Exceptional service in the national interest

Sandia National Laboratories

SAND2017-4646C

June 14, 2017

Full stress tensor measurement using fluorescence spectroscopy

SEM Annual Conference

S. J. Grutzik[1], S. P. Meserole[2], K T. Strong[3], T. E. Buchheit[3], M. C. Teague[3]

[1]Component Science and Mechanics (1556)

[2]Electronic, Optical, and Nano Materials (1816)

[3]Material Mechanics and Tribology (1851)

Sandia National Laboratories

Albuquerque, New Mexico USA

U.S. DEPARTMENT OF ENERGY

National Nuclear Security Administration

Stress measurement with photoluminscent spectroscopy

- Position of R1 and R2 stimulated emission peaks depend on stress, temperature, and doping concentration

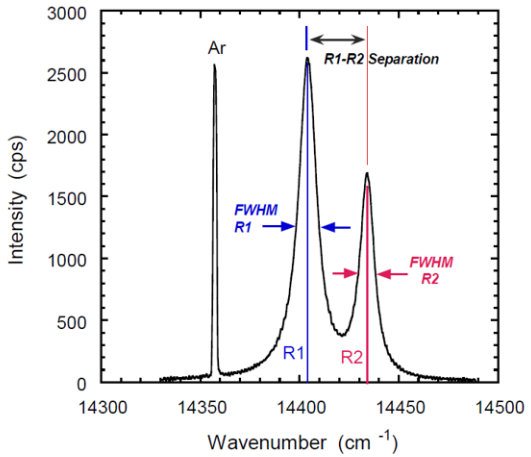
$$\begin{aligned}\nu^{(i)} &= \nu_0^{(i)} + \Delta\nu_\sigma^{(i)} + \Delta\nu_T^{(i)} + \Delta\nu_{Cr}^{(i)} \\ &= \nu_0^{(i)} + \Pi_{jk}^{(i)}\sigma_{jk} + \alpha^{(i)}(T - 298.8) + \beta^{(i)}C_{Cr}\end{aligned}$$

- In crystal basis, only diagonal elements of $\mathbf{\Pi}$ are nonzero

$$\mathbf{\Pi}^{(i)} = \begin{bmatrix} \Pi_{aa}^{(i)} & 0 & 0 \\ 0 & \Pi_{aa}^{(i)} & 0 \\ 0 & 0 & \Pi_{cc}^{(i)} \end{bmatrix}$$

- Early work used a diffuse laser spot, sampling many grains, and an isotropy assumption

$$\begin{aligned}\Delta\nu^{(i)} &= \frac{1}{3}\left(\Pi_{11}^{(i)} + \Pi_{22}^{(i)} + \Pi_{33}^{(i)}\right)(\sigma_{11} + \sigma_{22} + \sigma_{33}) \\ &= \Pi_M \sigma_M\end{aligned}$$



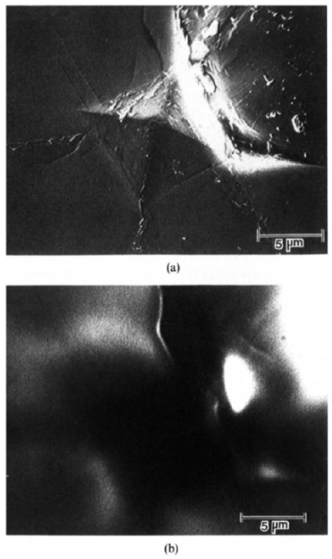


Fig. 1. Secondary electron micrograph (a) and corresponding CL image (b) of a 200 g Vickers hardness indentation in (1120) surface of sapphire. Note that the center of the indent is non-luminescent.

Single crystal measurement

- If single crystal, isotropy assumption no longer applies
- Michaels and Cook[1] showed that because of (approximately) hexagonal symmetry, peak shift can be expressed as

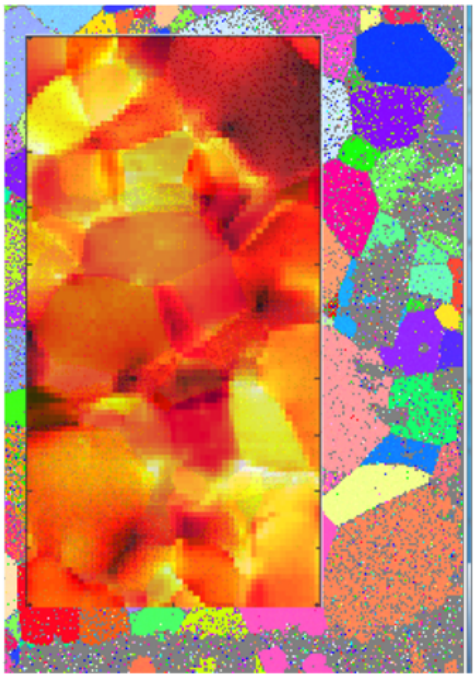
$$\begin{aligned}\Delta\nu^{(i)} &= \Pi_{11}^{(i)}\left(\sigma_{11}+\sigma_{22}\right)+\Pi_{33}^{(i)}\sigma_{33}\\ &= \left(2\Pi_{11}^{(i)}+\Pi_{33}^{(i)}\right)\left(\sigma_{11}+\sigma_{22}+\sigma_{33}\right)+\left(\Pi_{33}^{(i)}-\Pi_{11}^{(I)}\right)\left(2\sigma_{33}-\sigma_{11}-\sigma_{22}\right)\\ &= \Pi_{M}^{(i)}\sigma_{M}+\Pi_{S}^{(i)}\sigma_{S}\end{aligned}$$

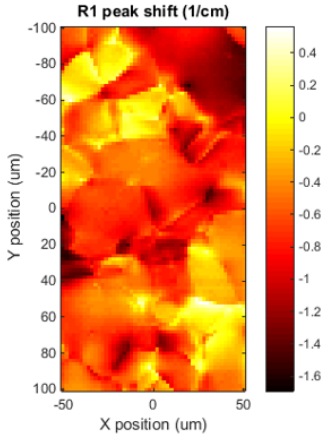
  where $\sigma_M = \left(\sigma_{11}+\sigma_{22}+\sigma_{33}\right)/3$ and $\sigma_S = \left(2\sigma_{33}-\sigma_{11}-\sigma_{22}\right)/3 = \sigma_{33}-\sigma_M$
- σ_M is mean stress and invariant
- σ_S is like a deviatoric stress with respect to the c axis and *not* invariant
- So we can use both R1 and R2 peak shifts to solve for σ_M and σ_S

