

# Residual Stresses in SOFC Materials and Components

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Solid Oxide Fuel Cell Workshop  
Pittsburgh, PA, July 2015

# Acknowledgments

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This work was sponsored by the US Department of Energy, Office of Fossil Energy, SECA Core Technology Program at ORNL.

We appreciate guidance and support from NETL program managers Rin Burke and Briggs White.



# Outline

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- Background
- Techniques for measuring Residual Stresses
  - Diffraction (x-rays, neutrons)
  - Raman Spectroscopy
  - Piezospectroscopy
  - Digital image correlation
- Summary and Future Work

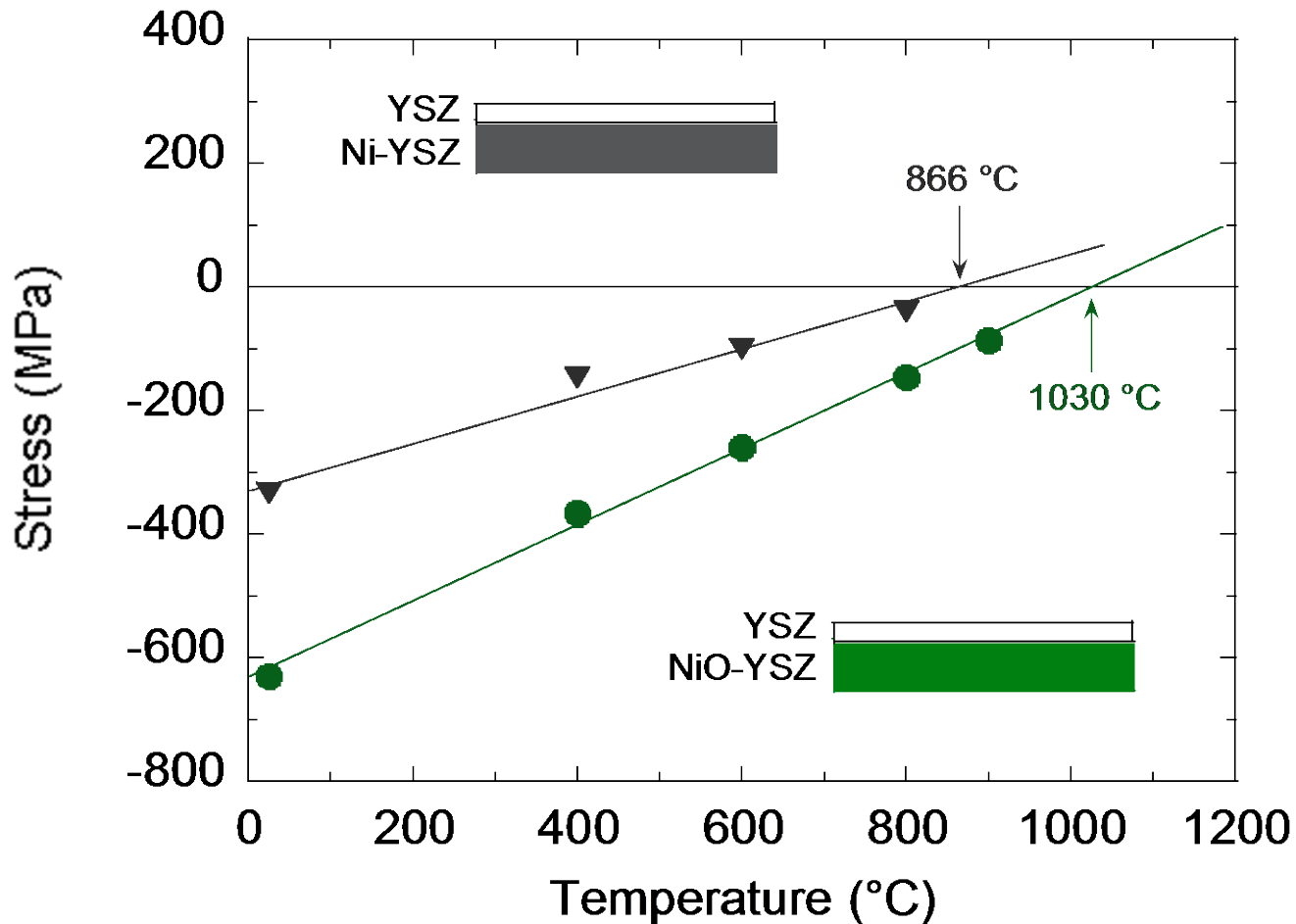
# Background

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- The reliability of materials and components for SOFCs is determined by their state of stress, which includes contributions from:
  - residual stresses

# Residual and “Reduction” Stresses (X-ray diffraction)

800°C Reduction in 4%H<sub>2</sub>-96%Ar



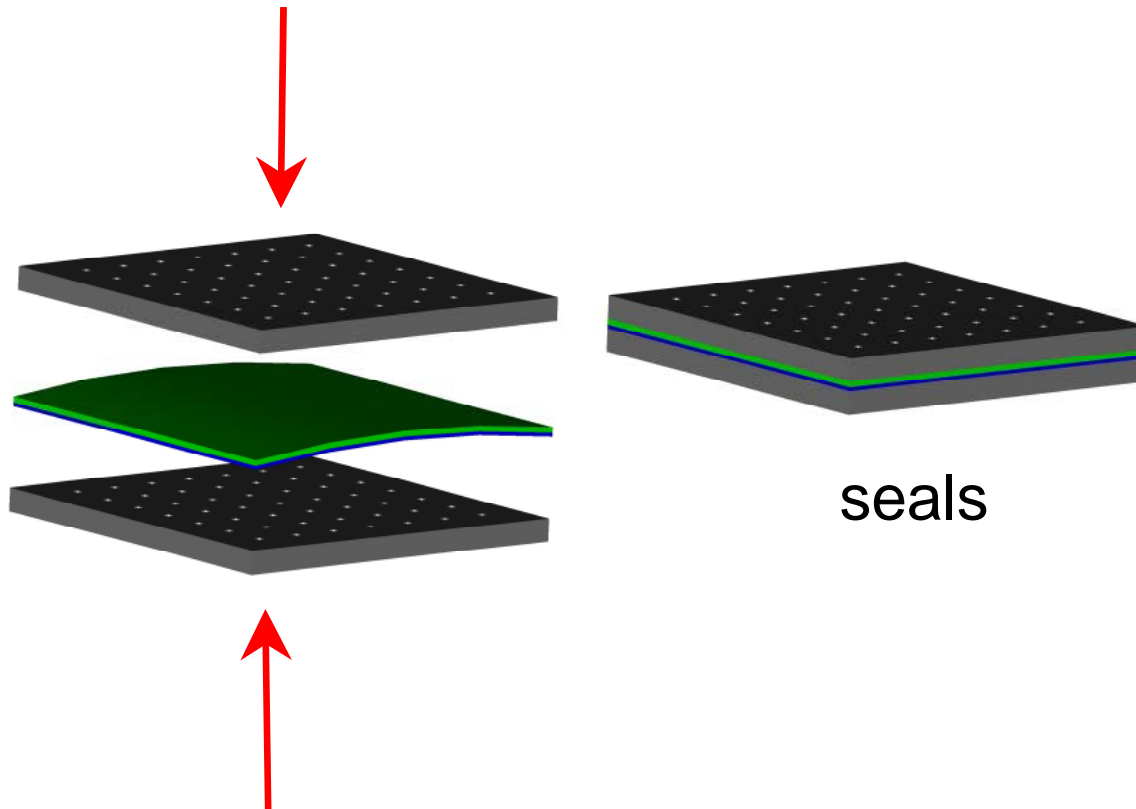
# Background

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- The reliability of materials and components for SOFCs is determined by their state of stress, which includes contributions from:
  - residual stresses
  - **assembly stresses**

