Improved Sample Notching for the Pennsylvania Notched Test (PENT)

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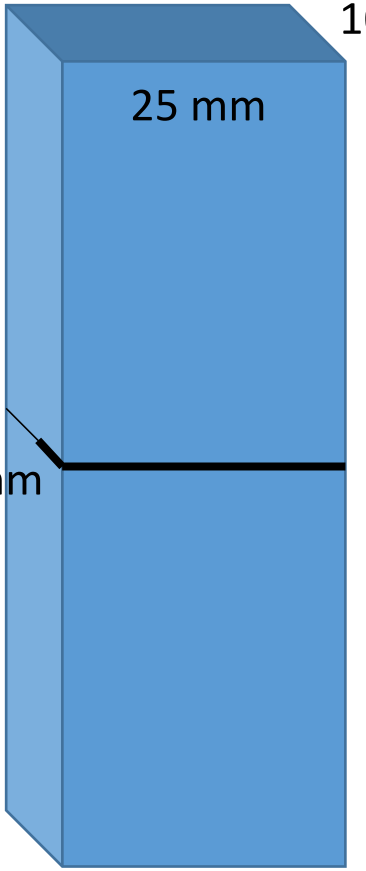
BACKGROUND

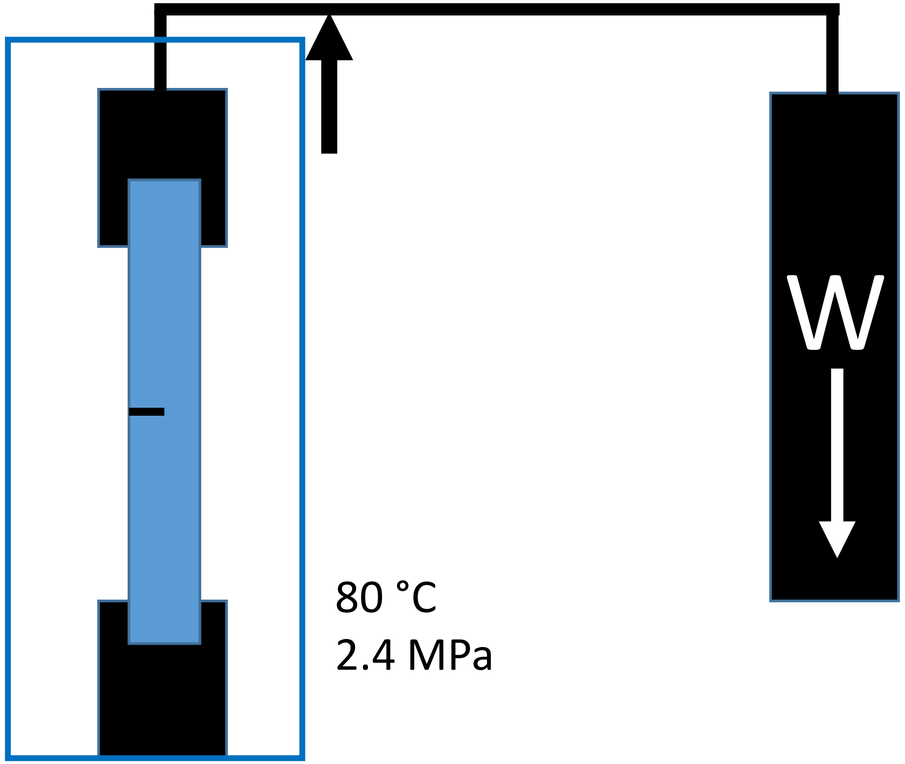
PEnnsylvania **N**otched **T**est :

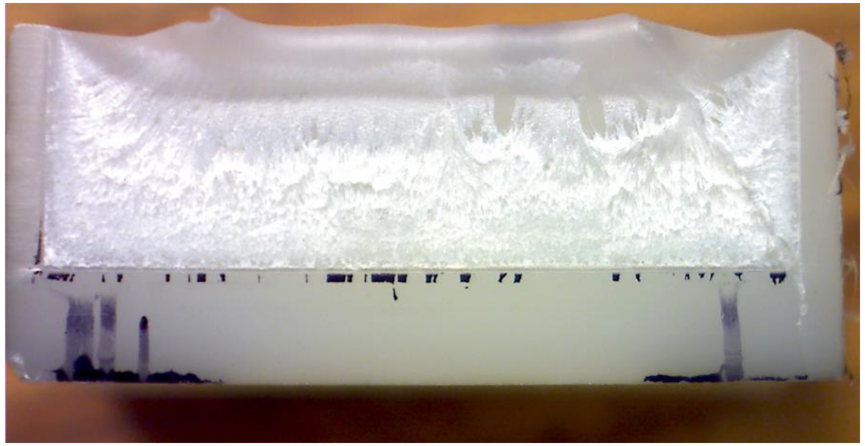
(developed by Norman Brown c.1992)

PENT - test for pipe grade materials, looking at the resistance to slow crack growth

Good samples will run for > 1 year (+10,000 hrs)



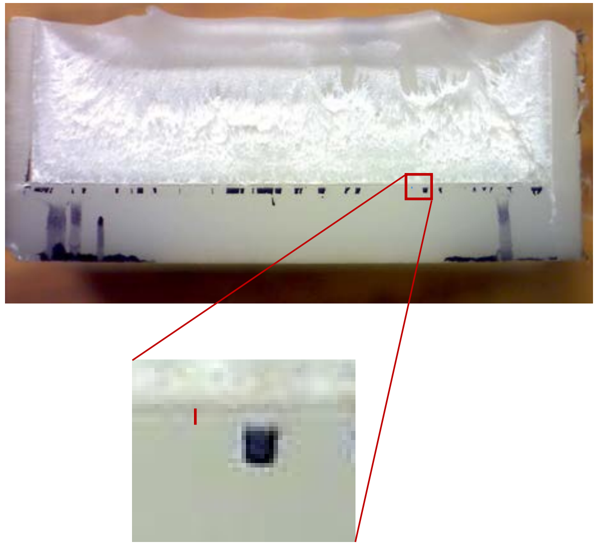




Early 2016 we received a new PENT notcher

As part of our on-going improvement program, we did a ‘deep drill’ exercise on our PENT procedure where we look in detail at our test methodology. In particular we wanted to ensure we are continuing to be within the ASTM specifications

ASTM F1473 specifies that the notch depth should be accurate to +/- 0.05 mm



The notch depth is set by a calibrated micrometer on the notching machine – the zero point is set where the blade just contacts the sample and it is then backed off from a micro-switch to the required depth

We have developed a procedure that allows us to non-destructively measure the notch depth

EFFECT OF ERROR IN NOTCH DEPTH ON THE FAILURE TIME

In fracture mechanics the failure of a sample is not simply driven by the magnitude of the load applied but is also a function of the geometry of the sample and in particular the notch depth. These factors are captured in what is called the Stress Intensity Factor, K. The general form of the stress intensity factor, K, is:

$$K = \sigma\sqrt{\pi a}\, Y\left(\frac{a}{b}\right) \qquad \text{Eq. 1}$$

Where σ is the applied stress, a is the notch length, b is the sample thickness and Y is a geometrical factor. For the case of the PENT sample geometry, Y is given by:

$$Y = 1.12 - 0.231\left(\frac{a}{b}\right) + 10.55\left(\frac{a}{b}\right)^2 - 21.72\left(\frac{a}{b}\right)^3 + 30.39\left(\frac{a}{b}\right)^4 \qquad \text{Eq. 2}$$

The PENT test is run at a stress intensity value of 0.467 $MPa.m^{1/2}$. Obviously, if the notch depth is incorrect, this will change the actual stress intensity factor and hence affect the failure time.

