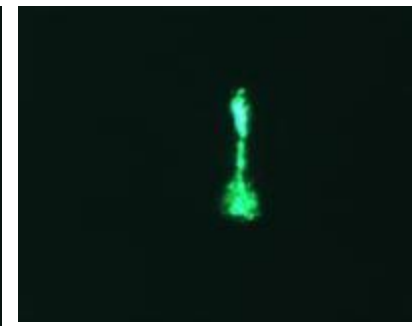
Run 3
B=53.556
E=160 ms



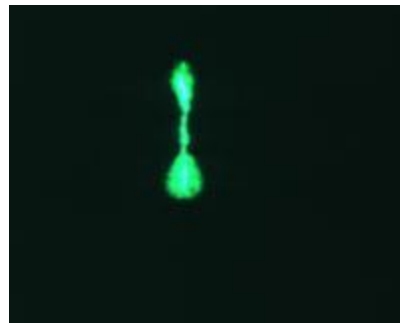
After Heat Treat
B=27.325
E=221 ms



After Clean
B= 39.0411
E= 253 ms



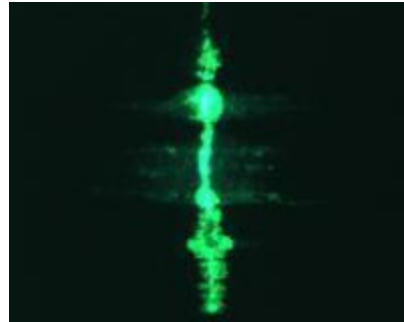
After ISU boil
B=26.69
E=211ms



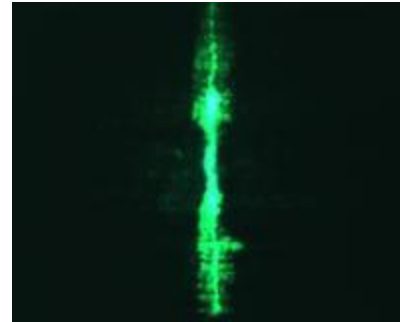
After ISU Acetone
B=29.484
E=229ms

02-550 .0116"

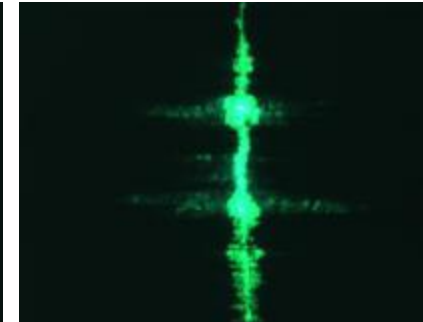
Group 4 Alkaline clean – water rinse – Acid Descaler– water rinse – oven dry – FPI – hot H₂O Boil – FPI – 1 Hour Acetone UT – FPI



Run 1
B=85.101
E=159 ms



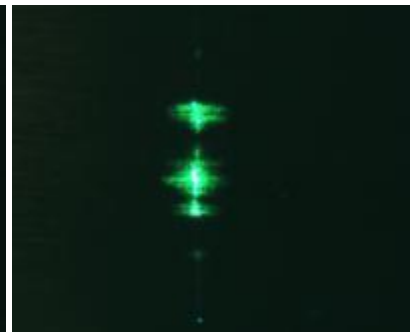
Run 2
B=82.203
E=164 ms



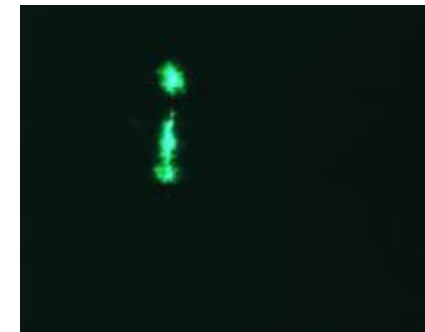
Run 3
B=82.696
E=149 ms



After Heat Treat
B=68.683
E=191 ms



After Clean
B= 0.3536
E= 25.4 sec



After ISU boil
B=18.07
E=311ms



After ISU Acetone
B=14.202
E=372ms

Phase III Conclusions

- Brightness of the samples after baseline tended to increase with increasing crack length
- After cleaning, brightness values of the samples decreased in most cases with the longer cracks above 0.070"
- Sample brightness values increased after boiling water and/or 1 hour acetone cleaning

