RUTGERS
THE STATE UNIVERSITY OF NEW JERSEY

Ceramic, Composite and Optical Materials Center

Effect of machining on residual stressing in ceramics

Principal Investigators: Adrian B. Mann & Richard A. Haber

Graduate Student: Benjamin Groth

Outline

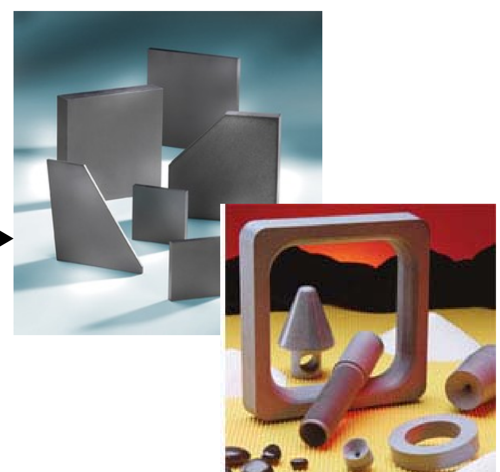
- Motivation and Objectives
- Reminder: Analysis Techniques
- Previous Work on Silicon Carbide

  (other previous work on metals, silicon and boron carbide will not be presented here – see previous center reports)
- Current Work on Silicon Carbide

Objective

Attempt to quantify changes in material structure and properties due to common machining processes, as well as link data obtained from Raman spectroscopy to mechanical data obtained from nanoindentation.

- Develop a technique for effective analysis of samples using Raman spectroscopy and nanoindentation.
- Identify the source of variation throughout polished samples on a local and bulk scale.
- Extend this into the analysis of more “realistic” sample surfaces.



Plus:

- *Residual stresses*
- *Phase transformations*
- *Voids & flaws*
- *Microstructural changes*

Raman Spectroscopy

- Renishaw InVia Raman system
  - 633nm HeNe laser
  - 514nm Argon laser
  - 785nm Diode laser

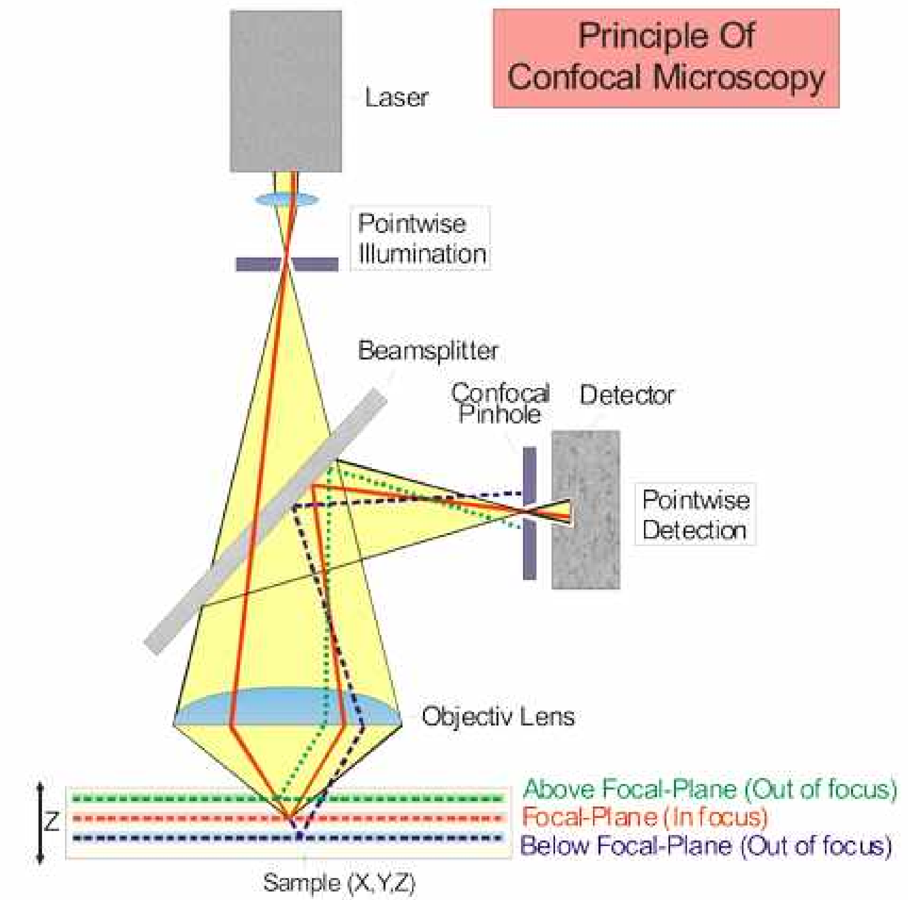
- Spatial resolution
  - Defined by physical equations:

$$\textit{Spatial Resolution} = \frac{0.61\lambda}{NA}$$ *where NA is the numerical aperture*

  - Actual resolution is typically ~1-2 μm or less

Raman Spectroscopy

- Depth of Penetration
  - Laser penetration goes as 1/α, the absorption coefficient
  - Using different laser wavelengths, can sample varying depths of the material of interest.
- Confocal Microscopy
  - Use of a pinhole (small aperture) in front of detector limits thickness of focal plane
  - This increases the depth sensitivity of the instrument

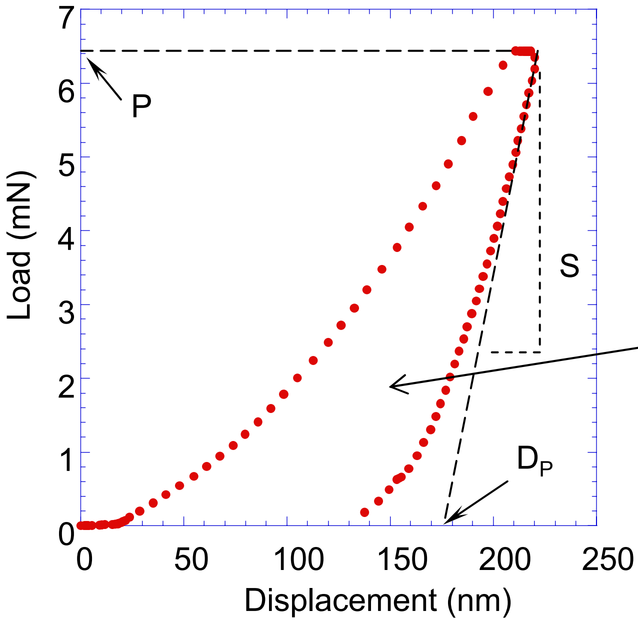


Gerhard Berth: http://fb6www.uni-paderborn.de/

Nanoindentation

- Hysitron Triboindenter 900
  - Berkovich Tip
    - Radius of Curvature: ~150nm
  - Low-load Transducer
    - 50uN to 10mN
    - <1nN resolution
- Automated controller
  - Ability to set up many indents at once
  - Feedback control for drift correction
- Indent Spacing
  - Spacing of indents is effectively limited by the size of the indent.

Quantifying hardening and stresses due to machining using nanoindentation



Work of indentation (elastic and plastic) is area under the loading curve, plastic work is area between curves.

Contact area A is common source of error (especially in rough or stressed samples)

Calculation of Elastic Modulus (E) and Hardness (H)

$E^* = S/2 \sqrt{\pi/A}$

$H = P/A$

Where: $A = A(D_P)$ and $\frac{1}{E^*} = \frac{1-\upsilon_1^2}{E_1} + \frac{1-\upsilon_2^2}{E_2}$

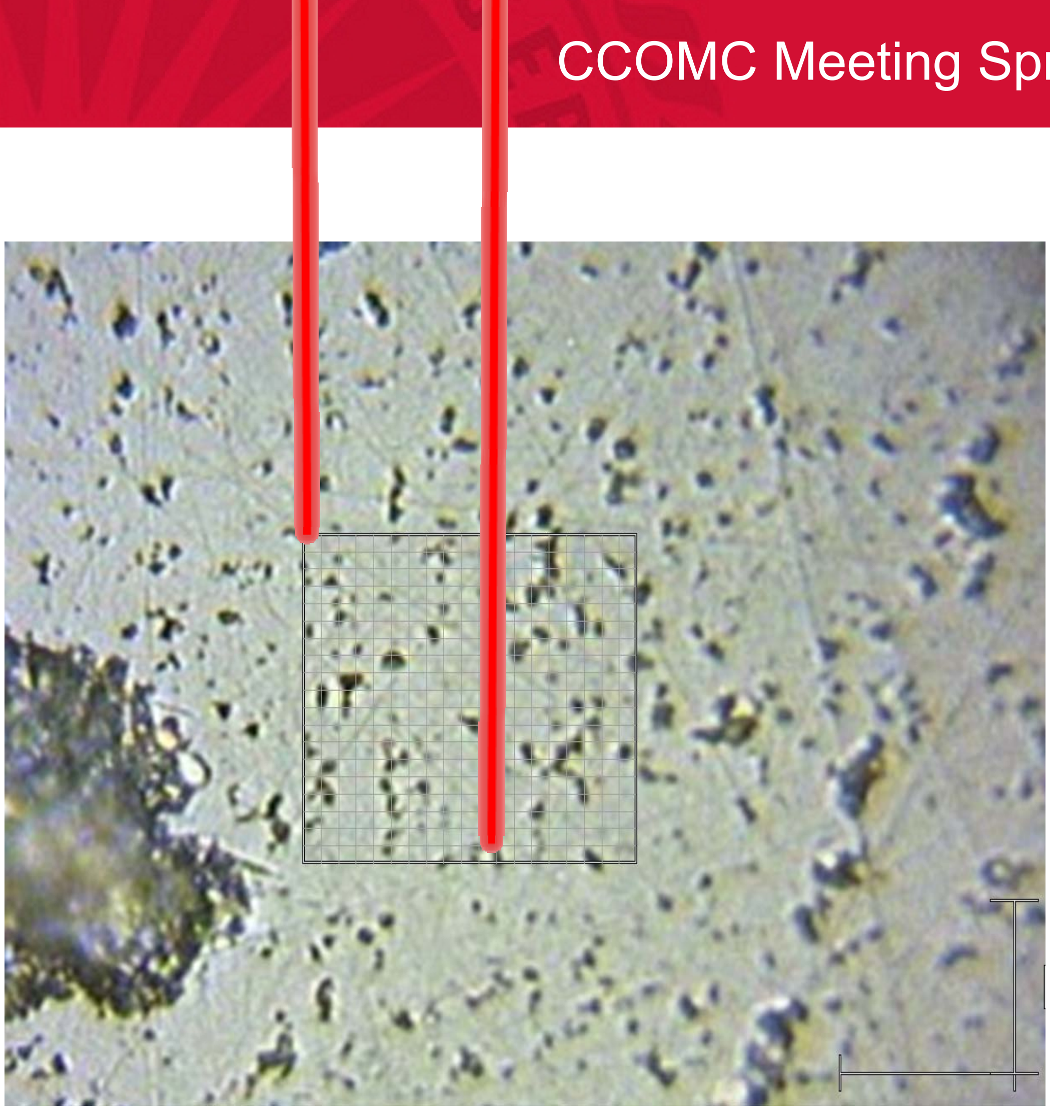
Can eliminate error using:

$E^{*2}/H = \pi S^2/4P$

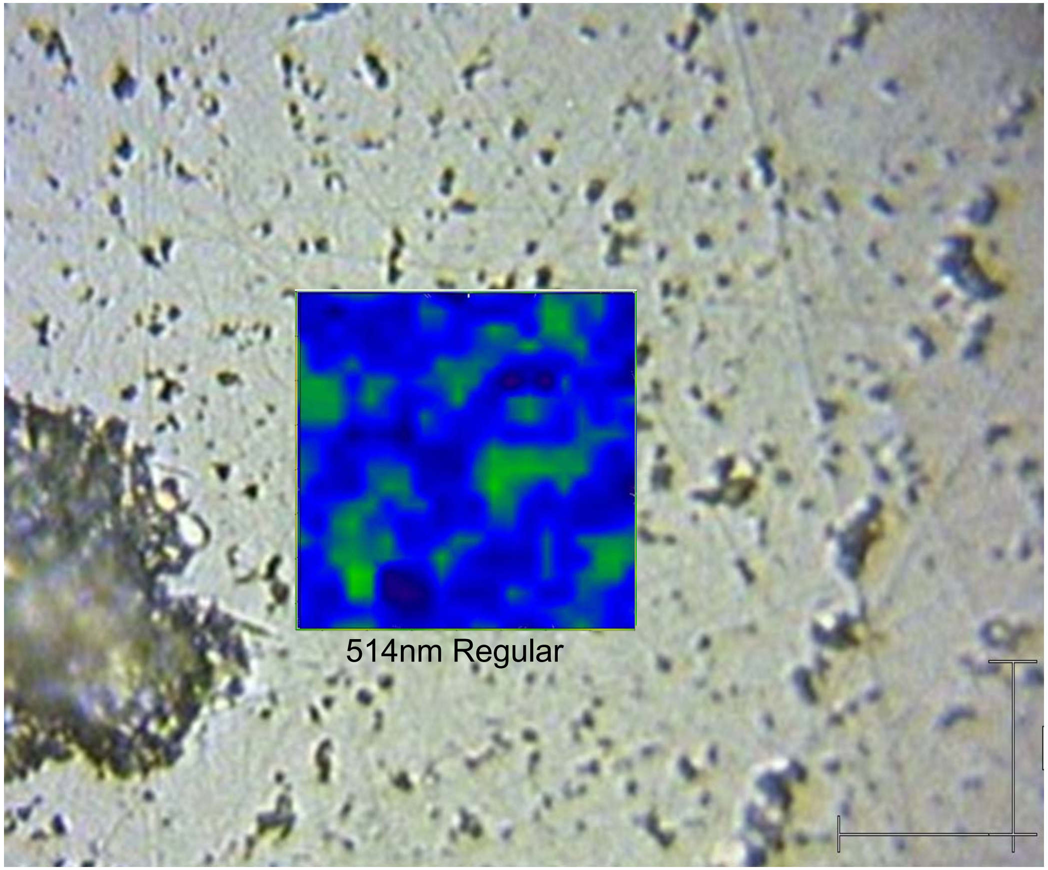
Which is independent of A

W.C.Oliver, G.M.Pharr, Journal of Materials Research, 7 (1992) 1564-1583.
W.C.Oliver, G.M.Pharr, Journal of Materials Research., 19 (2004) 3 – 20.

