

Jacquelyn Loven  
Notes

12 June 2017

- It doesn't make sense to not do the reference plane reset on each image because then the software makes you guess where the plane is visually, given a laser intensity profile. Each reference plane has been reset by excluding cracks and possible pileup regions as much as possible (loose) and by choosing the smallest possible rectangle around the indent without touching the indent (tight).
- Areas smaller than 5000 pixels are ignored to get just the indent.
- Just setting the reference plane is not enough to reliably catch indents in microscope slides at loads lower than 1961 mN. Also, primary radial cracks are picked up by the profiler as being part of the indent are when it's just reference planed: Gaussian blur or height cutoff needed?
- For loads 1961 mN and above, how does where you choose your reference plane matter? Tight is an average of  $-3.27 \pm 7.16$  % different in area from loose.

13 June 2017

- Now to remove the effect of cracks in the area, I'm cutting the DCL/ BCL level down. Setting DCL = 1.00% and BCL = 99.5%.
- After doing this, the lower loads (less than 1961 mN) still need some more processing to get an area.
- Some notes about pictures of Caila's indents:

<b>39.75 sample</b>	<b>Primary radials</b>	<b>Secondary radials</b>	<b>Median</b>	<b>Shallow laterals</b>	<b>Cone</b>
<b>490</b>			?		
<b>980</b>			?	X	
<b>1961</b>	X	X	?	X	
<b>2942</b>	X	X	?	X	
<b>4903</b>	X	X	?	X	
<b>9807</b>	X	X	?	X	

<b>45.14 sample</b>	<b>Primary radials</b>	<b>Secondary radials</b>	<b>Median</b>	<b>Shallow laterals</b>	<b>Cone</b>
<b>490</b>			?		
<b>980</b>		X	?		
<b>1961</b>	X	X	?		
<b>2942</b>	X	X	?		
<b>4903</b>	X	X	?	X	
<b>9807</b>	X	X	?	X	

<b>48.51 sample</b>	<b>Primary radials</b>	<b>Secondary radials</b>	<b>Median</b>	<b>Shallow laterals</b>	<b>Cone</b>
<b>245</b>			?		
<b>490</b>			?		
<b>980</b>		X	?		
<b>1961</b>	X	X	?		
<b>2942</b>	X	X	?	X	
<b>4903</b>	X	X	?	X	

<b>60.61 sample</b>	<b>Primary radials</b>	<b>Secondary radials</b>	<b>Median</b>	<b>Shallow laterals</b>	<b>Cone</b>
<b>980</b>	X	X	?		
<b>1961</b>	X	X	?	X	

<b>85.98 sample</b>	<b>Primary radials</b>	<b>Secondary radials</b>	<b>Median</b>	<b>Shallow laterals</b>	<b>Cone</b>
<b>980</b>			?	X	
<b>1961</b>			?	X	
<b>2942</b>	X		?	X	X
<b>4903</b>	X	X	?	X	X
<b>9807</b>	X	X	?	X	X

<b>89.93 sample</b>	<b>Primary radials</b>	<b>Secondary radials</b>	<b>Median</b>	<b>Shallow laterals</b>	<b>Cone</b>
<b>980</b>			?	X	X
<b>1961</b>	X	X	?	X	
<b>2942</b>	X	X	?	X	X
<b>4903</b>	X		?	X	

<b>100 sample</b>	<b>Primary radials</b>	<b>Secondary radials</b>	<b>Median</b>	<b>Shallow laterals</b>	<b>Cone</b>
<b>245</b>			?	X	
<b>490</b>			?	X	
<b>980</b>	X		?	X	
<b>1961</b>	X	X	?	X	X
<b>2942</b>	X	X	?	X	X
<b>4903</b>	X		?	X	
<b>9807</b>	X	X	?	X	

- Cone cracks start at 90% silica at high loads (2942 mN).
- Also I really don't see the indentation size effect (ISE) upon looking again at old data.

14 June 2017

- I definitely see median cracks, or at least ones that extend to the center of the indent at loads of 1961 on microscope glass. Since they don't come out the other side, it's also possible they're just extended radial cracks.
- 2 nm gold not enough. 8-10 nm carbon on top also not enough. It looks dirty but not opaque. Another 8-10 nm on top still isn't good enough.