In papers by Lu and Brown[1,2], they state a general form for the failure time of a PENT specimen is:

$$t_f = \boxed{Rs}\, K^{-n} \boxed{\exp\left(\frac{Q}{RT}\right)} \qquad \text{Eq. 3}$$

Where Rs is the resistance to crack growth, K is the stress intensity factor, n is a constant (varies from ~2.5 to ~4 and is most typically ~3), Q is the activation energy, R is the Gas Constant and T is the temperature. Thus, *for a given resin*, we can write:

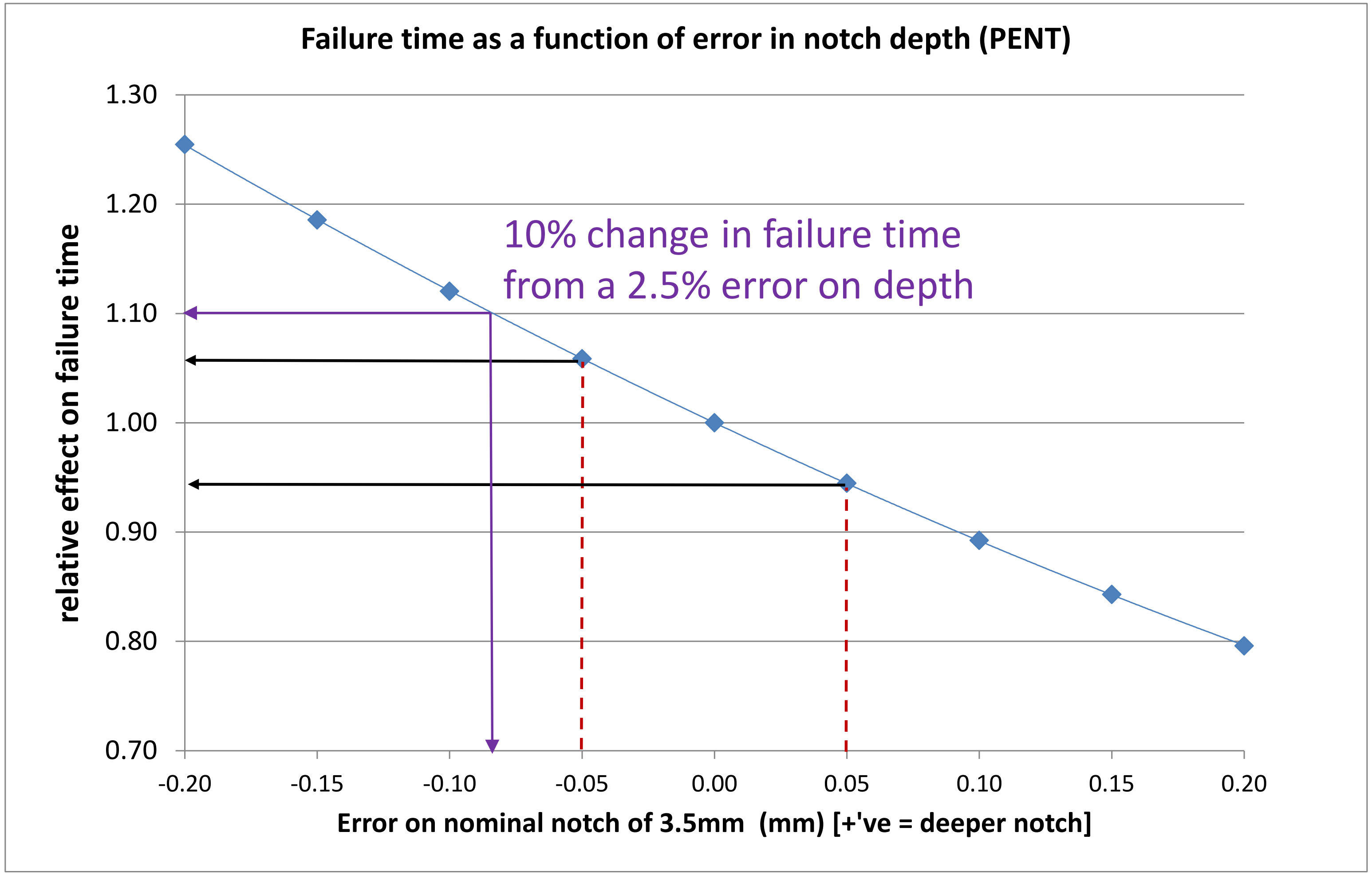
$$t_f = A\, K^{-3} \qquad \text{Eq. 4}$$

Using Equations 1 and 2 we can calculate the change in K for a given error in the notch depth. Using Equation 4 we can then estimate the effect this will have on the failure time. We normalize the change in failure time to the failure time at the correct notch depth of 3.5 mm (on a 10 mm thick sample), i.e. t_f is defined as = 1 for a notch of 3.5 mm.

1. X. Lu and N. Brown, J. Mater. Sci., **25**, 29 (1990)
2. X. Lu and N. Brown, J. Mater. Sci., **26**, 612 (1990)

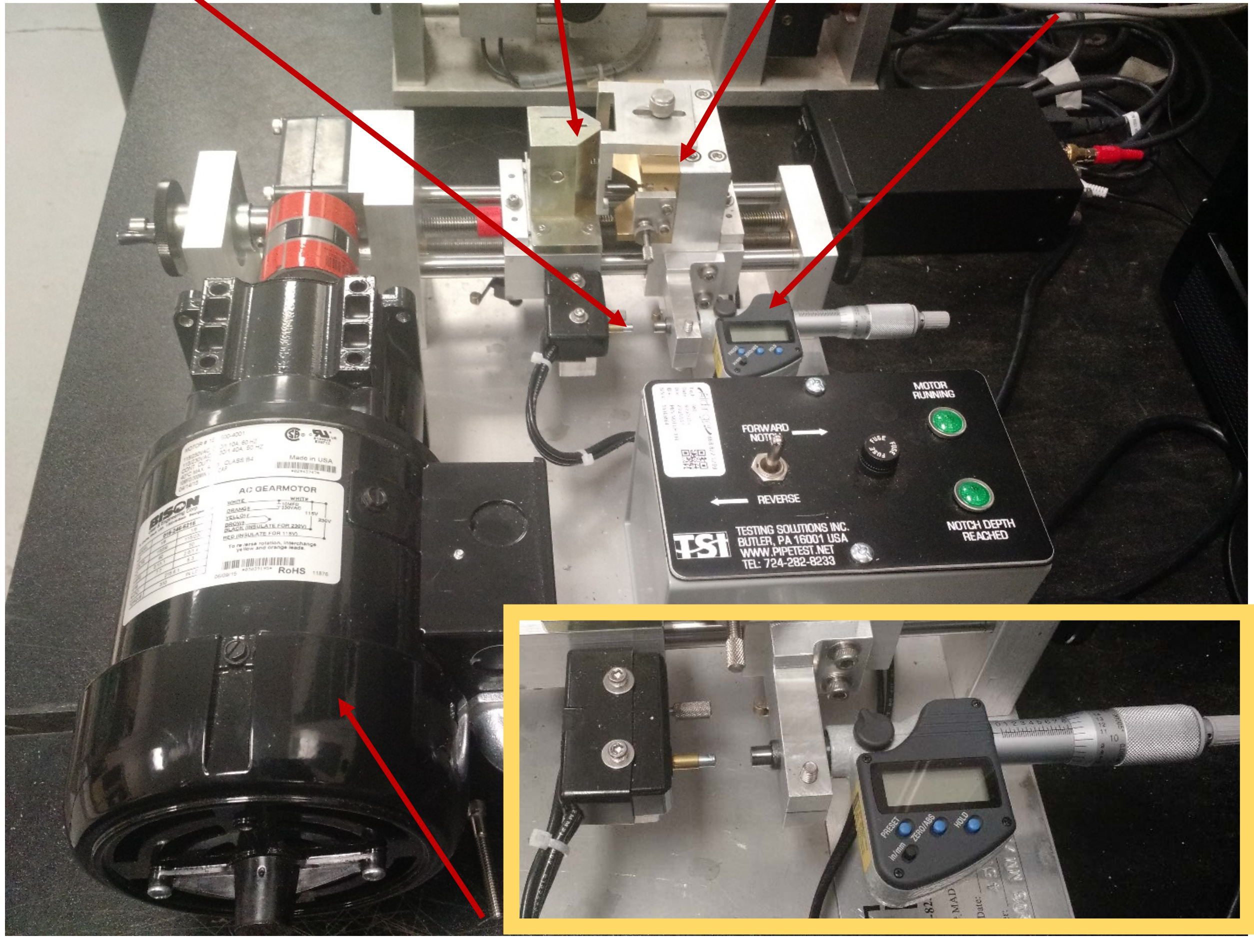
PEnnsylvania **N**otched **T**est : PENT

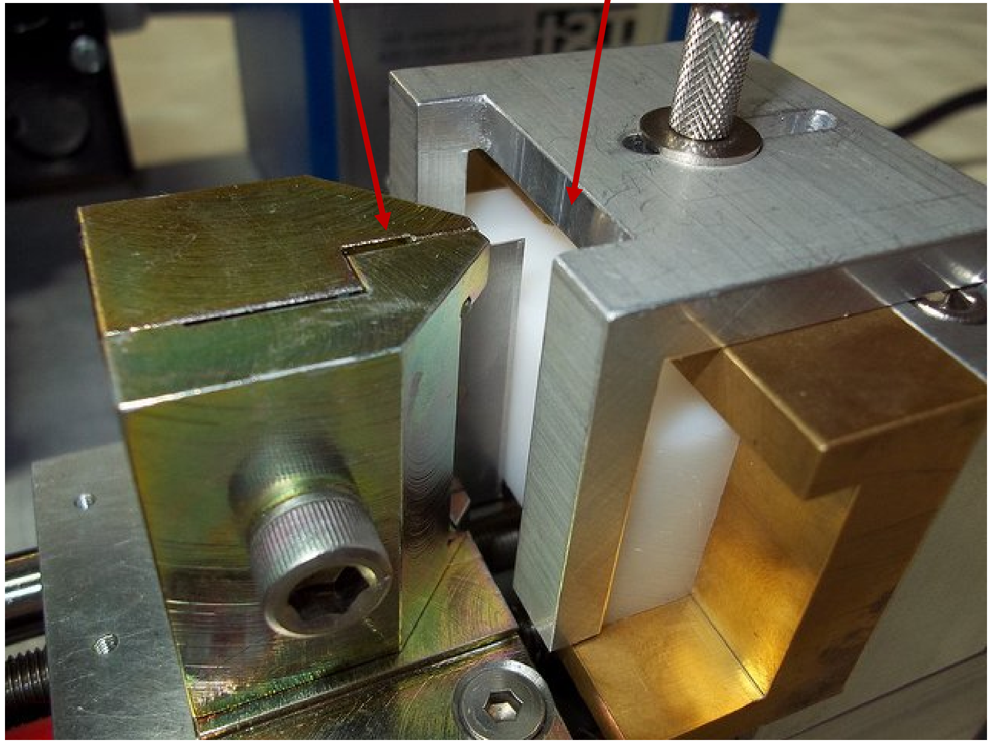
The failure time is very sensitive to the notch depth – we can estimate the expected error for a given notch depth error