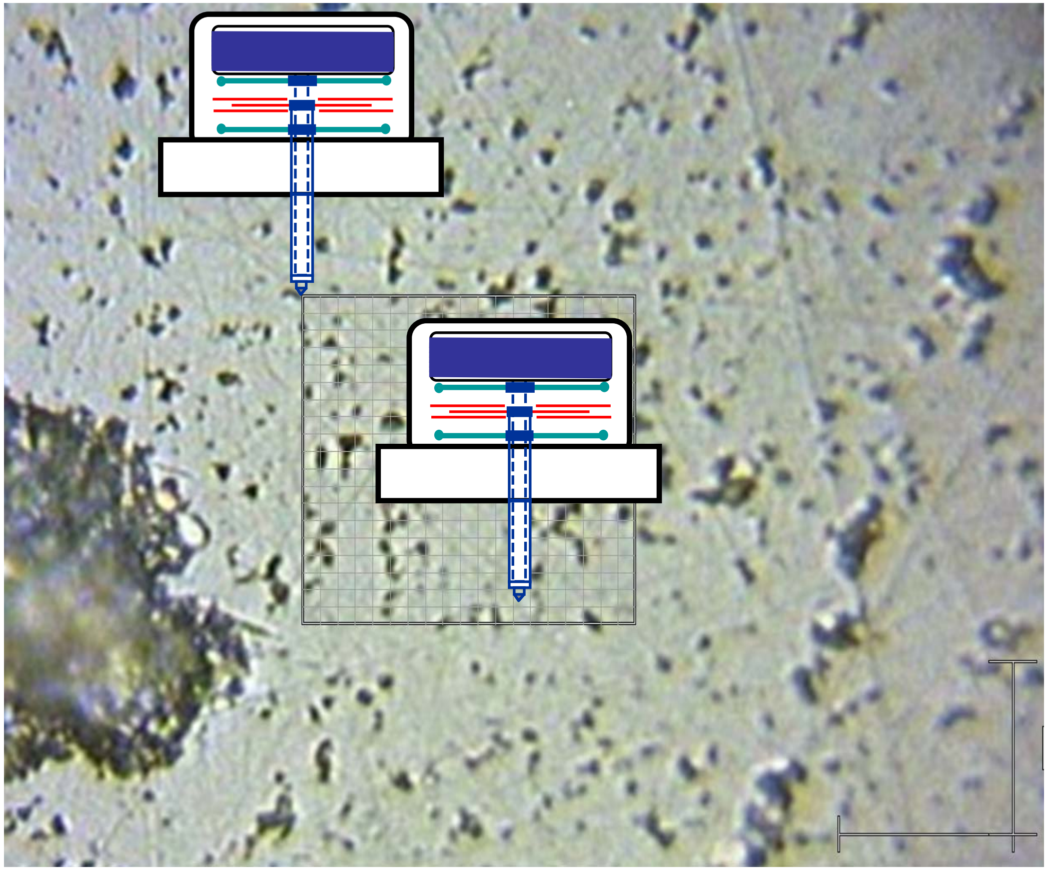
Spectral Maps



Spectral Maps – 2 laser wavelengths with 2 system settings

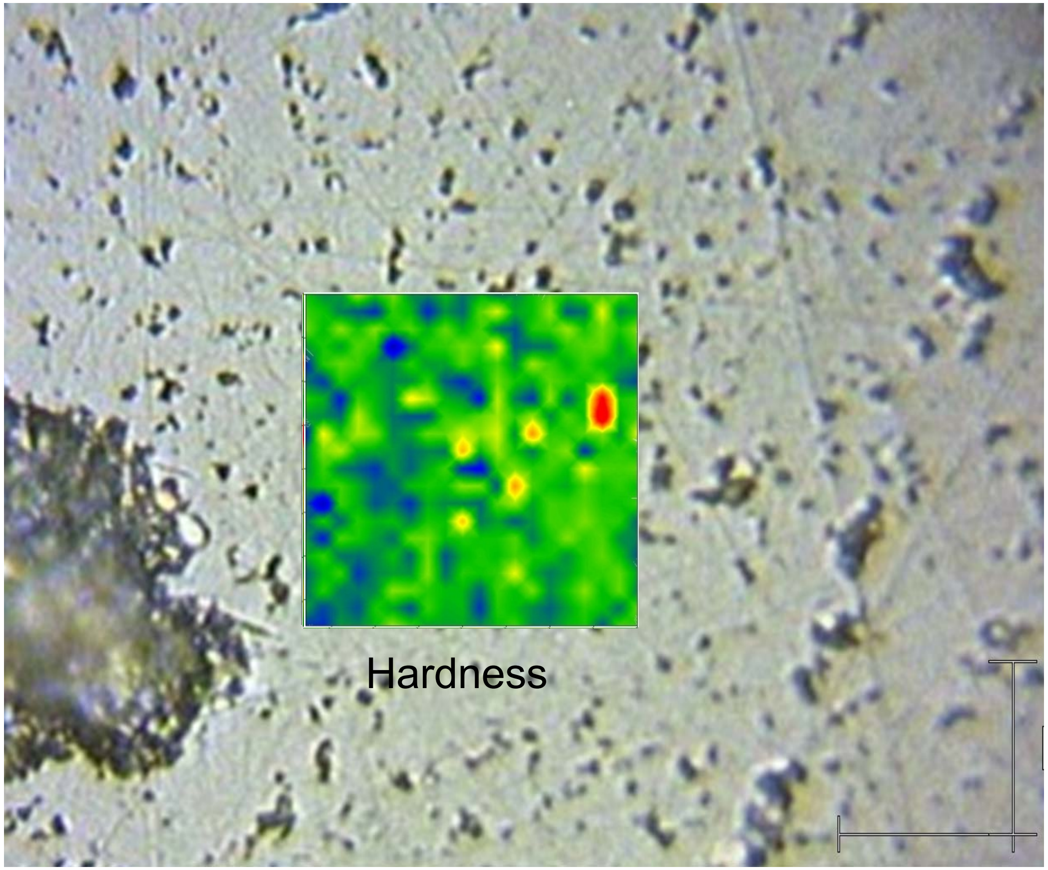


Mechanical Maps



Mechanical Maps

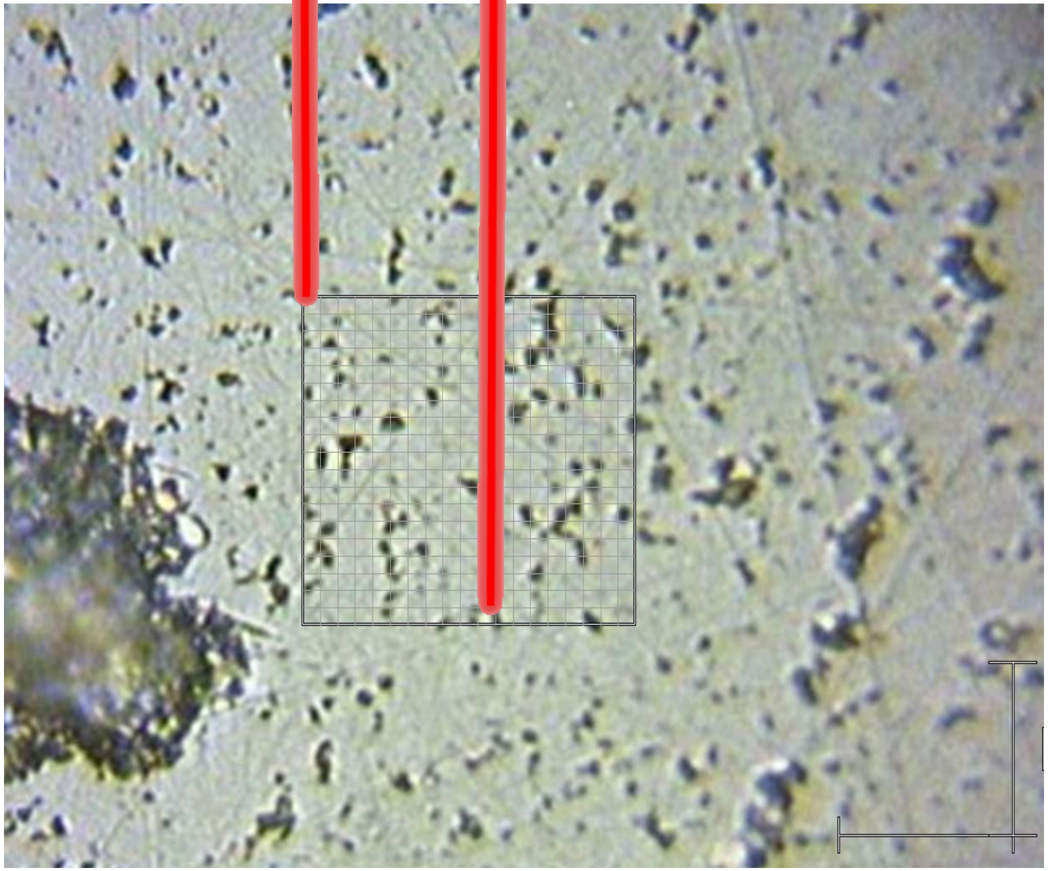
Modulus



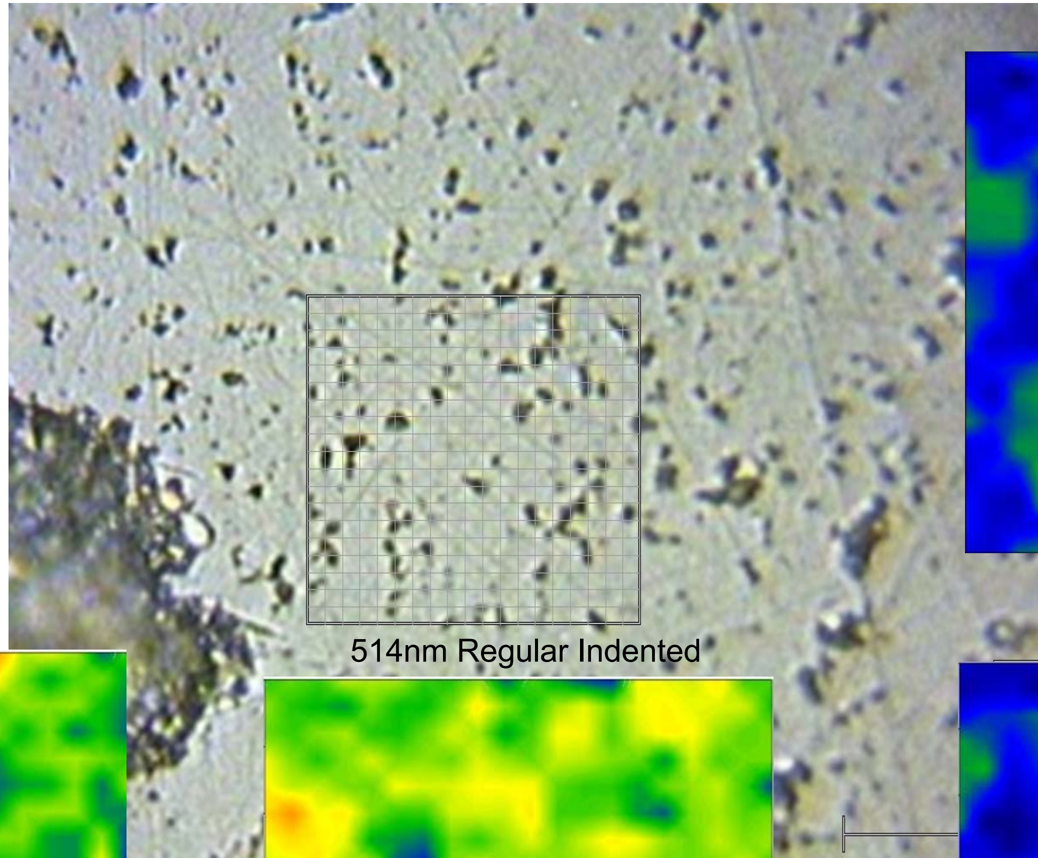
Hardness

Work

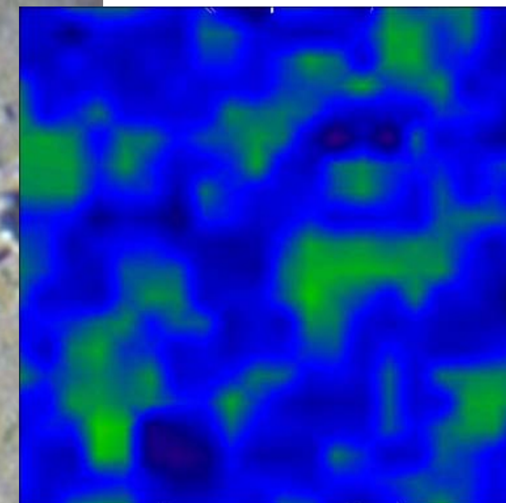
Spectral Maps on Indented surfaces



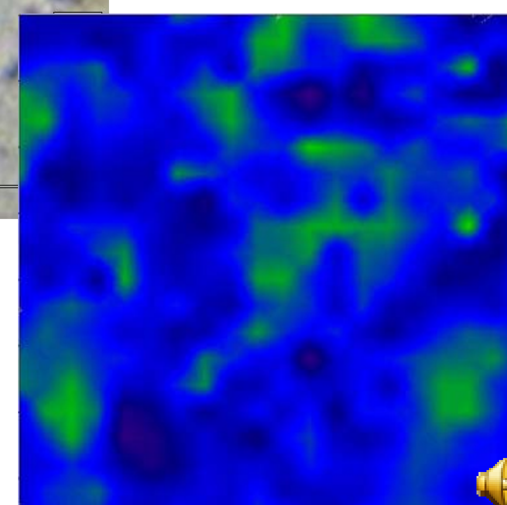
Spectral Maps – 2 laser wavelengths with 2 system settings