15 June 2017

- When I plane the profiles, the side that had the "largest pileup" changes. Planing looks like it changes the heights a lot.

16 June 2017

- I have no idea why my data is so bad for how planning affects pileup. Possibly the DCL/BCL cut is removing noise, and the pileup that I saw is just noise. But I need to do that in order to remove cracks from the area measurement.
- Today I think it is feasible to get plots of how cutoff height of the plane changes area measured. I don't need to do any cutoff heights for the larger indents, so perhaps I should just ask for a higher mag lens.
- Exhausting one carbon pencil thing is doing the trick, on top of the other several depositions. If viewed under 10-20x on the microscope, the indent kind of looks black. Much easier to see the edges.
- I have successfully dumped another pencil's worth of carbon onto the sample. I think the pictures are a little better? Difficult to tell.
- Looks like doing the height cutoff for the smaller indents loses about 10% of their area. I think I need a higher mag lens.
- Discuss w/ Nicole:  
[DONE] Get a higher mag lens  
[DONE] How to measure pileup better because how do I know if it's noise or not? Hold off until you get bigger pileups.  
Coating with carbon is sort of working- other ideas?  
Where does it make sense to do the planing, like what's an estimate for pileup width?  
[DONE] What do median cracks look like? Like radials.
- TODO:  
[DONE] Ask Darren about higher mag lens  
[DONE] Metallize samples 10 nm, try indenting before and after  
[DONE] Email Steve Kiske, cc Darren about AFM training (Asylum AFM).  
Use markers to show where indents are. 2 indents, get xyz file, do planing and see if you get the same as the profiler.

19 June 2017

- I put down 10.8 nm of gold on 14 June B. It looks gorgeous! This might work.
- I put down 14.4 nm of gold on Sample June 19. It hasn't been indented yet.

20 June 2017

- I am indenting sample June 19 that had 14.4 nm gold. 4 mm and 4 mm off each side from the corner is where it starts. 1 mm apart. 5 indents, 5 loads.
- Spraying the sample roughly with 70% isopropanol helps make the gold pileup inside the indent edges go down. Still, there's some noise on the edges due to the gold.

21 June 2017

- The evaporator is broken today. In the meantime, I will try to code for noise reduction on data from smaller samples. Ideally, this data would look like what the AFM says.
- The evaporator is fixed! I'm thinking 20 - 25 nm of gold should do the trick.
- Woops! I put down an extra 105.5 nm of gold on sample 14 June B (now total 116.3 nm). Christ. I also didn't rotate it. Well, it looks pretty defined in the optical microscope. Unusually, I don't really see a pincushion shape and instead see mostly square indents. Possibly it is too thick a layer and is obscuring their shapes?
- I should ask about electroless plating. Asked Phil- now ask John.
- On sample 14 June B I added another 78.6 nm (total 194.9 nm). I don't really notice a difference in how the optical microscope sees it...
- I sputtered for ~1 min 10 secs onto sample 14 June, Au-Pd. It's supposed to be a more uniform layer. It's quite black too.
- Dark field images are GORGEOUS. I need to bring my camera tomorrow and take images and see if my area detector can pick them up. Furthermore, there's no significant difference in dark field between 10 and 200 nm, which is great, because now I can just make some samples with a lot less gold.

23 June 2017

- Testing the different glasses with indents, I get a different crack threshold from Caila's data last year, but I get that 85% silica is weirdly crack resistant.

26 June 2017

- My data may be due simply to the fact that the glass is polished a lot nicer than it was for Caila.
- My error analysis shows that area changes linearly with cutoff height. After doing the derivation for a square pyramid with tip angle  $\theta$ , I get  $\left(\frac{dA}{dh}\right)_\theta = 4 h \tan^2 \theta$ , so it indeed looks like a change in h will make the same change in A.
- [DONE] Plot my crack fraction data like Caila's data. Just primaries, and also both.  
[DONE] Do Nicole's 3 samples and do indentations to see effects of surface roughness, full gamut: [DONE] indent and profile them and update Nicole.

[DONE] Profile all new indents. See if roughness is different from Caila's data.

[DONE] Look for new indenter tip.

[DONE] Put data on drive.

[DONE] Take dark field images.

Something useful this weekend would be to write a program to identify cracks.

[DONE] AFM if possible.

[DONE] See if cracks change over time.