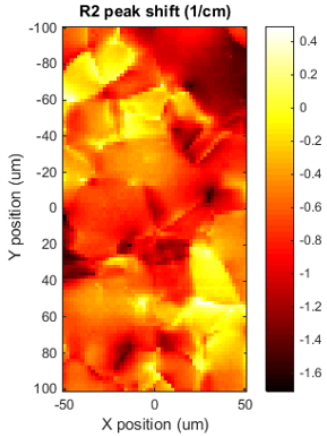
$$\begin{bmatrix}\Delta\nu^{(1)}\\ \Delta\nu^{(2)}\end{bmatrix} = \begin{bmatrix}\Pi_{M}^{(1)} & \Pi_{S}^{(1)}\\ \Pi_{M}^{(2)} & \Pi_{S}^{(2)}\end{bmatrix}\begin{bmatrix}\sigma_M\\ \sigma_S\end{bmatrix}$$

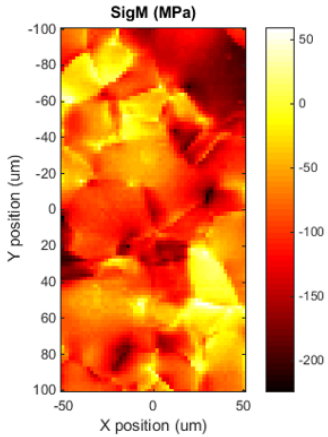
Polycrystal stress measurement

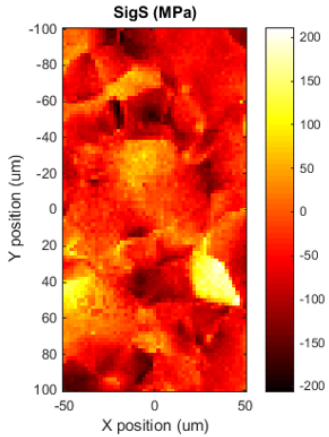
- Using a focused laser spot and an idealized specimen, we can resolve stresses to 2 µm length scale
- In order to interpret σ_M, we must know grain orientation











Proposed full stress tensor solution

- PLS is a surface measurement $\rightarrow$ traction free conditions apply
- With 2 peak shifts, $\boldsymbol{\sigma} \cdot \mathbf{n} = \mathbf{0}$, we have 5 constraints on 6 stress components. Strain compatibility could provide the final constraint.
- Instead, push problem to $\mathbf{u}$ space and solve for u_1 and u_2 given $\nu^{(1)}$ and $\nu^{(2)}$,

$$\begin{bmatrix} \Delta\nu^{(1)} \\ \Delta\nu^{(2)} \end{bmatrix} = \begin{bmatrix} \Pi_{11}^{(1)} & \Pi_{22}^{(1)} & \Pi_{12}^{(1)} \\ \Pi_{11}^{(2)} & \Pi_{22}^{(2)} & \Pi_{12}^{(2)} \end{bmatrix} \begin{bmatrix} \tilde{C}_{1111} & \tilde{C}_{1122} & \tilde{C}_{1112} \\ \tilde{C}_{1122} & \tilde{C}_{2222} & \tilde{C}_{1222} \\ \tilde{C}_{1112} & \tilde{C}_{1222} & \tilde{C}_{1212} \end{bmatrix} \begin{bmatrix} \frac{\partial}{\partial x_1} & 0 \\ 0 & \frac{\partial}{\partial x_2} \\ \frac{1}{2}\frac{\partial}{\partial x_2} & \frac{1}{2}\frac{\partial}{\partial x_1} \end{bmatrix} \begin{bmatrix} u_1 \\ u_2 \end{bmatrix}$$

$$\boldsymbol{\nu} = \boldsymbol{\Pi}\tilde{\mathbf{C}}\mathbf{D}\mathbf{u}\,,$$

- Because x_1, x_2, x_3 does not in general align with crystal a, b, c, $\Pi_{12}^{(i)} \neq 0$
- $\tilde{\mathbf{C}}$ is the traction-free, in-plane stiffness matrix
- This a surface analysis of a 3-D problem $\rightarrow$ stress equilibrium cannot be used
- The above equation is exact (until the derivatives are discretized)
- $\Delta\boldsymbol{\nu}$ effectively gives a linear combination of derivatives of $\mathbf{u}$ so we integrate to get $\boldsymbol{\epsilon}$ and $\boldsymbol{\sigma}$

$\boldsymbol{\Pi}$ and $\tilde{\mathbf{C}}$ calculation

- $\boldsymbol{\nu} = \boldsymbol{\Pi} : \boldsymbol{\sigma}$ and $\boldsymbol{\nu}$ is frame invariant $\rightarrow \boldsymbol{\Pi}$ is a tensor
- Determine $\tilde{\mathbf{C}}$ by partitioning $\mathbf{C}$ into in-plane and out-of-plane parts

$$\begin{bmatrix} \boldsymbol{\sigma}^{\mathrm{i}} \\ \boldsymbol{\sigma}^{\mathrm{o}} \end{bmatrix} = \begin{bmatrix} \mathbf{C}^{\mathrm{ii}} & \mathbf{C}^{\mathrm{io}} \\ \left(\mathbf{C}^{\mathrm{io}}\right)^{\mathsf{T}} & \mathbf{C}^{\mathrm{oo}} \end{bmatrix} \begin{bmatrix} \boldsymbol{\epsilon}^{\mathrm{i}} \\ \boldsymbol{\epsilon}^{\mathrm{o}} \end{bmatrix}$$

$$\begin{bmatrix} \boldsymbol{\epsilon}^{\mathrm{i}} \\ \boldsymbol{\epsilon}^{\mathrm{o}} \end{bmatrix} = \begin{bmatrix} \mathbf{S}^{\mathrm{ii}} & \mathbf{S}^{\mathrm{io}} \\ \left(\mathbf{S}^{\mathrm{io}}\right)^{\mathsf{T}} & \mathbf{S}^{\mathrm{oo}} \end{bmatrix} \begin{bmatrix} \boldsymbol{\sigma}^{\mathrm{i}} \\ \boldsymbol{\sigma}^{\mathrm{o}} \end{bmatrix}$$

- For example,

$$\mathbf{C}^{\mathrm{ii}} = \begin{bmatrix} C_{1111} & C_{1122} & C_{1112} \\ C_{1122} & C_{2222} & C_{1222} \\ C_{1112} & C_{1222} & C_{1212} \end{bmatrix}$$

- Under traction-free conditions, $\boldsymbol{\sigma}^{\mathrm{o}} = \mathbf{0}$, so $\tilde{\mathbf{C}}$ is given by either

$$\tilde{\mathbf{C}} = \left(\mathbf{S}^{\mathrm{ii}}\right)^{-1}$$

$$\tilde{\mathbf{C}} = \mathbf{C}^{\mathrm{ii}} - \mathbf{C}^{\mathrm{io}} \left(\mathbf{C}^{\mathrm{oo}}\right)^{-1} \left(\mathbf{C}^{\mathrm{io}}\right)^{\mathsf{T}}$$