ASTM F1473 specifies that the notch depth should be accurate to +/- 0.05 mm (1.4%)

NOTCHER

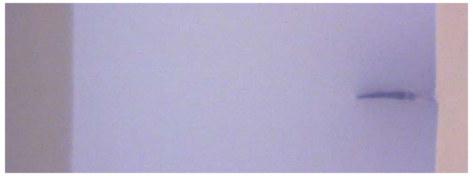




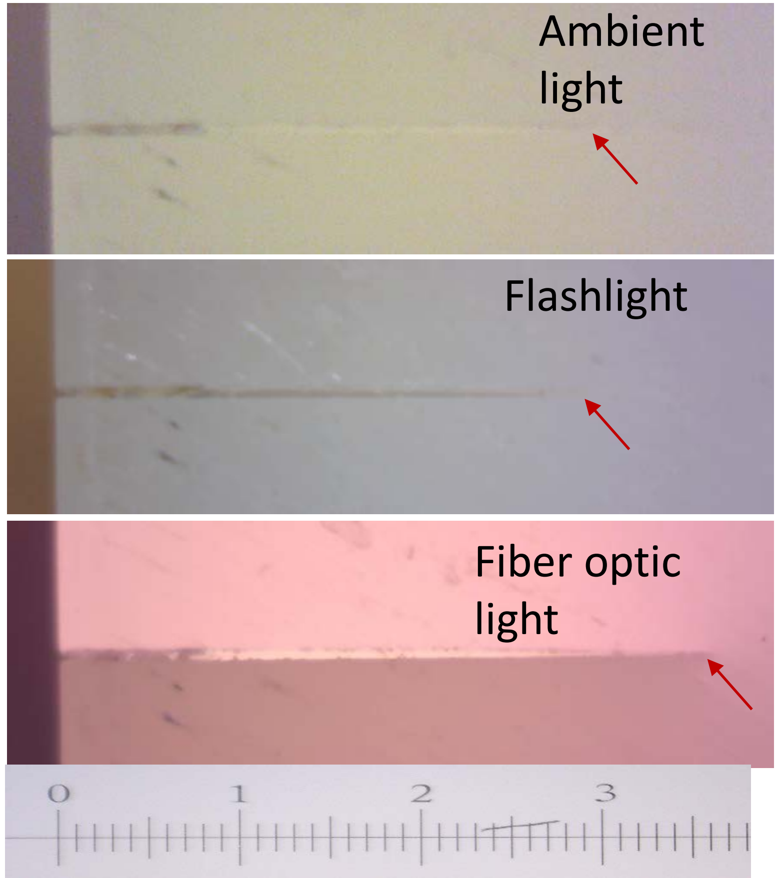
Slow - ~ 20 minutes to notch 3.5 mm

NOTCH MEASUREMENT

First, we needed to figure a way to measure the notch without pulling it apart. We use a benchtop microscope to verify the notch depth for other tests requiring a notch - we ‘stain’ the notch with ink.

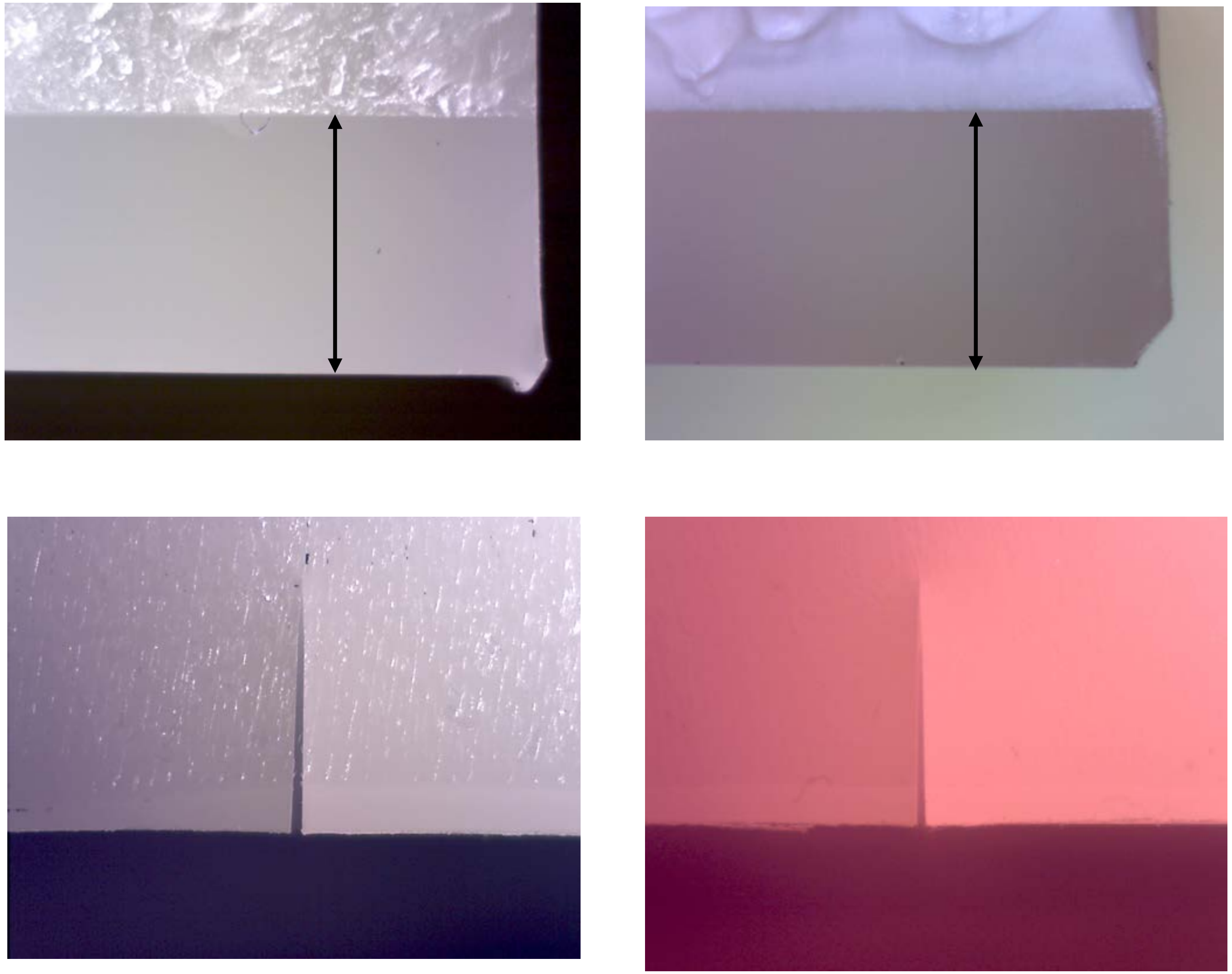


Can’t do that here in case the solvent affects the sample. We tried several microscope options but ultimately found that we could use the same benchtop microscope with appropriate lighting:



NOTCH MEASUREMENT

Validated our method - measured the same samples on our microscope and on cryo-fractured sample surface



