



# "An eigenstrain analysis of mechanical properties of nanostructured ceramic coatings by synchrotron probe"

Thomas Tsakalakos

Dept. of Materials Science and Engineering, Rutgers University  
M. Croft<sup>1</sup>, Physics Dept., Rutgers University

K. Akdogan, A. Ignatov, N. Ahmedi, L. Balarinni, J. Giminez, M. McNeley,<sup>1</sup>

Zhong<sup>2</sup>, N. Jisrawi<sup>3</sup>, J. Skaritka<sup>2</sup>, N. Simos<sup>2</sup>, T. Fast<sup>3</sup>, R. Sadangi<sup>3</sup>, V. Shukla<sup>3</sup>

<sup>1</sup>Department of Physics, Rutgers University, Piscataway, NJ 08854;

<sup>2</sup>National Synchrotron Light Source, Brookhaven National Laboratory, Upton, NY; 11973

<sup>3</sup>Mat Sc & Eng. Dept, Rutgers University, Piscataway, NJ; 08854

<sup>4</sup>Naval Research Laboratory, 4555 Overlook Ave., SW, Washington, DC 20375

L. Kabacoff      Office of Naval Research  
G. Kim,          Perpetual Technologies, Canada  
E. Lavernia      University of California, Davis  
R. Rigney        A&A INc , S. Brunswick NJ  
K. Scandell      MT NAVSEA

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# Report Documentation Page

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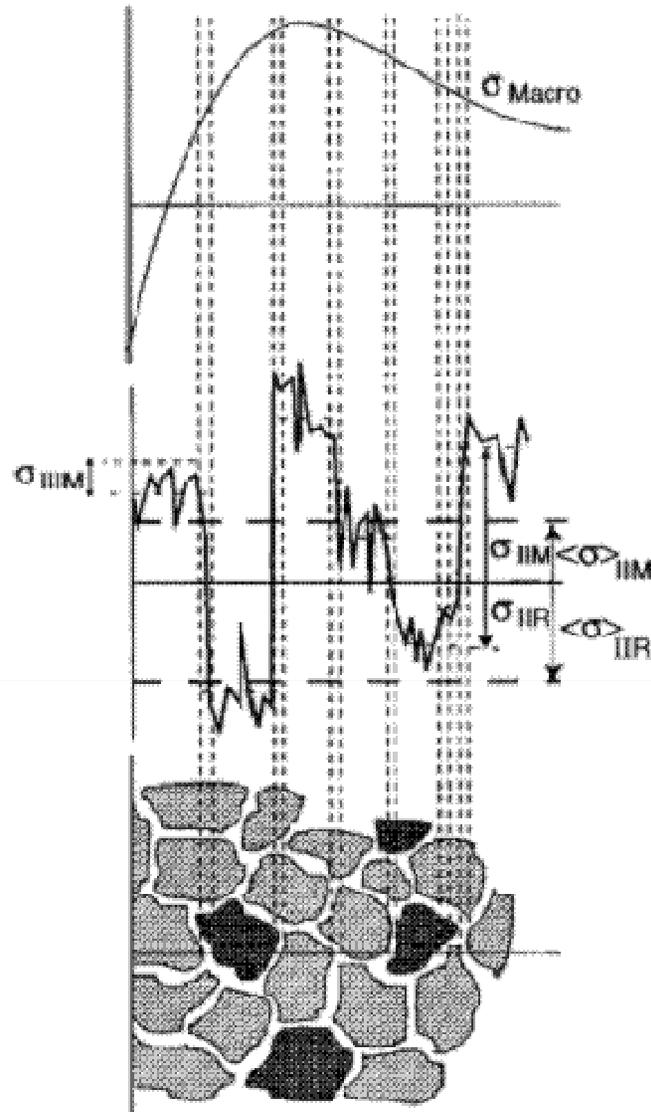
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# Outline



- Introductory Remarks
- Residual Stresses
- Synchrotron EDXRD Strain & Phase Mapping
- Thermal Sprayed Nanostructured Coatings
- In situ four-point bending experiments
- Eigenstrain Analysis
- Reliable Life prediction
- Applications to Engineering Systems