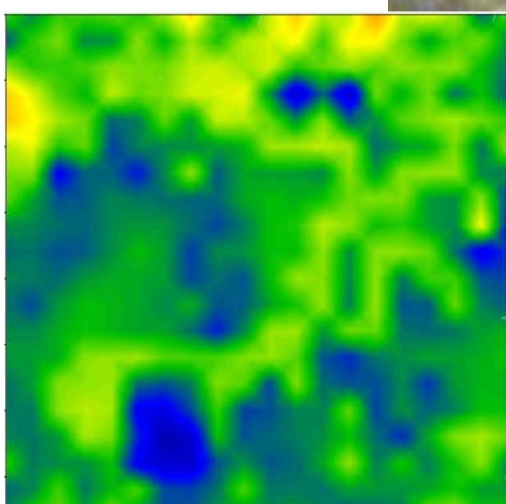
633nm Regular Indented



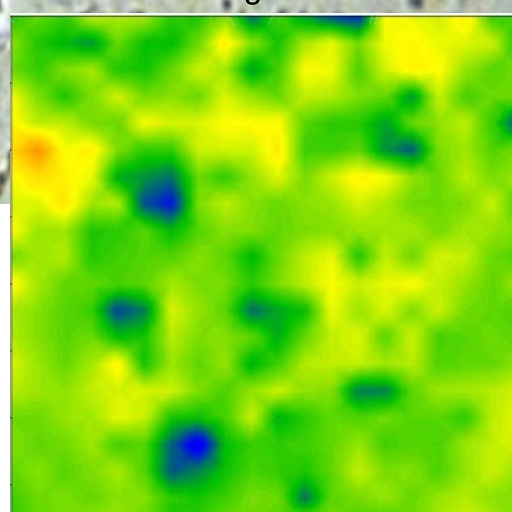
633nm Confocal Indented



514nm Confocal

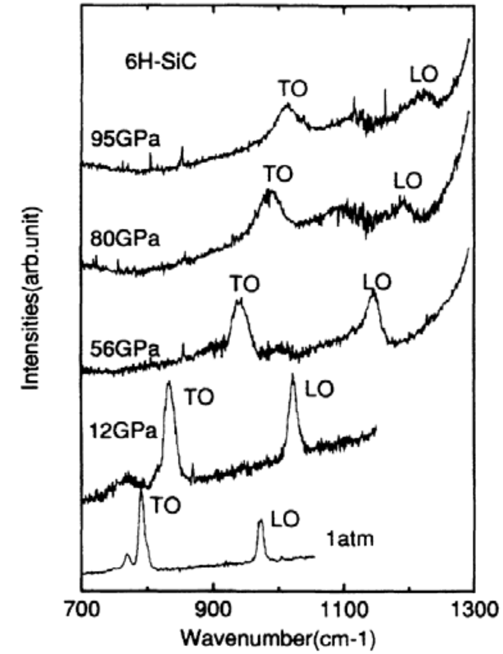


514nm Regular Indented



Stress Analysis

- Work done by many to analyze effect of stress on Raman spectra
- Stress evolves in spectral data through peak shifting, broadening, flattening.
- Peak of interest for 6H-SiC is the TO-Peak located around 789 cm^{-1}



$$\omega_{TO}(cm^{-1}) = 789.2 + 3.11\sigma - 0.009\sigma^2$$

- Equation used to describe shift vs. stress with empirically fit parameters, where ω_{TO} is the shift in the TO peak position and σ is the stress measured in GPa

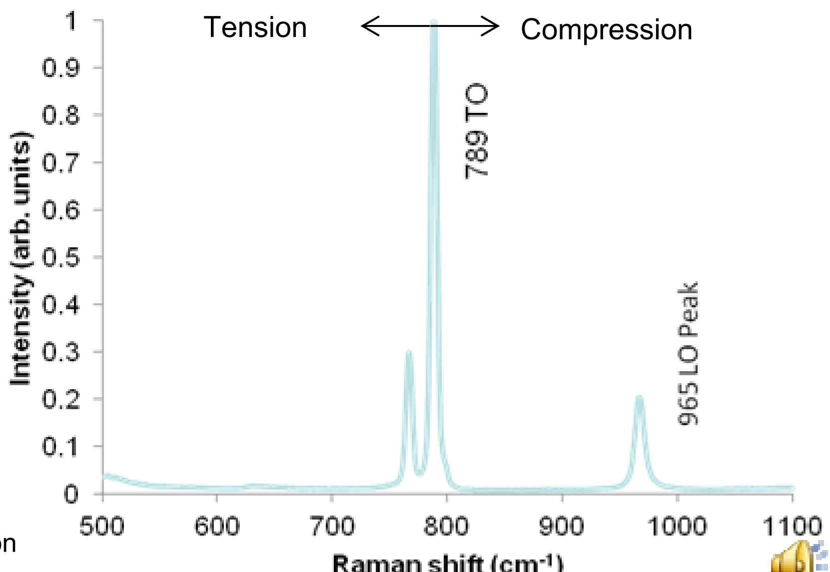
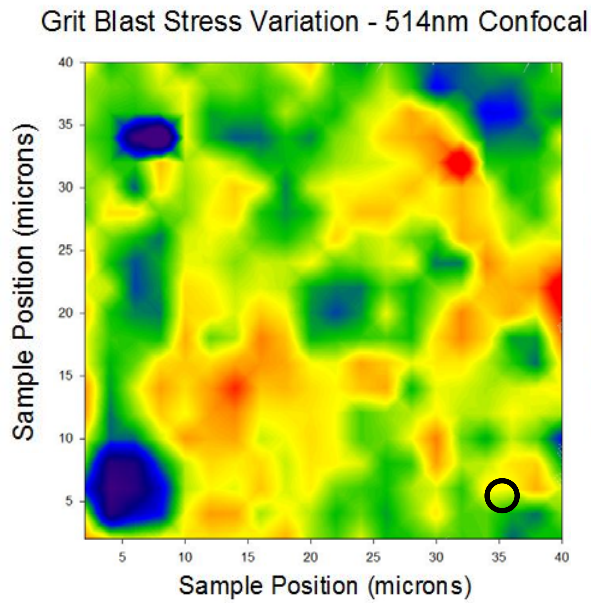


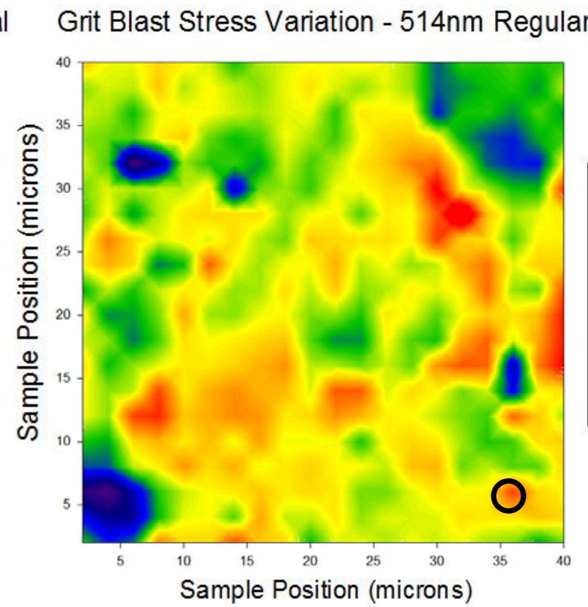
Image and Equation from: J. Liu and Y. K. Vohra, "Raman modes of 6H polytype of silicon carbide to ultrahigh pressures: A comparison with silicon and diamond," Physical review letters, vol. 72, no. 26, pp. 4105–4108, 1994.

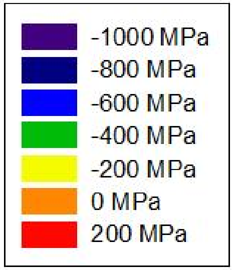
Current Silicon Carbide work

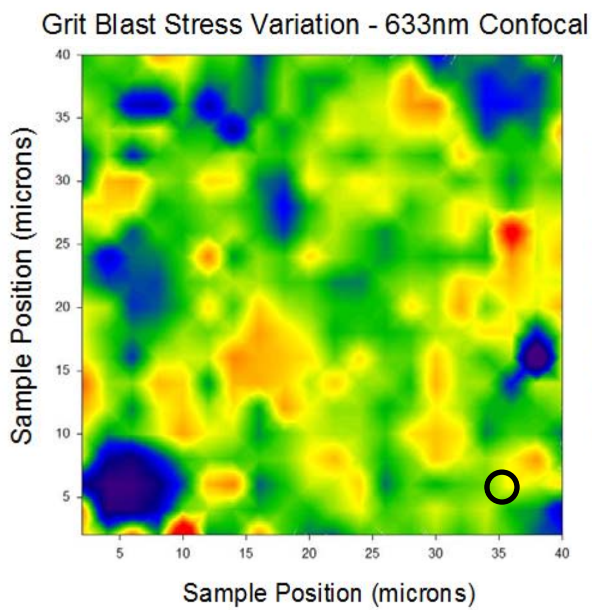
- 6H-Silicon Carbide samples were obtained with four levels of polishing:
  - "Standard" Finish
  - Grit Blast surface
  - Rotary ground surface
  - Mirror Polish surface
- Stress analysis performed on these samples showed variation both between the samples and within the depth of each sample.
- Efforts were made to understand these variations through further analysis of the Raman spectra and mechanical property maps from indentation

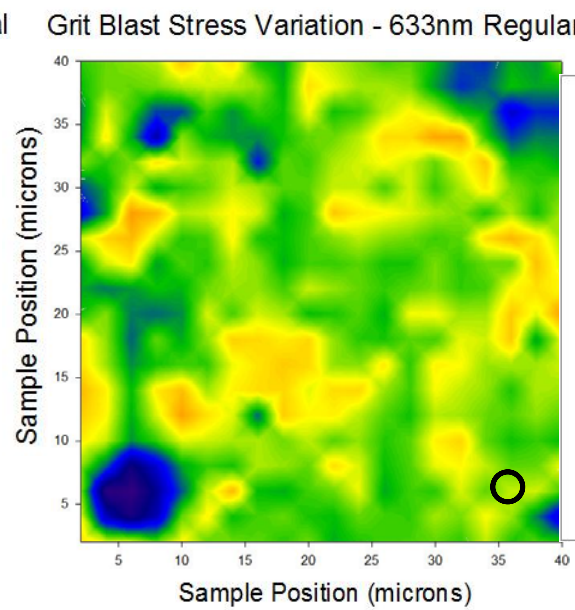
Grit blast

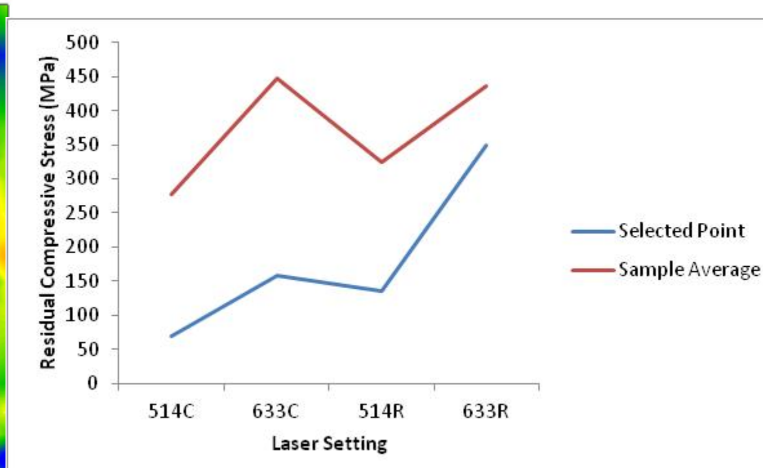

Grit Blast Stress Variation - 514nm Confocal
Sample Position (microns)
Sample Position (microns)


Grit Blast Stress Variation - 514nm Regular
Sample Position (microns)
Sample Position (microns)