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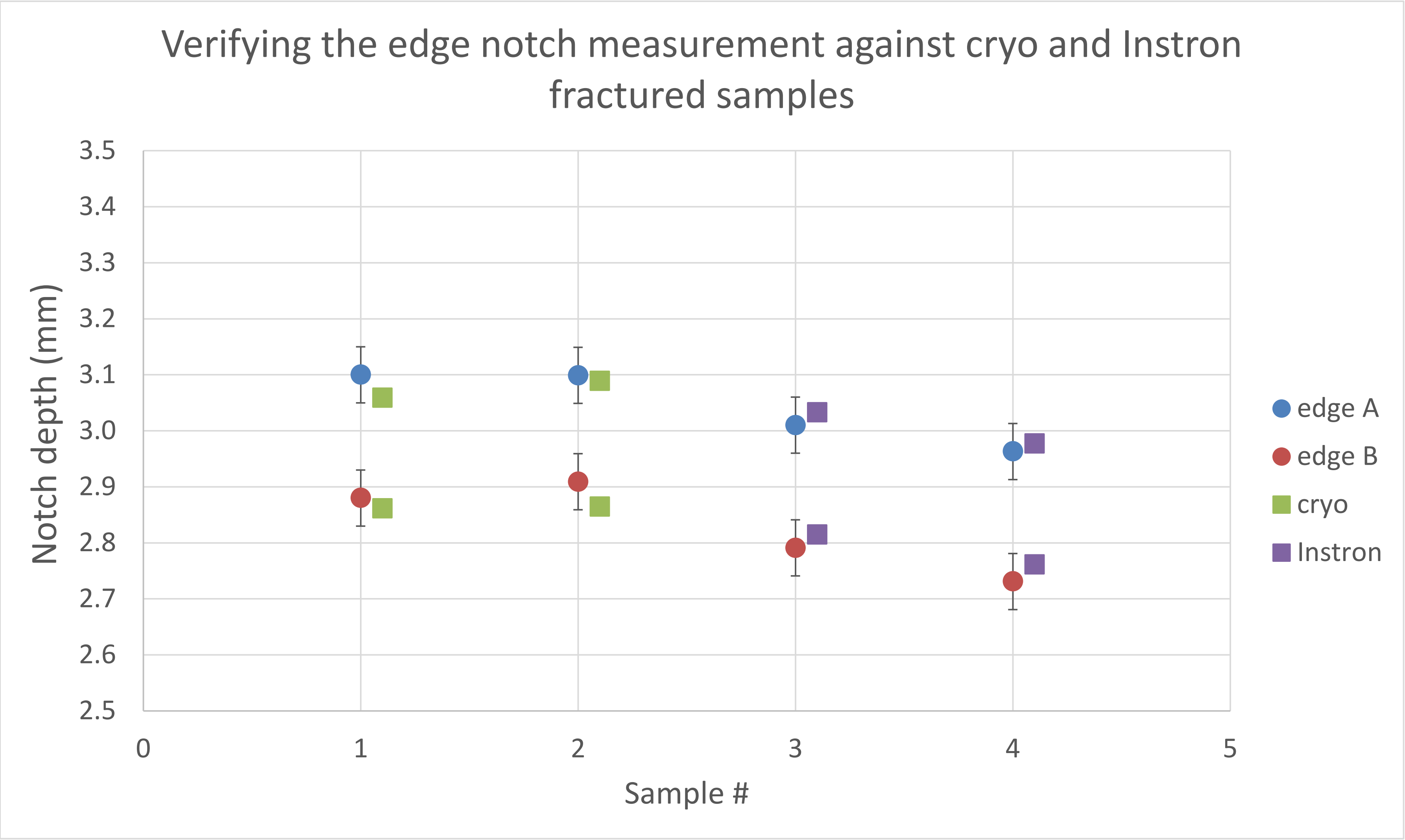


Average difference is ~ 0.03 mm

NOTCH MEASUREMENT

Note: There is an inherent uncertainty on the notch measurement since the selection of the start and end points is subjective

Image taken and analyzed by our microscopy group.

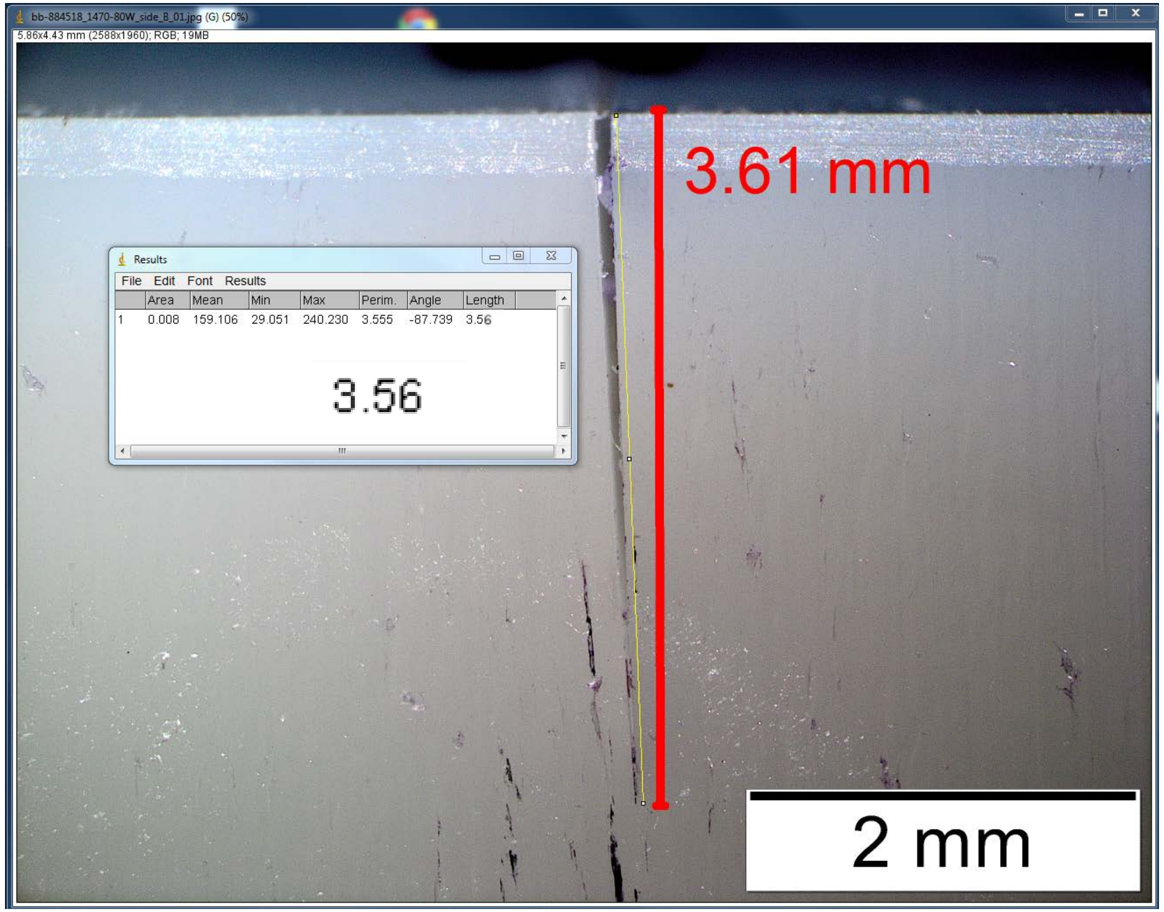
Also analyzed with ImageJ by POC

Microscopy = 3.61 mm

POC ImageJ = 3.56 mm

Delta = 0.05 mm (+/- 0.025 mm)

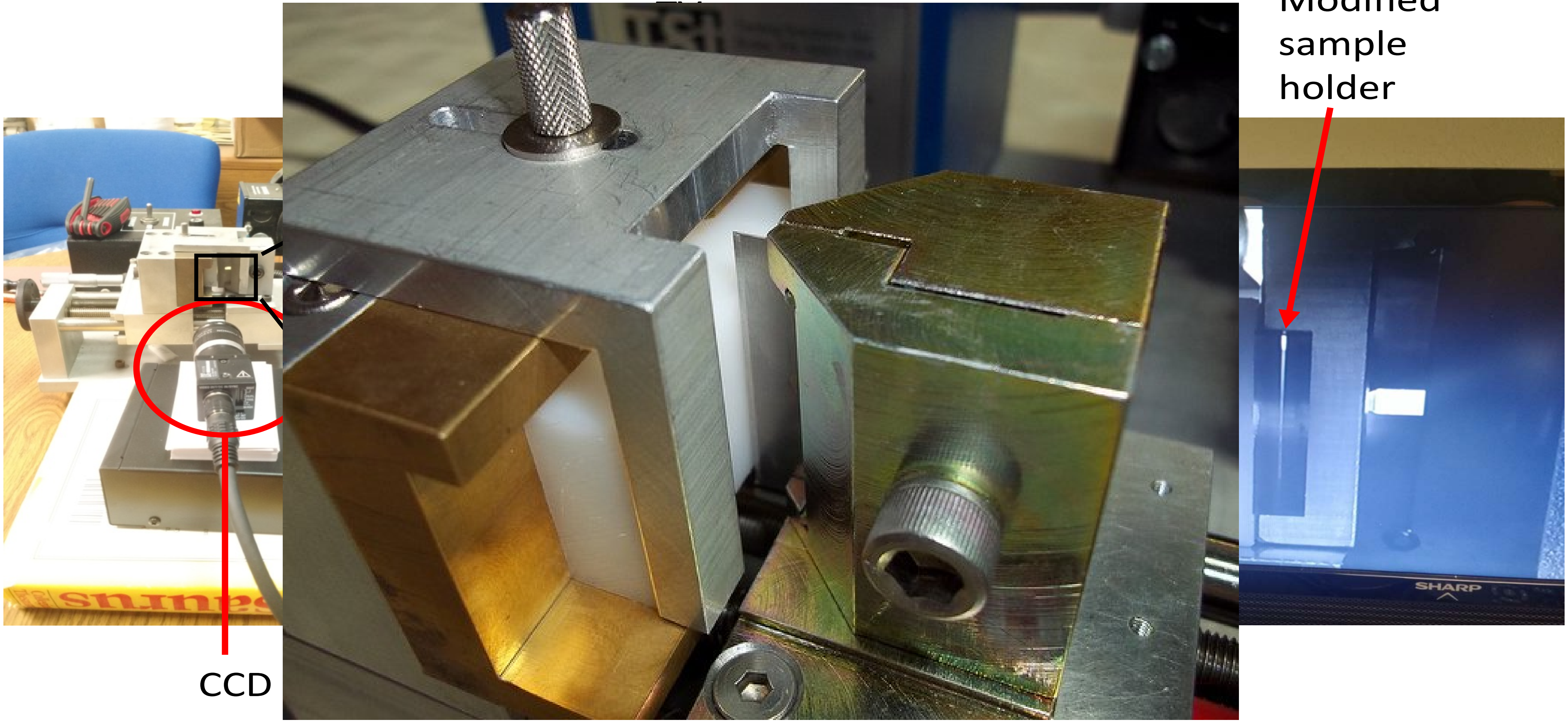
0.05 mm is the allowed tolerance from the ASTM standard



