

# Sspherical Nanouindentation Stress-Strain Analysis in MATLAB (Spin)

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# Code was made to..

- Make the analysis faster – less time spent by user.
- Make the analysis more robust – select an answer on quantifiable criteria.
- Provide a way to estimate the variance in the answer based on the statistics of multiple acceptable answers.
- Provide a pathway to analysis automation.
- Analyze data from indentation collected using the MTS or Agilent Nanoindenter (XP head) with CSM option on metals. The analysis protocol is not limited to this system or metals.

# Workflow

- Step 1: Load and Analyze Data.
- Step 2.1: Filter out poor answers, look at histogram plots of different variables and values which indicate goodness of answer. Look at the indentation stress-strain curve for different answers.
- Step 2.2 Make a new filter using the sliders on the histogram plot. Re-run according to the new filter.
- Step 3: Save data, parameters, analyses, and an indentation stress-strain plot

# Step 1: Perform Analysis

- Inputs
  - Data location and file name
  - Experimental parameters (e.g., indenter radius)
  - Some search and analysis parameters (e.g. array of segment sizes)
- Perform
  - load data (once)
  - zero-point regression (many times)
  - modulus regression (many times)
  - elastic stress-strain calculation (many times)

# Step 1: 'seg\_sizes'

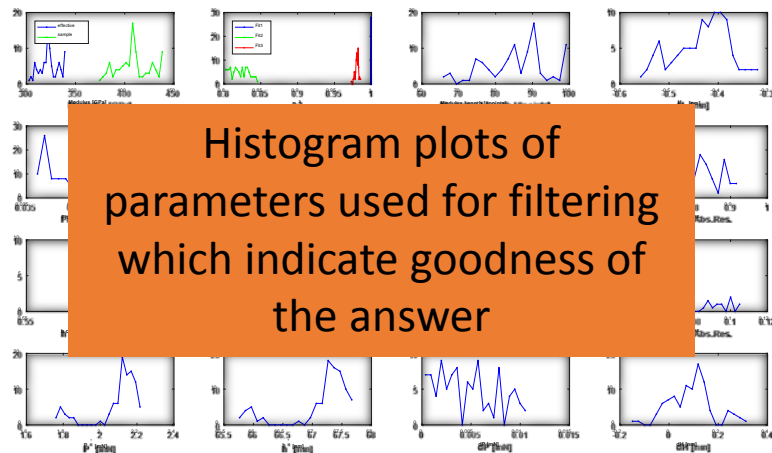
- Are all possible answers considered? Sort-of
  - Ex: a test with 1500 data points. An array of segment sizes is defined. One could define the array by [3:1:1500] which would look at all possible segment lengths.
  - The permutations of segments which are analyzed is
    - 1:3, 2:4,..., 1497:1499,1498:1500
    - 1:4, 2:5,..., 1496:1499,1497:1500
    - ....
    - 1:1499, 2:1500
    - 1:1500
  - The zero-point and modulus regression happens for the same segment permutation. You cannot take 10:100 for the zero point and 50:200 for the modulus. However, in reality, you often have 10:100 for the zero point and 50:100 for the modulus because the zero-point corrected data for the modulus regression may have negative values which are imaginary in the regression. However the permutation in this case is still 10:100, it just happens to discard 10:49 for the second regression. This is something that is also tracked.
  - A more practical segment array would be 20:20:400, but it will depend on the material and indenter size.