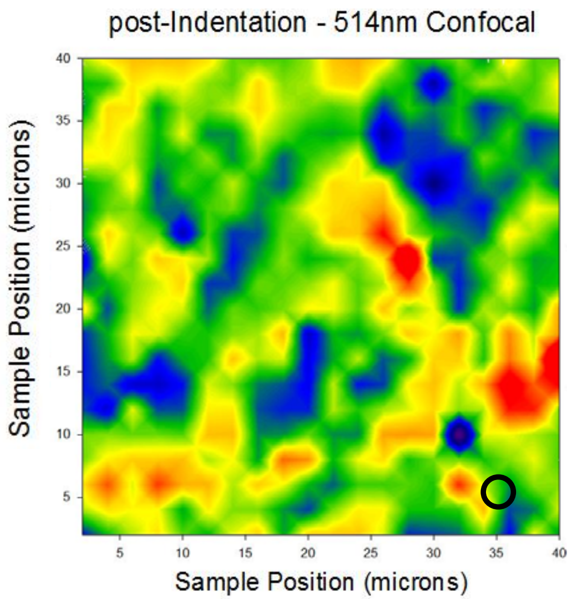

-1000 MPa
-800 MPa
-600 MPa
-400 MPa
-200 MPa
0 MPa
200 MPa

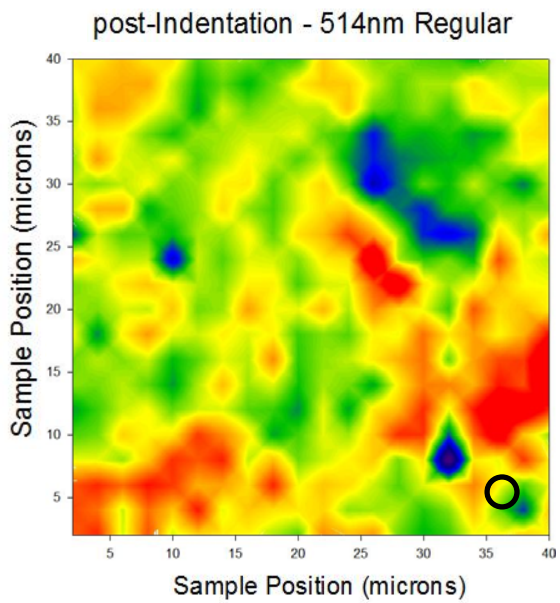

Grit Blast Stress Variation - 633nm Confocal
Sample Position (microns)
Sample Position (microns)

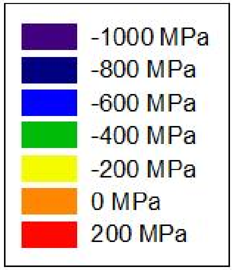

Grit Blast Stress Variation - 633nm Regular
Sample Position (microns)
Sample Position (microns)

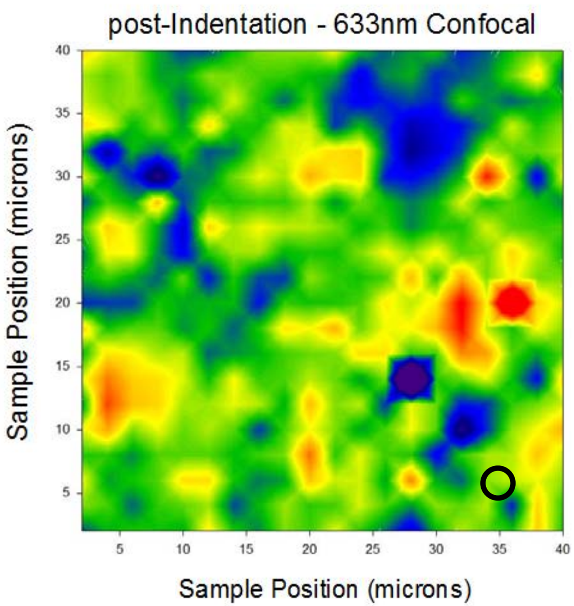

Residual Compressive Stress (MPa)
Laser Setting
514C
633C
514R
633R
Selected Point
Sample Average

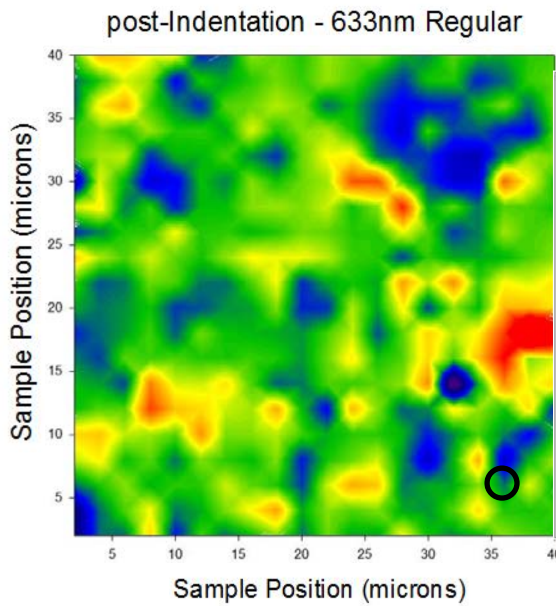
Grit blast

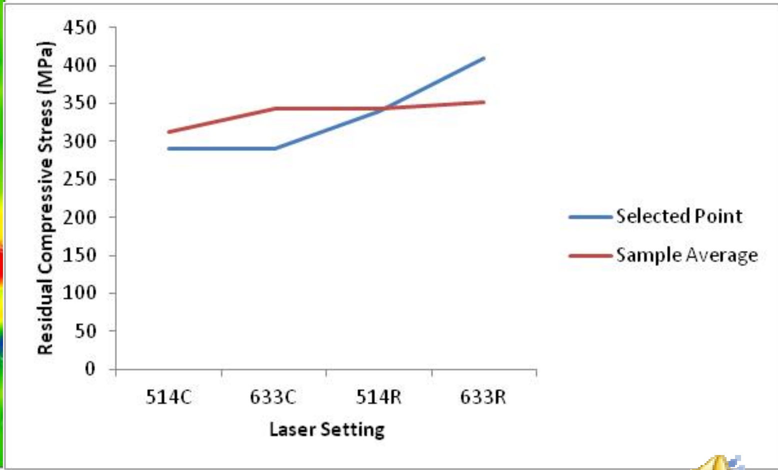

post-Indentation - 514nm Confocal
Sample Position (microns)
Sample Position (microns)
5 10 15 20 25 30 35 40


post-Indentation - 514nm Regular
Sample Position (microns)
Sample Position (microns)
5 10 15 20 25 30 35 40


-1000 MPa
-800 MPa
-600 MPa
-400 MPa
-200 MPa
0 MPa
200 MPa

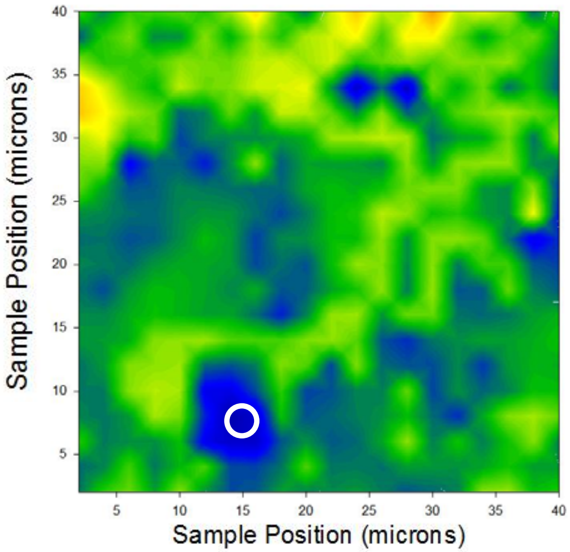

post-Indentation - 633nm Confocal
Sample Position (microns)
Sample Position (microns)
5 10 15 20 25 30 35 40


post-Indentation - 633nm Regular
Sample Position (microns)
Sample Position (microns)
5 10 15 20 25 30 35 40

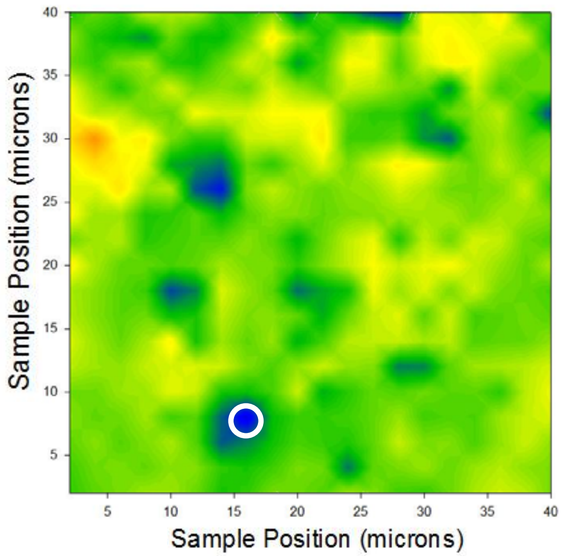

Residual Compressive Stress (MPa)
0 50 100 150 200 250 300 350 400 450
514C 633C 514R 633R
Laser Setting
Selected Point
Sample Average