M and R denote matrix and reinforcement respectively

- Residual stress fields can be categorised according to characteristic length scales  $l_{0,I}$ ,  $l_{0,II}$ , and  $l_{0,III}$  over which they self-equilibrate: for type I,  $l_{0,I}$  represents considerable fraction of component; for type II,  $l_{0,II}$  is comparable to grain dimensions, while for type III,  $l_{0,III}$  is less than grain diameter

## TYPE OF RESIDUAL STRESSES MEASURED BY EDXRD

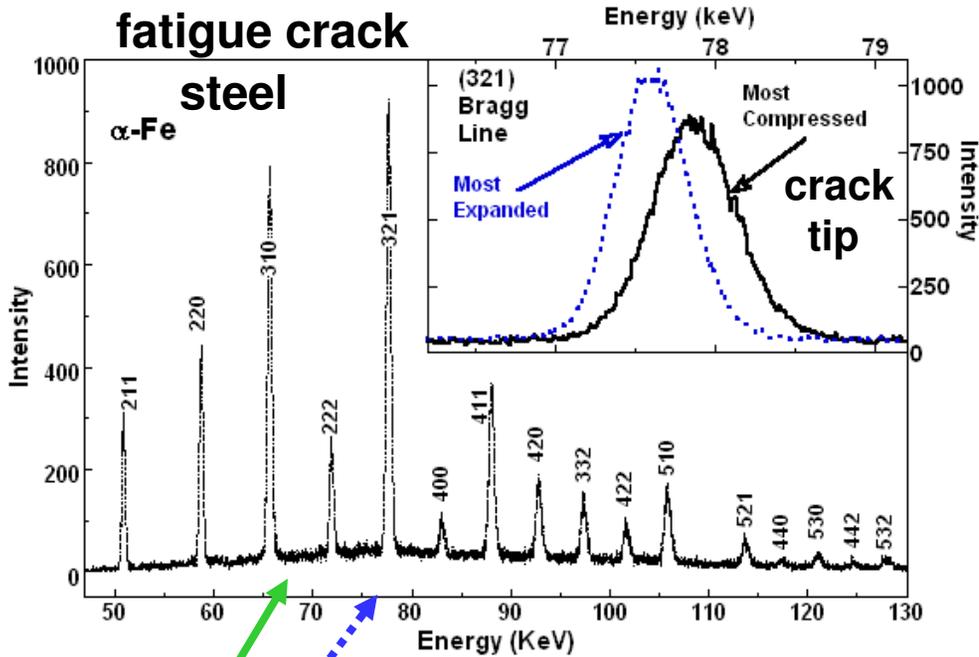
Type I  $\sigma_I$  Macroscale

Type II  $\sigma_{II}$  Mesoscale

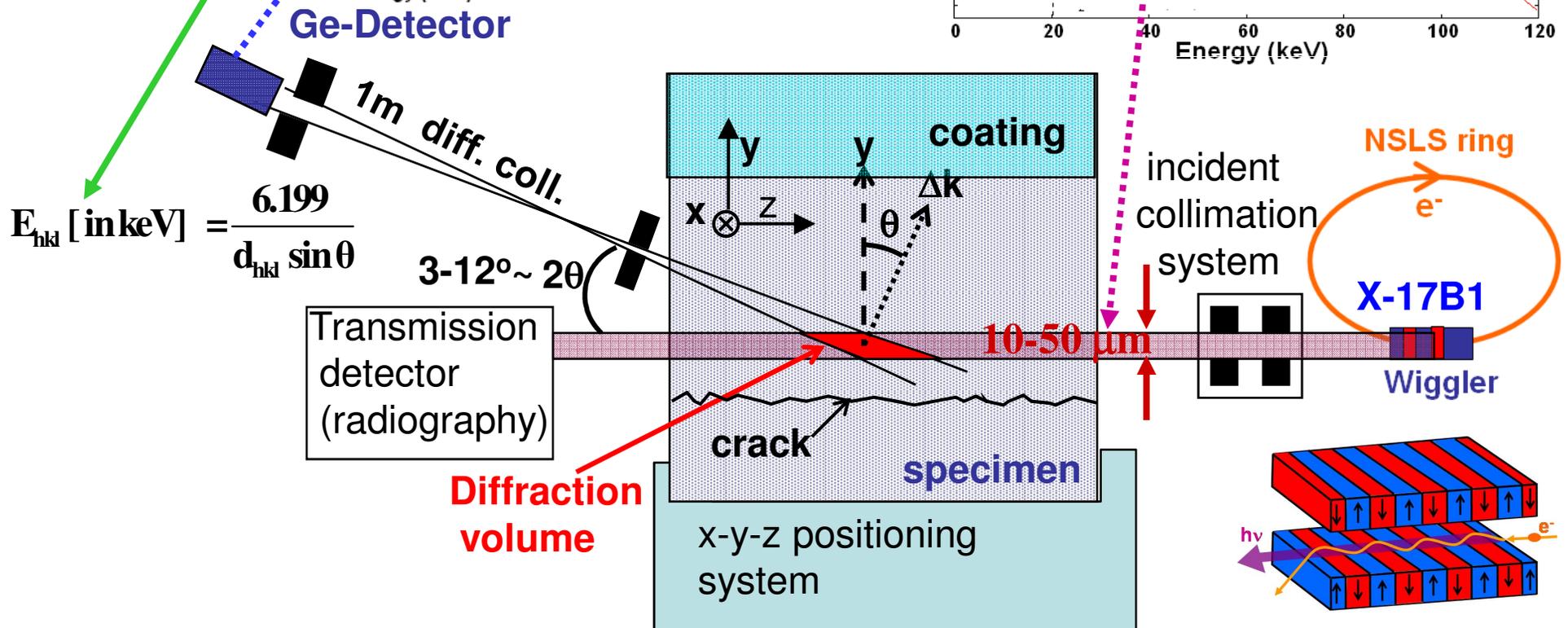
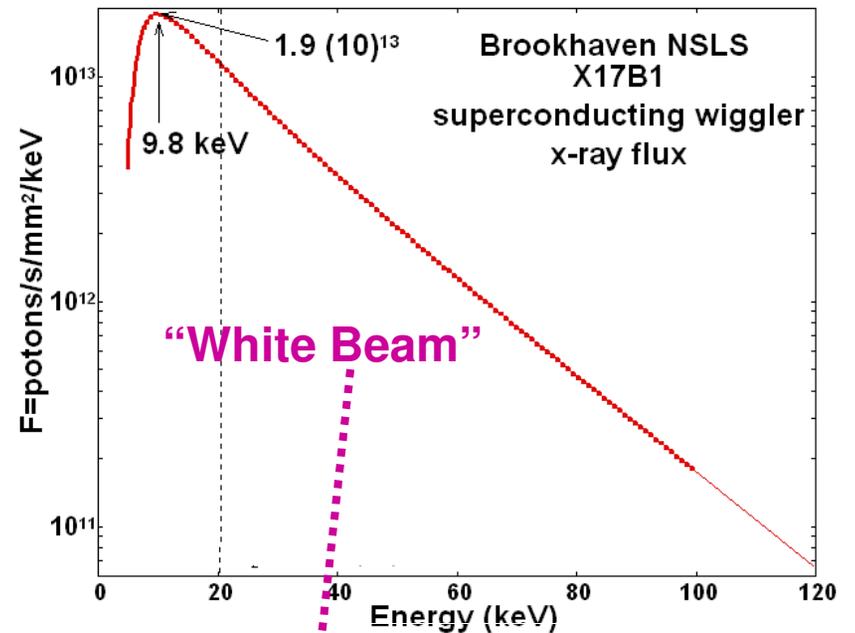
Type III  $\sigma_{III}$  Nanoscale