# Step 1: 'CSM'

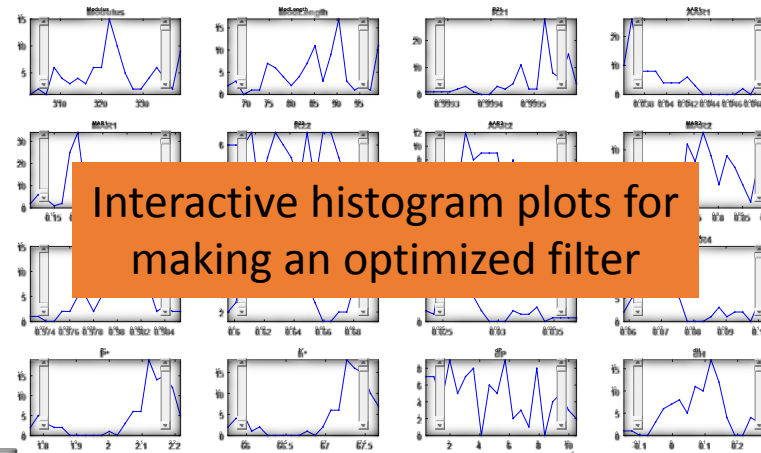
- There are known corrections to the load, displacement, and harmonic contact stiffness due to data collection protocols.
  - S.J. Vachhani, R.D. Doherty, S.R. Kalidindi. (2013) Acta Materialia 61: 3744-3751.  
<http://dx.doi.org/10.1016/j.actamat.2013.03.005>
  - G.M. Pharr, J.H. Strader, W.C. Oliver. (2009) J. Mater. Res.  
<http://dx.doi.org/10.1557/JMR.2009.0096>
- It is up to the user to decide if these are needed.
- The variable CSM controls whether the corrections are applied
  - CSM = 0, no corrections
  - CSM = 1, corrections to just the load and displacement
  - CSM = 2, corrections to the load, displacement, and harmonic contact stiffness

# Step 2.1: Filtering

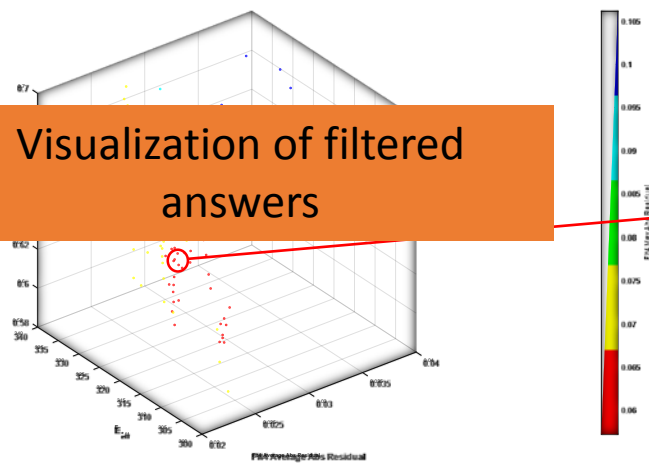
- Inputs
  - All the potential answers from step 1 (typically ~20,000).
  - Parameters for determining indentation properties (e.g., pop-ins,  $Y_{ind}$ )
  - Parameters for filtering out bad answers.
- Perform
  - Filtering
  - Visualization and interaction of filtered answers (typically ~1,000)
  - Calculation of indentation stress-strain curve for selected answers (when needed)
  - Calculation of indentation stress and strain for all filtered answers (when needed)



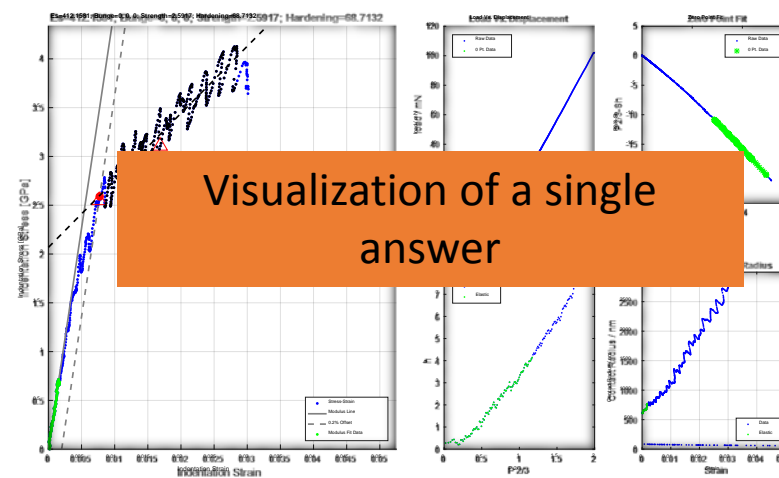
Histogram plots of parameters used for filtering which indicate goodness of the answer