Summary Conclusions

- Hot water boil removed soluble material that was reducing fluorescence (residual alkaline)
- Neither the hot water boil or the one hour acetone had any effect on the titinate
- Since the hot water rinse is the last step in the cleaning process, is the rinse water as clean as it could be? Is there a way to measure the cleanliness of the rinse water?

Continued Work

- Increase the dwell time to see if the longer, tighter crack are more responsive. (results included in Phase III slides)
- Boil the Phase II samples to see if they could be recovered from the contamination. (results included in Phase II slides)
- Process non heat treated samples in alkaline cleaner and check for indications (results in following slides)

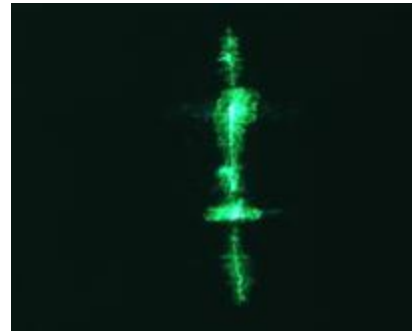
Results of non HT samples after alkaline clean

- 552 shows loss of crack ends in both post alkaline cleaning runs
- 553 was not recovered after the alkaline runs
- 554 lost some brightness after the cleaning, approximately 40%

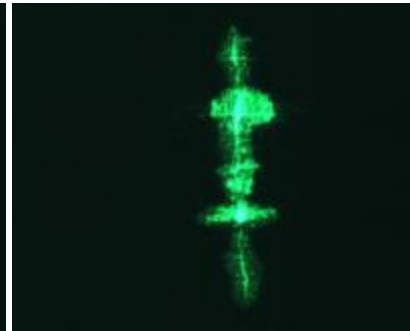
02-552



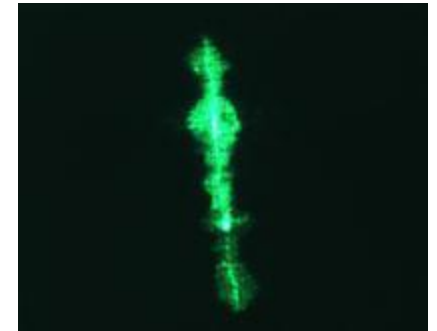
CASIX



B=39.365
E=248ms



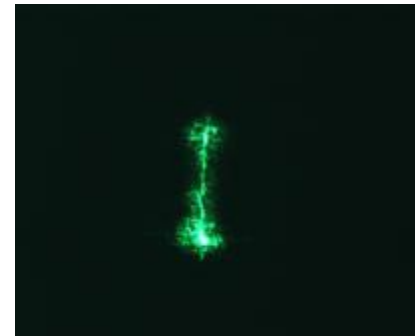
B=58.271
E=220ms



B=50.644
E=255ms



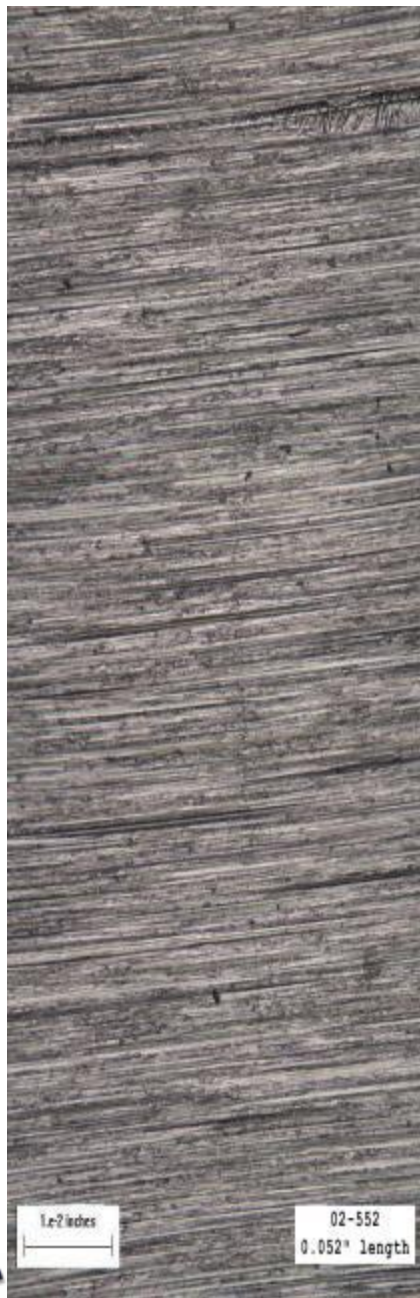
After alkaline clean
B=7.7267
E=1.07s



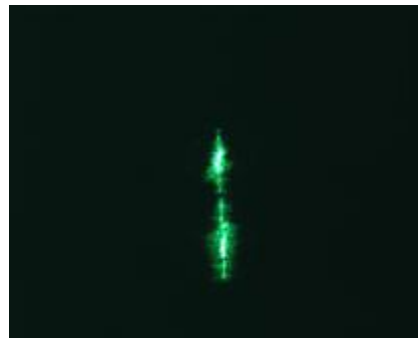
After FPI and Boil
B=15.935
E=443ms

L=0.0965"

02-553



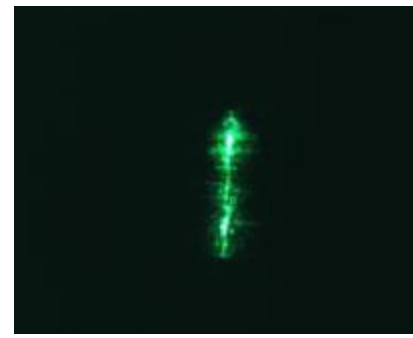
CASIX



B=13.274
E=448ms

After alkaline clean
No Indication

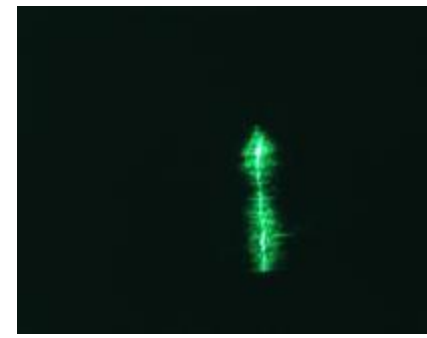
B=0
E=0ms



B=15.696
E=379ms

After FPI and Boil
No Indication

B=0
E=0ms



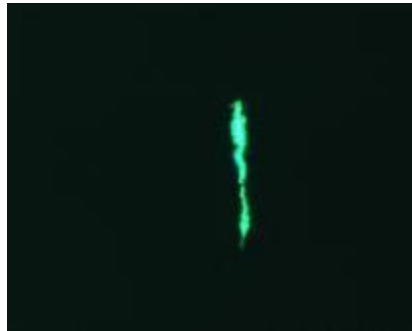
B=15.770
E=476ms

L=0.052"

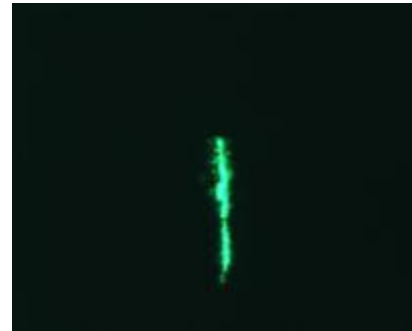
02-554



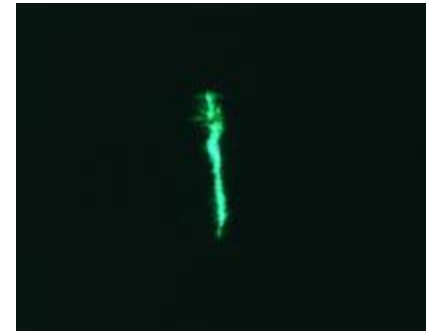
CASIX



B=21.433
E=179ms



B=22.352
E=203ms



B=20.007
E=201ms



After alkaline clean
B=13.247
E=402ms



After FPI and Boil
B=13.465
E=254ms

L=0.051"