27 June 2017

- This is gross. Caila's data corresponds to a surface polish of  $\sim 1 \mu\text{m}$ , whereas my samples were polished at  $0.5 \mu\text{m}$ .
- Something I didn't do to the samples that are polished differently: I didn't go to the lowest load on all of them, which would be good to have since it could tell me if small indents are affected by surface more than larger ones.

29 June 2017

- Taking gold palladium pictures under 20x in the darkfield microscope yields pretty clear images. I will ask for a camera for the scope.
- I also took pictures of the gold 200 nm. It also looks good.
- The surface of the 12 June carbon sample looks weird, like it's covered in plates.

30 June 2017

- Before I start AFM, I know I can only do a few samples, so I need to choose. The problem right now is that the laser profiler can't accurately measure the sizes of small indents due to thermal noise. My job is to see if my correction (moving the plane of reference down about 0.015 microns) alters the result in a linear fashion. I already have profiles of every indent except fused silica. Ideally, I AFM all loads to see the size of the small loads and verify the profiler's reading of the large loads. I don't have time for that. I should choose one glass sample, read two indents each of 245.2 and 490.3 mN indents, and maybe two of the 2942 mN indents.
- Okay, well it was impossible to find the small indents. Especially since they were so close to the edge of the sample. I am gonna come in with microscope slides with my small indents in the center for Thursday.
- Looks like the silica sample wasn't polished? It looks like there are two sets of indents side by side.

4 July 2017

- No difference in hardness between differently polished samples. But cracks differ.

5 July 2017

- So what's the plan? I need to remember that if my evaporator project isn't complete in the next week, it doesn't make it in the paper. I need to prove

- that it works. So, if I do a sample, laser profile it and get areas, and prove that my evaporator technique gets you closer to the real area than without it then it's real progress. I also need to prove that this technique gets you closer to the real areas for the smaller indents, which I don't actually know. AFM will hopefully tell me that tomorrow.
- Remainder of this week's TODO:
    - [DONE] Check roughnesses of the glass of the 3 polished samples and see how that compares to Caila's data.
    - Possibly a computer program to detect cracks.
    - [DONE] Analyze the data I got last week to see how cracks change over time for the newly polished samples from Corning.
    - [DONE] Create samples for AFM tomorrow and for profiling and evaporating for proof of concept.
    - [DONE] Run new images through original image processing software to see if it picks them up. Needs work LOL.
  - So for the actual samples for AFM, etc.:
    - [DONE] One sample with the two smallest loads and one of the larger loads (maybe just the whole gamut?) on microscope glass. Profile this ahead of time. Mark clearly where the indents are.
    - One fully indented sample (all loads), which is profiled, analyzed with reticule method, and then evaporated for proof of concept. Requires working evaporation technique. Can be done next week.
    - [DONE] Five samples with three indents for each load for evaporator practice. Profile some of these ahead of time.
    - [DONE] Ask Nicole about how to get a microscope camera.
  - Samples 5 July A, B, C, D, and E and each have 3 indents each for 9807, 4903, 980.7, and 490.3. Sample 5 July is for AFM.
  - Roughness parameters in the Keyence software:
    - Sa is mean height. Extension of linear Ra.
    - Sz is max height from lowest part to highest part. Extension of linear Rz.
    - Sdr is how much extra surface area you get from the roughness as opposed to a plane (0).
    - Spc is the inverse of the size of curvature of objects sticking out of the surface.
  - Inconclusive what the "real" roughness of Caila's indents is (it's hard because I didn't clean them prior to profiling). My newly polished samples, nominally at 0.5  $\mu\text{m}$ , check out and seem to be between 0.5  $\mu\text{m}$  and 1  $\mu\text{m}$ .
  - Samples D and E have yet to be profiled.
  - Doesn't look like there's anything particularly interesting with the cracks over time on my newly polished samples from Corning. Cracks over time increase for the four highest silica content glasses, and even then they don't increase by that much. Not sure why it's so much more dramatic for the older samples.

6 July 2017

- AFM all day today! Woo. For whatever reason the AFM says indents that the profiler says are  $\sim 3 \mu\text{m}$  deep are actually like  $1.2 \mu\text{m}$  deep. Not sure what the deal is... I'll just take the data and ask Nicole after.
- I did two AFM profiles each on the 2942 mN, 490.3 mN, and 245.2 mN. Analysis will have to be another day since someone else has the AFM now.

