

# Tip radii effects on scratch testing of ultra- thin films

*A focus on the scratch testing  
of low-k films and long term  
repeatability of results*



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# Content

Review of the scratch process

Examination of scratch failures in low-k films using sharp cube-corner tips

Examination of tip radii effects in scratch testing of ultra-thin films

Results from a process that has been developed to generate scratch tips with consistent and well controlled geometries (tip radii on the order of 350nm or smaller)

A three step scratch process to provide a history of deformation during the scratch process.

# THE SCRATCH PROCESS



# Testing Method

## Ramp-load scratch test

Test type: progressive/ramp-load scratch tests

Scratch length: 100  $\mu\text{m}$

Scratch Velocity: 20  $\mu\text{m/s}$

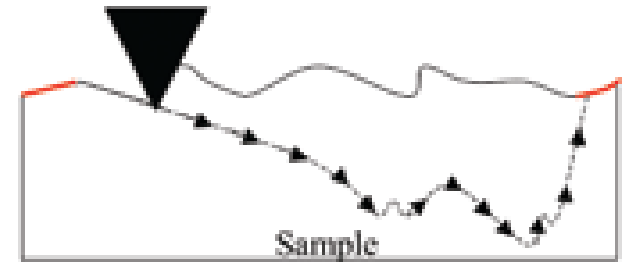
Maximum Scratch Load: 1 mN

Scratch Tip: Cube-corner

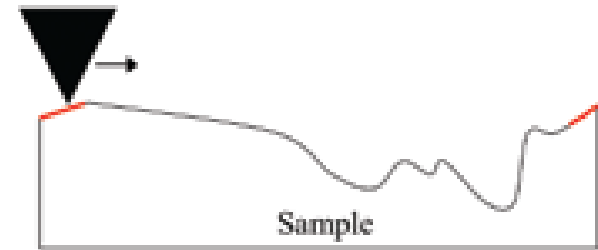
Scratch Direction: Face-forward



Step 1: Original Surface Scan

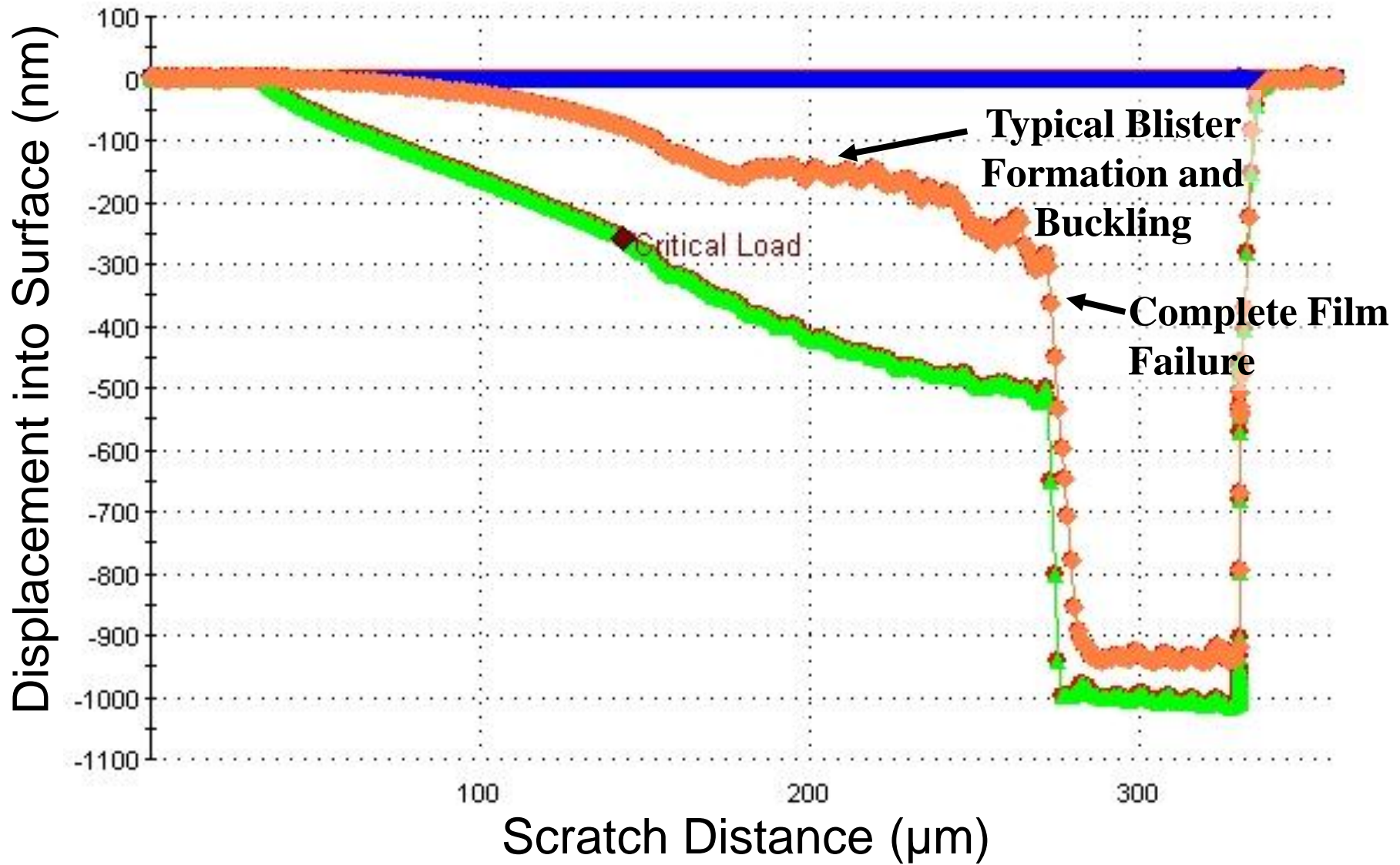


Step 2: Scratch Cycle



Step 3: Residual Deformation Scan

# Failure in the Scratch Curves

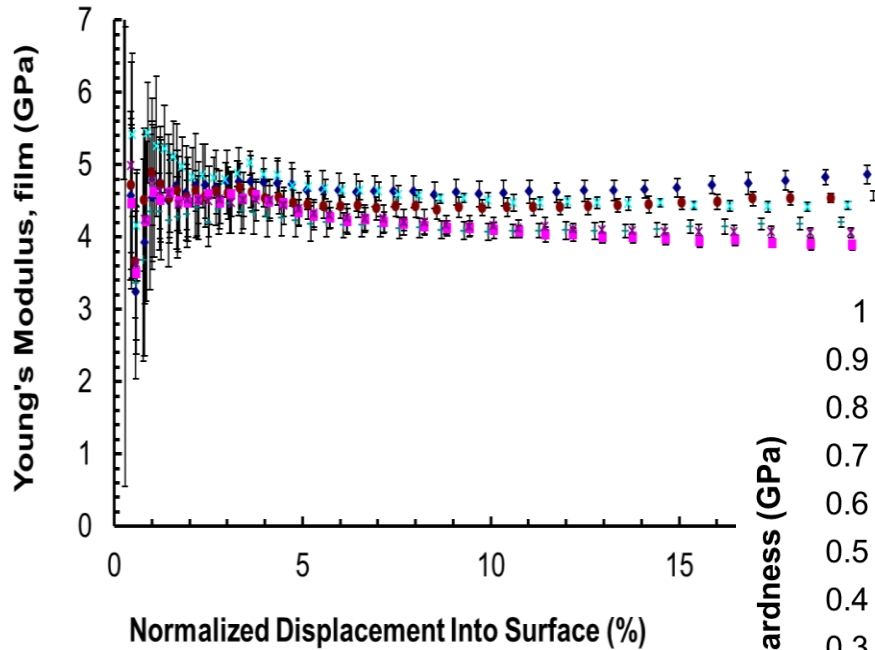


Driving Force: Providing further evaluation beyond elastic modulus and hardness for low-k films.