Next Steps (14 May 08)

- Boil the 3 samples (552, 553, 554) for 60 minutes and recheck FPI
- Produce 6 new crack samples, 3 w/60 mil cracks, 3 w/100 mil cracks. These 6 samples will be reprocessed the same as before, but with a 30 minute hot DI water rinse at the end.
- Results will be discussed in a call before moving on to reformulating the alkaline cleaner



Questions ?

Cleaning procedure for Recovery Attempts

Organization One Cleaning Procedure – Phase I

Cleaning sequence is as follows:

1. Aqueous clean 20% alkaline general purpose oil, grease and carbon remover at 70 deg C for 30 minutes.
2. Cold water wash and pressure rinse.
3. Condition scale in alkaline deoxidizer at 600 g/l, 90 deg C for 60 minutes.
4. Immerse in 400 - 500 g/l Nitric acid, at room temperature for 30 minutes.
5. Cold water wash and pressure rinse at 1500 psi.
6. Dry off from hot deionized water, 80 deg C. min.

Cleaning procedure for Recovery Attempts

Organization Two Cleaning Procedure – Phase I

Process 1:

1. Immerse in alkaline liquid all-purpose cleaner at 10 %/vol for 30 minutes at 162 F with mechanical agitation.
2. Immerse in flowing DI water for 2 to 4 minutes
3. Immerse in liquid alkaline permanganate scale conditioner for 60 minutes at 190 w mechanical agitation. (Part 1 and Part 2 each run between 15 and 25 %/vol respectively)
4. Immerse in flowing DI water for 2 to 4 minutes
5. Immerse in 20 %/vol sulfuric acid at 130 F for 5 minutes with mechanical agitation.
6. Immerse in flowing deionized water for 2 to 4 minutes
7. Oven dry (air circulating) at 200 F for 30 minutes

Cleaning procedure for Recovery Attempts

Organization Two Cleaning Procedure – Phase I

Process 2:

1. Immerse in alkaline liquid all-purpose cleaner at 10 %/vol for 30 minutes at 162 F with mechanical agitation.
2. Water Rinse
3. Alkaline Permanganate Oxide Conditioning - Standard concentration for hot line cleaning
4. Water Rinse
5. Acid Stripping Solution for Ti
6. Water Rinse

Cleaning procedure for Recovery Attempts

Organization Two Cleaning Procedure – Phase I

Process 3:

1. 20 min in Molten Salt @ 800o F. Dark brown scale was gone and specimens were pretty much metallic color.
2. 5 min water quench
3. 5.5 minutes in 380 g/l HNO₃-7 % clear, colorless liquid acidic compound (fluoride is ~12 g/l). Etch coupon showed 0.5 mils stock per surface removed.
4. Cold water rinse
5. Hot water dip and air dry

OEM 1 Cleaning Process Nov 07

– Phase II

- Due to availability there was a change in the Nitric acid strength from 50% to 25% w/v.
 - I don't believe this is significant from a Ti cleaning / descaling standpoint although I would have preferred to remain with the 50% for consistency.
- The bars went through 2 cycles through the process to try and remove as much discoloration as possible.
- The Process sequence was as follows:
 - Aqueous degrease
 - Immerse in 600g/l alkaline descaler at 90 deg C for 1 hour.
 - Cold water swill and air / water blast.
 - Immerse in 25% Nitric acid at room temperature for 30 minutes.
 - Cold water swill and air / water blast.
 - Dry off from hot deionized water at 80 deg C min
 - Repeat for one cycle.