- Using block matrix inversion, these forms are equivalent

$$\begin{bmatrix} \mathbf{a} & \mathbf{b} \\ \mathbf{b}^{\mathsf{T}} & \mathbf{c} \end{bmatrix}^{-1} = \begin{bmatrix} \left(\mathbf{a} - \mathbf{b}\mathbf{c}^{-1}\mathbf{b}^{\mathsf{T}}\right)^{-1} & -\left(\mathbf{a} - \mathbf{b}\mathbf{c}^{-1}\mathbf{b}^{\mathsf{T}}\right)^{-1} \mathbf{b}\mathbf{c}^{-1} \\ -\mathbf{c}^{-1}\mathbf{b}^{\mathsf{T}} \left(\mathbf{a} - \mathbf{b}\mathbf{c}^{-1}\mathbf{b}^{\mathsf{T}}\right)^{-1} & \mathbf{c}^{-1}\mathbf{b}^{\mathsf{T}} \left(\mathbf{a} - \mathbf{b}\mathbf{c}^{-1}\mathbf{b}^{\mathsf{T}}\right)^{-1} \mathbf{b}\mathbf{c}^{-1} + \mathbf{c}^{-1} \end{bmatrix}$$

Numerical strategy: total regularization

- Using central difference differentiation the resulting linear problem is extremely ill-conditioned
- Strain at a point is a function of displacement at only neighboring points $\rightarrow$ "letter" points and "number" points are only coupled at edges

a	1	b	2	c	3
4	d	5	e	6	f
g	7	h	8	i	9

- This results in large, oscillatory spurious displacements in solution
- Use total regularization to penalize these spurious solutions and minimize the functional F to find $\eta = f'$

$$F(\eta) = \alpha R(\eta) - \left| \int_0^{\eta} \eta(\xi)\, d\xi - f \right|_2$$

- To keep displacements small, we set $R(\boldsymbol{\epsilon}) = |\mathbf{Du}|_2 = |\boldsymbol{\epsilon}|_2$ and minimize

$$F(\mathbf{u}) = \alpha\, |\mathbf{Du}|_2 + \left| \boldsymbol{\Pi}\tilde{\mathbf{C}}\mathbf{Du} - \Delta\boldsymbol{\nu} \right|_2 .$$

Numerical strategy: Least squares differentiation

Sandia National Laboratories

- Peak shifts measurements are noisy from point to point
- Standard finite difference interpolates a quadratic polynomial between 3 points
- We fit a p-term 2-d polynomial to a $n \times m$ grid of nearest points

$$P(x_1, x_2) = a_0 + a_1 x + a_2 y + a_3 xy + a_4 x^2 + a_5 y^2 + a_6 x^2 y + a_7 x y^2 + a_8 x^2 y^2 + \ldots$$

- First x_1 and x_2 partial derivatives are given by a_1 and a_2 respectively
- The grid is centered for interior points and skewed toward the center for edge points.
- A 5×5 grid with a 9-term polynomial and a 7×7 grid with a 25 term polynomial have similar performance

Least squares differentiation example

- Consider the point just to the right of the upper left grid point. The interpolated, 3 point central difference coefficients for the first x_1 derivative would be:

$$\begin{array}{ccccc} \cdot & \times & \cdot & \cdot & \cdot \\ \cdot & \cdot & \cdot & \cdot & \cdot \\ \cdot & \cdot & \cdot & \cdot & \cdot \\ \cdot & \cdot & \cdot & \cdot & \cdot \\ \cdot & \cdot & \cdot & \cdot & \cdot \\ \vdots & & & \ddots & \end{array} \quad \cdots \qquad \begin{array}{ccccc} \text{-1/2} & 0 & 1/2 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 \\ \vdots & & & & \ddots \end{array} \quad \cdots$$

- The least squares finite difference coefficients with a 9 term polynomial and a 5×5 grid would be:

$$\begin{array}{ccccc} -\frac{527}{1225} & \frac{93}{2450} & \frac{62}{245} & \frac{527}{1225} & -\frac{93}{2450} \\ -\frac{153}{1225} & \frac{27}{2450} & \frac{18}{245} & \frac{153}{1225} & -\frac{27}{2450} \\ \frac{51}{1225} & -\frac{9}{2450} & -\frac{6}{245} & -\frac{51}{1225} & \frac{9}{2450} \\ \frac{17}{245} & -\frac{3}{490} & -\frac{2}{49} & -\frac{17}{245} & \frac{3}{490} \\ -\frac{51}{1225} & \frac{9}{2450} & \frac{6}{245} & \frac{51}{1225} & -\frac{9}{2450} \\ 0 & 0 & 0 & 0 & 0 \\ \vdots & & & & \end{array} \quad \begin{array}{c} 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ \ddots \end{array} \quad \cdots$$

Error analysis

- $\mathbf{\Pi}$ and $\tilde{\mathbf{C}}$ depend on grain orientation, so an error in orientation will cause an error in the solution
- A single orientation error causes a rank-3 update to $\mathbf{K} = \mathbf{\Pi C D}$

$$\left(\mathbf{K} + \mathbf{U}\bar{\mathbf{C}}\mathbf{V}\right)^{-1} = \mathbf{K}^{-1} - \mathbf{K}^{-1}\mathbf{U}\left(\bar{\mathbf{C}}^{-1} + \mathbf{V}\mathbf{K}^{-1}\mathbf{U}\right)^{-1}\mathbf{V}\mathbf{K}^{-1}$$