IMPROVED OPTICS

Improved optics to the system to better view the sample\blade interaction

Modified sample holder



CCD camera

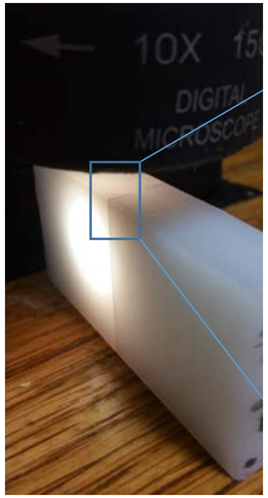
INITIAL RESULTS – NEW NOTCHER USING OPTICS

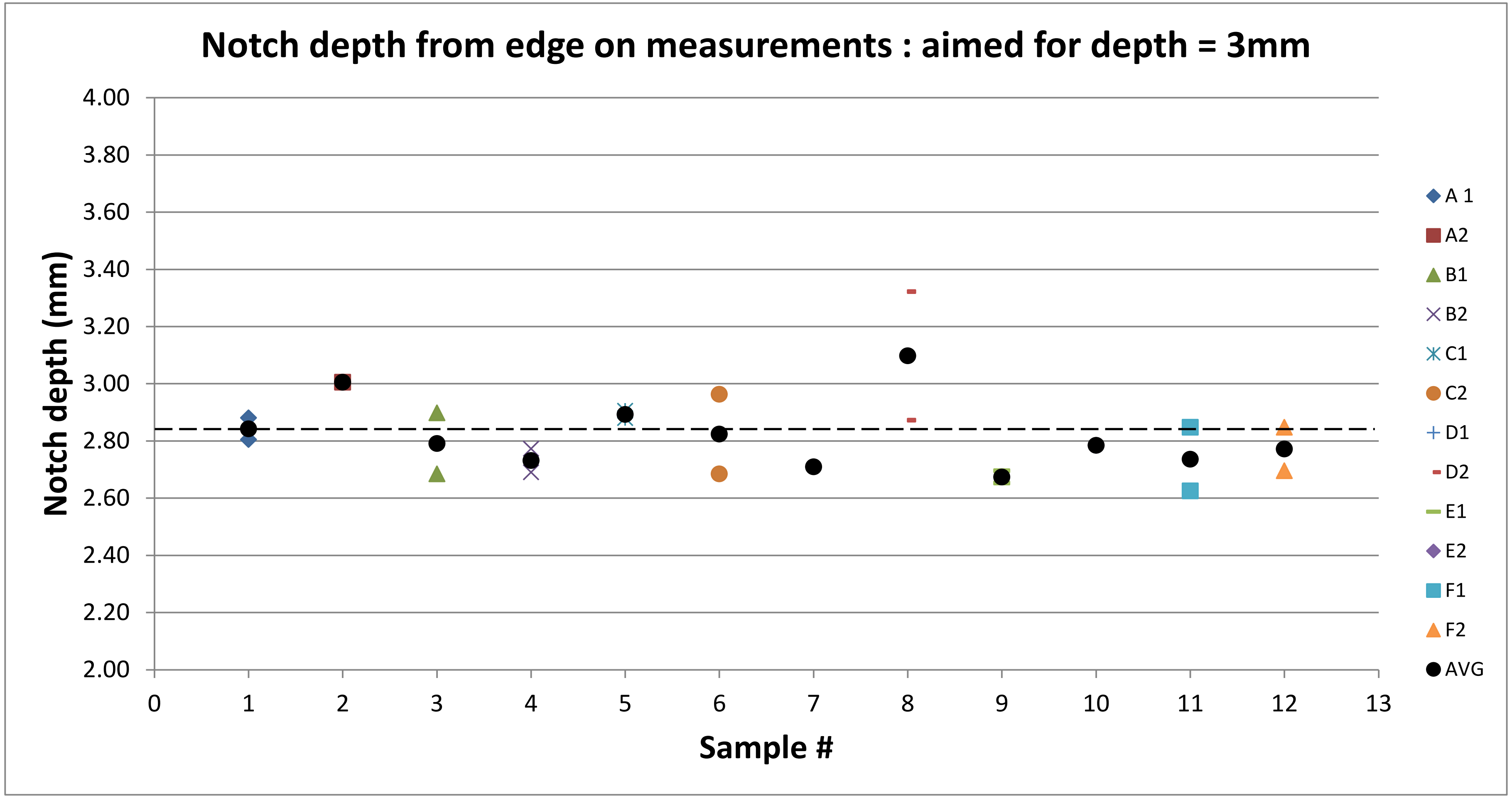
Notched 12 samples with an aimed for depth of 3 mm : We can measure the notch on both the top and bottom edge – gives two readings per sample.

The notch depths were approximately +/-0.2 mm

They were shallow by about 0.18mm (average depth ~ 2.82 mm)

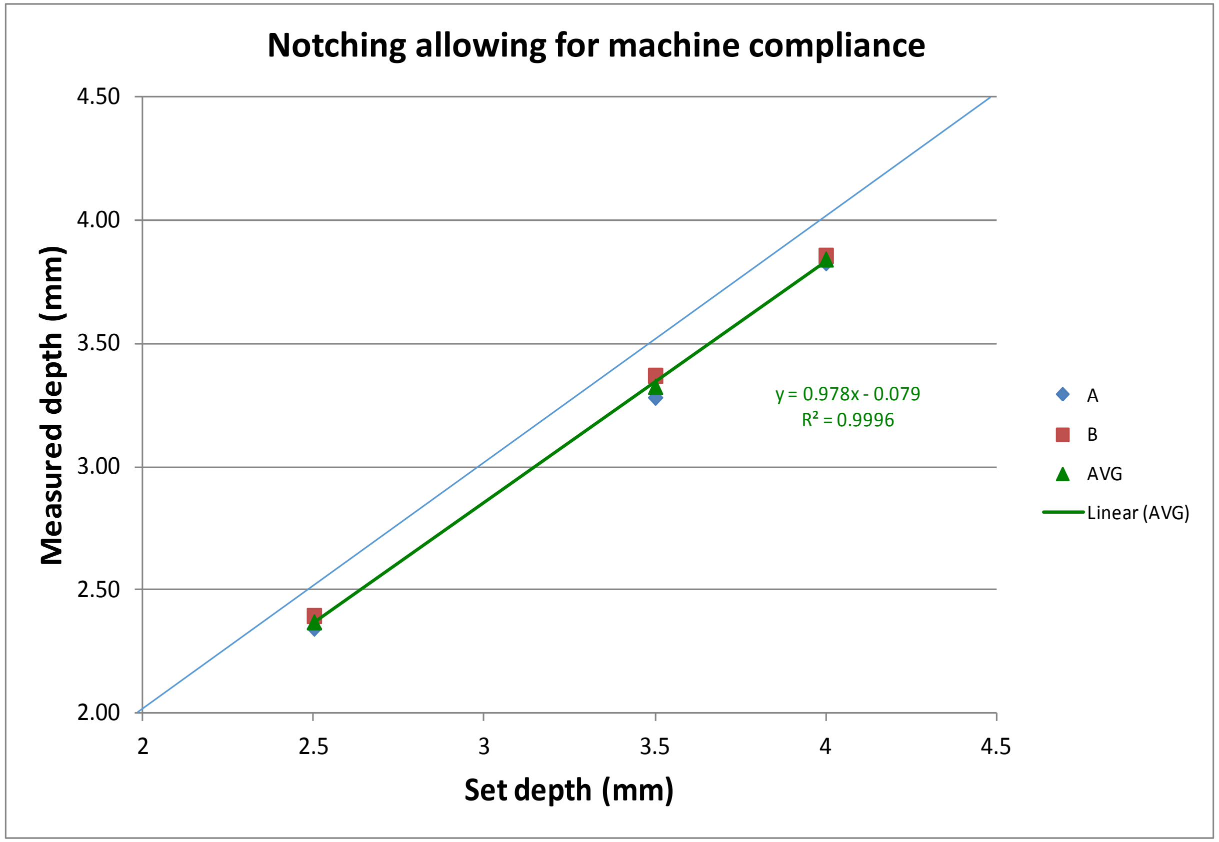
Offset was thought due to machine compliance