Interactive histogram plots for making an optimized filter



Visualization of filtered answers



Visualization of a single answer



## Step 2.1: 'Filt'

- The filtering process is essentially limiting the space of answers. For example, you made decide that the answer must have r-squared values between 0.7 and 1.0 for each linear regression. In addition you may only want answers with an effective modulus between 100 and 200 GPa if you are expecting a value of 150 GPa.
- It is up to the user to define a consistent criteria for defining bounds on the answer space without applying a bias.
- The filter variables are in a table on the following slides with descriptions, expected values, and comments. You are not limited to these variables.

## Step 2.1: 'Filt'

Filter Variable Name	Description	Ideal value	Comments
Modulus	The effective modulus	It can be calculated based on the material and indenter properties.	It's not Young's modulus nor should you use Young's modulus to calculate the effective modulus if your material is anisotropic. See Patel and Kalidindi (2014). Acta Materialia. <a href="http://dx.doi.org/10.1016/j.actamat.2014.07.021">http://dx.doi.org/10.1016/j.actamat.2014.07.021</a> and Vlassak and Nix (1994) J. Mech. Phys. Solids. 42(8): 1223-1245
R21	R-squared of zero-point regression	=1	R-squared is not the only measure of goodness of fit.
R22	R-squared of modulus regression	=1	R-squared is not the only measure of goodness of fit.
R23	R-squared of elastic indentation stress and strain	=1	R-squared is not the only measure of goodness of fit.

AAR1	Average absolute residual of the zero-point regression	=0	Measure of goodness of fit
AAR2	Average absolute residual of the modulus regression	=0	Measure of goodness of fit
AAR4	Average absolute residual between the calculated indentation modulus and the elastic indentation stress and strain	=0	Measure of goodness of fit
MAR1	Maximum absolute residual of the zero-point regression	=0	Measure of goodness of fit
MAR2	Maximum absolute residual of the modulus regression	=0	Measure of goodness of fit
MAR4	Maximum absolute residual between the calculated indentation modulus and the elastic indentation stress and strain	=0	Measure of goodness of fit