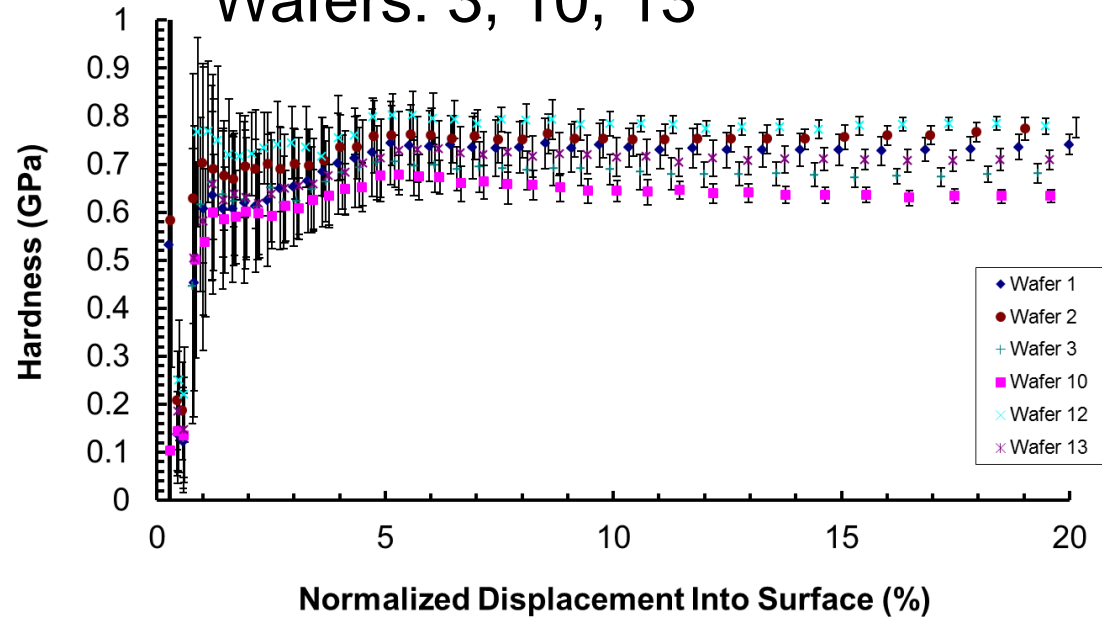
# SCRATCH FAILURES IN LOW-K FILMS



# What can we do to further distinguish processing effects on mechanical integrity?



Two distinct groups:  
Wafers: 1, 2, 12  
Wafers: 3, 10, 13

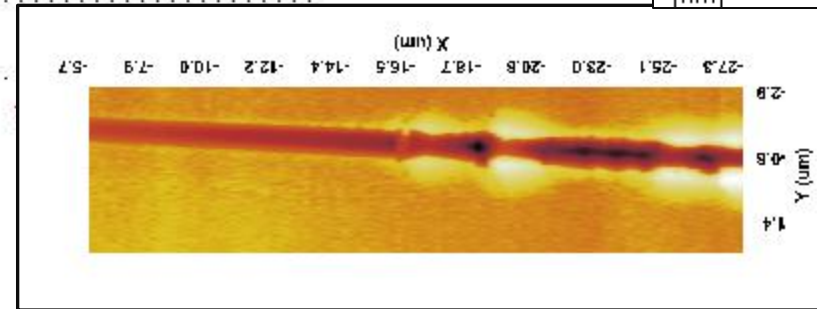
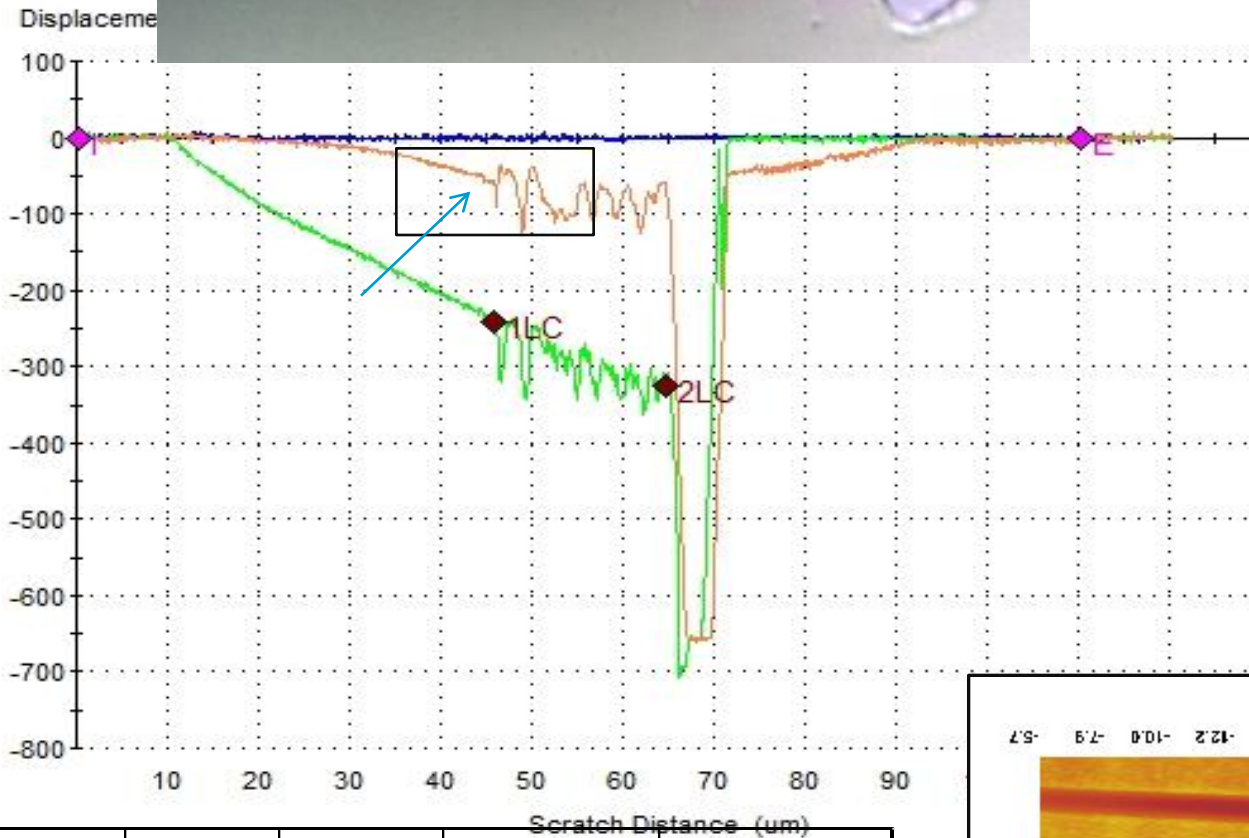