# Reliability of SOFCs (**Assembly Stresses**)

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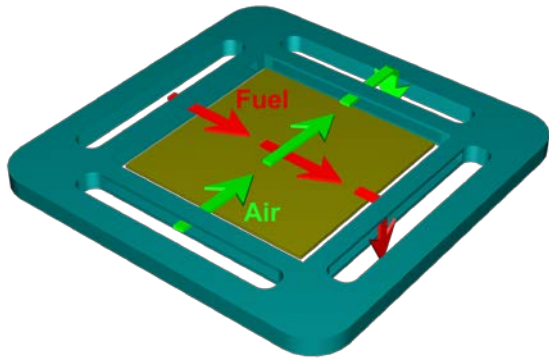
# Background

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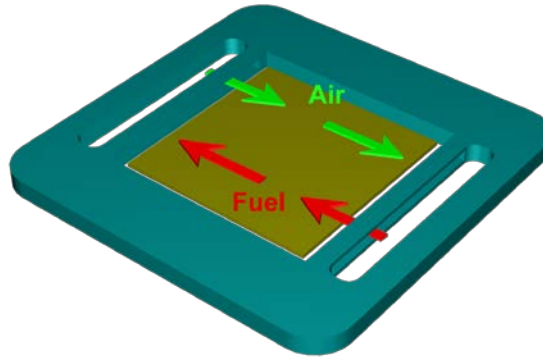
- The reliability of materials and components for SOFCs is determined by their state of stress, which includes contributions from:
  - residual stresses
  - assembly stresses
  - **operational stresses**



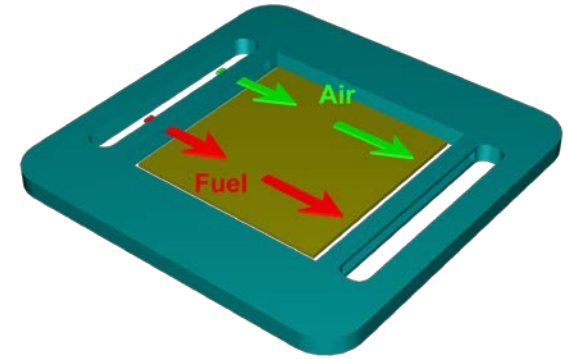
# Reliability of SOFCs (Operation-induced Stresses)



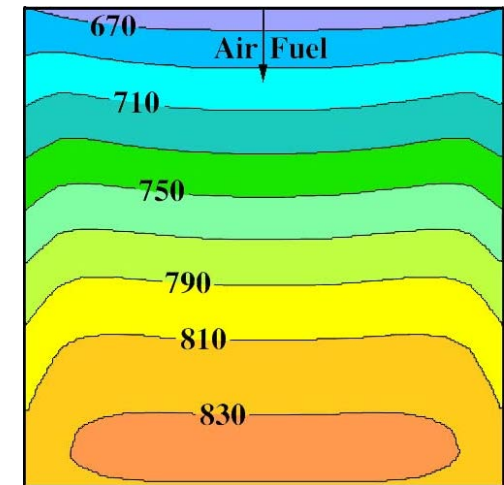
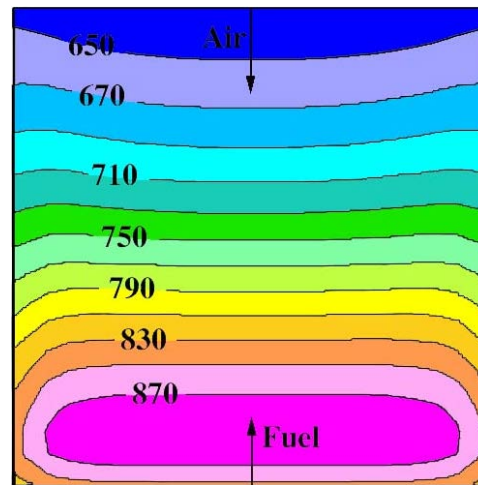
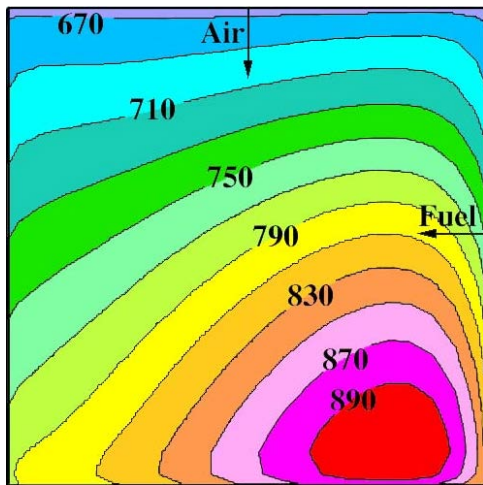
Cross-Flow



Counter-Flow



Co-Flow



# Background

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- Residual stresses arise from differences in the thermoelastic properties of materials when these are constrained to expand-contract as a function of temperature.
- The objective of this exercise is to identify and apply techniques to quantify residual stresses in SOFC materials and components and studying how these stresses evolve as a function of time and operational history.

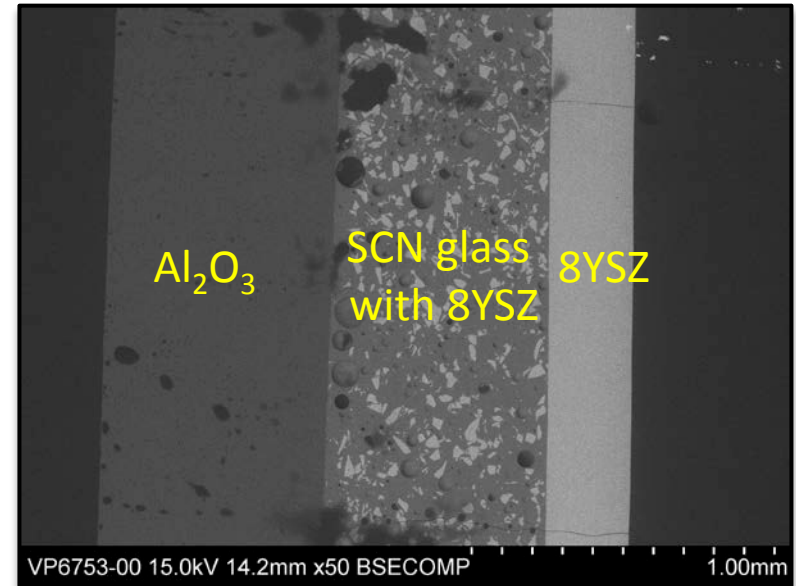
# Techniques for Measuring Residual Stresses

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- Diffraction (x-rays, neutrons)
- Raman Spectroscopy
- Piezospectroscopy
- Digital image correlation