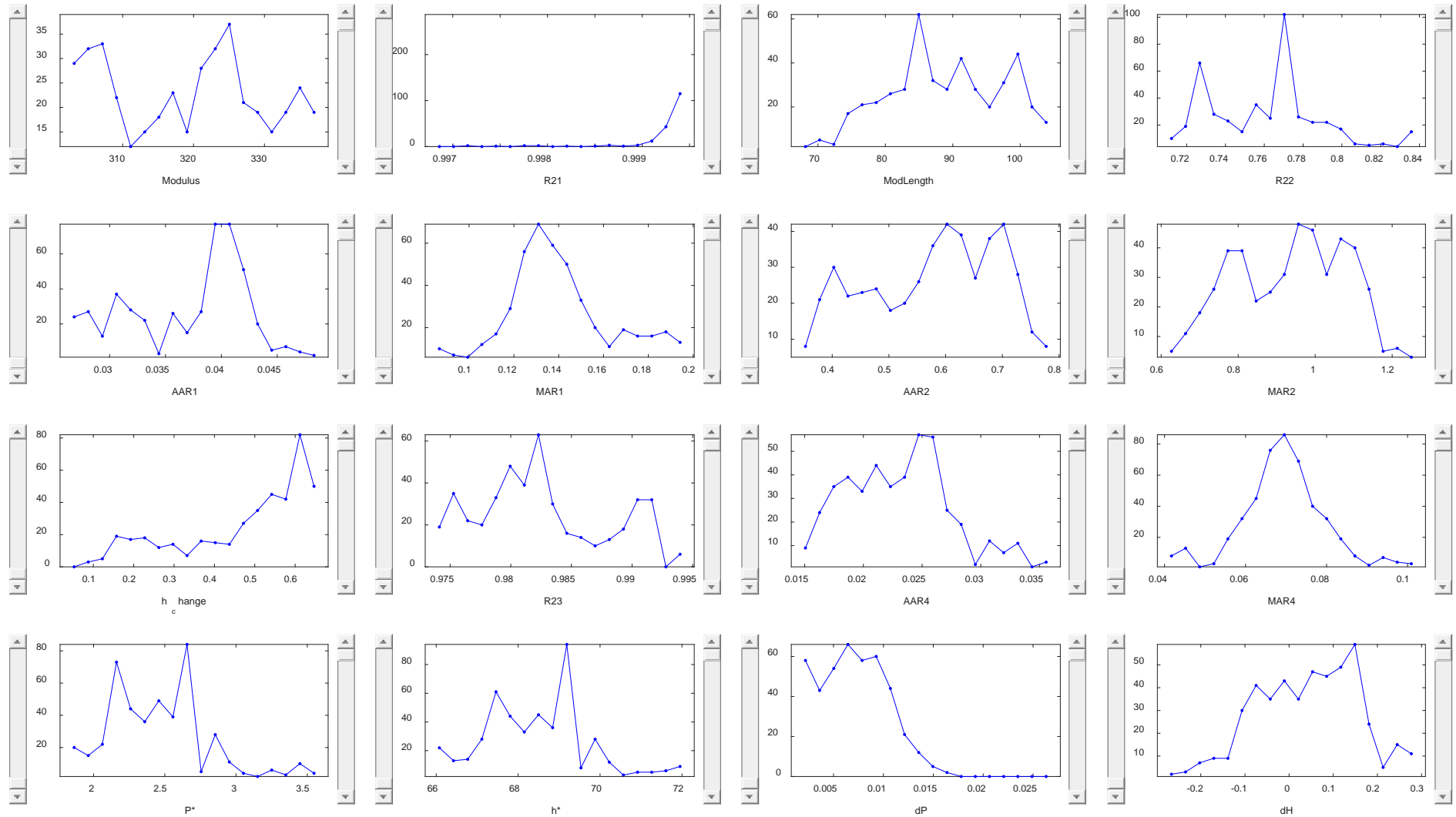
dH	The value of the first displacement data point in the modulus regression	=0	If is close to the origin, then the stress-strain curve will also be close to the origin.
dP	The value of the first load data point in the modulus regression	=0	If is close to the origin, then the stress-strain curve will also be close to the origin.
h_change	normalized displacement between the start of the zero-point regression segment and the first positive point (load) after the zero-point correction is applied	=0	The segment for the zero-point regression and modulus regression should be close to the same data since the zero-point regression assumes the data is the initial elastic loading segment that follows Hertz's equation. See p_change comments for what is meant by the first positive point.
p_change	normalized load between the start of the zero-point regression segment and the first positive point (load) after the zero-point correction is applied	=0	The segment for the zero-point regression and modulus regression should be close to the same data since the zero-point regression assumes the data is the initial elastic loading segment that follows Hertz's equation. Because the load is raised to the 2/3 power in the modulus regression, no negative load values can be used. Some of the corrected load values become negative after zero-point correction is applied.

P*	The zero-point load correction	n/a	The value will depend on the sample, indenter size, etc.
h*	The zero-point displacement correction	n/a	The value will depend on the sample, indenter size, etc. Sometimes large values are needed when the machine does a poor job of selecting the zero-point.
ModStart	The starting data point for the modulus regression	n/a	Will depend on your data collection frequency, etc. Typically you would want to provide an upper bound to make sure you are not analyzing data after plastic deformation has occurred.
SegStart	The starting data point for the zero-point regression.	n/a	Will depend on your data collection frequency, etc. Typically you would want to provide an upper bound to make sure you are not analyzing data after plastic deformation has occurred.
Hr	The y-intercept of the modulus regression or the residual height	0	It never seems to be near zero, but it should be small.
<i>Make a variable</i>	<i>If you find a parameter useful, make sure it is calculated and saved in FitResults</i>	<i>What should it be?</i>	<i>You can add it as a Filter variable in filterResults.m using the switch logic.</i>