# Scratch Results

## Wafer 1



LC1 – Recovery Spallation  
 LC2 – Gross Spallation



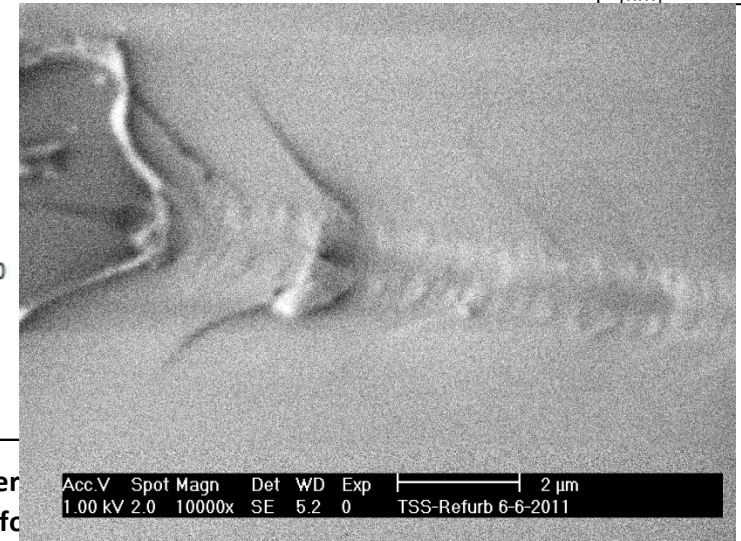
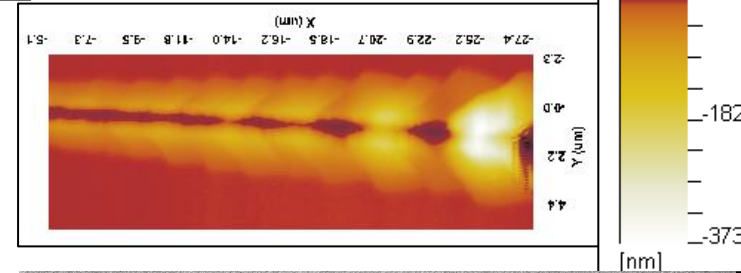
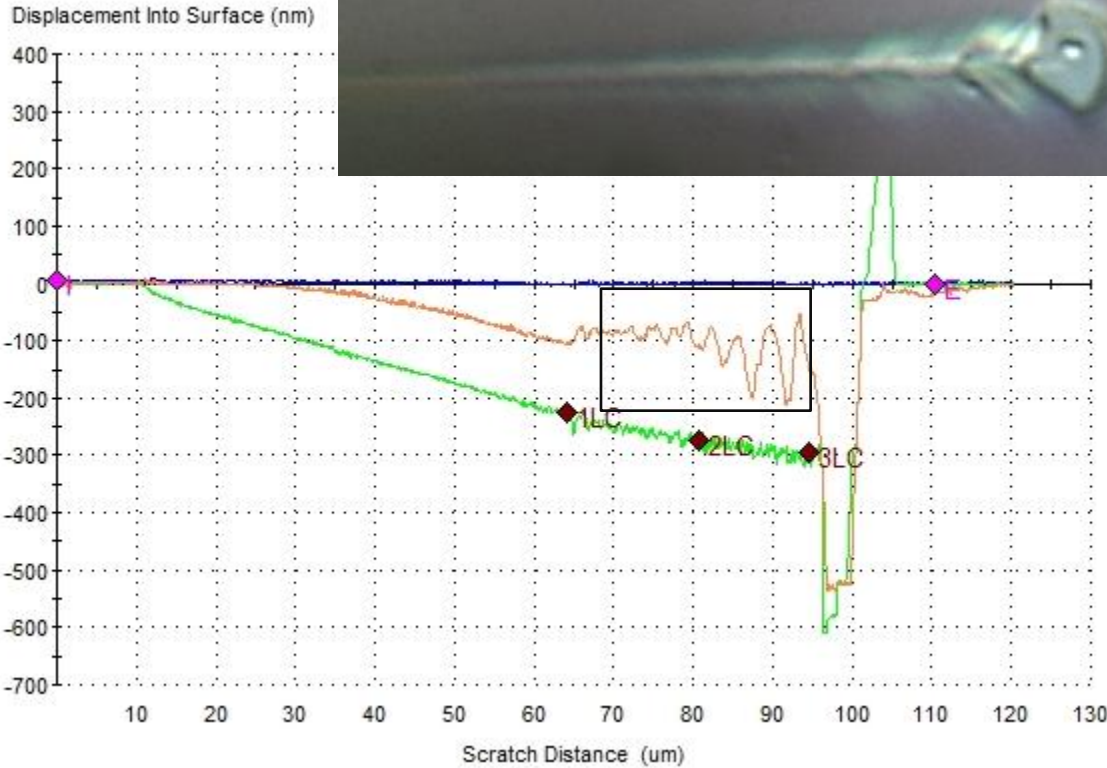
Film Thickness (nm)	LC1 (mN)	LC2 (mN)	Percent Elastic Deformation (%)	Percent Plastic Deformation (%)
622	0.38 ± 0.01	0.56 ± 0.04	86.1 ± 0.5	13.9 ± 0.5