# Experimental

sandwich specimen processed  
at 850°C



Layer	Thickness ( $\mu\text{m}$ )	CTE ( $\text{ppm}/^\circ\text{C}$ )	Young's Modulus (GPa)
$\text{Al}_2\text{O}_3$	760	8.1	400
8YSZ	290	8.5 ( $100^\circ\text{C}$ ) 10.5 ( $950^\circ\text{C}$ )	200
SCN+8YSZ	870	9.97	70

# Experimental

$$\sigma_s = \frac{2}{t_s^2} \left( 3z + 2t_s - \frac{2}{E_s} \sum_{j=1}^n E_j t_j \right) \sum_{i=1}^n E_i t_i (\alpha_i - \alpha_s) \Delta T$$

$$= \frac{E_s}{3r} (3z + 2t_s) - \frac{2}{3r} \sum_{i=1}^n E_i t_i,$$

$$\sigma_i = E_i \left[ \alpha_s - \alpha_i + 4 \sum_{j=1}^n \frac{E_j t_j (\alpha_j - \alpha_s)}{E_s t_s} \right] \Delta T$$

$$= -\frac{E_s t_s^2}{6t_i r_i} + \frac{2E_i t_s}{3r}.$$

C. H. Hsueh, J. Appl. Phys., Vol. 91, No. 12, 15 June 2002

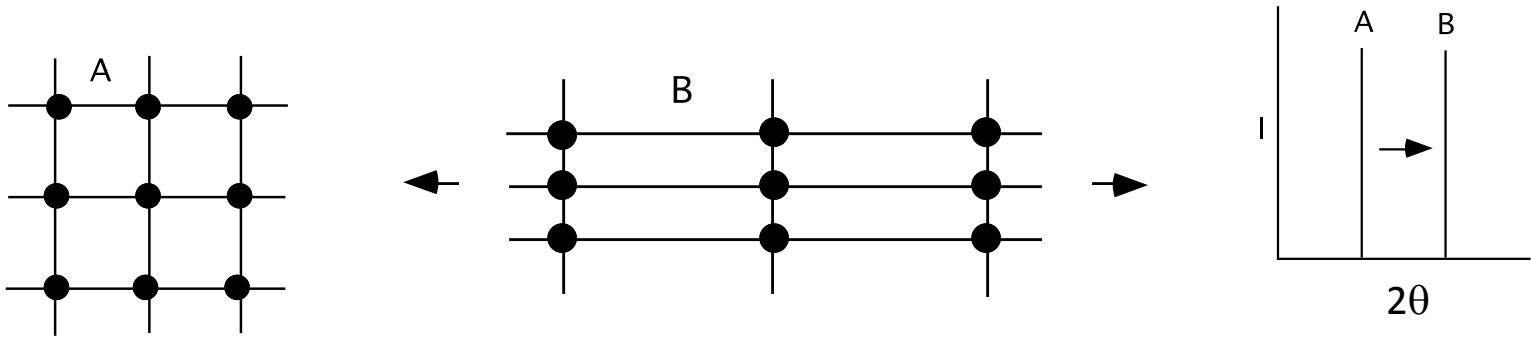
# X-ray Diffraction

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- Strains in a crystal lattice are measured (with respect to a stress-free condition) and the associated residual stress is determined from the elastic constants assuming a linear elastic distortion of the crystal planes.

# X-ray Diffraction

Strain (applied or residual) changes the interplanar and spacing  $\theta$  peak shift



- Bragg's Law

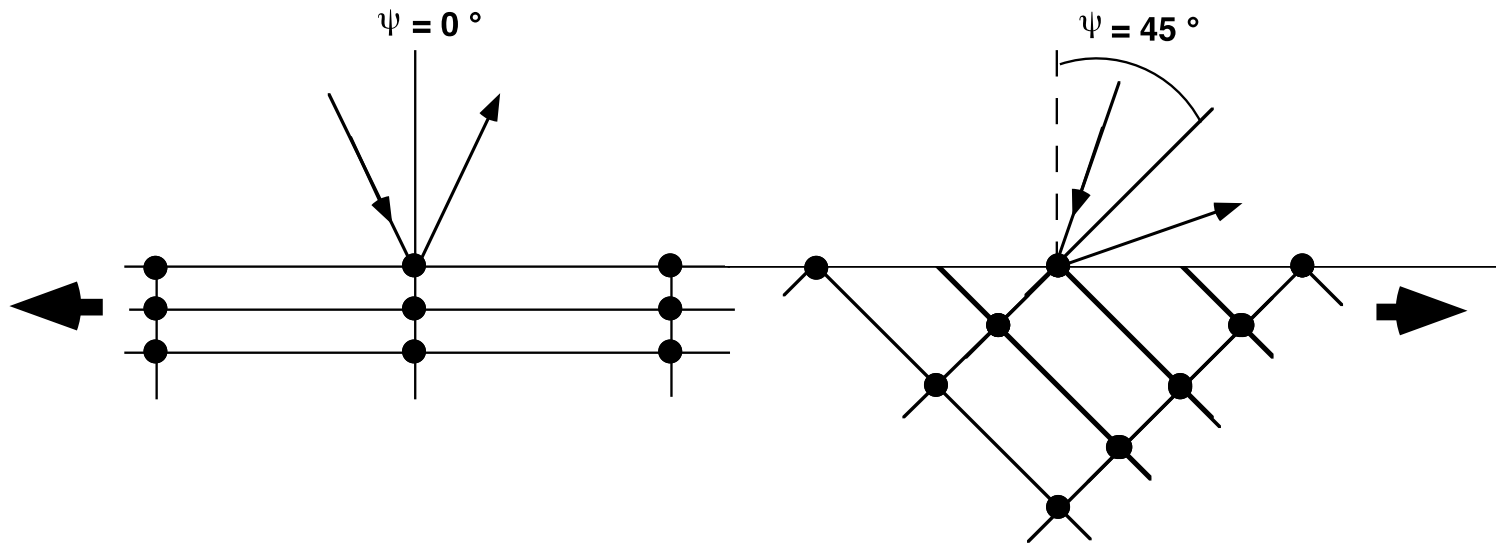
$$\lambda = 2 d \sin\theta$$

where  $\lambda$  = wavelength  
 $d$  = interplanar spacing  
 $2\theta$  = diffraction peak position