## Step 2.2: Filtering

- You have two options for iterating on the filter
  1. Manually type out the filter variables in the RunMe.m code
  2. Use the sliders on the histogram plot to make a filter
- In both cases, you will re-run SearchExplorer.m to update the answer space and plots.
  1. Use Filt if you manually changed the filter
  2. Use NewFilt if you made the filter from the histogram plot

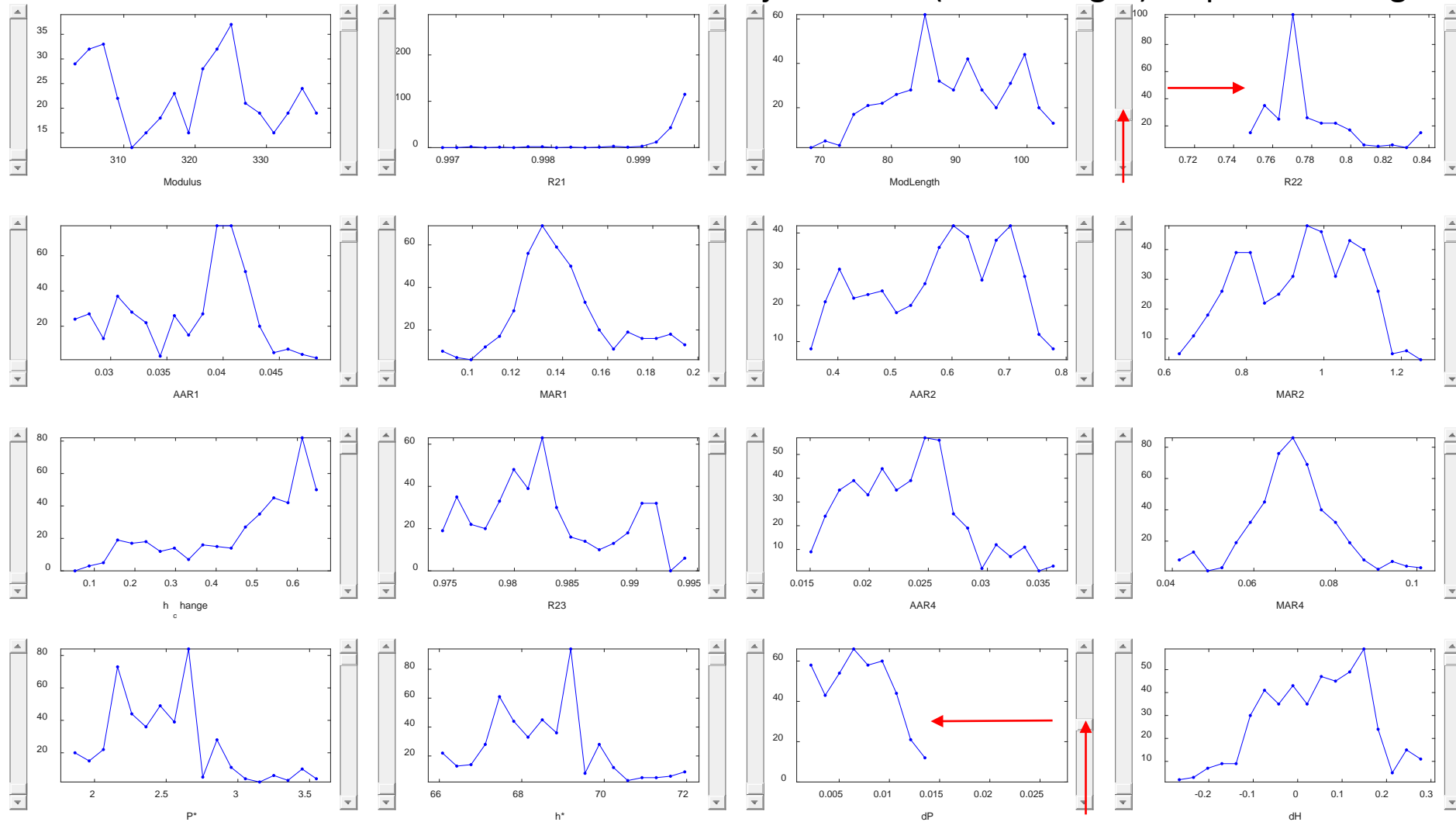
# Interactive histogram plot for making a new filter