# Scratch Results

## Wafer 2

LC1 – Buckling/Blistering  
 LC2 – Spallation Recovery  
 LC3 – Gross Spallation



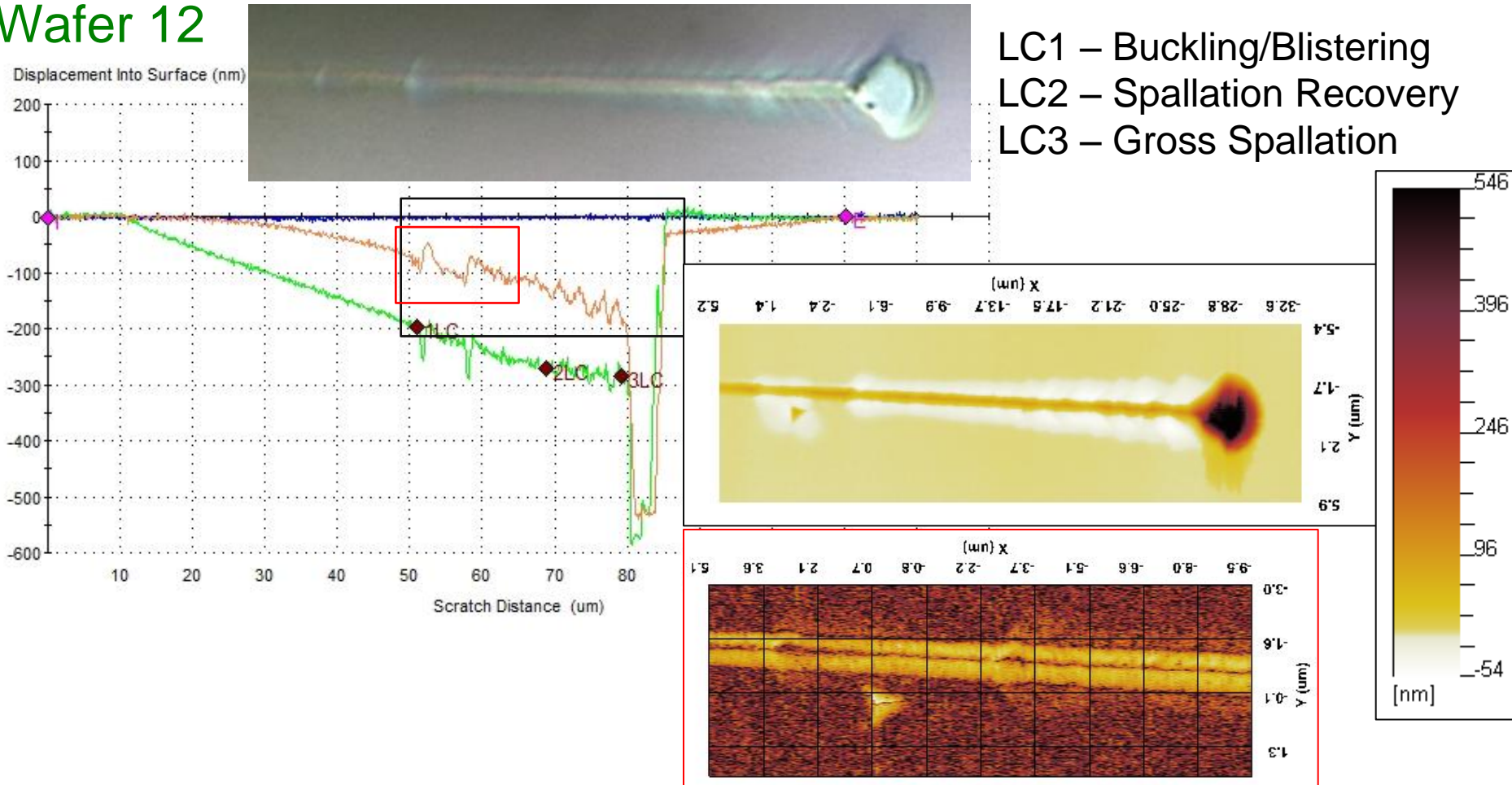
Film Thickness (nm)	LC1 (mN)	LC2 (mN)	LC3 (mN)	Percent Elastic Deformation (%)	Per Defo
617	0.54 ± 0.01	0.70 ± 0.03	0.83 ± 0.02	74.5 ± 2.1	25.5 ± 2.1



# Scratch Results

## Wafer 12

LC1 – Buckling/Blistering  
 LC2 – Spallation Recovery  
 LC3 – Gross Spallation



Film Thickness (nm)	LC1 (mN)	LC2 (mN)	LC3 (mN)	Percent Elastic Deformation (%)	Percent Plastic Deformation (%)
569	0.41 ± 0.01	0.57 ± 0.06	0.70 ± 0.01	79.9 ± 0.5	20.1 ± 0.5

# Low-k Results

## E and H and Scratch Tests

Test	Elastic Modulus _ Film (9.5 % to 10.5 %)	Hardness (9.5 % to 10.5 %)	Minimum Apparent Modulus	Minimum Hardness	Depth At Min E	Depth At Min H
	GPa	GPa	GPa	GPa	Angstrom	Angstrom
Wafer 1	4.6	0.74	5.01	0.66	261	347
Wafer 2	4.4	0.75	4.84	0.7	274	366
Wafer 12	4.5	0.79	5.14	0.73	330	343

Quick conclusion on this data:

1. The critical loads do not correlate with film thickness or hardness.

		mN	mN	mN	%	%
<b>Wafer 1</b>	Mean	0.376		0.562	86.1	13.9
622 nm	Std. Dev.	0.012		0.044	1.8	1.8
	% COV	3.26		7.76	2.06	12.79
<b>Wafer 2</b>	Mean	0.542	0.698	0.83	74.5	25.5
617 nm	Std. Dev.	0.006	0.025	0.019	2.1	2.1
	% COV	1.13	3.65	2.33	2.83	8.26
<b>Wafer 12</b>	Mean	0.409	0.573	0.7	79.9	20.1
569 nm	Std. Dev.	0.01	0.055	0.01	1.6	1.6
	% COV	2.38	9.64	1.5	1.96	7.77

Motivation: How does the tip radius affect the scratch results and how do we make this scratch process repeatable over time – You must make scratch tips with reproducible geometries.

## SCRATCH TIPS

\* V. Jardret, BN Lucas, W. Oliver. “Scratch durability of automotive clear coats: a quantitative, reliable, and robust methodology.” *JCT (2000)*

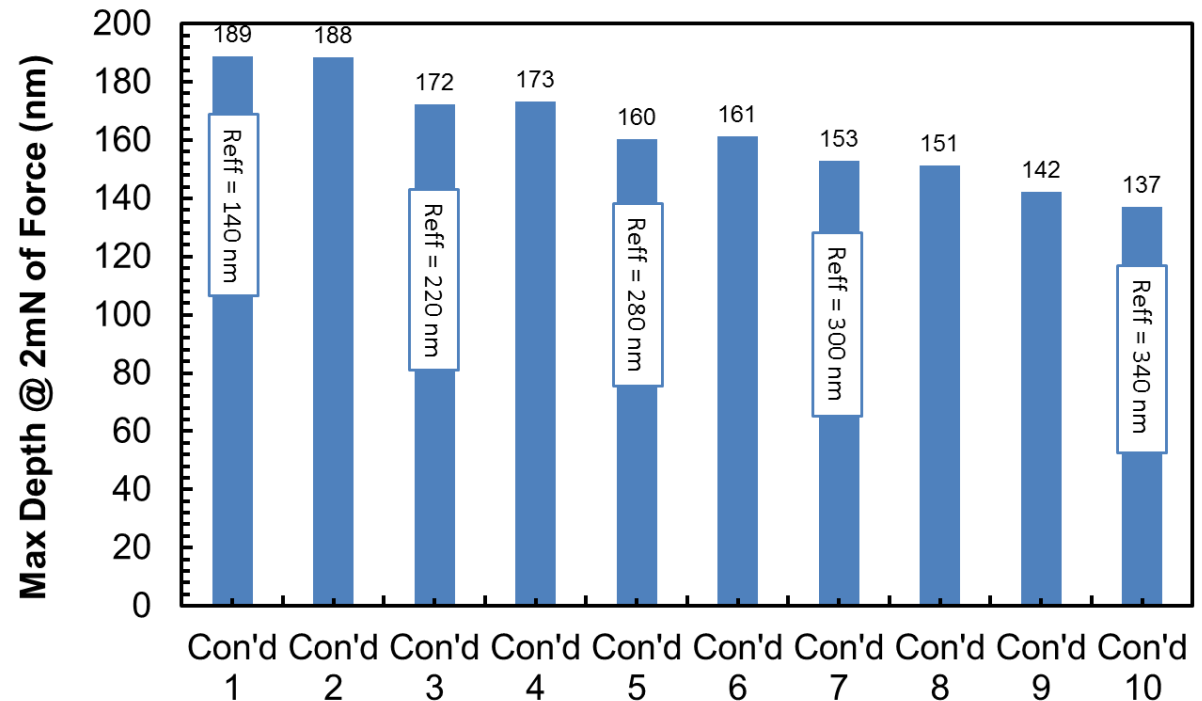


# Process of evaluation

Start with a diamond cube-corner tip that has a tip radius less than 50nm.