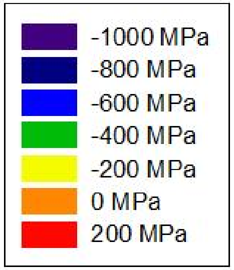
Mirror Finish

Mirror Finish Stress Variation - 514nm Confocal

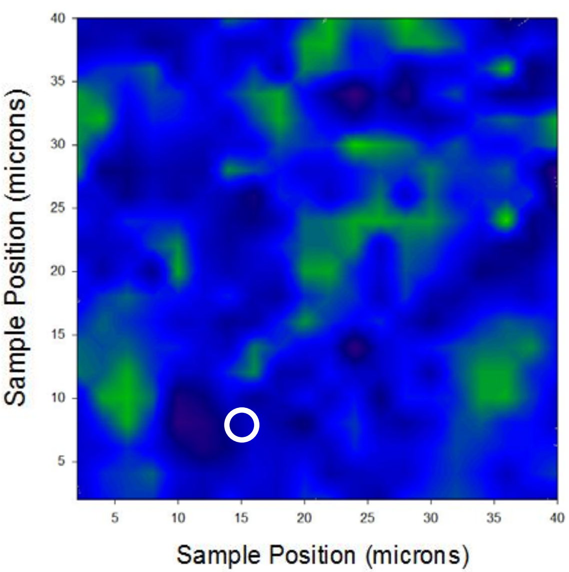


Mirror Finish Stress Variation - 514nm Regular

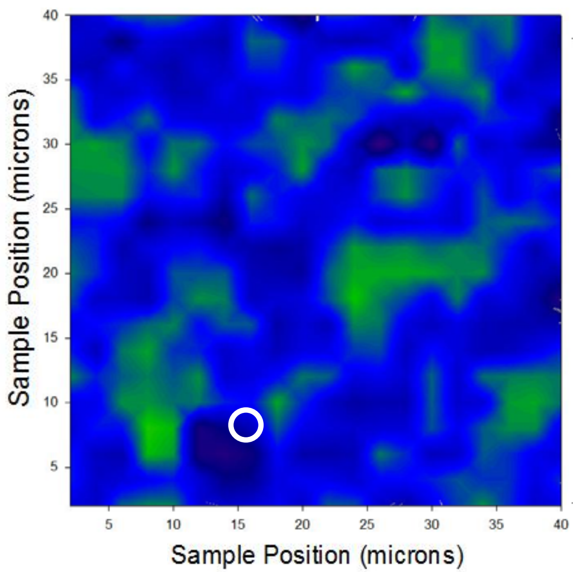


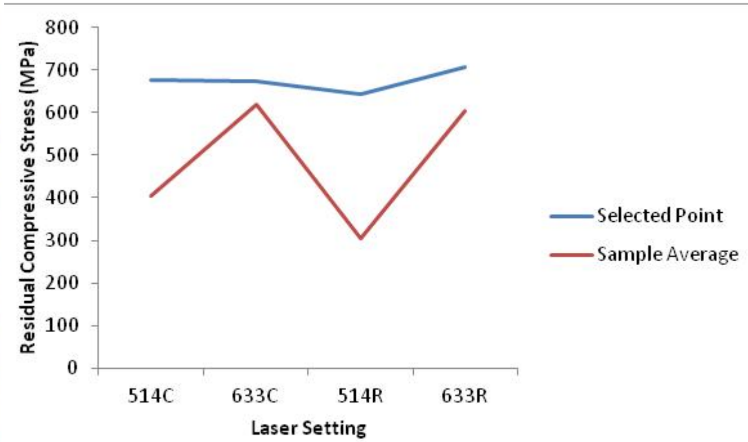


Mirror Finish Stress Variation - 633nm Confocal

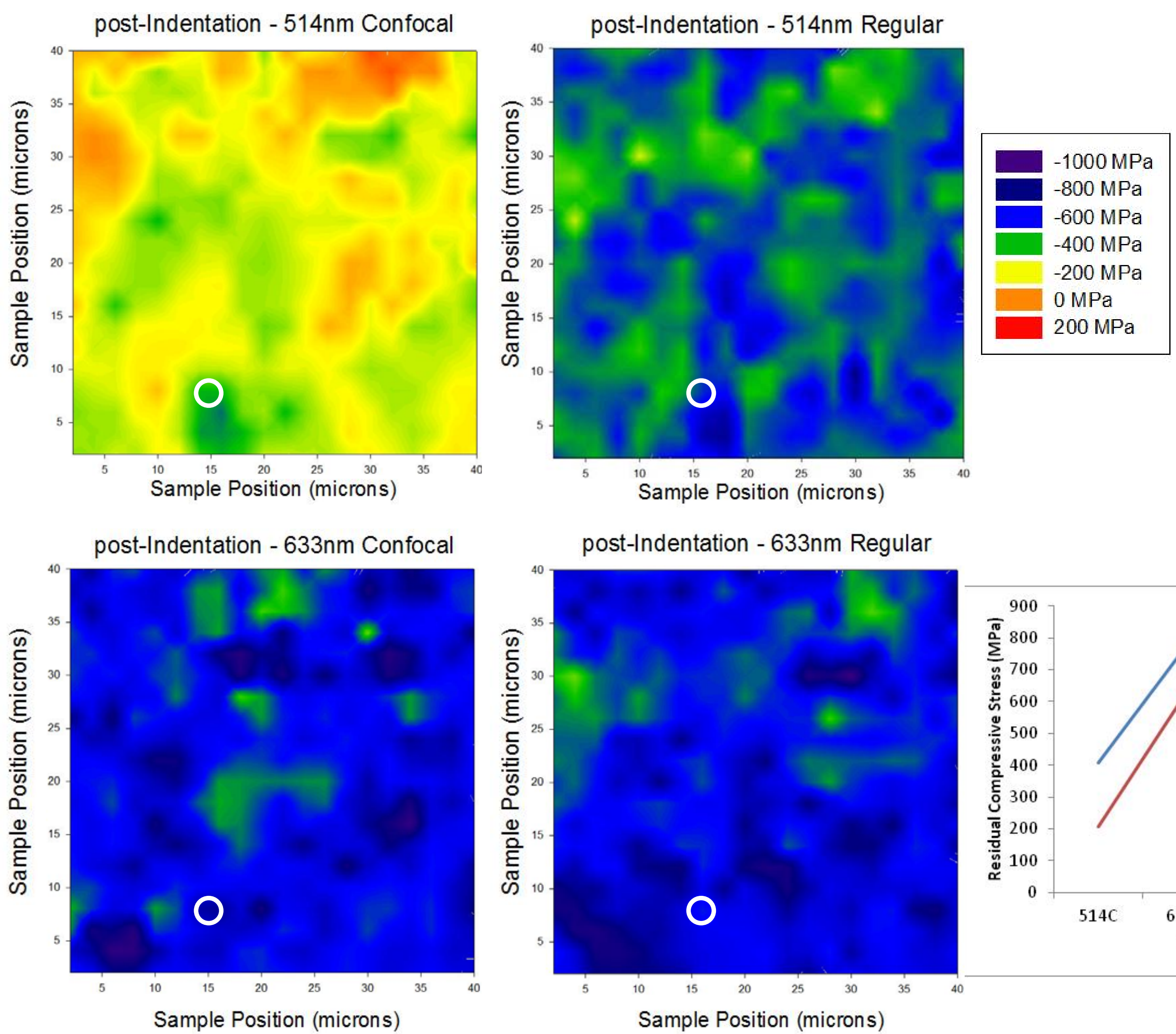


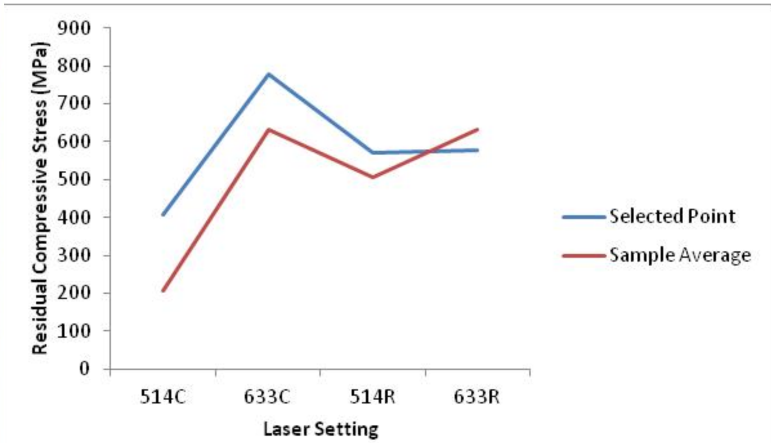
Mirror Finish Stress Variation - 633nm Regular