MACHINE COMPLIANCE

Ran several samples at different set depths and measured actual notch depth



Measured depth is less than the set depth by ~0.18 mm

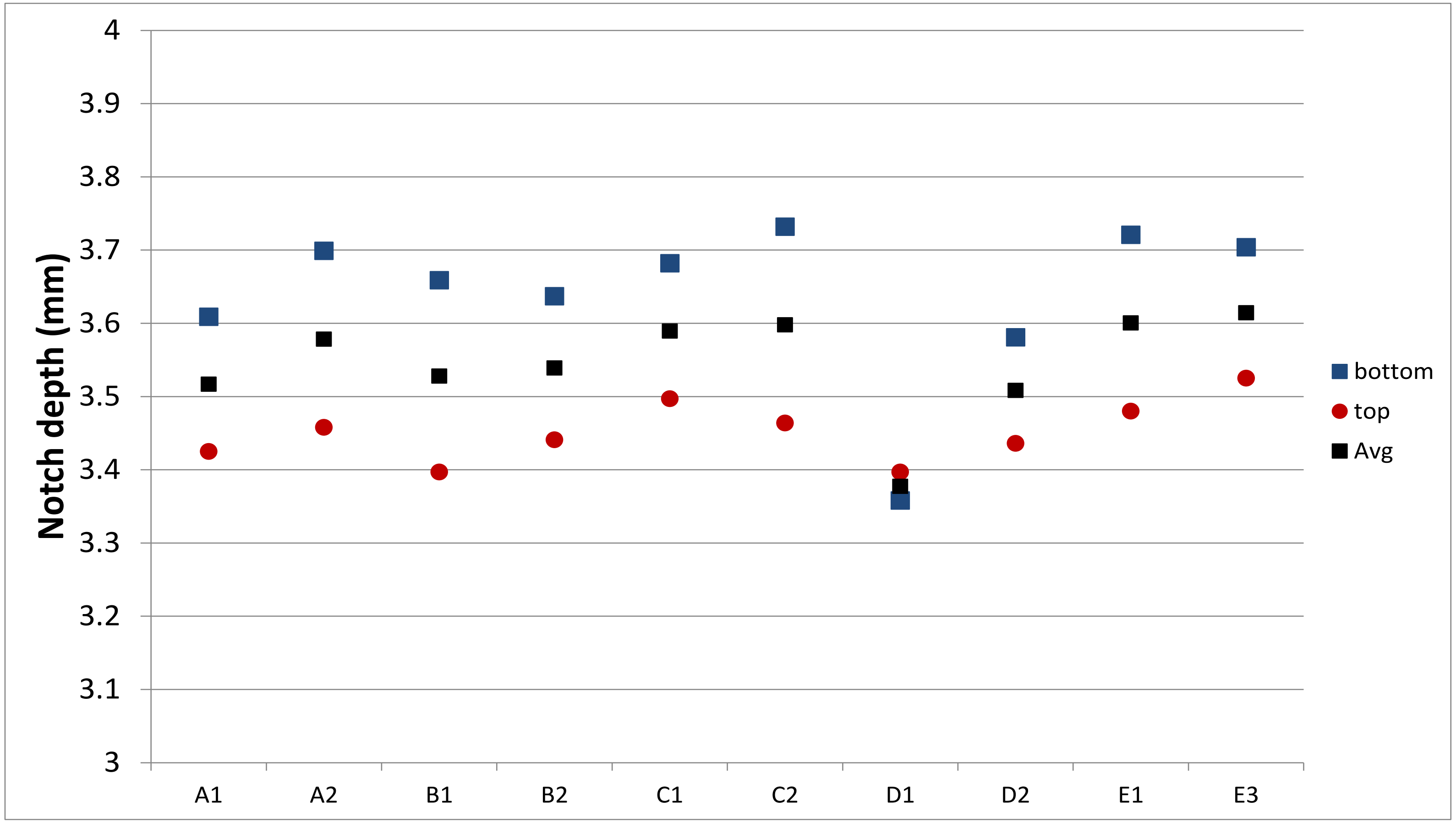
We allow for machine compliance by aiming for a deeper notch (compliance correction term)

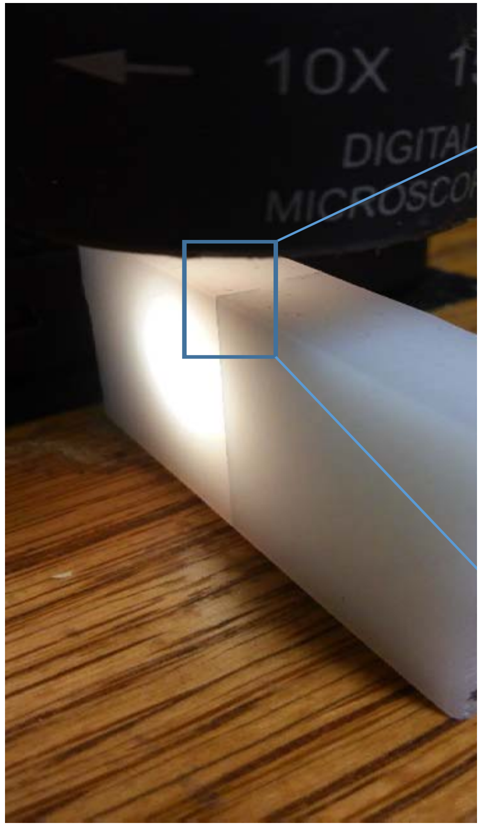
RESULTS USING THE COMPLIANCE CORRECTION TERM – aimed for depth = 3.5 mm

With compliance correction, the average notch depth is 3.55 +/- 0.05 mm. *So, within the ASTM tolerance (+/- 0.05 mm)* ☺

Over compensated on the compliance term by ~0.05 mm.

Top to bottom variation is large (~0.2 mm) -> blade is ending up not parallel to the sample





MODIFYING THE BLADE LOCKING PROCEDURE

Looking at the blade holder:

Backing ridge interfered with the clamp – removed it

Loosely clamp sample, get it parallel to surface and then lock down

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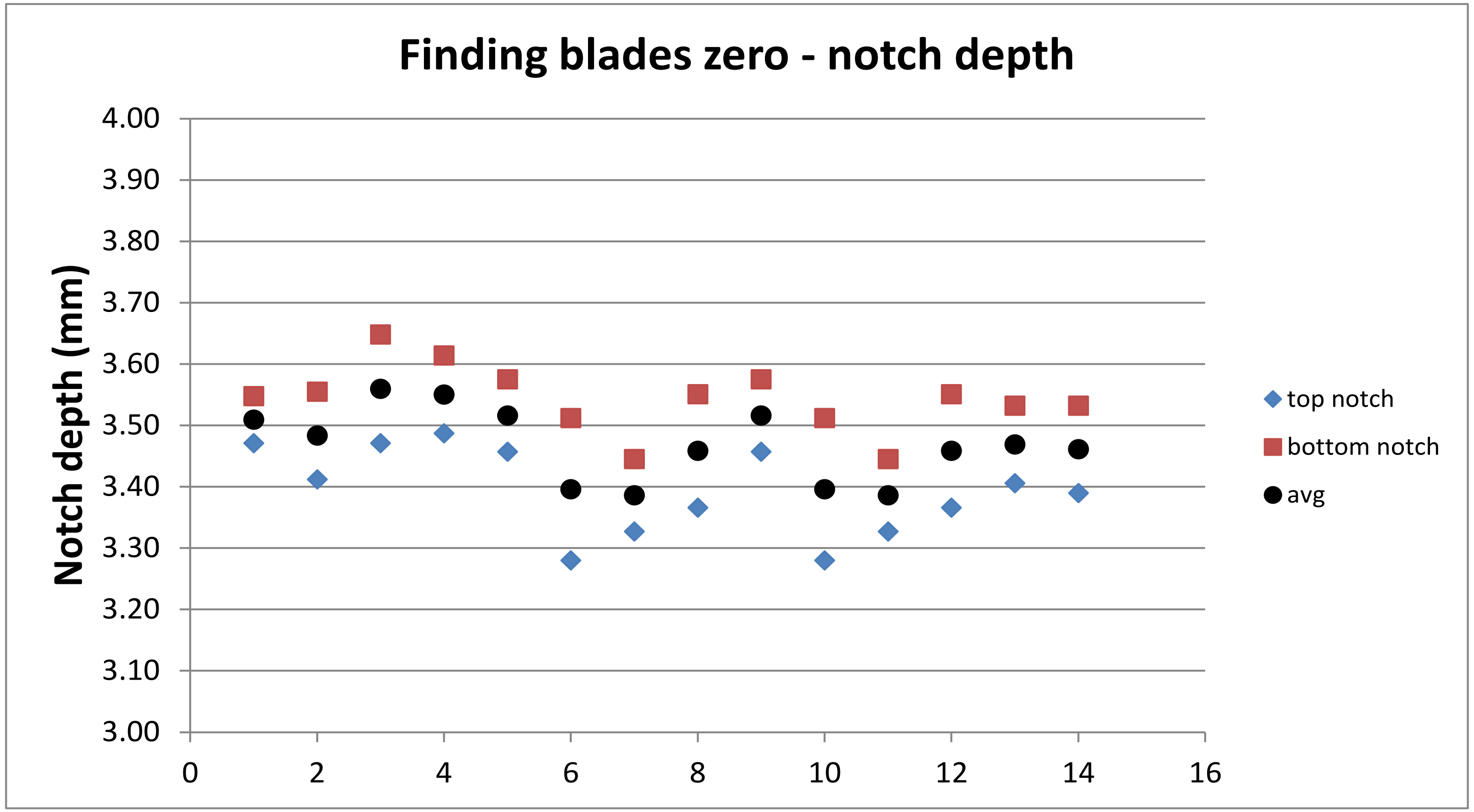
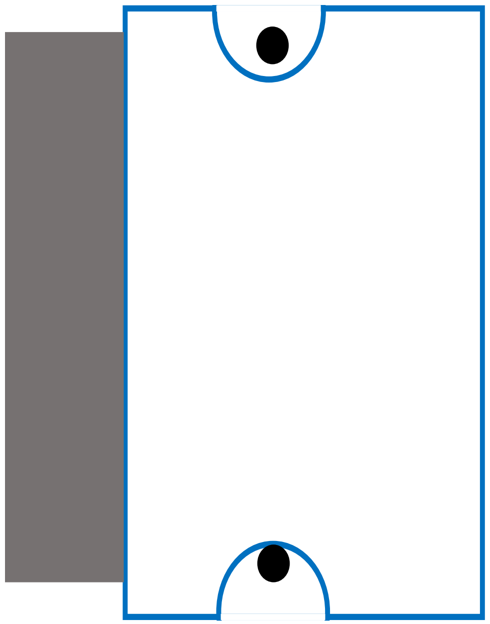
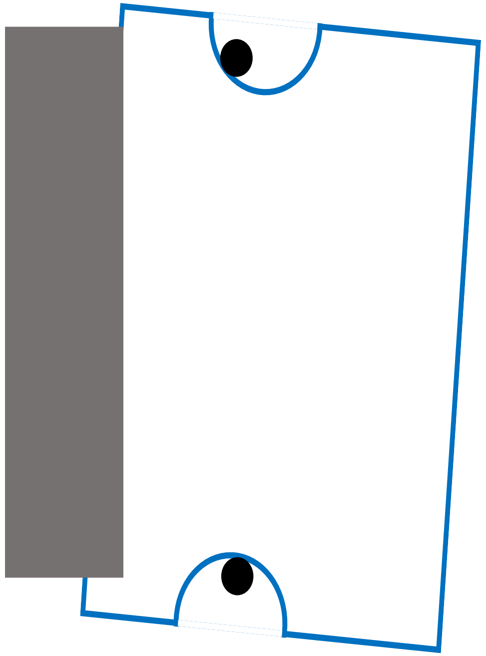


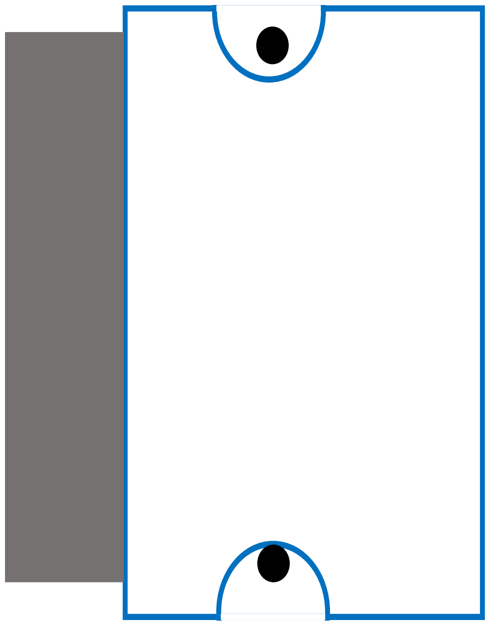
Helped a little (difference typically < +/- 0.1 mm) but still got movement (looks like the notch variation is now a little worse) ☹



Sample is parallel to surface when it is locked into place



However, under the high loads experienced during notching, the blade can rotate around the lower guide pin



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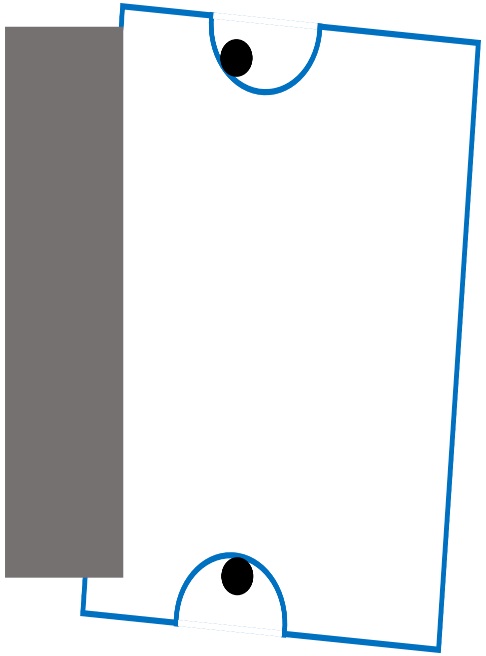

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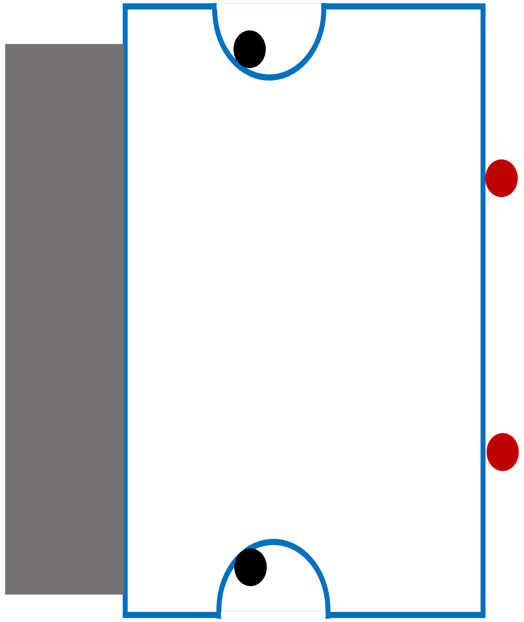
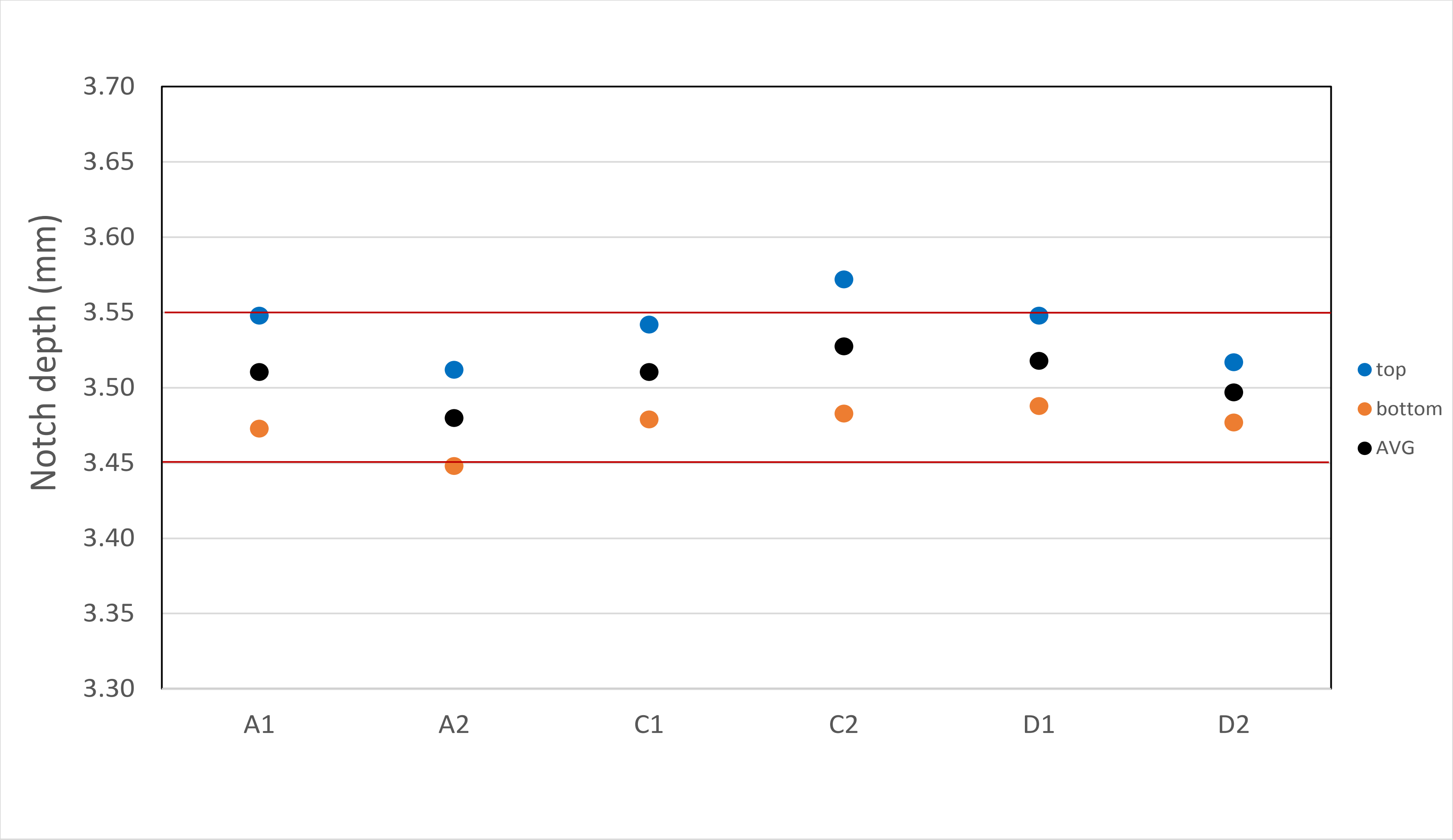