where and $\mathbf{U}$ and $\mathbf{V}$ are

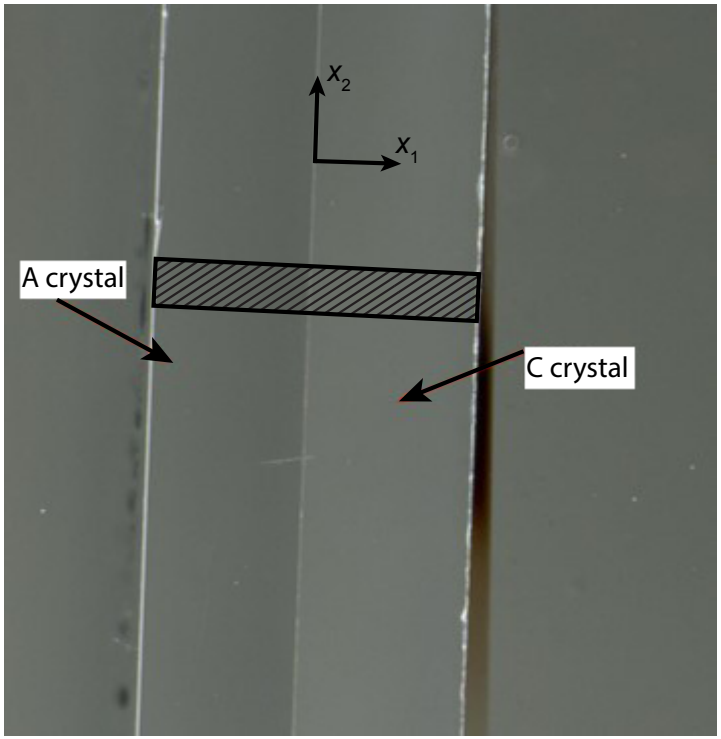
$$\mathbf{U} = \begin{bmatrix}\mathbf{0}_{2\times3} & \mathbf{0}_{2\times3} & \cdots & \bar{\mathbf{\Pi}} \cdots & \mathbf{0}_{2\times3} & \mathbf{0}_{2\times3}\end{bmatrix}^{\mathsf{T}}$$

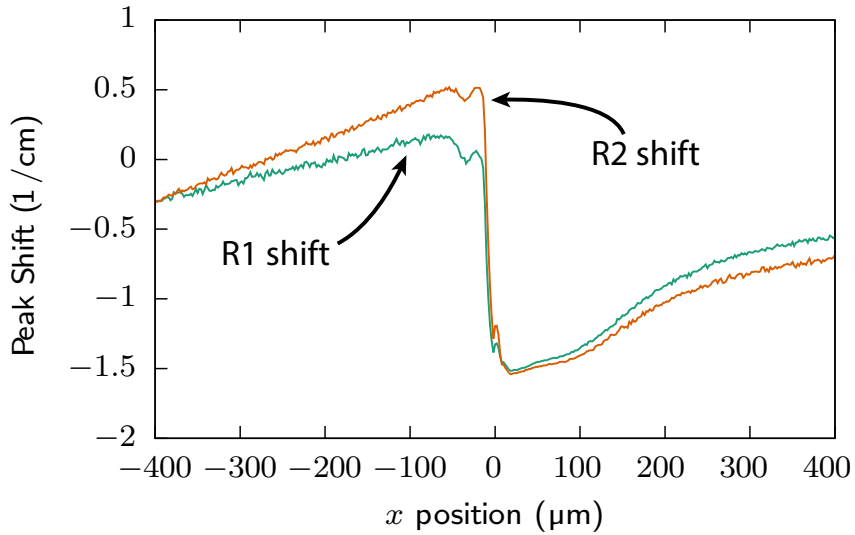
$$\mathbf{V} = \begin{bmatrix}\mathbf{0}_{3\times3} & \mathbf{0}_{3\times3} & \cdots & \mathbf{I}_3 \cdots & \mathbf{0}_{3\times3} & \mathbf{0}_{3\times3}\end{bmatrix}\mathbf{D}$$

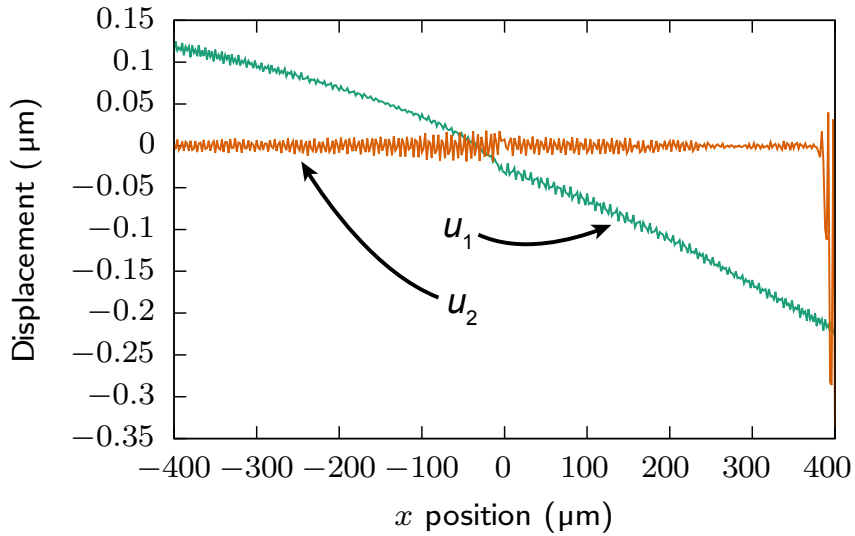
- $\bar{\mathbf{\Pi}}$ and $\bar{\mathbf{C}}$ are errors in $\mathbf{\Pi}$ and $\tilde{\mathbf{C}}$ due to an error in material orientation of the i-th spectroscopy measurement point.
- $\bar{\mathbf{\Pi}}$ and $\mathbf{I}_3$ occur in the i-th block matrix position of $\mathbf{U}$ and the first factor of $\mathbf{V}$ respectively. $\mathbf{V}$ is the i-th set of 3 rows of $\mathbf{D}$.
- $\mathbf{K}^{-1}\mathbf{U}$ then selects the i-th set of 3 rows of $\mathbf{K}^{-1}$ and $\mathbf{V}\mathbf{K}^{-1}$ selects n^2 sets of 3 columns of $\mathbf{K}^{-1}$ corresponding to the $\mathrm{n} \times \mathrm{n}$ set of points included in the Savitzky-Golay differentiator of point i.
- Computing a solution $\bar{\mathbf{u}} = \left(\mathbf{K} + \mathbf{U}\bar{\mathbf{C}}\mathbf{V}\right)^{-1}(\Delta\boldsymbol{\nu})$ then results in errors at only the i-th point and the points coupled to it by the Savitzky-Golay differentiator.