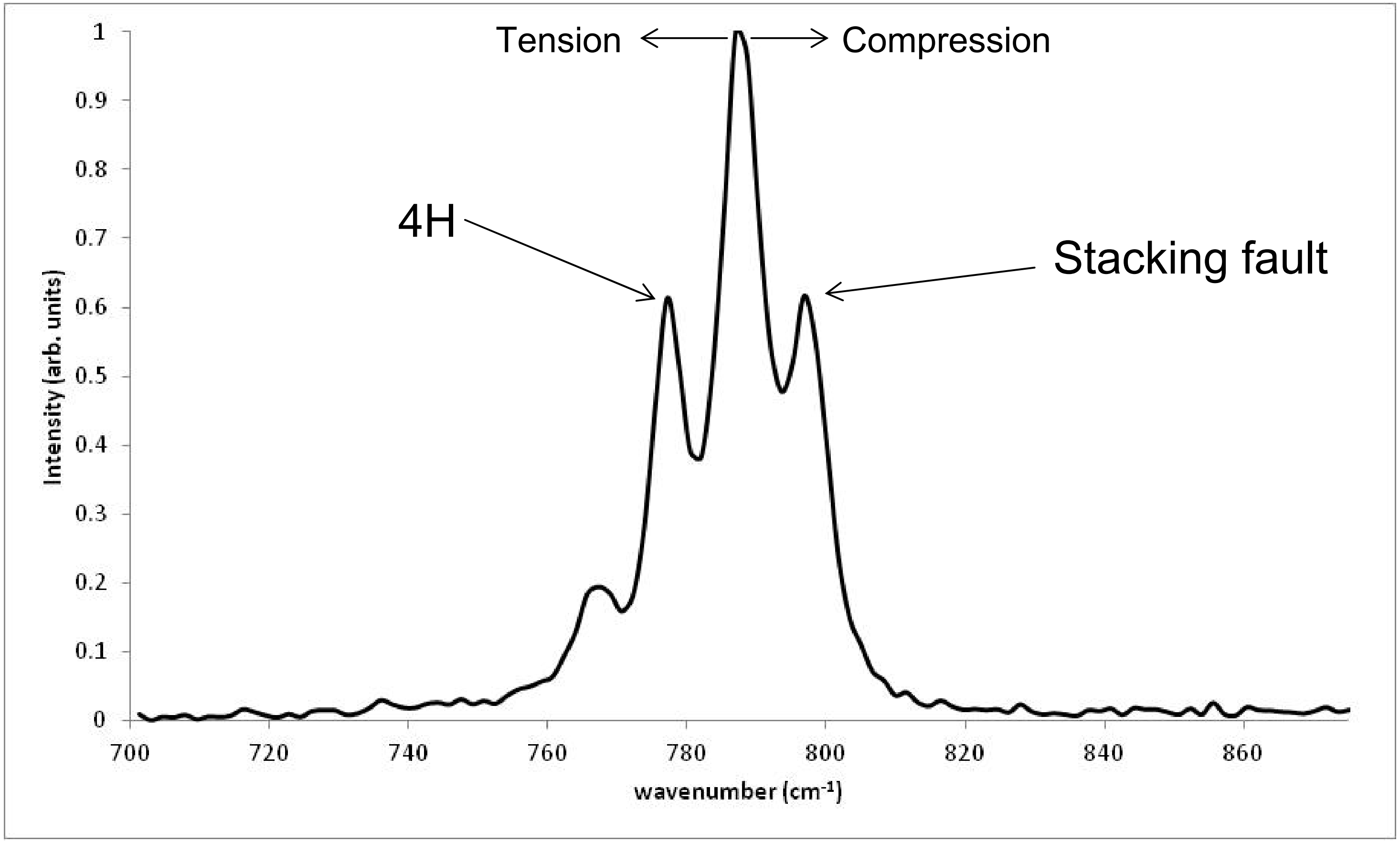


Mirror Finish

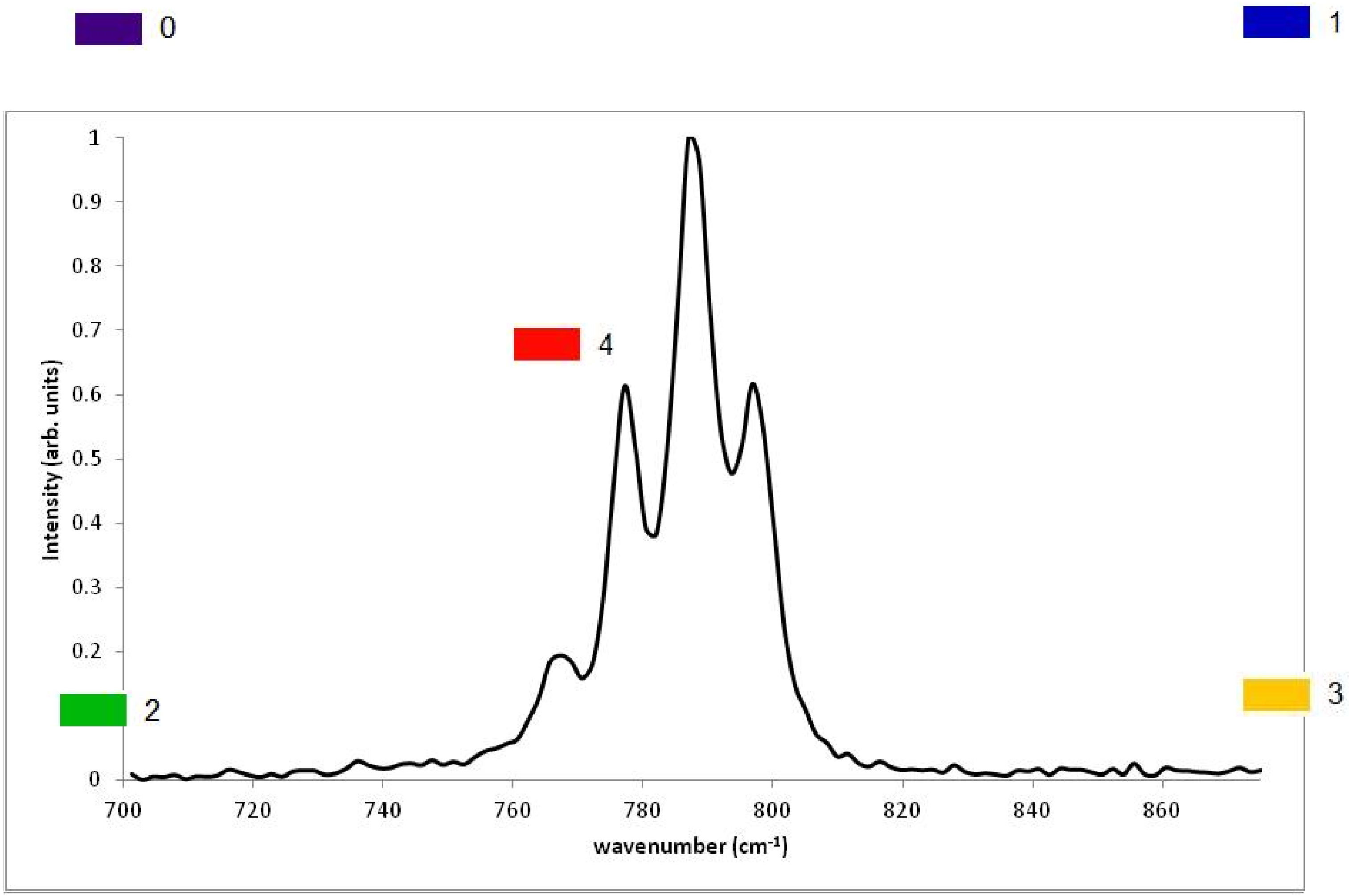




Possible Contributor to stress variation: Stacking Faults

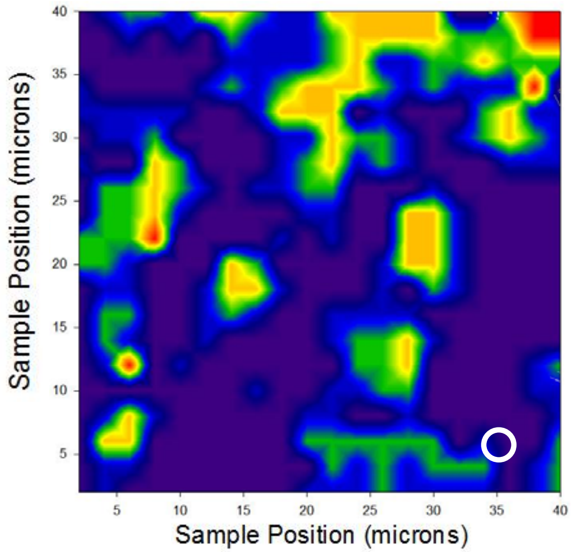


Possible Contributor to stress variation: Stacking Faults

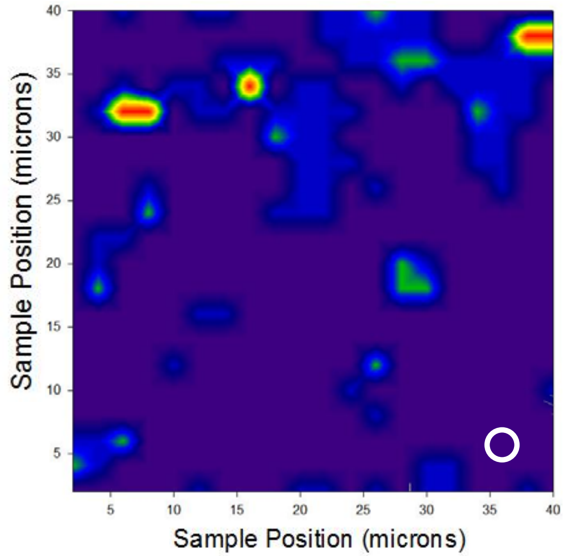


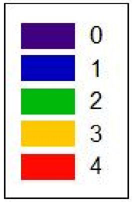
Grit blast

Stacking Fault Peak Intensity - 514nm Confocal

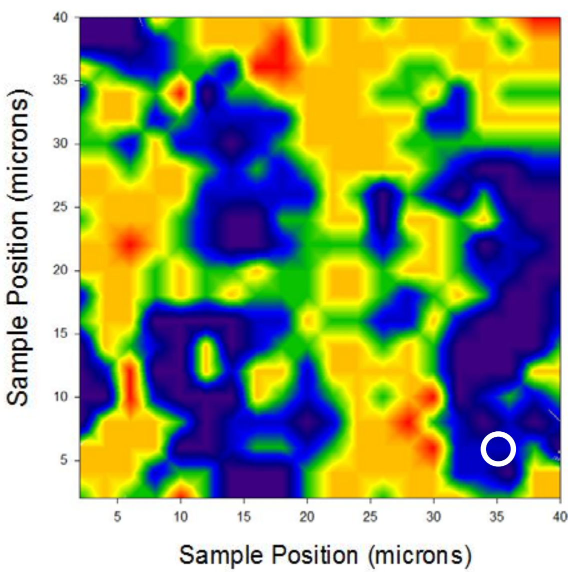


Stacking Fault Peak Intensity - 514nm Regular

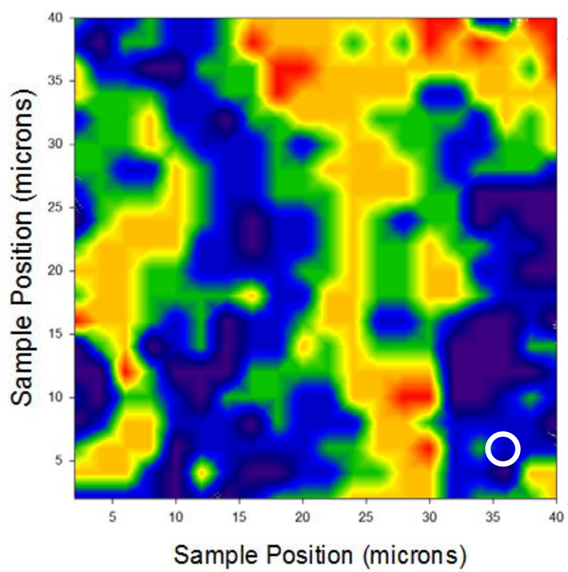


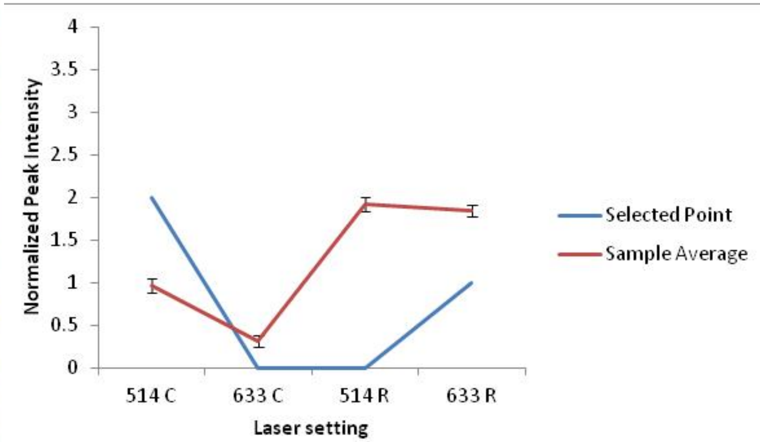


Stacking Fault Peak Intensity - 633nm Confocal



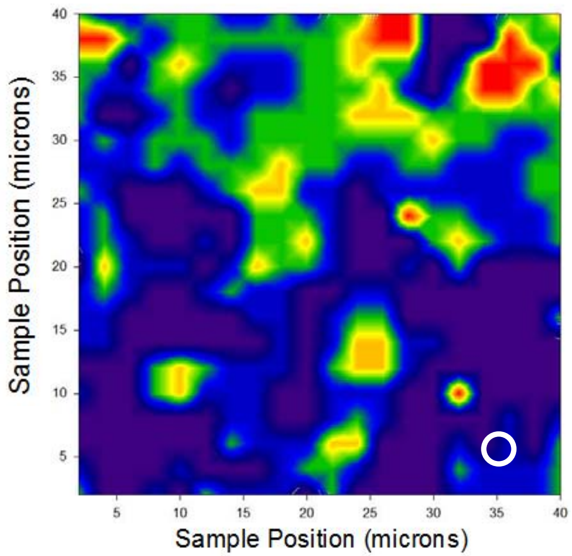
Stacking Fault Peak Intensity - 633nm Regular