Solution was to put two stop pins behind the blade. Once the sample is parallel to the blade, the blade is pushed slightly until it hits the stop pins – guarantees it stays parallel

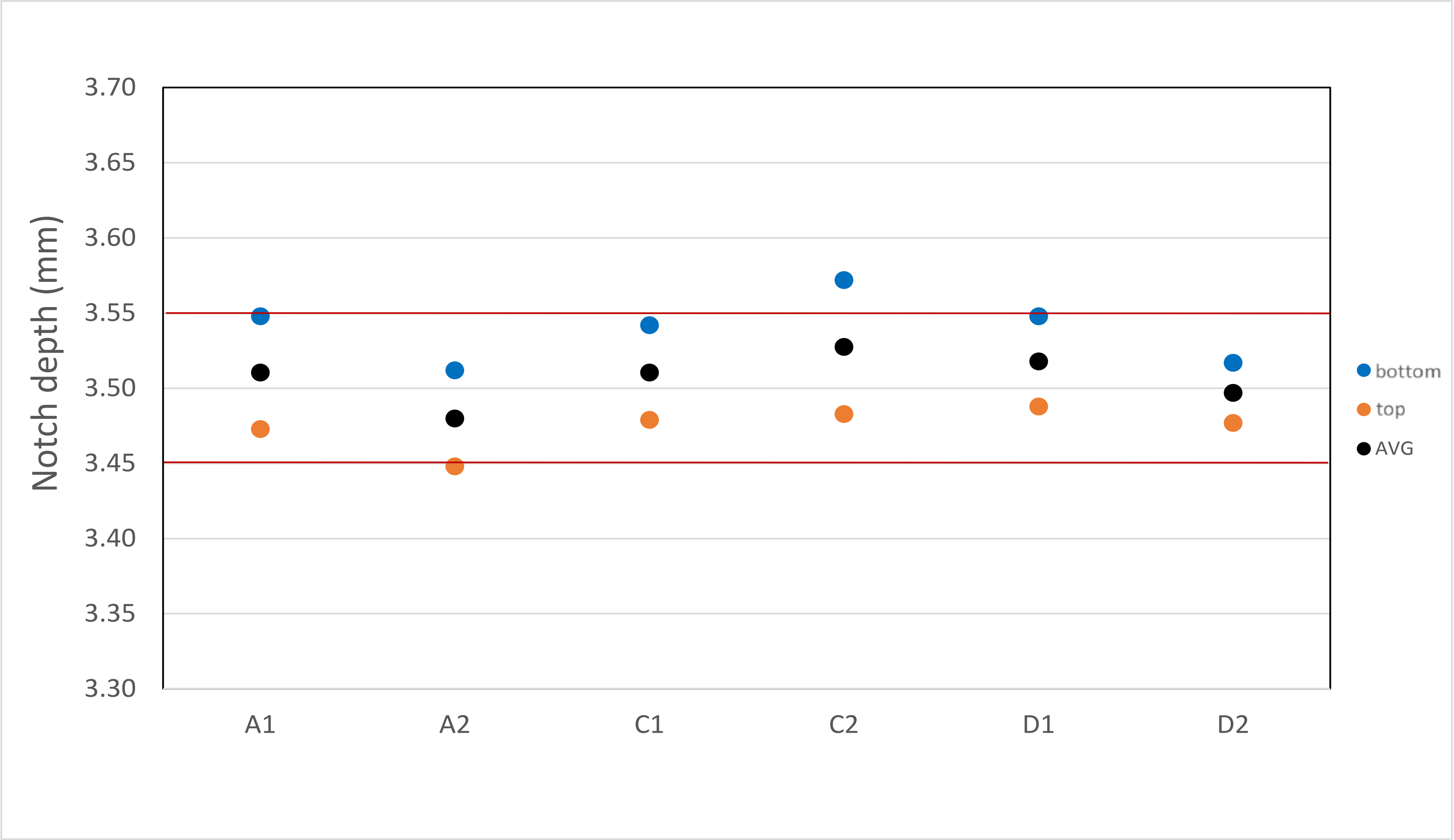
MODIFIED BLADE HOLDER

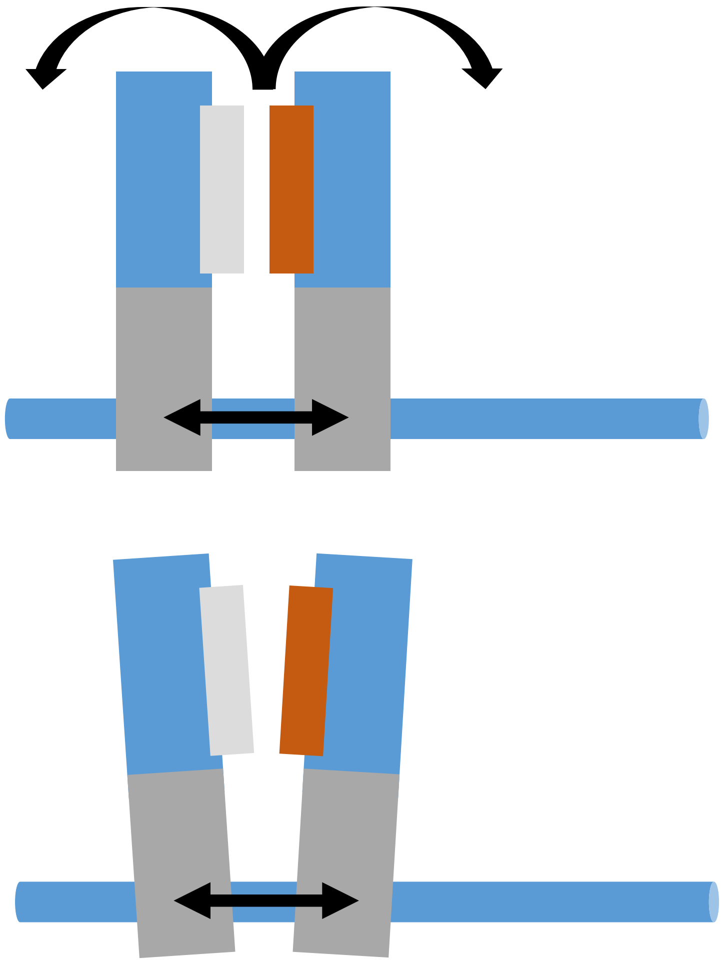
Final data set : Average notch depth is within the ASTM specs (+/- 0.05 mm) ☺
Difference between average and top and bottom notch is < 0.05 mm ☺



MODIFIED BLADE HOLDER

There is still some top-bottom variation (<+/- 0.05 mm)
Likely due to a torque on the system – difficult to engineer out, and acceptable





CONCLUSIONS/ACTIONS

OPERATORS:

The blade ridge can interfere with the placement of the blade – suggest removal

Machine compliance can lead to under-notching of the samples - addition of a compliance correction term can allow for this

VENDOR:

Work with the vendor to leverage learnings relative to the addition of pins to lock the blade parallel to the sample and holder modification to allow better viewing of the sample/polymer contact area

ASTM:

Work with ASTM to leverage these learnings into the published method