- Strain,  $\varepsilon = (d_B - d_A)/d_A$

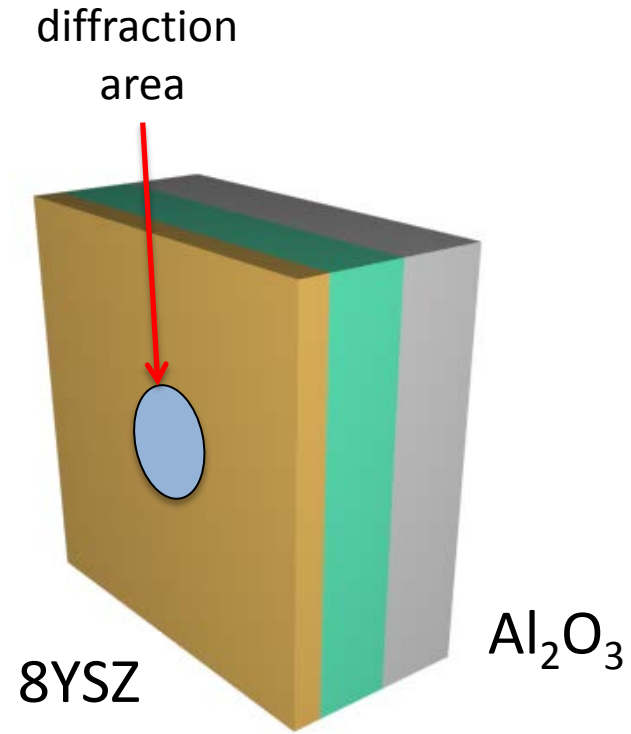
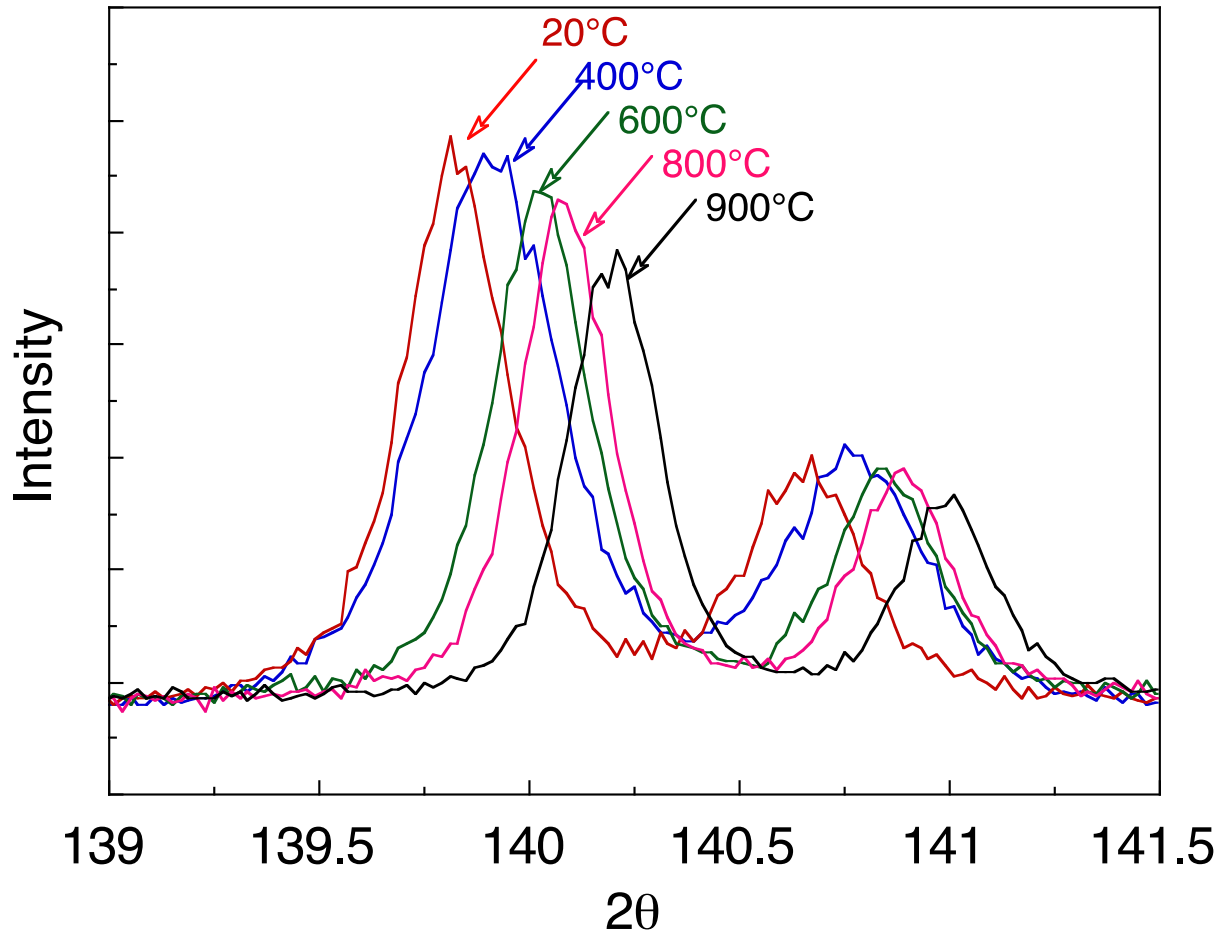
# X-ray Diffraction

- Sample tilting is required for accurate strain measurement with x-rays
- Peak position as a function of tilt angle,  $\psi$
- Slope of  $d$  (interplanar spacing) vs.  $\sin^2\psi$  is used to calculate strain.

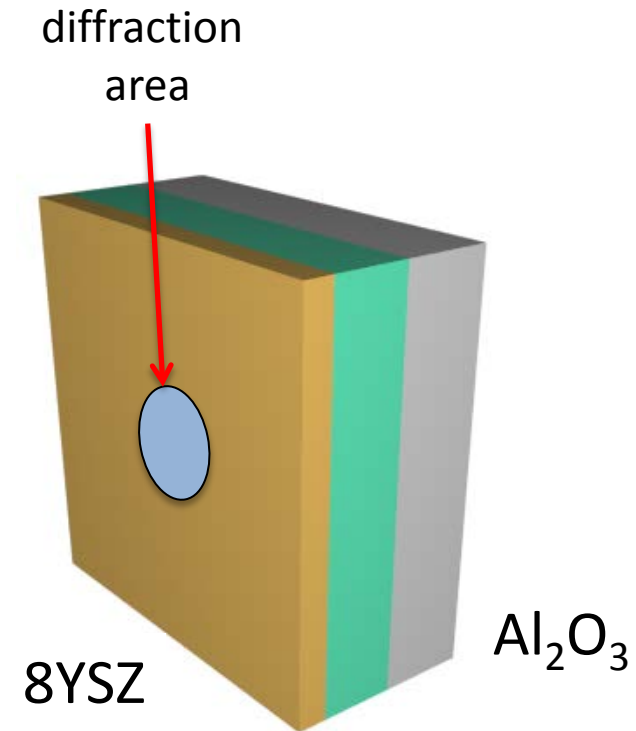
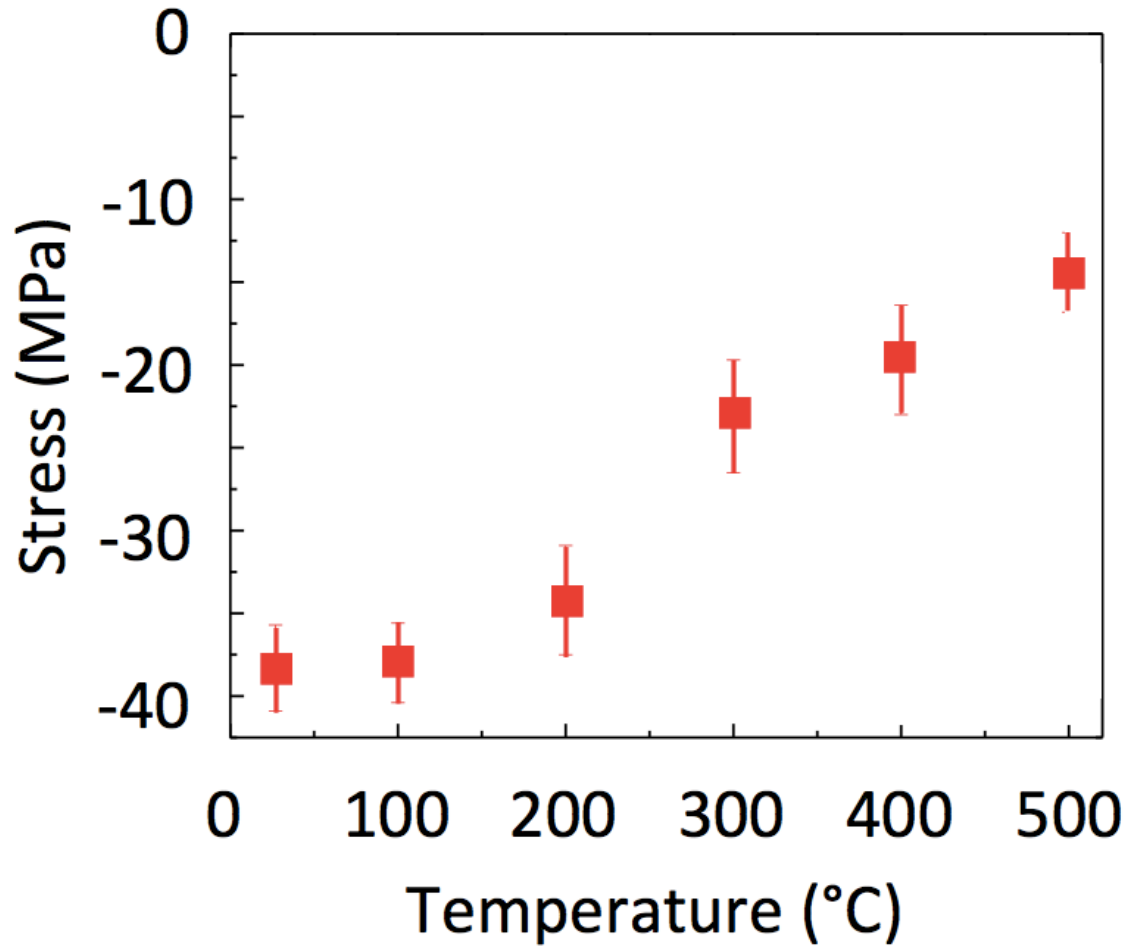