Bicrystal example

- For a simple proof-of-concept example, a bicrystal made from two single sapphire crystals bonded at 1600 °C with a glass frit layer
- Peak shifts measured in depicted area and results are averaged parallel to interface

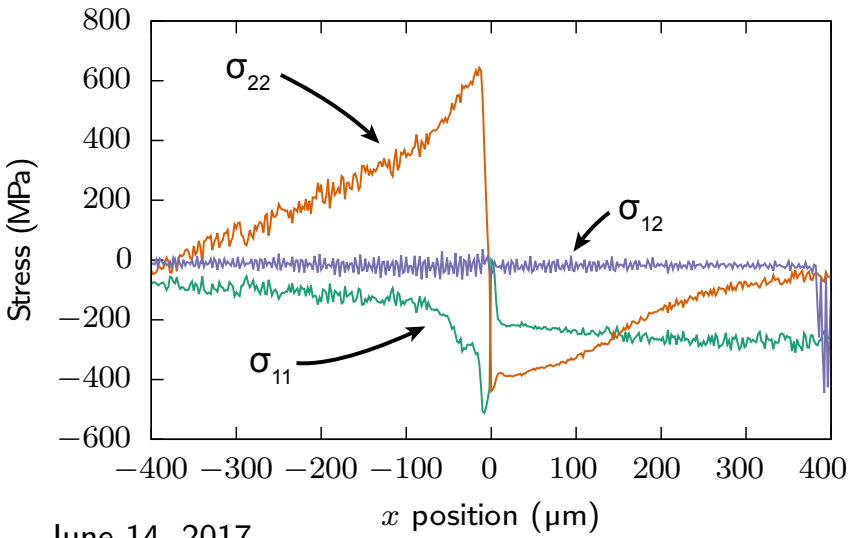


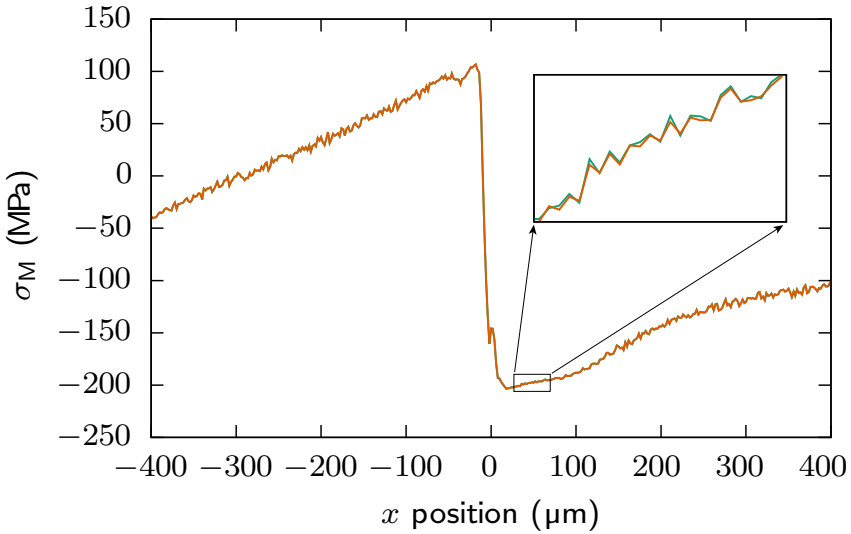


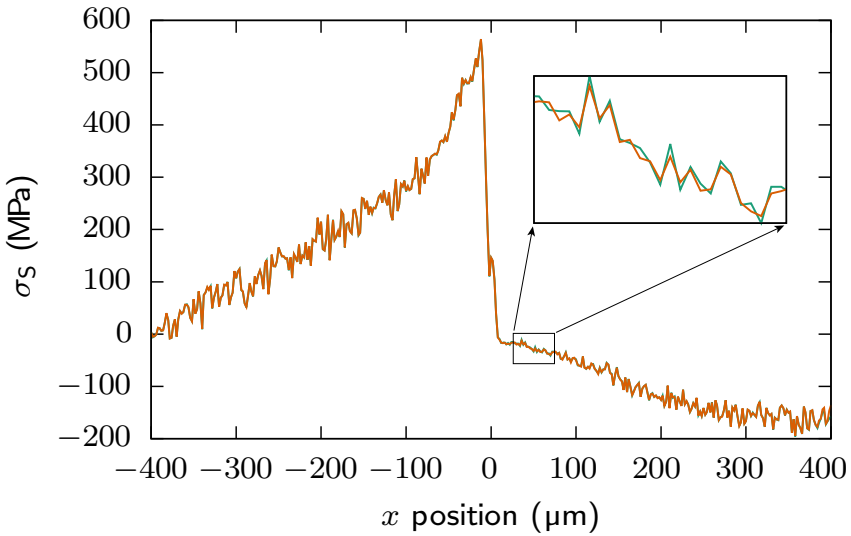


Bicrystal example

- Stresses can be calculated from solution using $\boldsymbol{\sigma} = \tilde{\mathbf{C}}\mathbf{D}\mathbf{u}$
- As an accuracy check, stresses are rotated to crystal basis where σ_M and σ_S are calculated and compared to the direct method using Π_M and Π_S
- Solution noise is from peak shift measurements, not numerical accuracy