7 July 2017

- Today I'm gonna try to get a working evaporation technique. I'll see if I can start with the gold-palladium mix.
- I put sample 5 July A under the gold-palladium thing for 60 seconds. 5 July B was put under for 30 seconds. 5 July C was put under for 90 seconds. All were masked so later I can tell how thick the coating is, I just need to take them to the profiler.
- Profiler is not good enough to discern the thicknesses of such thin coatings. Also AFM says the indents aren't even close to square, more like a weird squished rhombus. Time to go home...

10 July 2017

- Need to ask about getting a camera.
- Just kidding? Unclear. The DinoXcope camera might be good enough.

11 July 2017

- Plans for this week: I need to characterize the coatings that work best. To do that, I need software that works on at least one of the coatings. So today, I'll make that.  
Unfortunately, the scale bars in images are all gonna look different. Maybe a dialog box to trace over the scale bar? Maybe if they click on the scale bar I can grab  $\pm$  some percentage of each pixel value and grab the scale bar size automatically
- Software, TODO today:  
Dialog box to trace over scale bar and input its size  
Dialog box to select images, which should be named  
<LOAD> <NUMBER> <...>. <extension>  
Dialog box for selecting a location for the final text output file
- [DONE] Also need a camera for the dark field scope, ask for a quote.
- Nicole is gonna help me with starting outlines for three papers I'll help write. A method paper on metallizing glass, a short paper on how roughness affects crack resistance, and a paper detailing the hardness differences in various calcium aluminosilicates.
- Here's a wrench: OpenCV needs to somehow be packaged in my app, or I need to make some sort of dynamic site where people can upload their images. Agh!
- It could also be really cool to try to view the indents in UV or IR, where glass is opaque. Could be better than metallization.

12 July 2017

- I've been looking into cameras. The near-IR one is way too expensive. The Dino-Lite one seems good, but I want to make sure that it's significantly better than the one we have.
- I'm also working on getting OpenCV to work in Java. It will be easier to package libraries this way (? that's what Odell said).

14 July 2017

- Nicole has made me an outline for the paper. I should start putting my thoughts down this weekend.
- They've decided that if it takes 2 extra weeks to remove the IR filter then I shouldn't do that. I will call today and see if they can expedite the process for relatively cheap.
- Today on AFM I'm gonna try:  
A stiffer tip  
[DONE] Contact mode  
[DONE] Changing gain
- Today I will also:  
Rewrite my code in MATLAB if possible  
[DONE] Call Dino-Lite about expediting shipments. Ugh can they answer their phone!
- New tip bought (\$40): Olympus RC150VB, 0.03 N/m and 0.06 N/m spring constants.
- So turns out AC mode is bad for such deep indents. The air displaced by the oscillating dip dampens it and can prevent it from getting all the way into the hole. The contact tip is good.
- I fixed the issue I had before with the distorted indents. I was flattening parts of the image that weren't being flattened! Live and learn.
- Finished imaging the 12 indents. I just redid all of them, I could have used old images but they weren't named well. Analysis is this weekend.

16 July 2017

- So I have to write some code to find the areas from AFM images. Shouldn't be hard at all, I can make a mask of the surface that's like red or something.
- I have no idea what happened – the small indent pictures are the same as the medium ones. That can't really be possible unless I was suuuuper tired and either did the same set twice or overwrote them. I can still write an analysis program for the large ones before my next AFM time on Wednesday.
- Todo today:  
[DONE] Write program to get areas from AFM images.  
[DONE] Rewrite code in MATLAB

17 July 2017

- So I got the area defined from coated glass images in MATLAB. I just need to convert pixel count to a size by detecting the error bar.
- [DONE] I also need to write something quick to find AFM areas. Both should be accomplishable tomorrow.



- Then Wednesday I get the AFM images I missed and analyze those. (??)
- [DONE] Also need to order camera.