# X-ray Diffraction



# X-ray Diffraction



# Techniques for Measuring Residual Stresses

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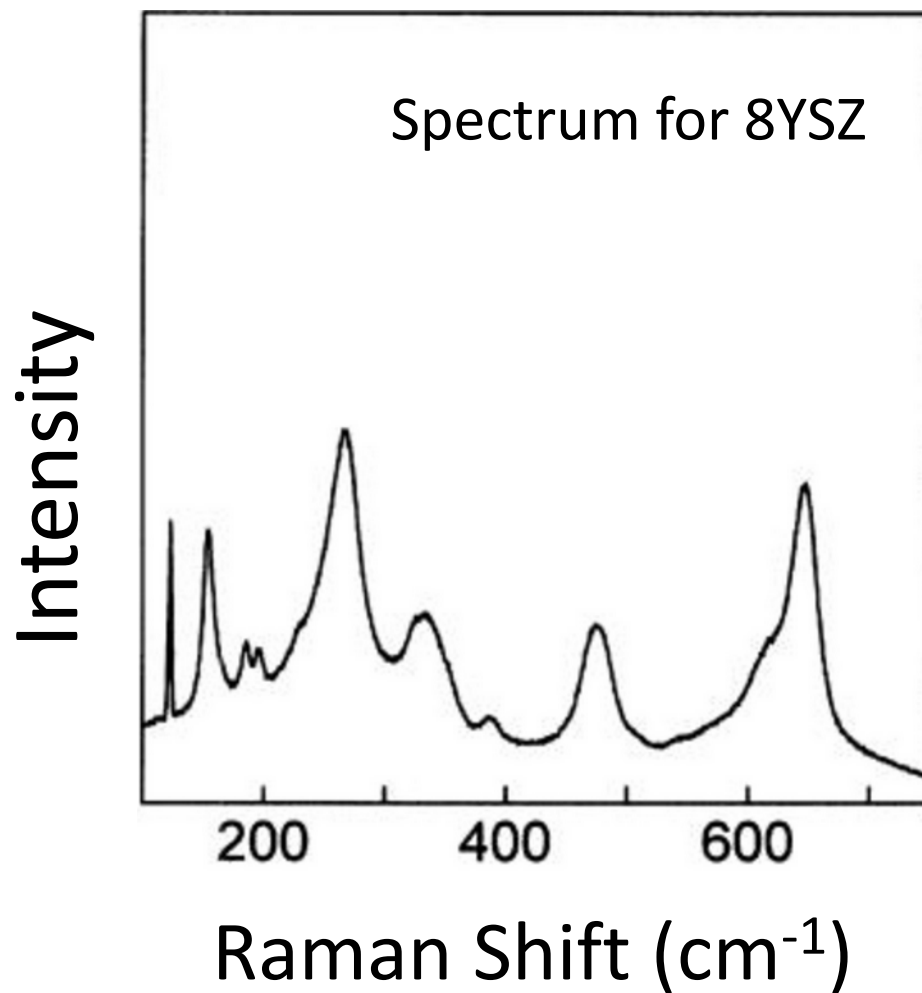
- Diffraction (x-rays, neutrons)
- Raman Spectroscopy

# Raman Spectroscopy

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- Raman scattering arises from the inelastic interaction between photons and phonons
- The frequency of the Raman signal,  $\nu$ , is related to the frequency of the lattice vibrations of the probed material