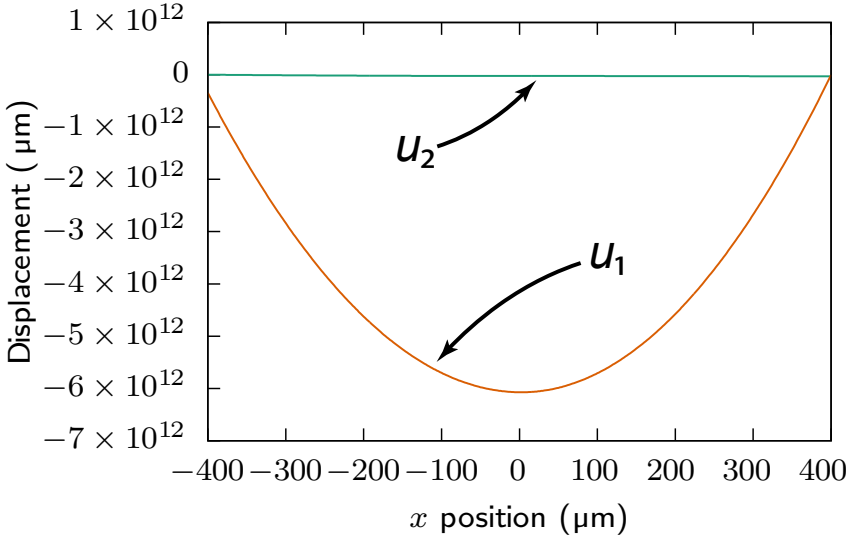




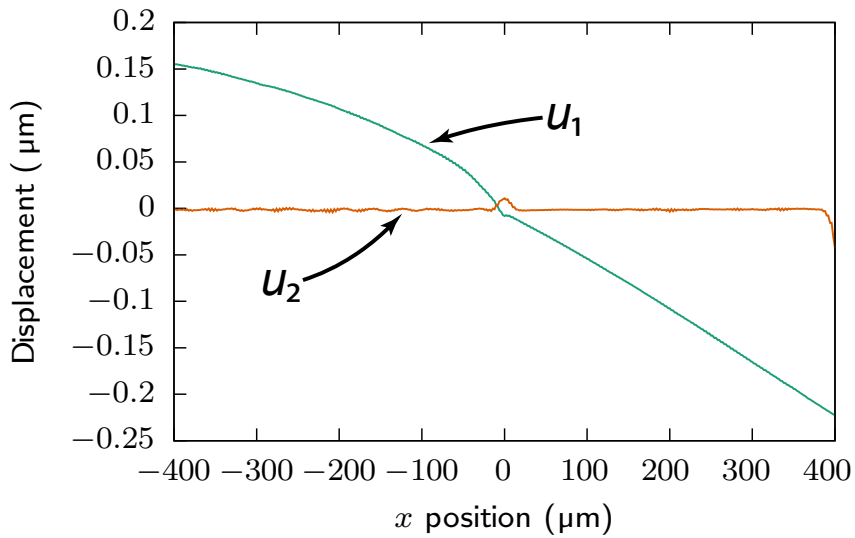


Other solution methods

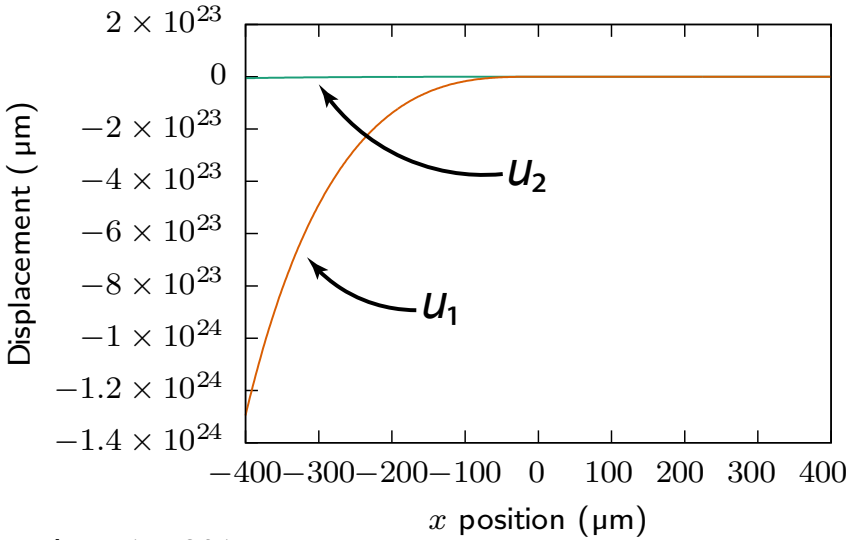
Basic linear solve



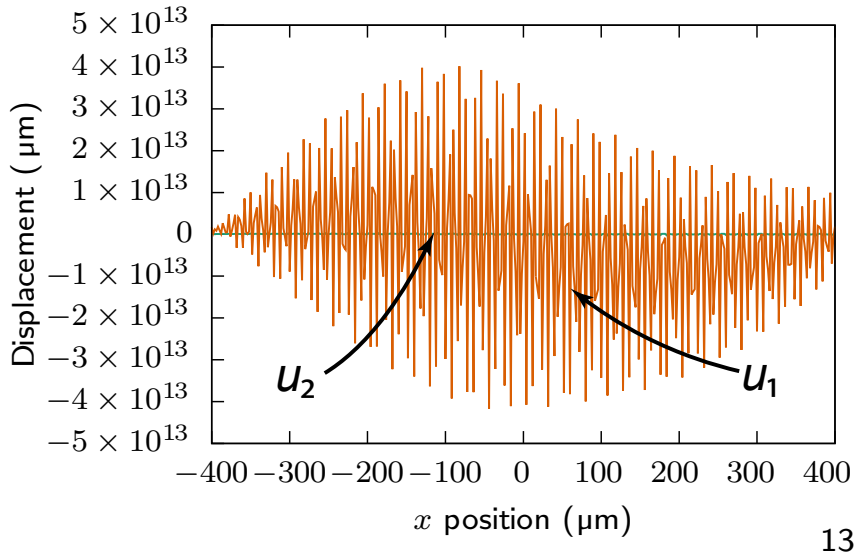
Total regularization only



Forward difference

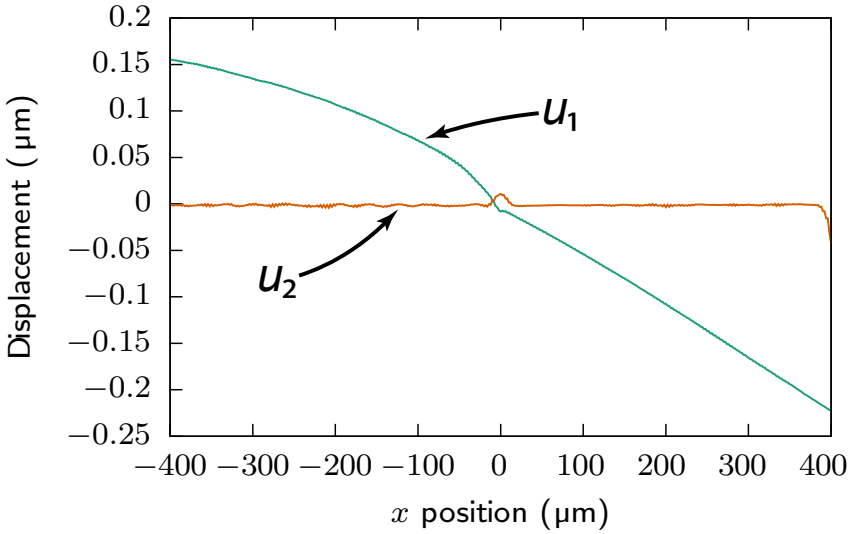


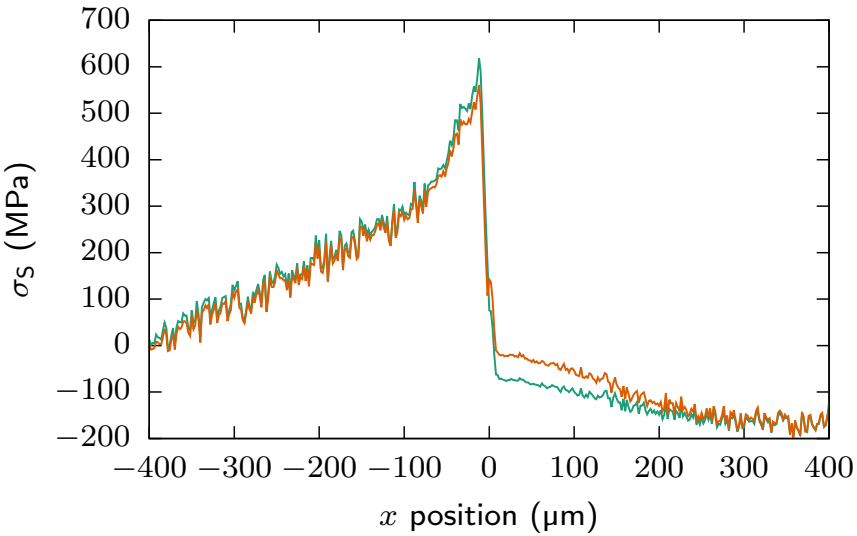
Least squares differentiation only



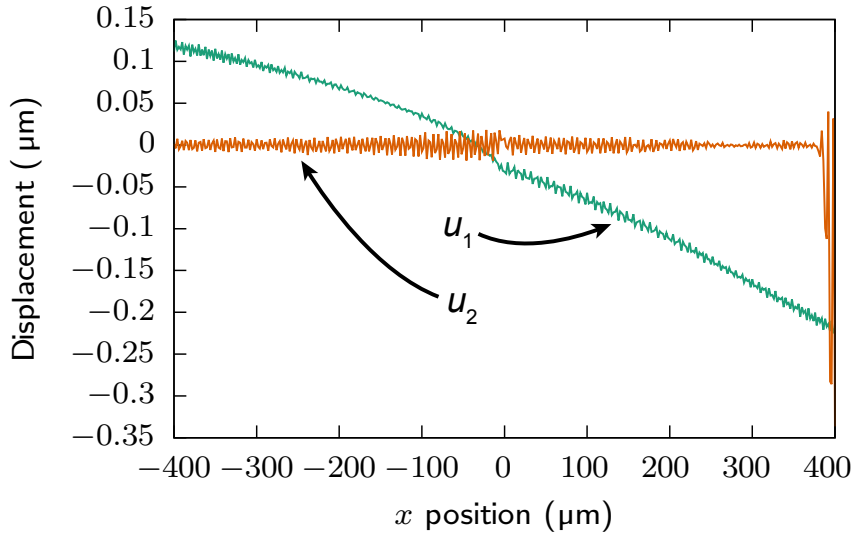
Regularization only