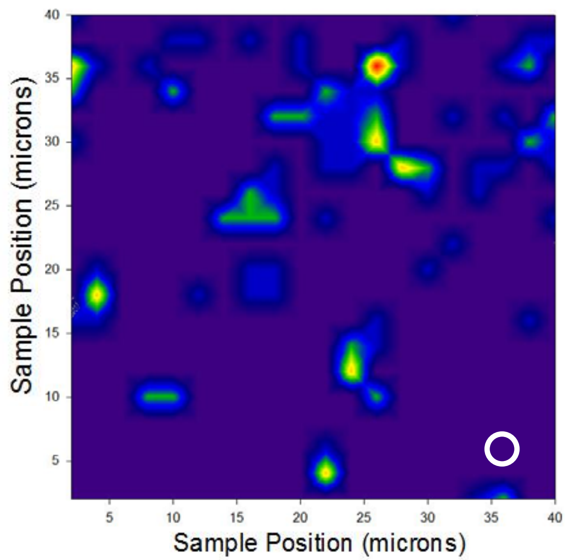


Grit blast

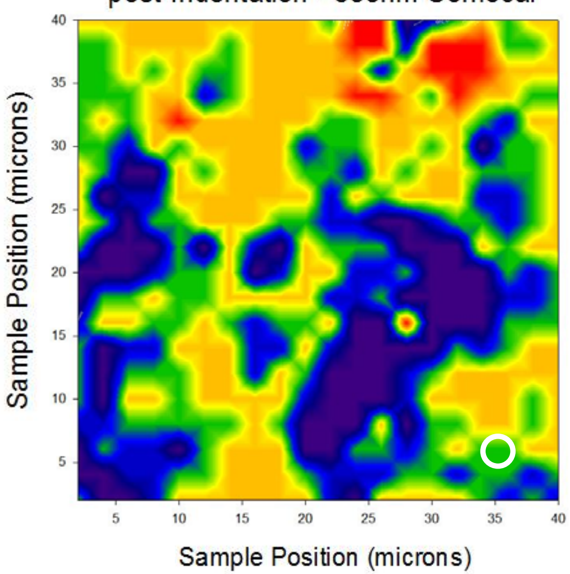
post-Indentation - 514nm Confocal



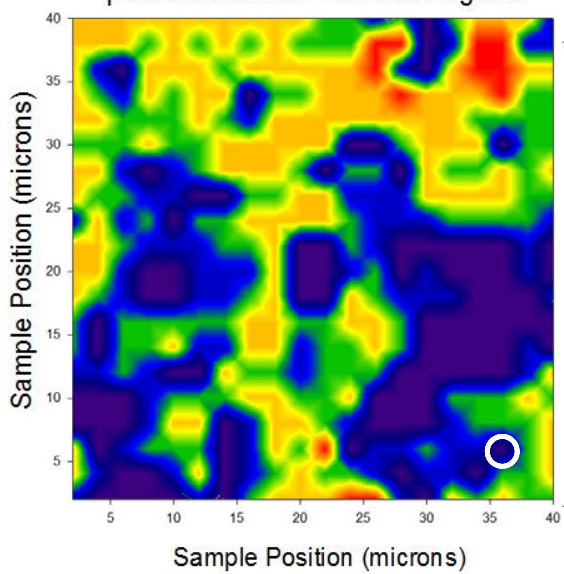
post-Indentation - 514nm Regular

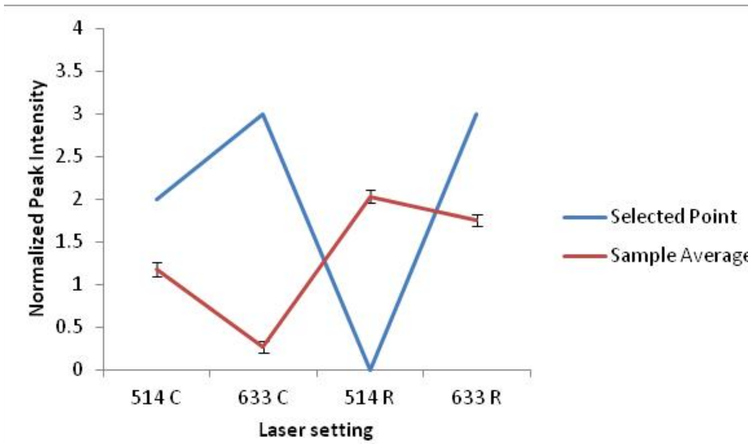


post-Indentation - 633nm Confocal

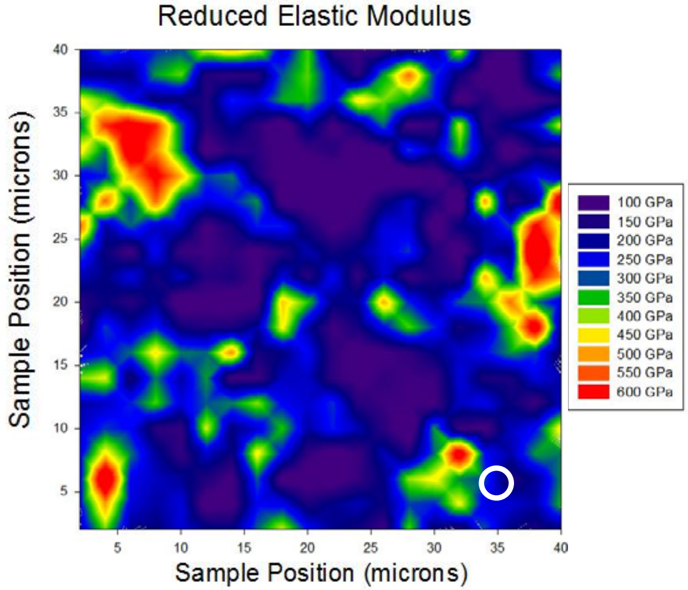


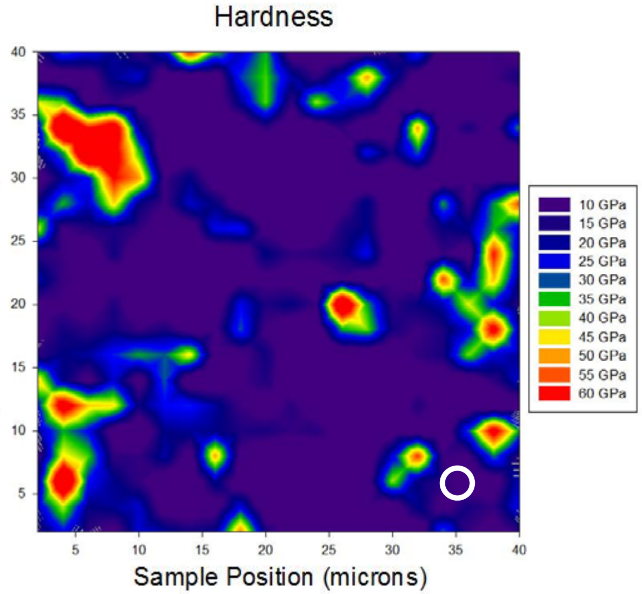
post-Indentation - 633nm Regular

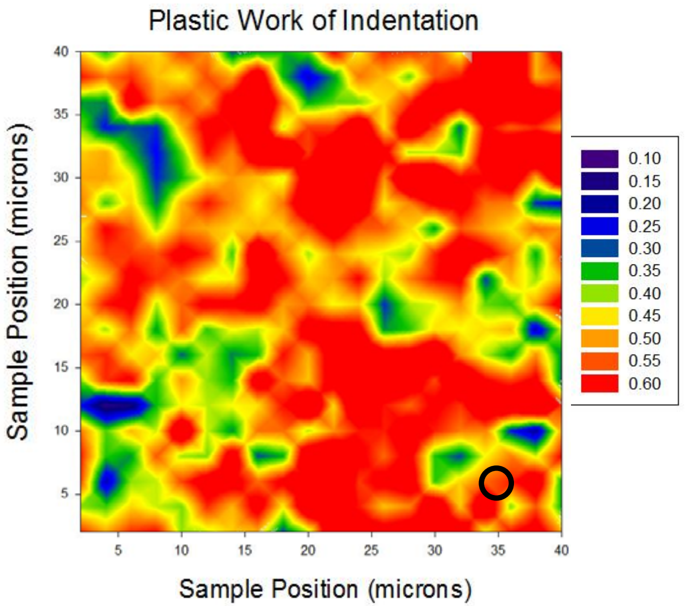


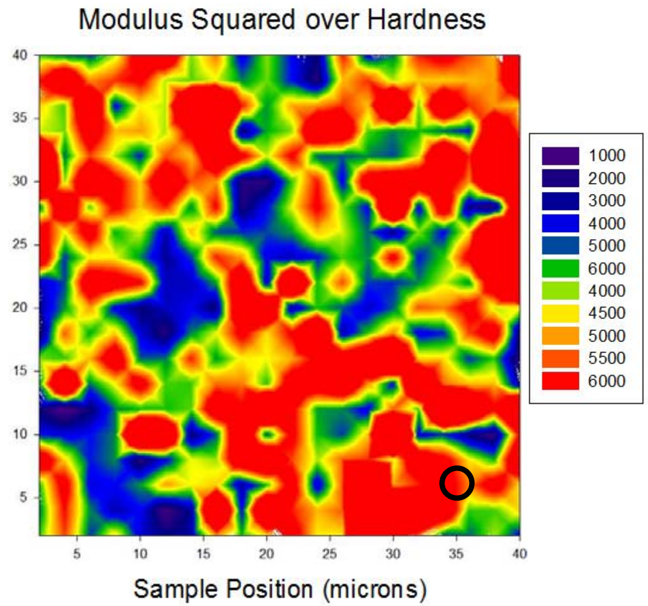


Grit blast







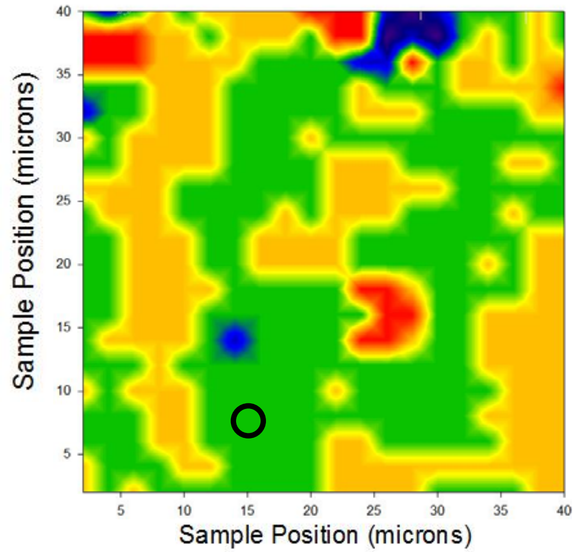


Selected Point:
Modulus: 324 GPa
Hardness: 23 GPa
Work:0.35
E^2/H:4550 GPa*

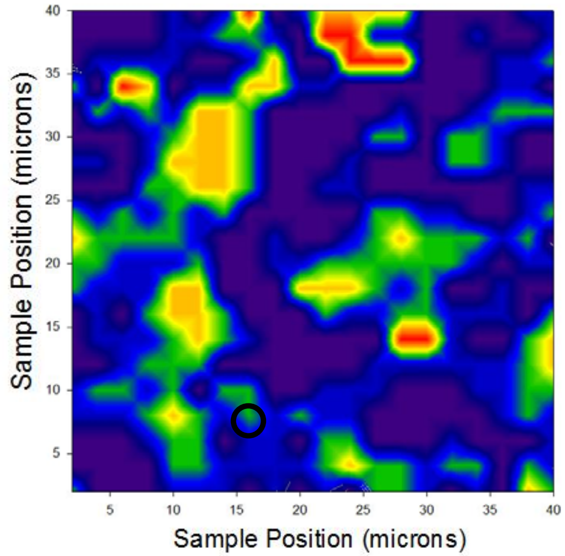
Sample Average:
Modulus: 237 GPa
Hardness: 15 GPa
Work:0.52
E^2/H:5063 GPa*

Mirror Finish

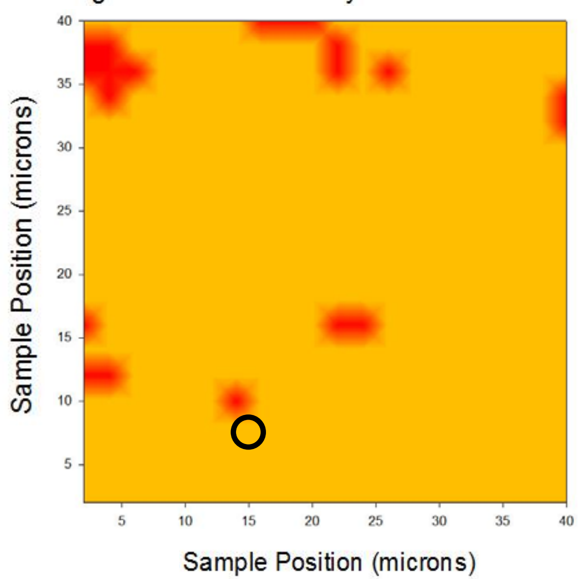
Stacking Fault Peak Intensity - 514nm Confocal



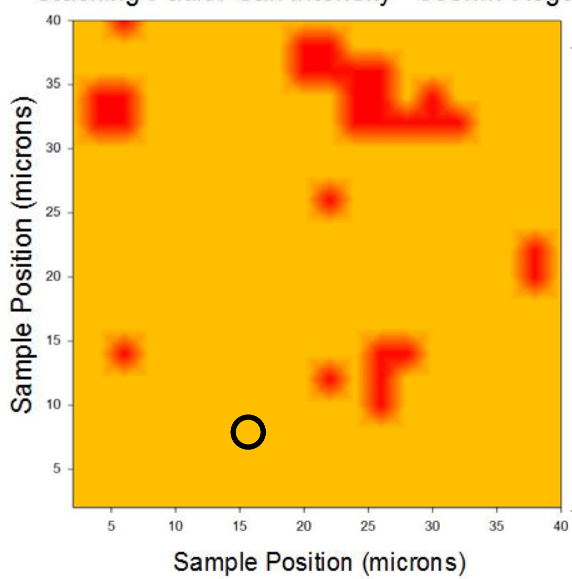
Stacking Fault Peak Intensity - 514nm Regular

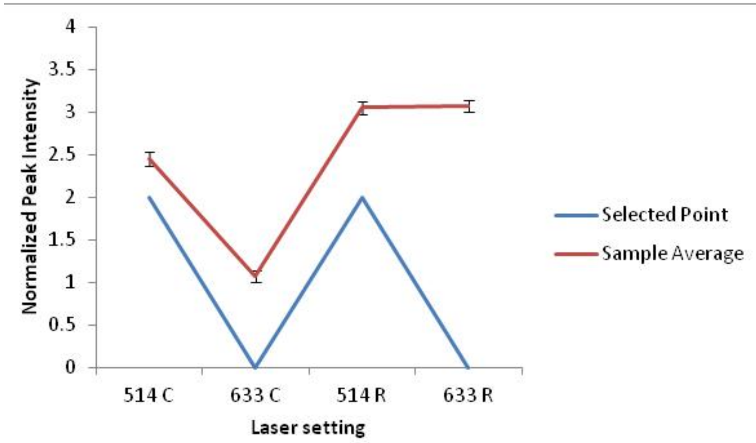


Stacking Fault Peak Intensity - 633nm Confocal

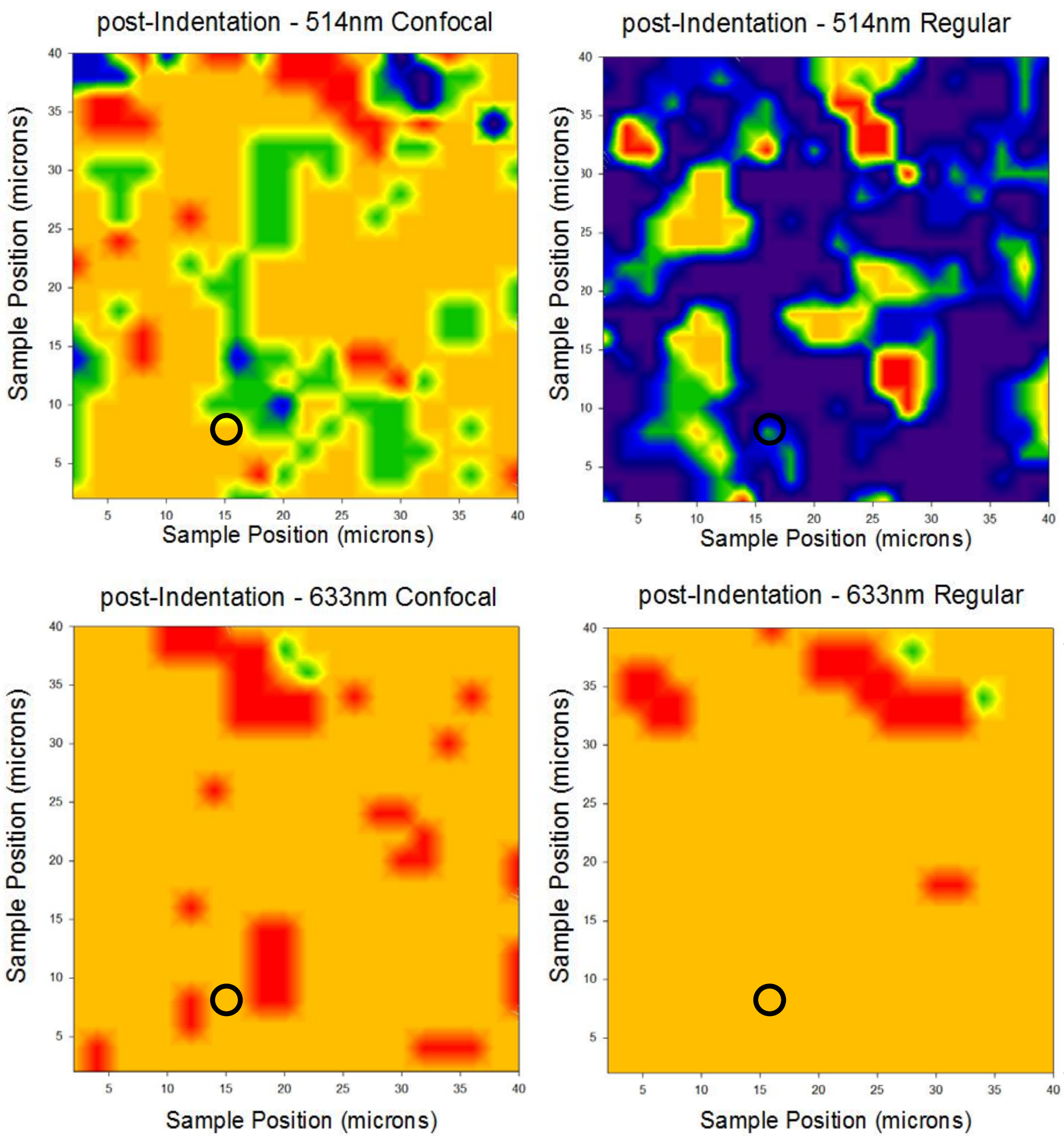


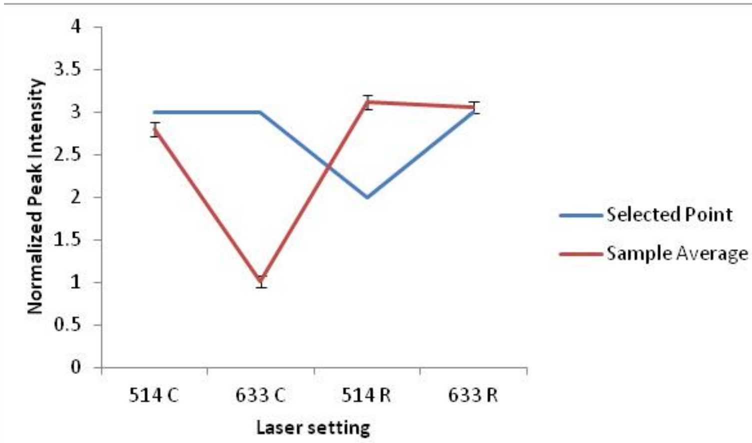
Stacking Fault Peak Intensity - 633nm Regular