# Raman Spectroscopy

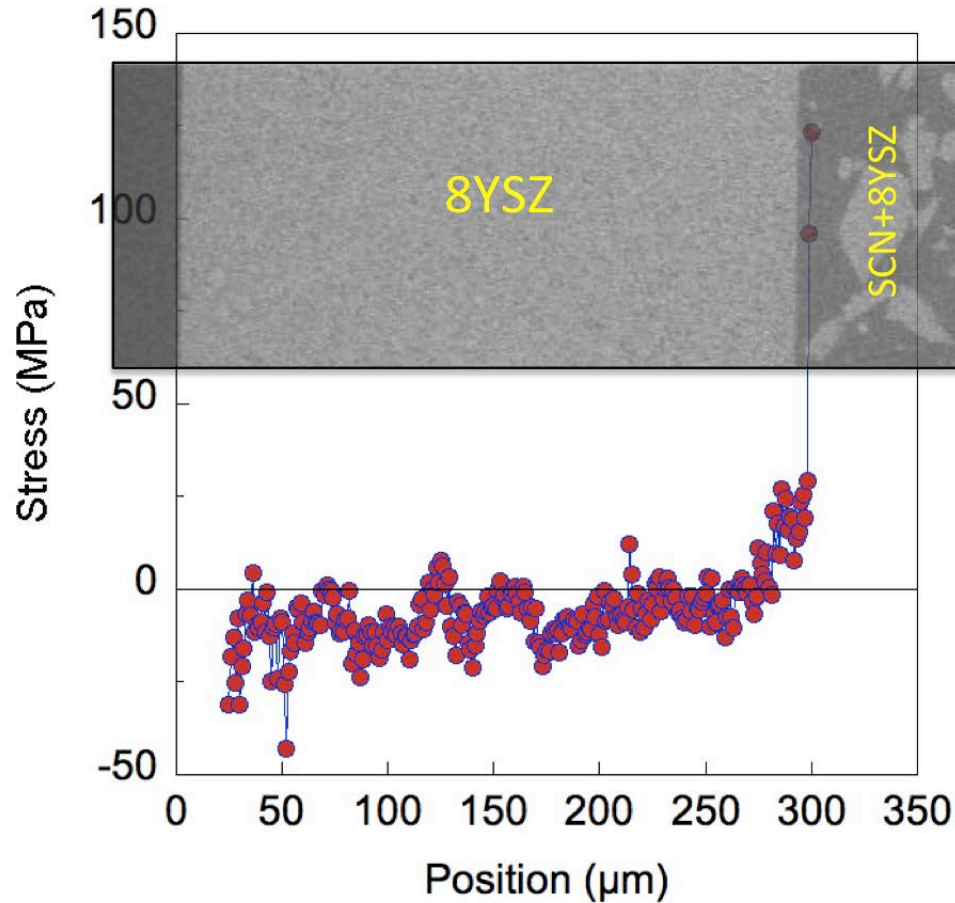


# Raman Spectroscopy

- Raman scattering arises from the inelastic interaction between photons and phonons
- The frequency of the Raman signal,  $\nu$ , is related to the frequency of the lattice vibrations of the probed material
- Because strain changes the frequency of the lattice vibrations, it will also shift the Raman frequency. By mapping the frequency shift,  $\Delta\nu$ , of Raman peaks at different positions on the sample, information on the local stress can be obtained

# Raman Spectroscopy

- $\mu$ Raman spectroscopy has high spatial resolution



# Techniques for Measuring Residual Stresses

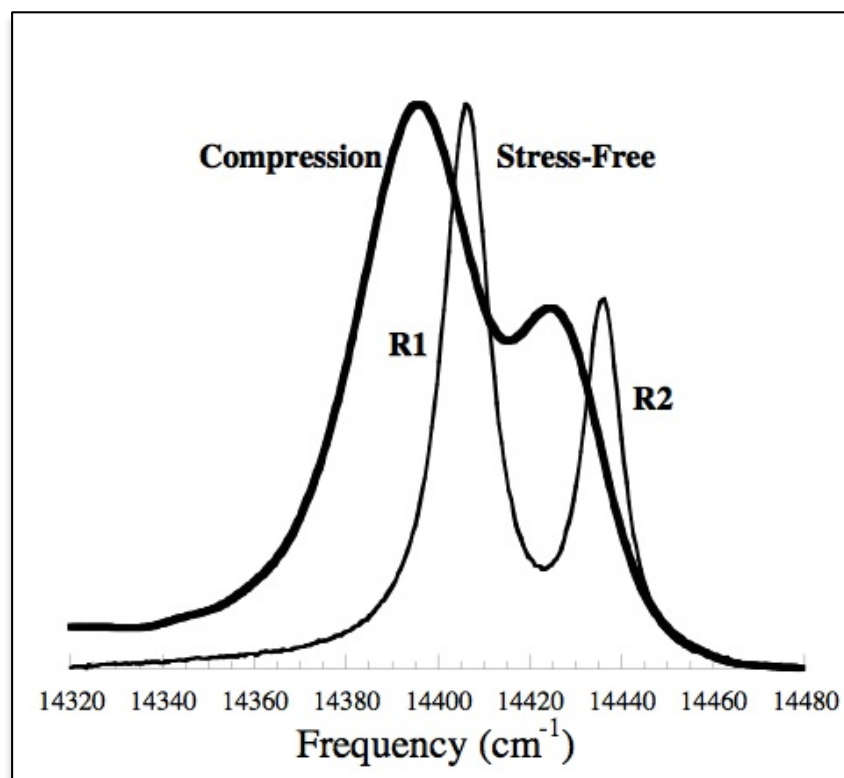
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- Diffraction (x-rays, neutrons)
- Raman Spectroscopy
- Piezospectroscopy



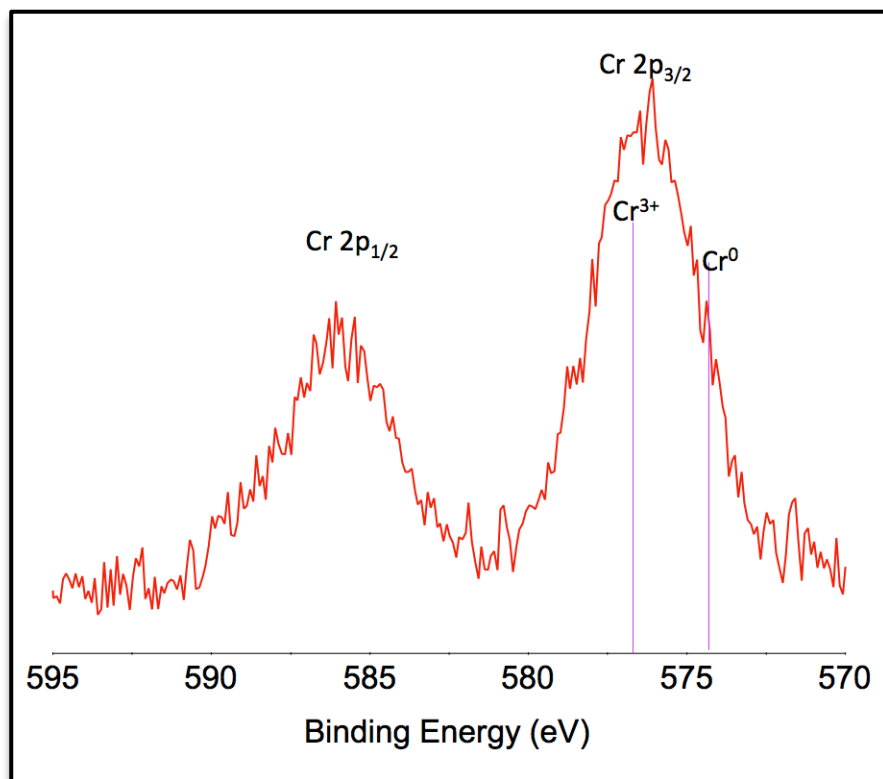
# Piezospectroscopy

Unfortunately the intensity of the Raman lines is very weak for many materials, with zirconia being an exception. Alternative approaches to measure local stresses using stimulated luminescence associated with  $\text{Cr}^{3+}$  have been proven successful.



# Piezospectroscopy

This XPS spectrum corresponds to 8YSZ after exposure to  $\text{Cr}_2\text{O}_3$  powders in air at  $800^\circ\text{C}$  for 1 hour, demonstrating the feasibility of doping oxides with chromium