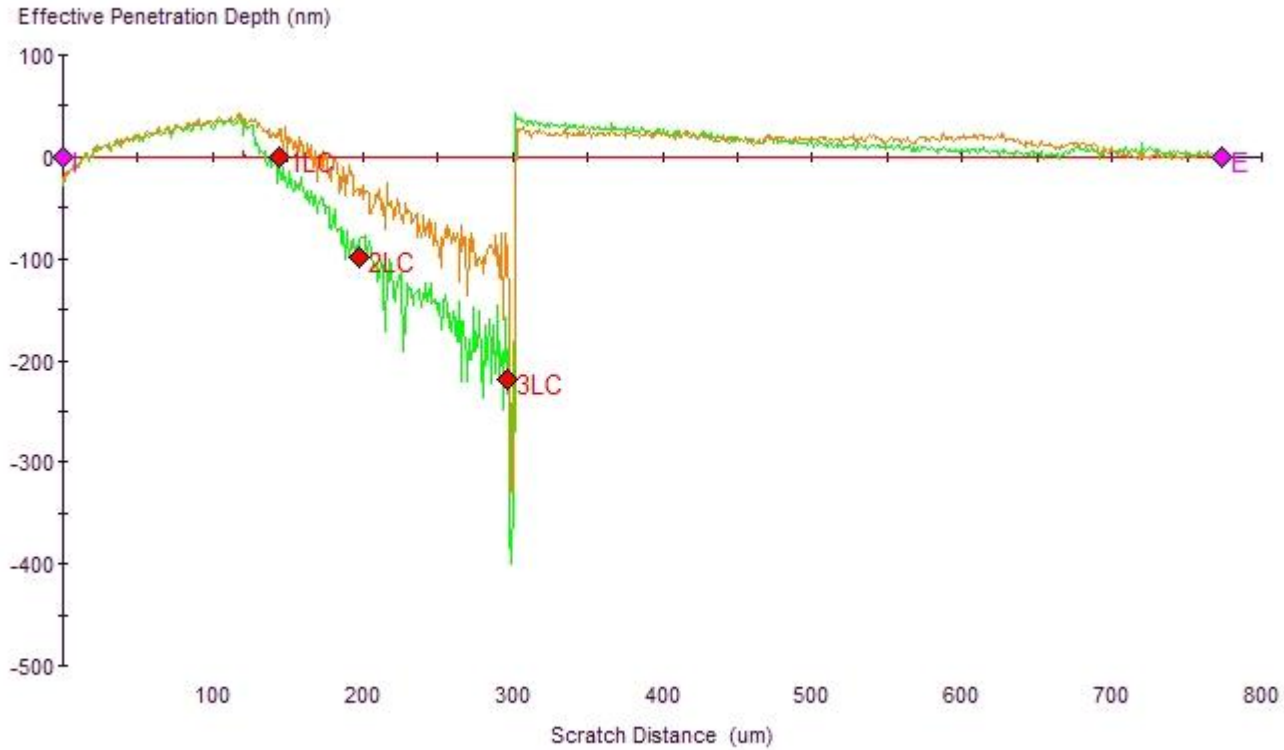
Gradually condition the tip – in the instrument - to create a larger tip radius and systematically perform scratch tests as the radius increases.

Maximum Penetration Depth in Fused Silica



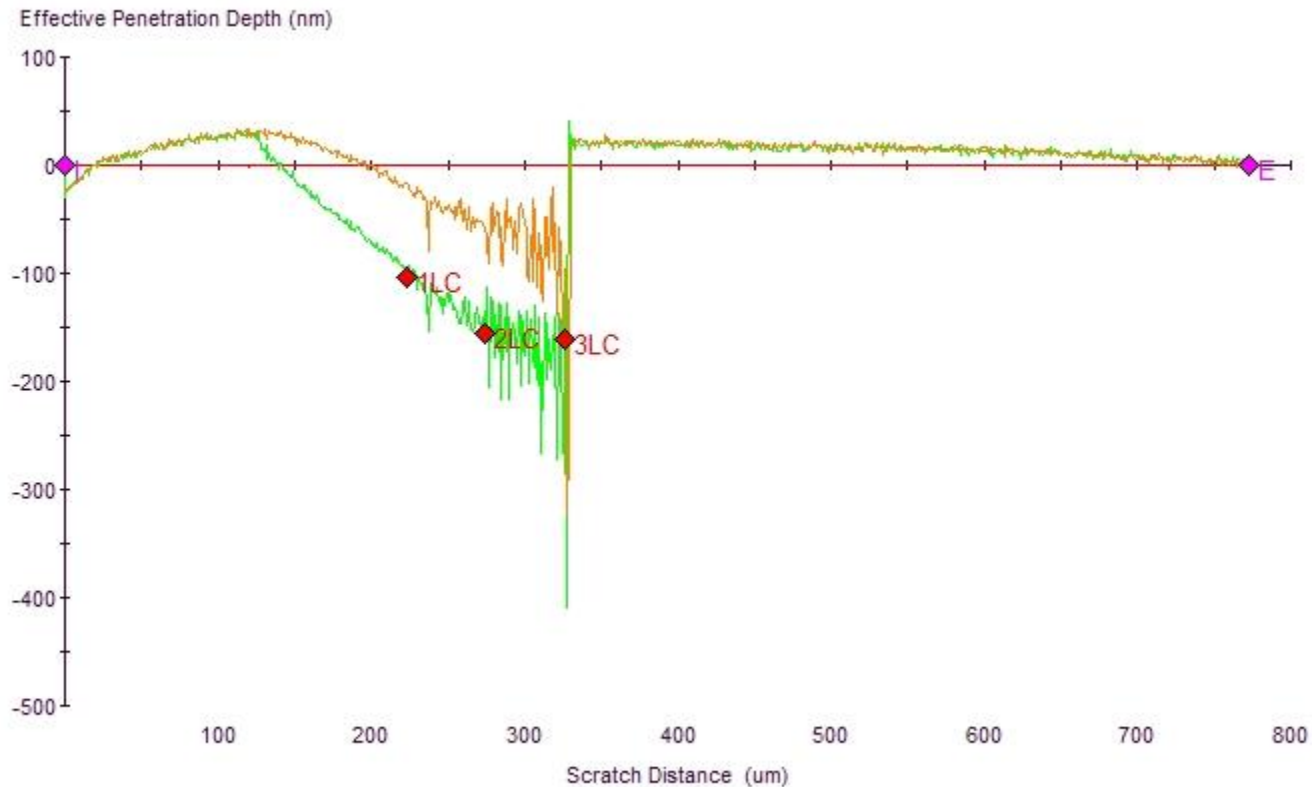
# Evolution of failure with tip blunting