Save New Filter

# Interactive histogram plot for making a new filter

Adjust sliders (left and right) > updates histogram plot



Save New Filter

Write variable NewFilt, then you re-run SearchExplorer.m with NewFilt



## Step 2: 'Plastic'

- The variable contains most of the variables needed to determine the indentation yield strength and work hardening from the indentation stress-strain curve.
- The main principle for yield is that it can be defined as a 0.2% offset or using a back-extrapolation when there is a small pop-in event.
- The main principle for hardening is that it can be determined from a linear fit to data in-between two strain offsets.
- It is up to the user to determine the best parameters and methods for determine strength and hardening from the indentation stress-strain curve. The variables and methods used in this code are described in the following slides.

## Step 2: 'Plastic'

Plastic.variable	Description	Scenario/ Value	Comments
method	How to determine the 0.2% strength	Linear, max, median, or mean	Once a range near the 0.2% offset has been determined, a linear fit, maximum, median, or mean calculation is performed. See YS_window for the range of data.
YS_offset	Offset strain for yield calculations	0.002	0.2% strain seems to be good for simulations and experiments (similar principle to uniaxial tests).
H_offset	Strain offsets for the first hardening calculation		The start should be greater than or equal to YS_offset. This hardening fit goes from H_offset(1) to H_offset(2).
H_offset2	The end strain offset of the second hardening fit		This value should be greater than H_offset(2). This hardening fit goes from H_offset(2) to H_offset_2
pop_in	The indentation strain burst value that is considered a pop-in	small # or Inf	If you don't want a pop-in to be detected = Inf.

pop_window	Number of data points for the strain burst calculation		3 seems to work well
C_dstrain	Tuning factor for the back extrapolation	Between 0 and 1	A value of 1 would force the back extrapolation to start after the strain has exceeded the strain in the pop-in (out of the pop-in shadow). A value of 0 starts the back extrapolation right after the pop-in (minimum stress point after the pop-in).
smooth_window	+/- number of data points for a moving average on the hardening fits		A value of 0 applies no moving average. A moving average is useful when the hardening or back extrapolation fit is very sensitive to the start and end points.
Eassume	Assumes a sample modulus for the calculation of contact radius		This is for troubleshooting the analysis when you think there should be an answer but you can't get the right modulus. A value of 0 will use the modulus from the regression fit. This should not be used for the final answer.