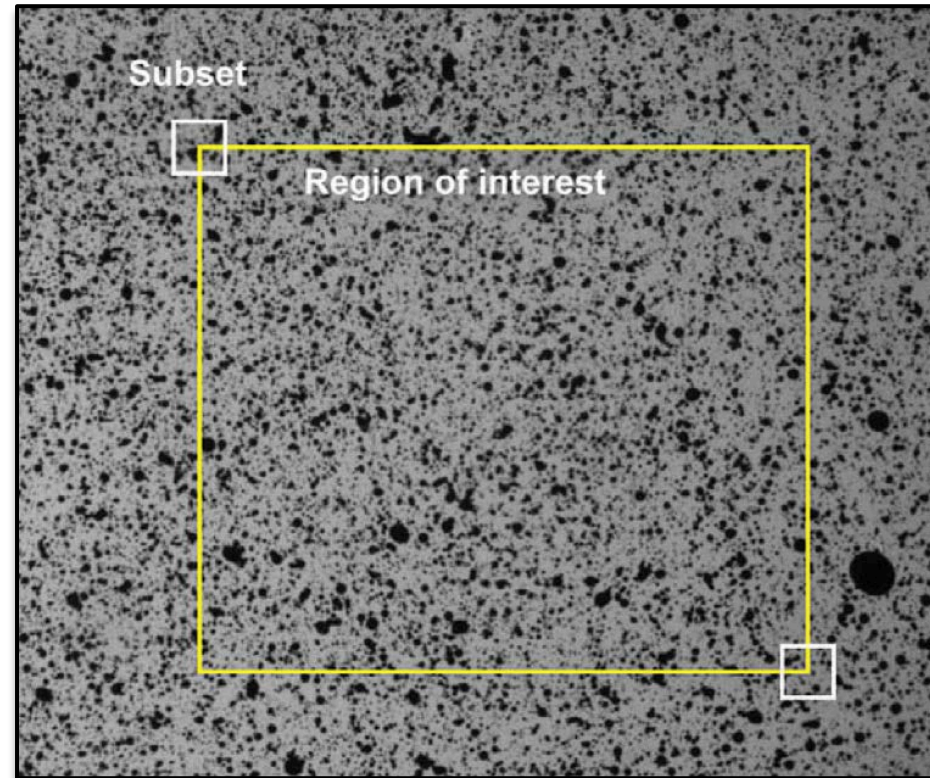
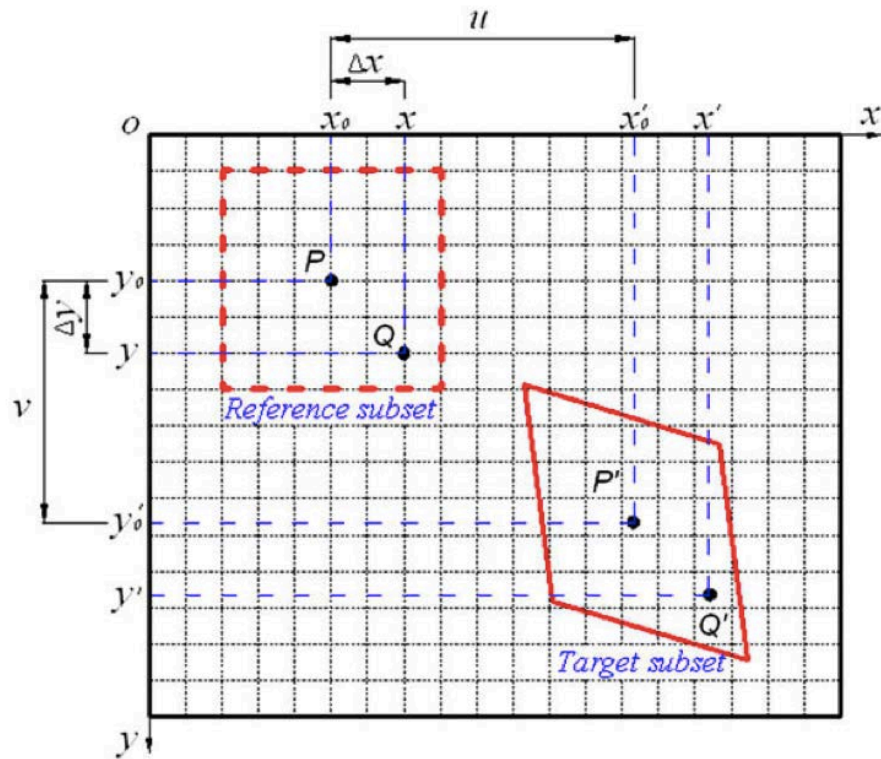
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- Diffraction (x-rays, neutrons)
- Raman Spectroscopy
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- Digital image correlation

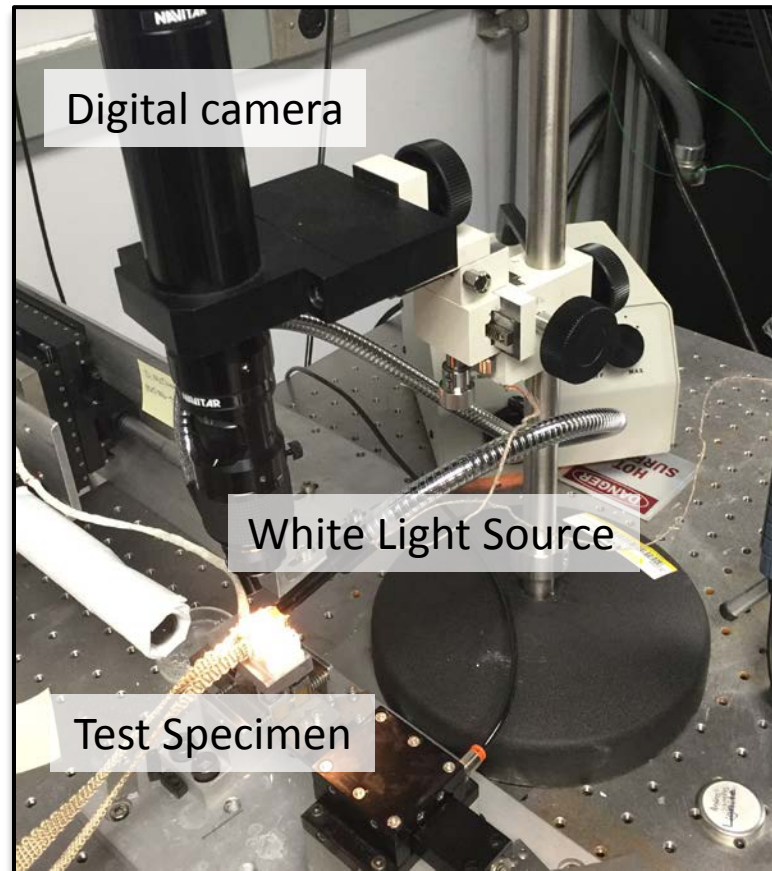
# Digital Image Correlation

DIC directly provides full-field in-plane deformation fields of the test planar specimen surface by comparing the digital images of the specimen surface acquired before and after deformation