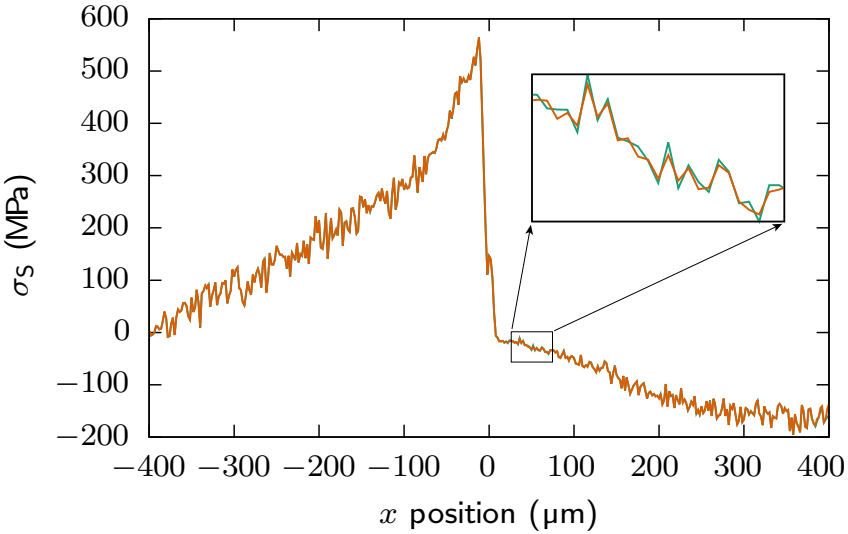
Using only regularization





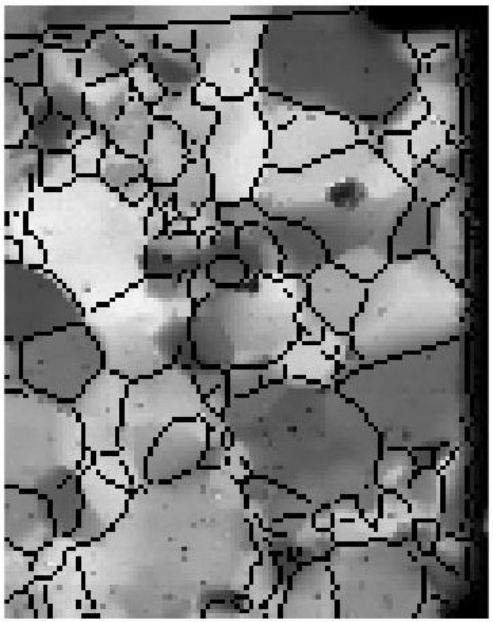
Regularization and least squares differentiation



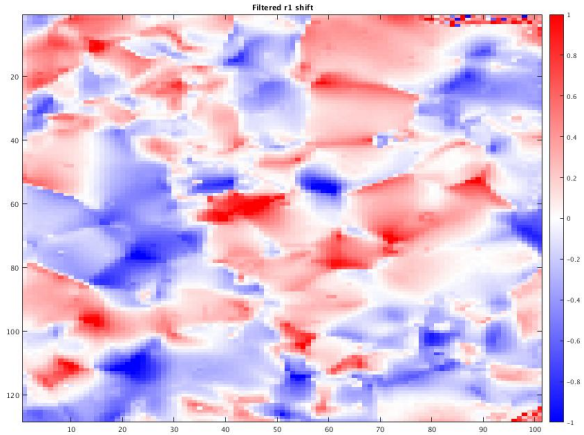


Polycrystal example

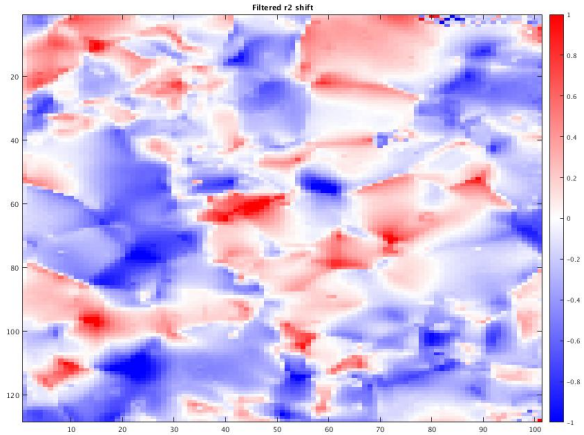
- $Al_2O_3 \cdot Cr$ polycrystals with 20 µm to 60 µm grains and 0.025 % Cr by weight
- Specimen fabricated to have low void and glassy phase density
- Use PLS intensity map to align EBSD orientation map