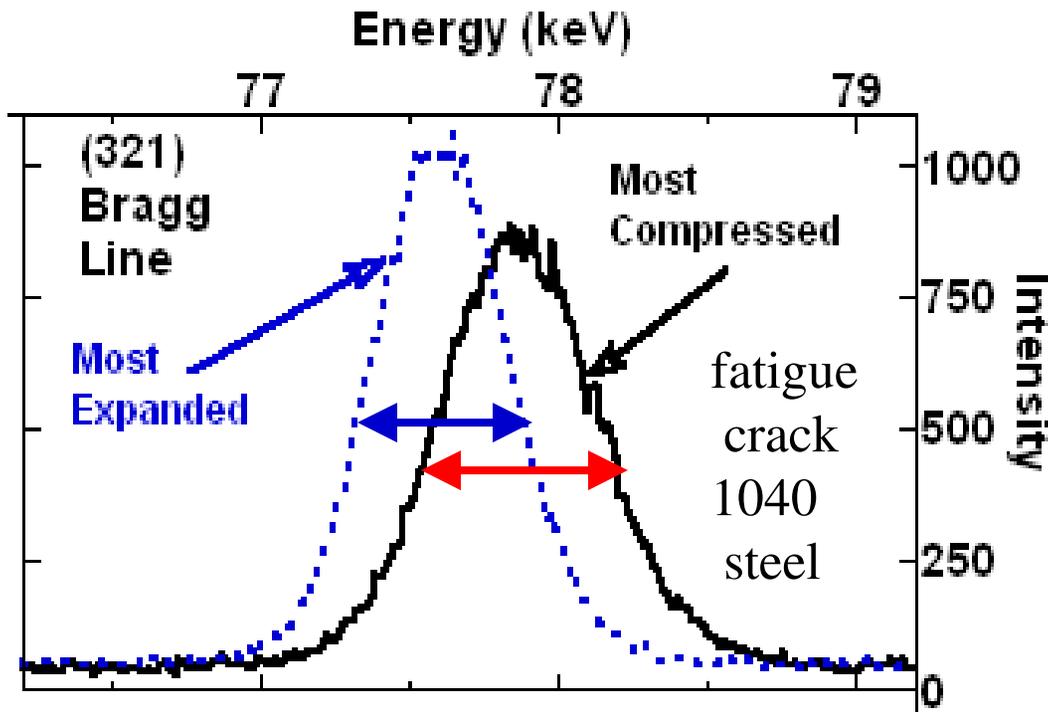
For a two phase material, the macrostress  $\sigma_I$  (type I) is continuous across phases, but the type II and III stresses are not. As a result, even when the sampling area is greater than the characteristic areas for type  $\sigma_{II}$  II and type  $\sigma_{III}$  III, non-zero phase-average microstresses can be recorded. Considering the stresses in two phases:

$$0 = f \langle \sigma_\alpha \rangle^{II} + (1-f) \langle \sigma_\beta \rangle^{II}$$
 where f is the volume fraction of phase  $\alpha$ .



## Energy Dispersive Diffraction





## EDXRD Bragg Line Width

$$E_{hkl} \text{ [ in keV ]} = \frac{b}{d_{hkl} \sin \theta}$$

$$b = 6.199 [\text{keV}(\text{\AA})]$$

Assume Gaussian fitting

$$(\delta E)^2 = (\delta E_{\text{det}})^2 + (\delta E_{\text{gs}})^2 + (\delta E_{\text{ms}})^2$$

radioactive &  
atomic fluorescence  
standards

$$\delta E_{\text{det}} = \frac{a}{E}$$

coherent scattering  
domain (<~grain) size

Debye-Sherrer

$$(\delta E_{\text{gs}})^2 = \left( \frac{bK}{d_{\text{gs}} \sin(\theta)} \right)^2$$

$K \sim 0.9$

micro-strain

$$\delta E_{\text{ms}} = 2e E$$

❖  $d_0$  values to determine strains.

❖ How:

❖ Find regions with Stress-free

❖ Find samples of Stress-free

❖ Use powders

❖ Use of equilibrium conditions:

❖ Multi phases

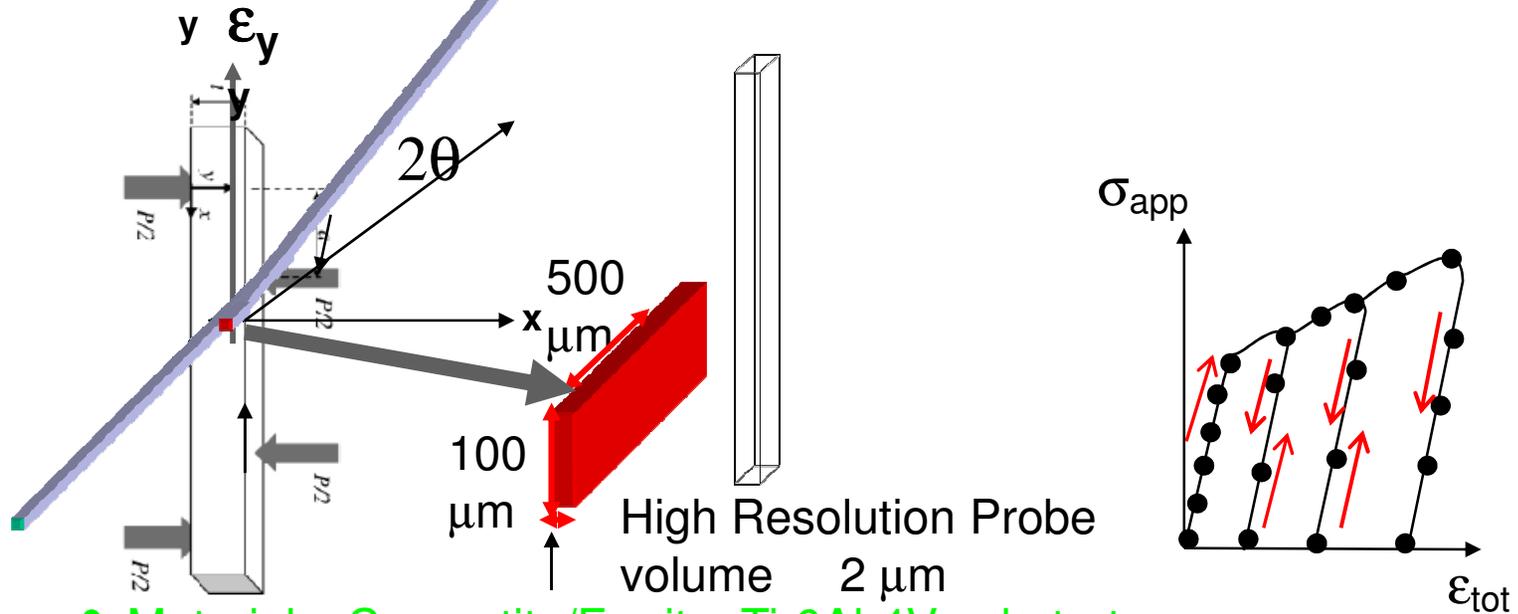
❖ Force/moment balance

## Constitutive equation

$$\sigma_{ij} = \frac{E}{1 + \nu} \left[ \varepsilon_{ij} + \delta_{ij} \left( \frac{\nu}{1 - 2\nu} \right) (\varepsilon_{11} + \varepsilon_{22} + \varepsilon_{33}) \right]$$