## New Cube-Corner Tip <50nm tip radius



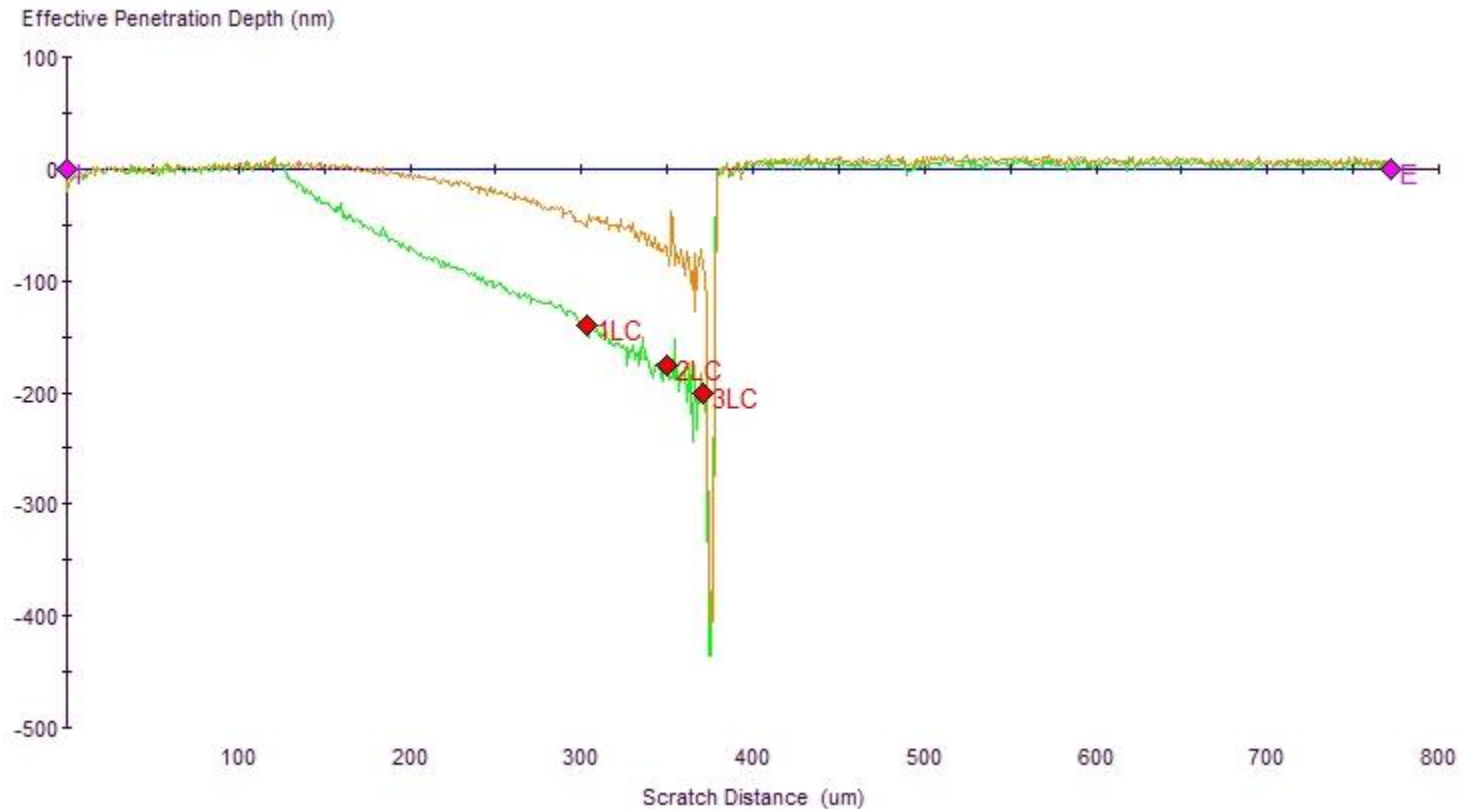
# Evolution of failure with tip blunting

Con'd 1: Max Depth 189 nm (Reff = 140nm)



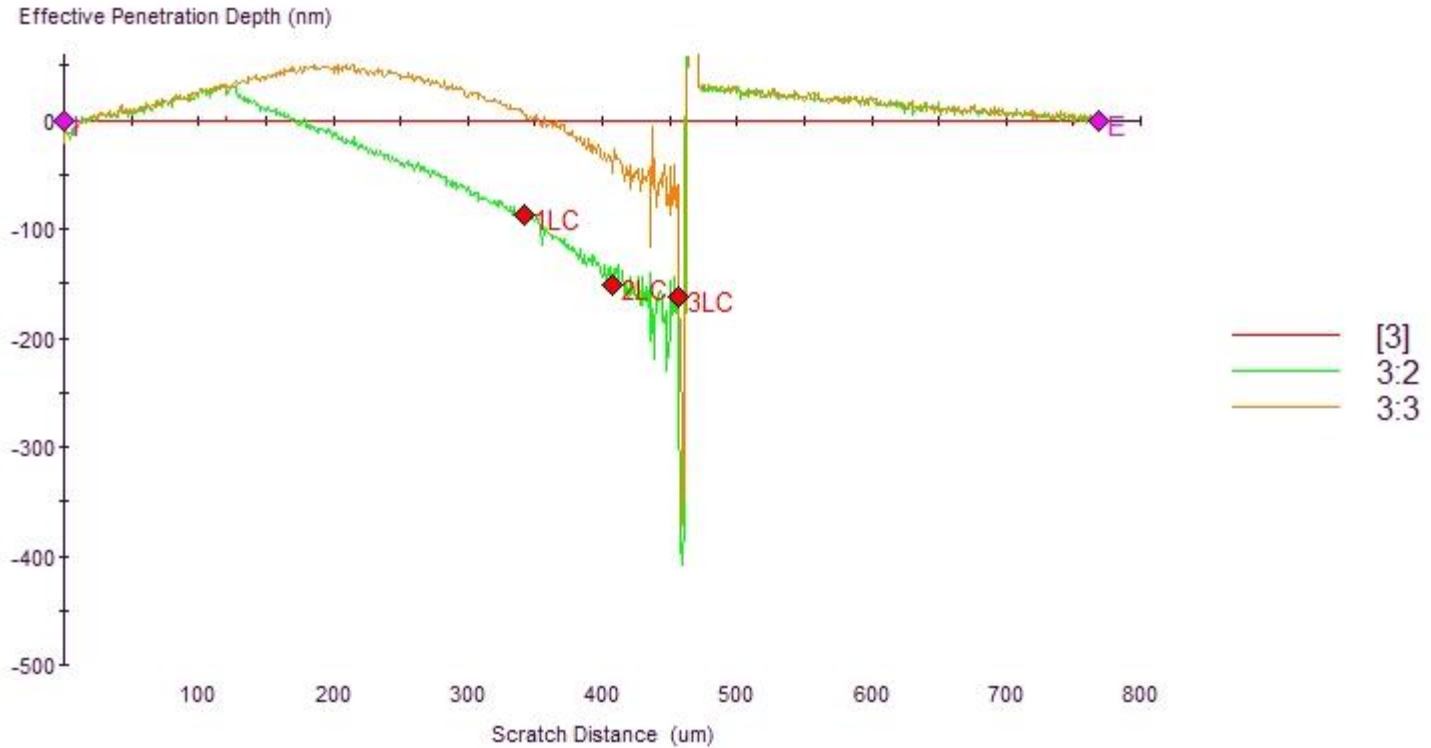
# Evolution of failure with tip blunting