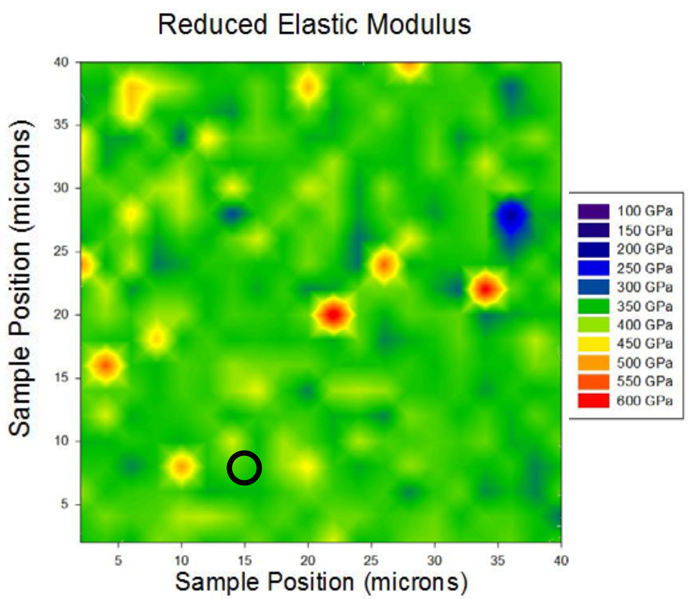


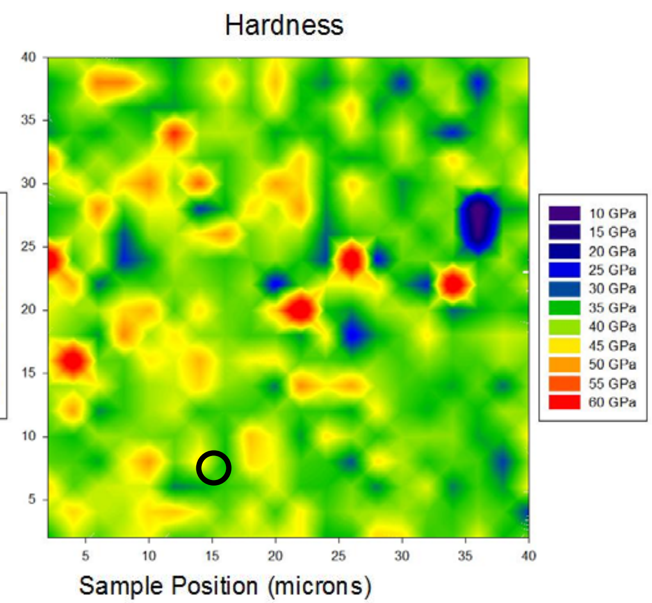


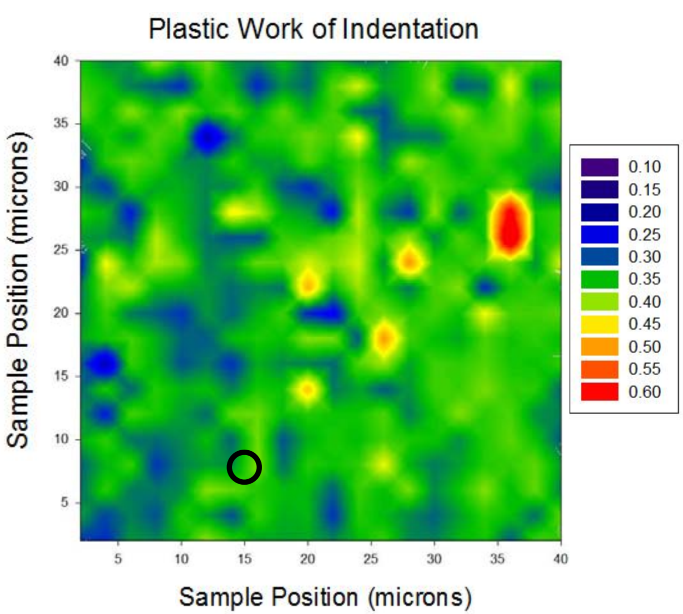
Mirror Finish

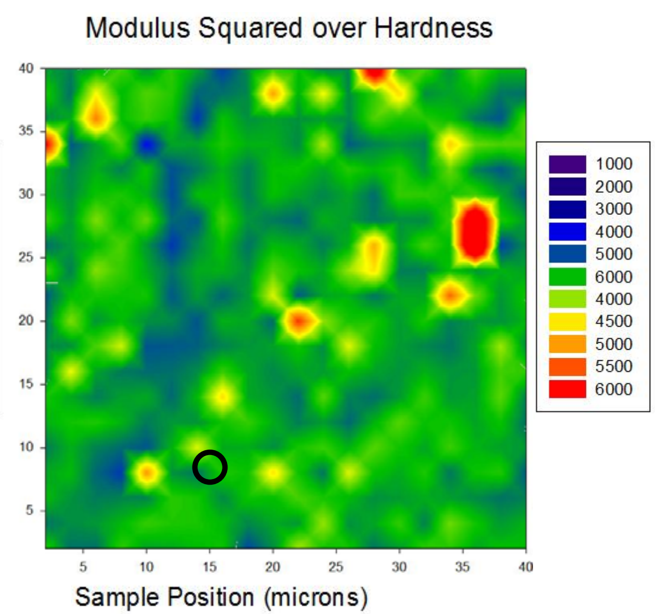












Mirror Finish

Selected Point:
Modulus: 353 GPa
Hardness: 35 GPa
Work: 0.39
E^2/H: 3591 GPa*

Sample Average:
Modulus: 367 GPa
Hardness: 39 GPa
Work: 0.35
E^2/H: 3499 GPa*

Conclusions

•Raman can be used to identify residual stress in machined silicon carbide by examining shift in TO (transverse optical) peak at 789 nm

Example:
mirror finish ave. compressive stress = 400 MPa
grit blast finish ave. compressive stress = 270 MPa
(using 514 nm laser in confocal setting)

•Changes in residual stress due to controlled indentation tests are detectable

•Complications exist because of variations in stress with depth

Example:
mirror finish ave. compressive stress = 400 MPa (514 nm confocal)
mirror finish ave. compressive stress = 310 MPa (514 nm normal)

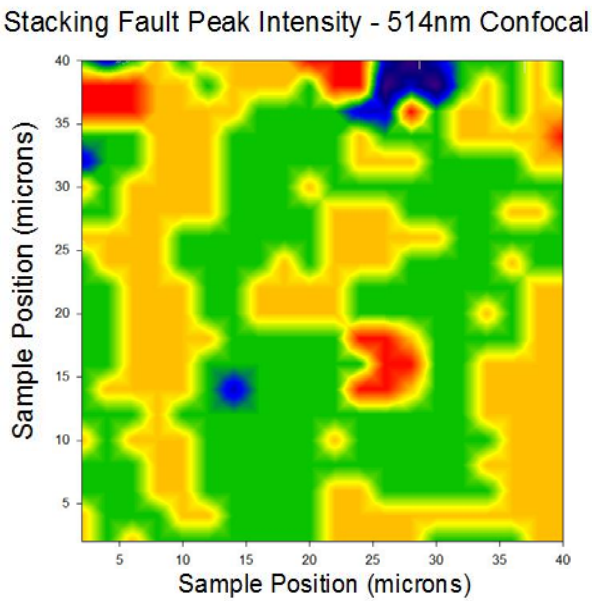
•Stacking faults and other phases (4H) further complicate analysis

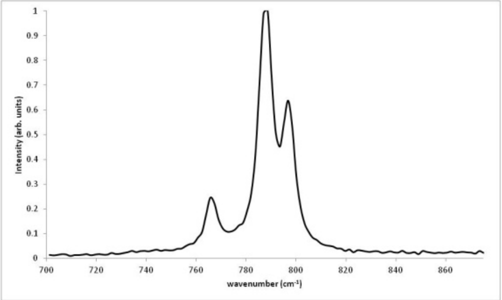
•Results of the analysis need to be correlated with performance criteria

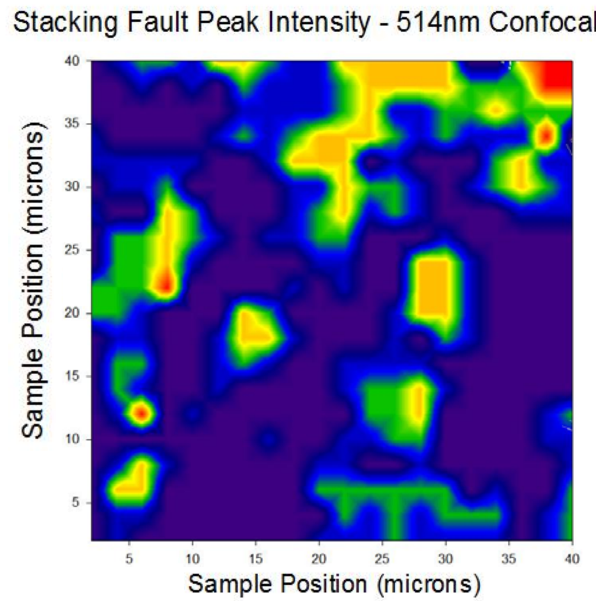
Future of project – *machining / silicon carbide*

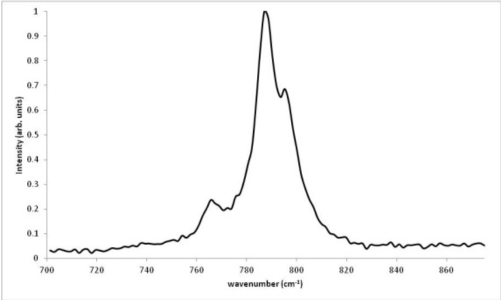
- Use confocal methods and surface enhanced methods to give better depth sensitivity.

- Correlate stress, stacking fault and phase analysis with performance data (e.g. fracture tests)



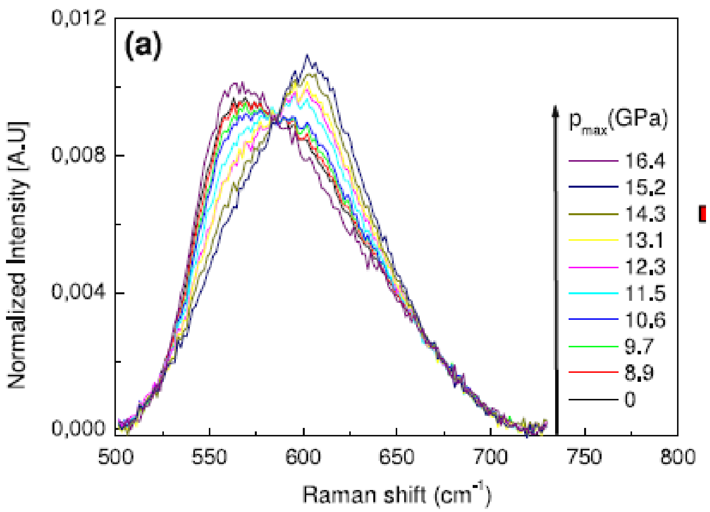






Future of project – *residual stress in ceramics*

- Extend to other ceramic systems: silica, silicates, glasses,….etc.



Silica – peak shift with hydrostatic pressure – Deschamps et al., J.Physics: Cond. Matter,v.23, 2010

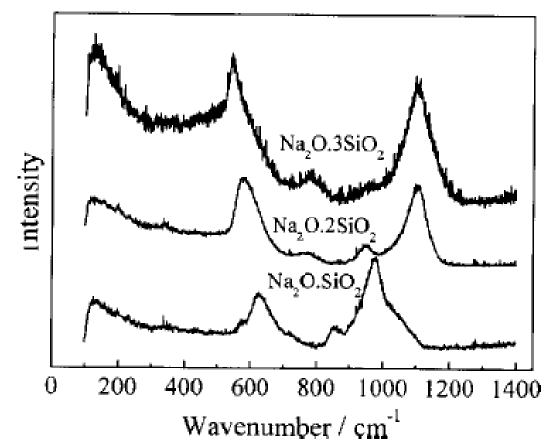
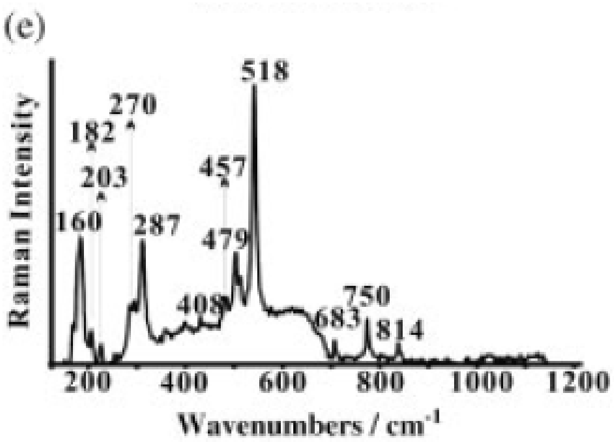


Figure 12. Raman spectra of binary sodium silicate glasses with different silica contents recorded under the same experimental conditions.

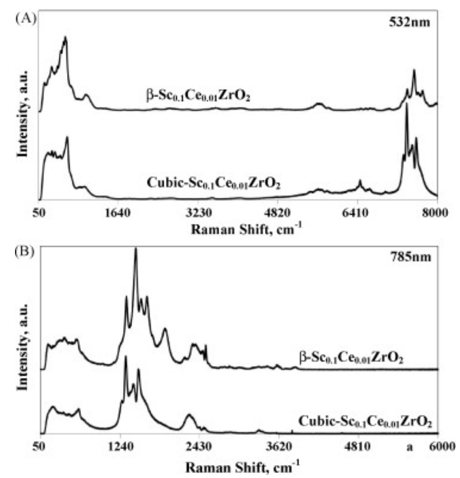
You et al., J.Raman Spec.,v.36, 2005

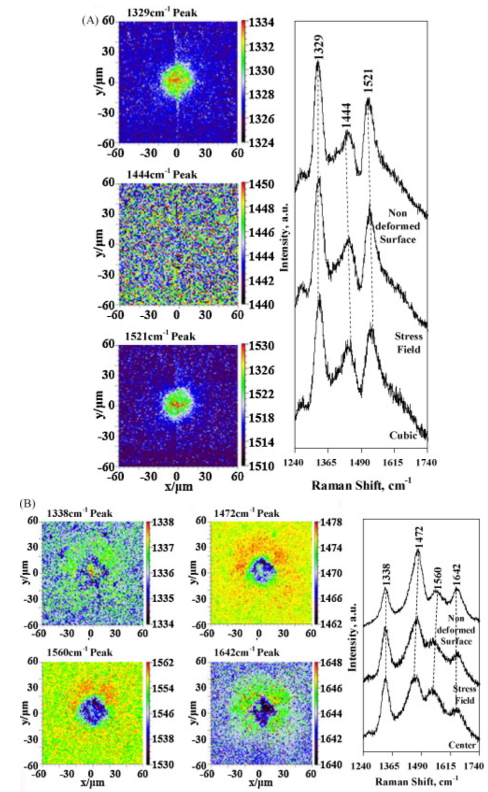
Future of project – *residual stress in ceramics*

- Examine residual stresses and phases in granular ceramics (specifically in individual grains and across grain boundaries): feldspar, porcelains,….etc.



Raman spectra of feldspar –
Liou et al., J.Raman Spec.,v.42, 2011





Raman and stresses in a ceramic ($Sc_{0.1}Ce_{0.01}ZrO_2$)
Lukich et al., J.Power Sources,v.195, 2010