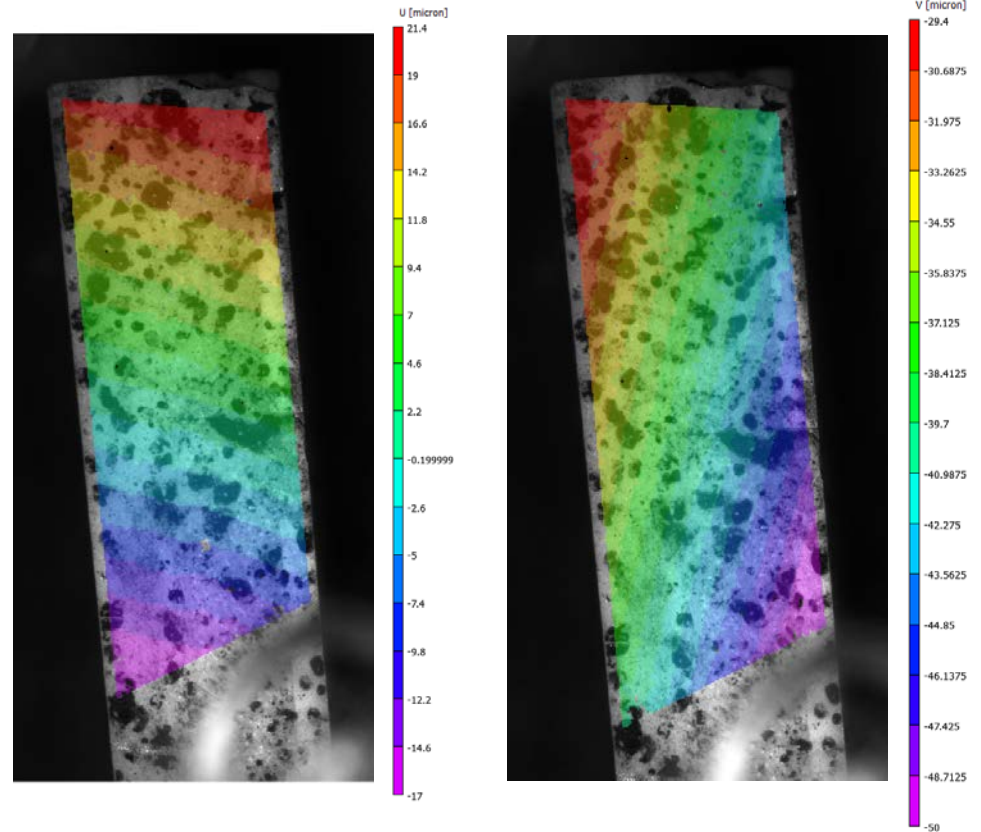
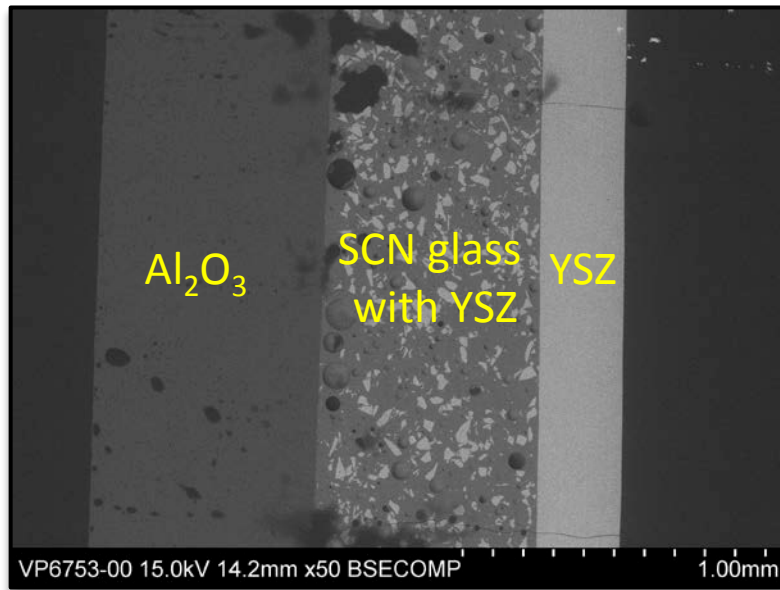
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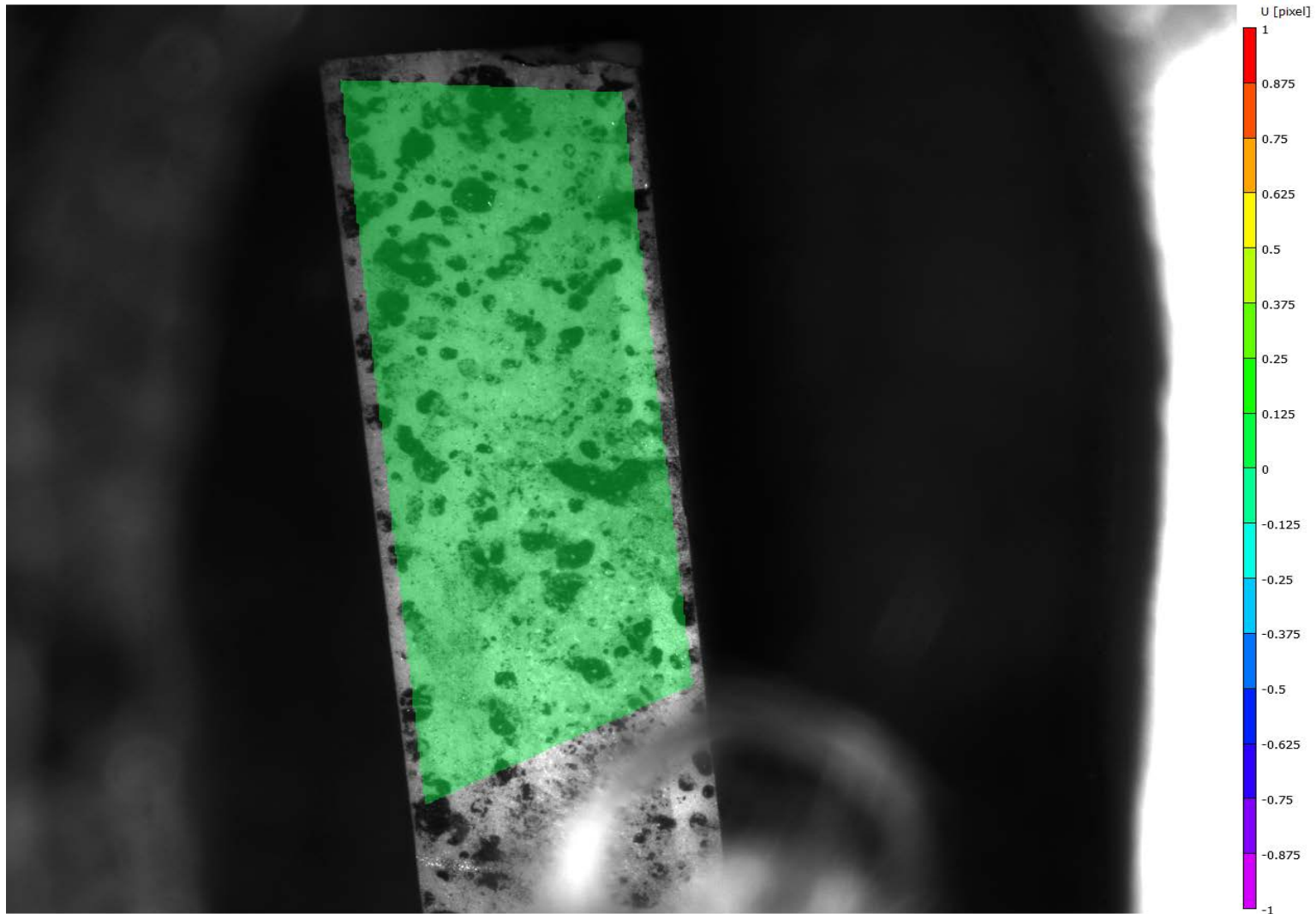


# Digital Image Correlation

## Displacement Fields (u, v)



# Digital Image Correlation



# Summary

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- Different techniques for measuring residual stresses are being investigated and adapted to SOFC materials and components. Some of these techniques have high spatial resolution, while others can be adapted to measure residual stresses as a function of temperature or, on the surface of components with complex geometries.
- Using a model system, the precision, advantages and disadvantages of these techniques will be determined
- Determination of residual stresses is essential to ensure the manufacturing of durable and reliable SOFCs



# X-ray Diffraction