Con'd 5: Max Depth 160 nm ( $R_{eff} = 280\text{nm}$ )

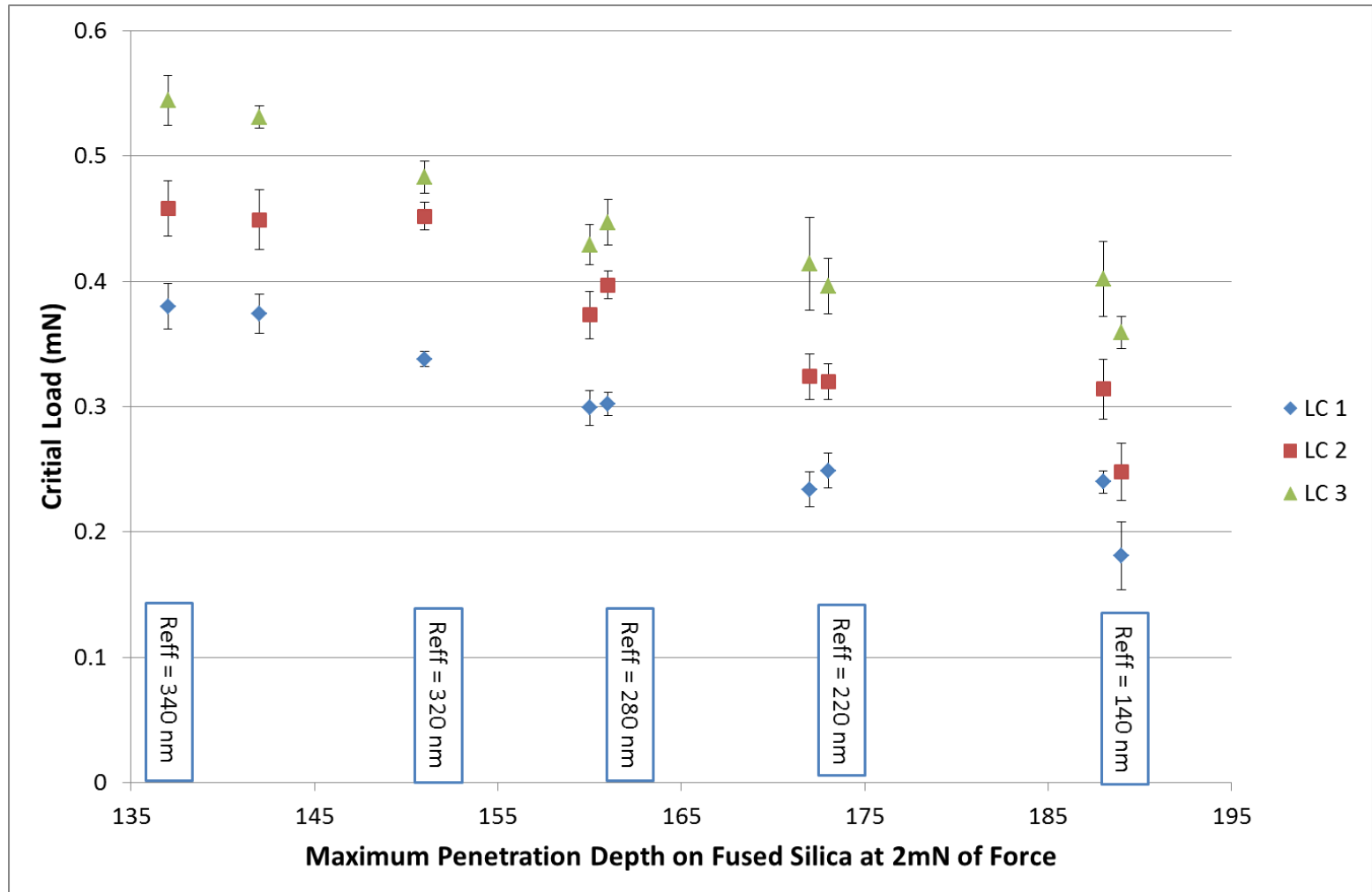


# Evolution of failure with tip blunting

Con'd 10: Max Depth 137 nm (Reff = 340nm)