18 July 2017

- [DONE] Gonna make the AFM program. Probably just in MATLAB.
- Probably I should invert the masks so they have the indent in red. Makes my life easier since the picture isn't exactly 40  $\mu\text{m}$  square.
- I'm not sure what the error bar is gonna be for the images, so I won't worry about it for the program that I made for the indents. I'll just make sure it's grabbing the right area.
- After accidentally making a program that just binarizes metallized images, it's possible that the metallization method is so good that that's all I need to do to them to get the indent size.
- Until I get AFM data and the new camera I don't think I have anything else to do today.
- Time to start the paper! Ahh

19 July 2017

- Wait why don't we measure fracture toughness based on crack length in the microindenter?
- Some relevant things for AFM: Used Asylum Research ALL PEEK cantilever holder, Olympus RC150VB 0.06 N/m tip.

Scan	Scan size	Scan rate	Scan angle	Set point	Integral gain	Points & Lines
	$[\mu\text{m}]$	$[\text{Hz}]$	$[\text{°}]$	$[\text{V}]$		
Smallest 1	9.20	0.50	45.00	1.065	5.56	256
Smallest 2	11.00	0.50	45.00	1.000	5.56	256
Smallest 3	9.70	0.50	45.00	0.798	5.56	256

24 July 2017

- Analyzing AFM data.
- Also the new images in the new camera look great, but there's a lot of reflection at 20x. I am now using 10x, hopefully resolution is good enough!
- It makes sense to compare diagonal lengths and diagonal depths. Makes sense to compare lengthwise depths but not cross sections because I can take them at any angle.

26 July 2017

- Working on the paper. I finished Abstract, Intro, and Methods. I am about to go take photos for the metallization results. I am thinking that because of the way I chose a different threshold for the AFM such that 75% or more indents were defined, I should maybe do the same for the laser profiler. Changes the methods to better mimic what I did on AFM instead of being something different and arbitrary.

- [DONE] Oh right, I also need to evaporate onto the sample I used for AFM. I should clean it prior. Which will take off the Sharpie marks but should be okay.
- [DONE] I also need to take a picture of a reticule.
- Tonight I write results (should be just one table and a couple sentences?) and Discussion (sources of error) and Conclusions. Should also drop in some sources.
- Remember to mask the slide to get a step edge on the coating! Sample 5 July D does not have a step edge but was sputtered for 60 seconds.
- Emailed Don for Au-Pd composition.
- Goddammit. The Desk II sputterer gives a different coating every time I use it (!?!!) so I need to make more samples and test out the Desk V to see what I gotta do.
- Sample 26 July A has a 60 second sputter. 26 July B has 30 second sputter. 26 July C has 45 seconds. Sample 26 July D, 26 July E, and 7 July B have 38-second sputters. I was trying to replicate the coating on 5 July A by looking at its color. 26 July F is blank but has a Kapton tape mask.
- Okay so this is super dumb. I was saying "darkfield" all this time when it was lightfield. My results are just from another microscope. Which is okay. They're still not amazing, but I've clearly eliminated a lot of disturbance from cracks. I think I will try to dump some more Au-Pd.
- I also need to CLEAN THE CAMERA. Somehow it's already grimy.
- Dumping 240 seconds of Au-Pd on sample 26 July F. It is opaque but not under light. I'll try one of the less thickly coated samples from earlier today now. 7 July B is put under 202 more seconds for a total of 240 seconds.
- 240  $\mu\text{m}$  is good.
- Okay so it looks like the profiler overestimates areas and depths as much as 30%. Yikes. I'm skeptical of areas I got from AFM but I trust the depths and they're less. I should write a program tomorrow in MATLAB to get the depths from images. Maybe I'll just do that tonight.
- Need to AFM step edge. Because laser profiler can't pick it up.
- Also sorry everyone but I don't think the ISE is a thing.

27 July 2017

- // TODO today:
  1. Write program to analyze reticule image
  2. Coat AFM sample
  3. Take photos of AFM sample, clean camera
  4. Run program on each image.
  5. Get image for paper of area defined
- Maybe if instruments didn't break I could actually get through my // TODO lists.

1 August 2017

- Looks like the sputterer (even after being set to  $6 \times 10^{-4}$  Torr) is at  $6.5 \times 10^{-3}$  Torr.

- I have to redo AFM data because the thresholds I was using were too coarse and I can get closer to the surface.
- The crud on the lens is messing up readings for small indents. I need to edit them a bit before running the program on them.

9 August 2017

- This is annoying. I need higher opacity on the red for the indent images. Field trip to Clark!
-