## Step 3: Saving the Results

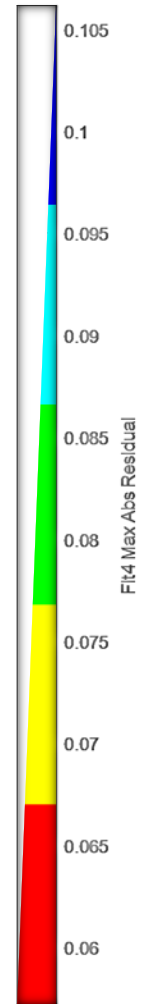
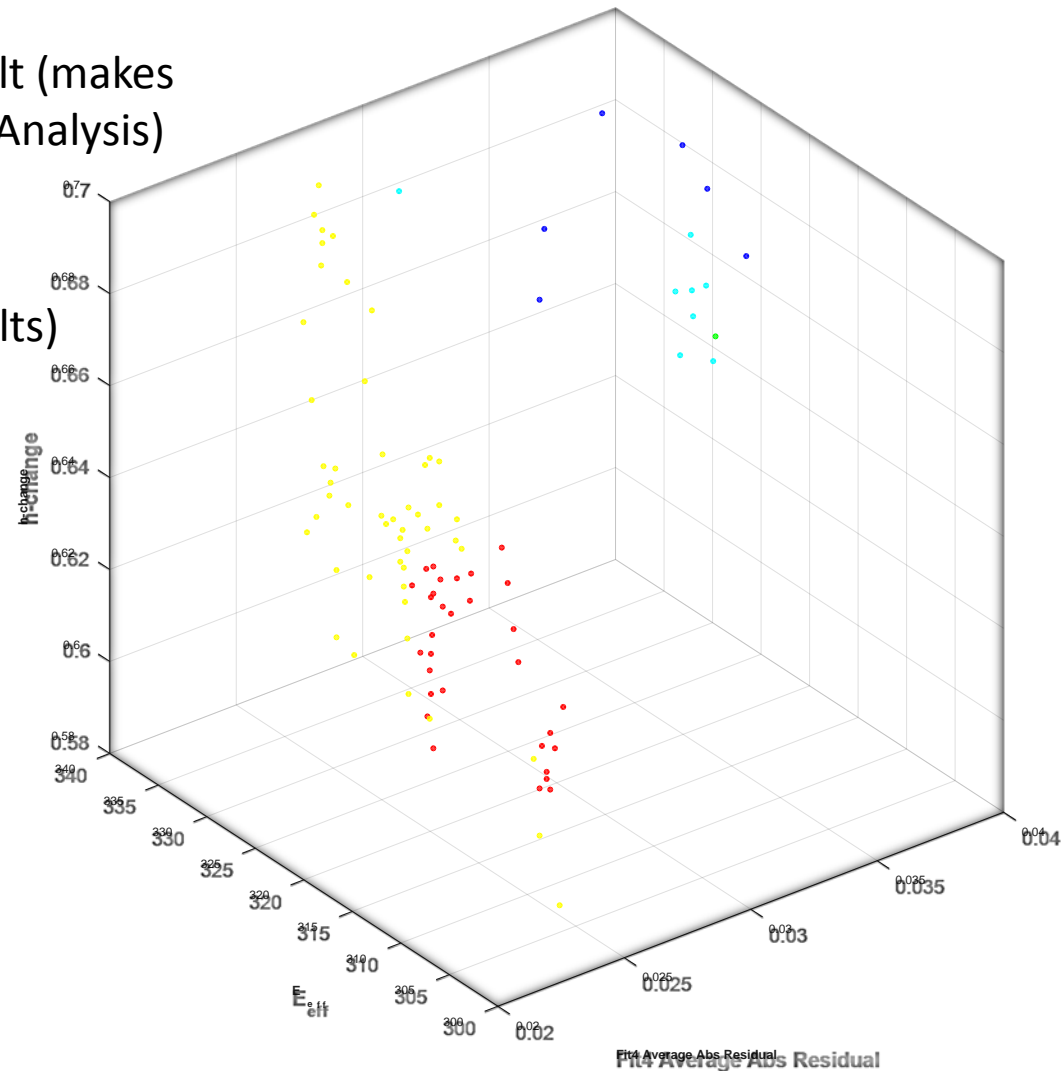
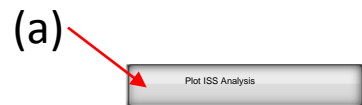
- Save data, analyses, parameters, etc. This is just saving the MATLAB workspace. It can be loaded again for further analysis, plotting, calculations, etc.
- Save the indentation stress-strain plot for the representative answer. This makes reviewing the data easy, but this figure isn't used for much else.
- Make your own scripts to tabulate data from multiple tests, do additional analysis, make plots, etc. This is easy since the MATLAB data is structured the same for each test you save.

(a) Plot the indentation stress-strain curve for the selected answer

(b) Calculate the selected result (makes the variable Stress\_Strain\_Analysis)

(c) Calculate all the results (makes the variable Stress\_Strain\_Search\_Results)

The buttons just generate variables. You still have to save the workspace to save your data.



# Example Data

- The parameters in RunMe are setup to run the example test (Example\_Test.xlsx). Make sure this works before trying your own data. Change the parameters for this example to see how the process, answer, etc. will change.

# ReadMe

- The ReadMe file is meant to be a glossary of the functions so that you can find where something is calculated or modify the code.