R1 peak shift map



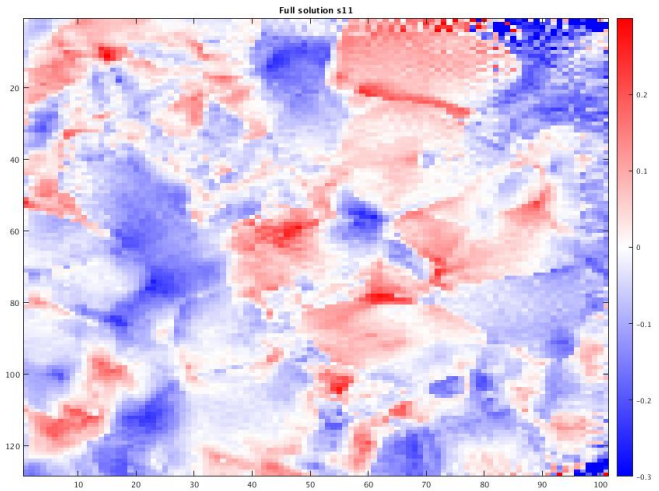
R2 peak shift map



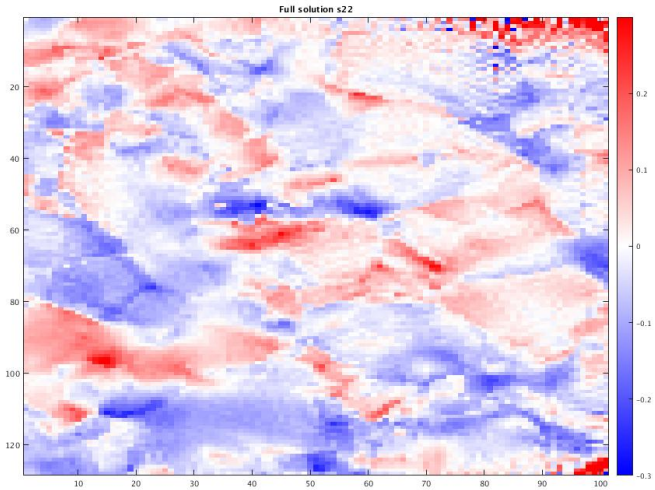
Polycrystal example

- When calculating stress, more residual noise in shear map
- Errors are visible near grain boundaries (as expected)

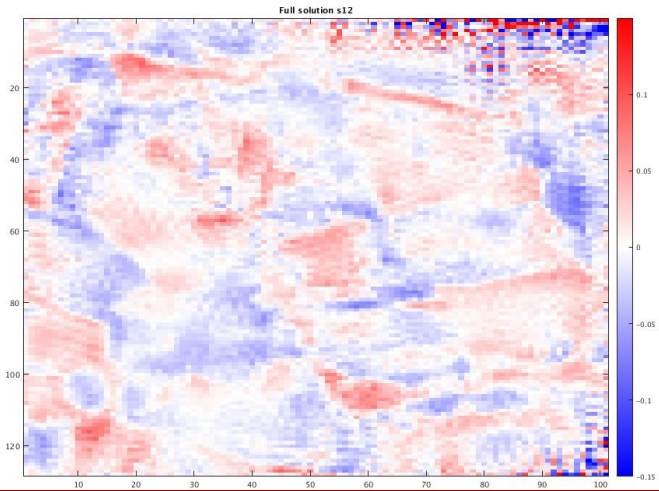
σ_{11} map



σ_{22} map



σ_{12} map



Other things I tried

- Solving in $\boldsymbol{\epsilon}$ space with strain compatibility $\rightarrow$ What BCs to use with $\epsilon_{11,22} + \epsilon_{22,11} = 2\epsilon_{12,12}$? What happens to these BCs when solving in $\mathbf{u}$ space?
- Use a pseudoinverse in $\boldsymbol{\epsilon}$ to find solution space and pick that which minimizes $\boldsymbol{\epsilon}$
- Principal component analysis
- Preconditioning
- Regularization is a linear optimization, can we solve that directly?

$$\min_{\mathbf{u}} \left[\alpha \mathbf{u}^{\mathsf{T}} \mathbf{D}^{\mathsf{T}} \mathbf{D} \mathbf{u} + \left(\mathbf{\Pi C D u} - \Delta \boldsymbol{\nu} \right)^{\mathsf{T}} \left(\mathbf{\Pi C D u} - \Delta \boldsymbol{\nu} \right) \right]$$

$$\mathbf{u} = \left[\mathbf{D}^{\mathsf{T}} \left(\alpha \mathbf{I} + \mathbf{C} \mathbf{\Pi}^{\mathsf{T}} \mathbf{\Pi C} \right) \mathbf{D} \right]^{-1} \mathbf{D}^{\mathsf{T}} \mathbf{C} \mathbf{\Pi}^{\mathsf{T}} \left(\Delta \boldsymbol{\nu} \right)$$

- Is the problem physically ill-conditioned?

$$\Delta \boldsymbol{\nu} = \lambda \mathbf{u} = \mathbf{\Pi C} \left[\nabla \mathbf{u} + \left(\nabla \mathbf{u} \right)^{\mathsf{T}} \right]$$

$$\mathbf{u} = \mathbf{b} \cdot \exp \left(\mathbf{a} \otimes \mathbf{x} \right)$$

$\rightarrow$ For an area with uniform properties, only two eigenmodes

Final thoughts

Sandia National Laboratories

- We have demonstrated a method to use traction-free conditions to obtain full stress tensor from two peak shift measurements
- First order central difference method is numerically unstable and input data is noisy
- We use regularization to avoid these issues, but now uniqueness is an issue. Is the regularized solution the correct solution?
- Regularization turns a linear solve into an optimization, currently implemented in serial in Matlab $\rightarrow$ very slow
- Is there a better approach to dealing with the finite difference numerical instability?
- Is this problem well-posed?
  $\rightarrow$ From the point of view of integrating $\Delta\boldsymbol{\nu}$ to get $\mathbf{u}$, two peak shifts are not enough for $\mathfrak{u}_1(\mathfrak{x}_1, \mathfrak{x}_2)$ and $\mathfrak{u}_2(\mathfrak{x}_1, \mathfrak{x}_2)$
  $\rightarrow$ $\boldsymbol{\Pi}\mathbf{CD}$ is very ill-conditioned, but *not* singular
  $\rightarrow$ For a small problem where interior points are more coupled to edges, problem is well-behaved

Questions?

Sandia National Laboratories

U.S. DEPARTMENT OF ENERGY

Honeywell

THE POWER OF **CONNECTED**

National Nuclear Security Administration

Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.