# Evolution of critical load with tip blunting



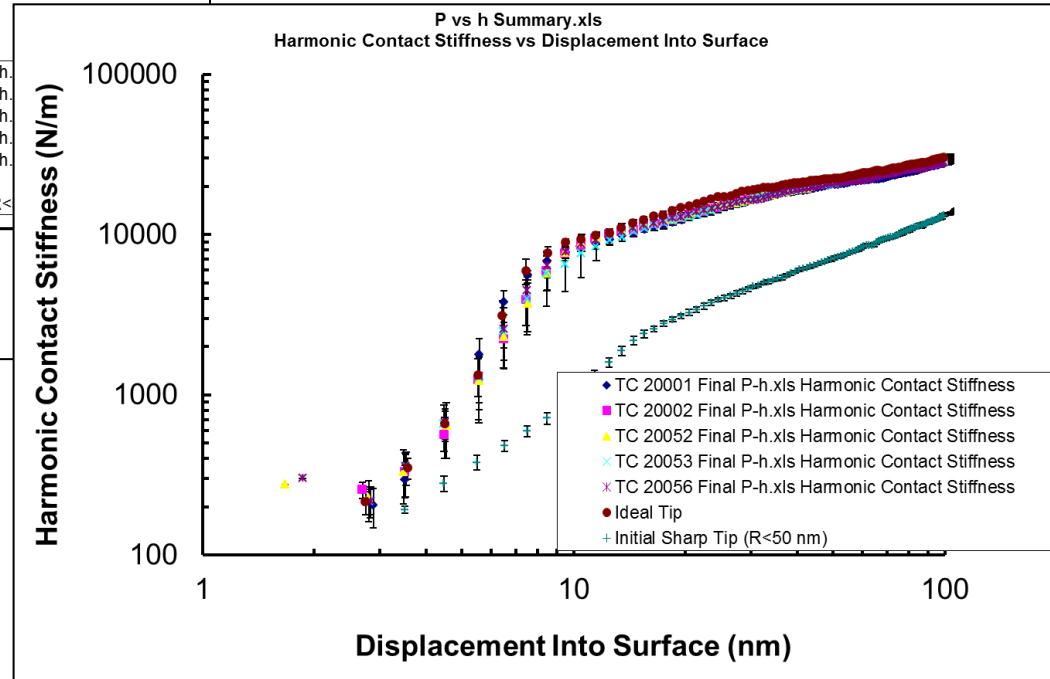
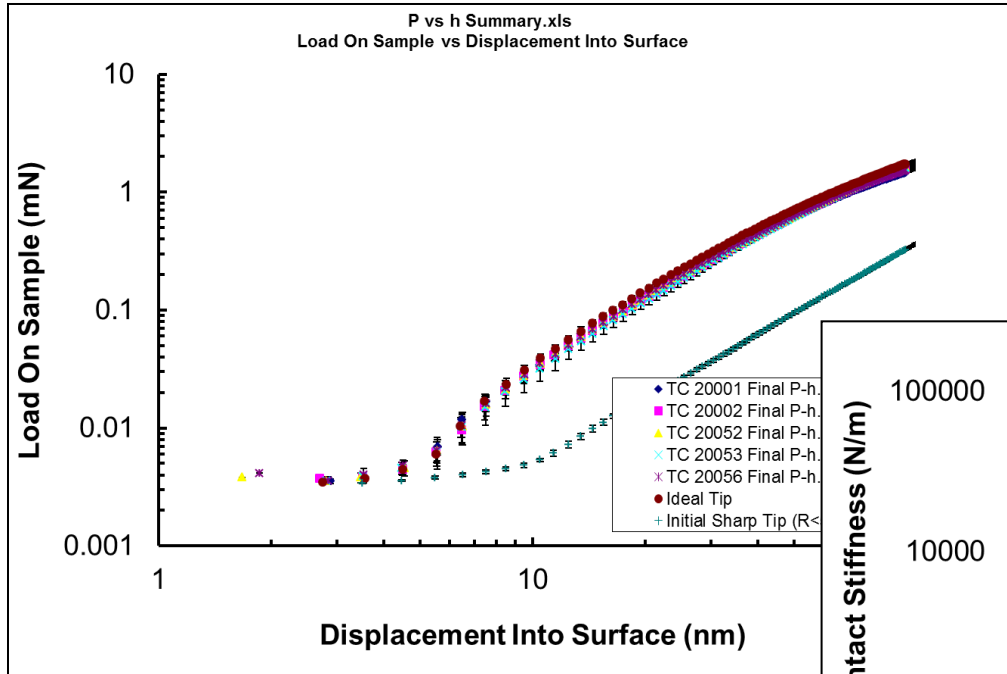
# Reproducibility of the process

Tests Performed on Reference Film	
	LC3 Critical Load Value (mN)
<b>TC 20001</b>	
<b>Mean</b>	0.864
<b>Std. Dev.</b>	0.013
<b>% COV</b>	1.46
<b>Minimum</b>	0.847
<b>Maximum</b>	0.884
<b>TC 20002</b>	
<b>Mean</b>	0.872
<b>Std. Dev.</b>	0.023
<b>% COV</b>	2.62
<b>Minimum</b>	0.832
<b>Maximum</b>	0.895
<b>TC 20053</b>	
<b>Mean</b>	0.866
<b>Std. Dev.</b>	0.017
<b>% COV</b>	1.97
<b>Minimum</b>	0.842
<b>Maximum</b>	0.892
<b>TC 20056</b>	
<b>Mean</b>	0.865
<b>Std. Dev.</b>	0.019
<b>% COV</b>	2.24
<b>Minimum</b>	0.847
<b>Maximum</b>	0.914

Ideal Tip - Historical Average	
	Critical Load Value (mN)
<b>Mean</b>	0.905
<b>Std. Dev.</b>	0.053
<b>% COV</b>	5.8%
<b>Minimum</b>	0.835
<b>Maximum</b>	0.965

\* Max penetration depth in Fused Silica @ 2mN:  
130nm and 140nm

# Multiple tip results using this process



# Conclusions

The scratch process clearly shows the evolution of failure in the ultra-thin film samples.

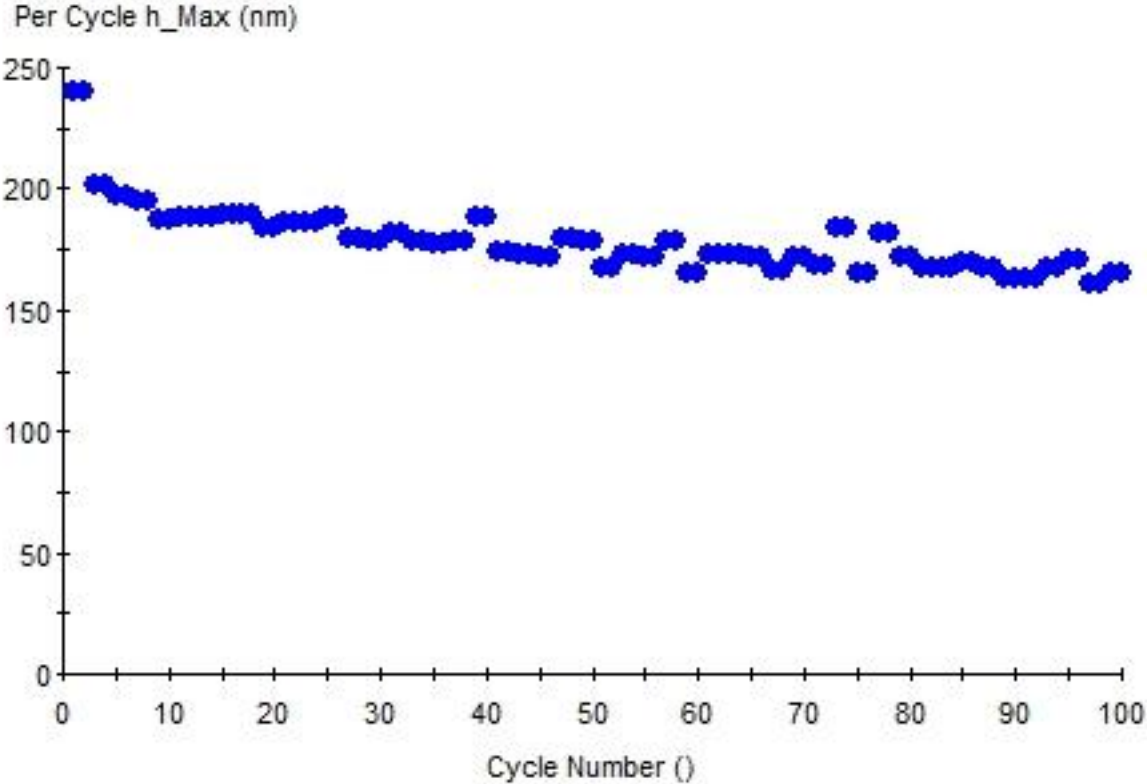
Tip radii have been shown to have a large effect on film failure.

Tips can be conditioned to provide the same progression of failure

The tips should be evaluated by examination of the  $P$  vs.  $h$  curves.



# Progression of a tip during the first conditioning cycle



[1]