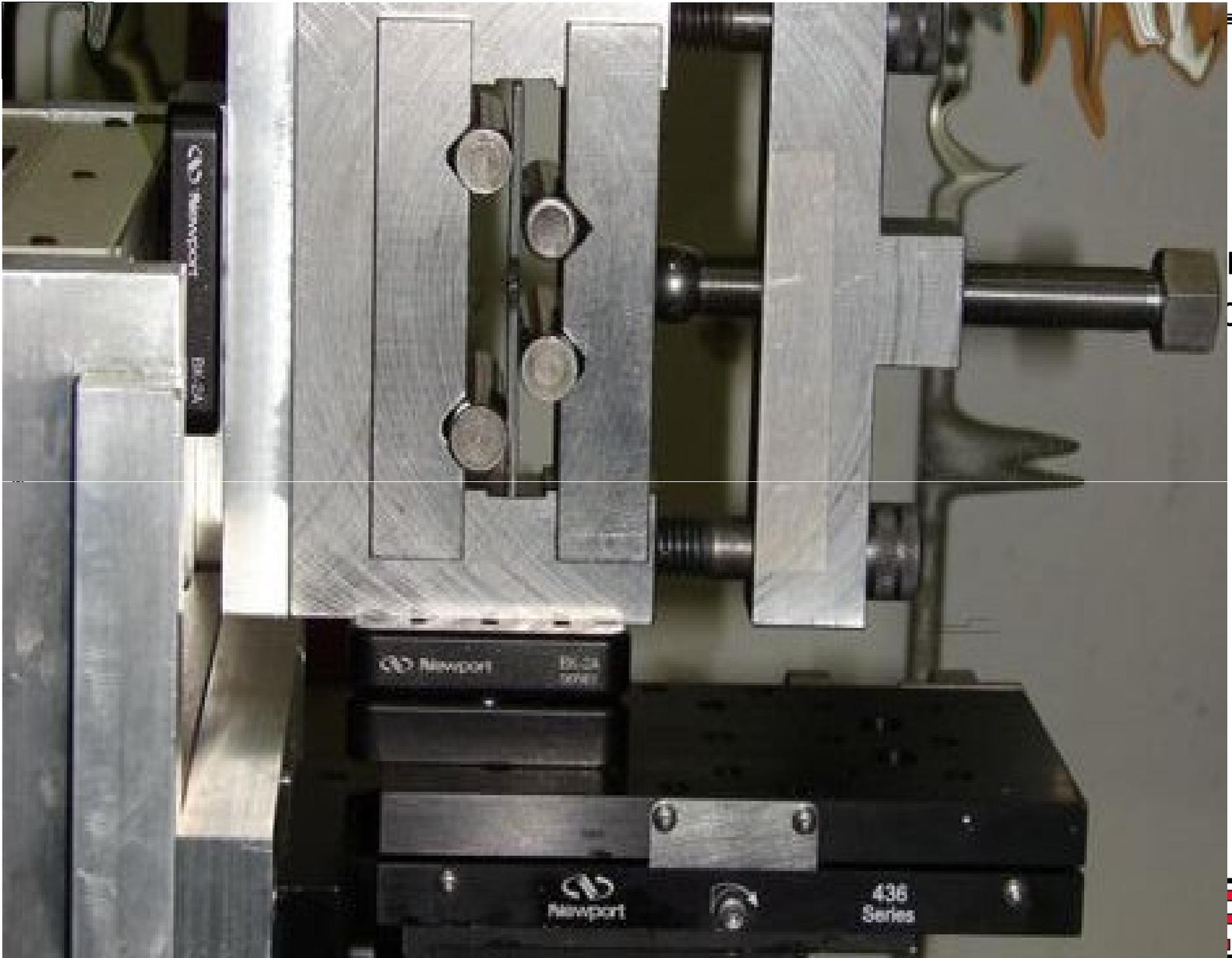


# In-Situ Study of the Development of Intergranular/Interphase Strains



- Materials: Cementite/Ferrite; Ti-6Al-4V substrate
- New Alumina/Titania coatings with new bond coating
- New Titania (Rutile) coating, Amorphous Fe-C-B alloy coating
- Diamond Like Coatings, Aluminum alloy coatings,
- 4-point bending - unload cycle
- homogeneous plastic deformation macro/micro scopically
- Measured:
- Lattice strain response for individual phases and reflections.
- Total deformation by an in-situ 4-point bending





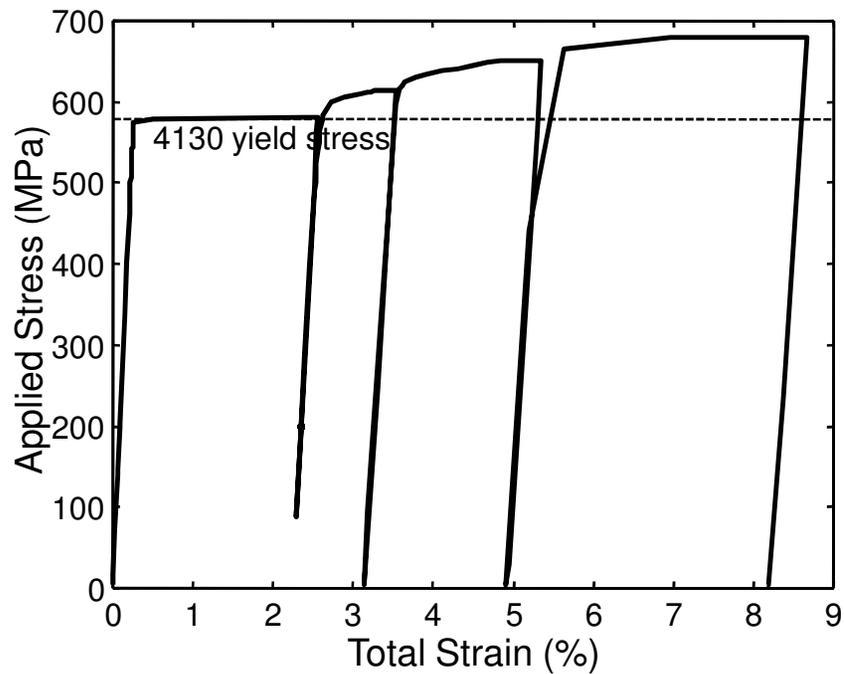
rain  
on





## 4130 Steel

2 phases: cementite + ferrite

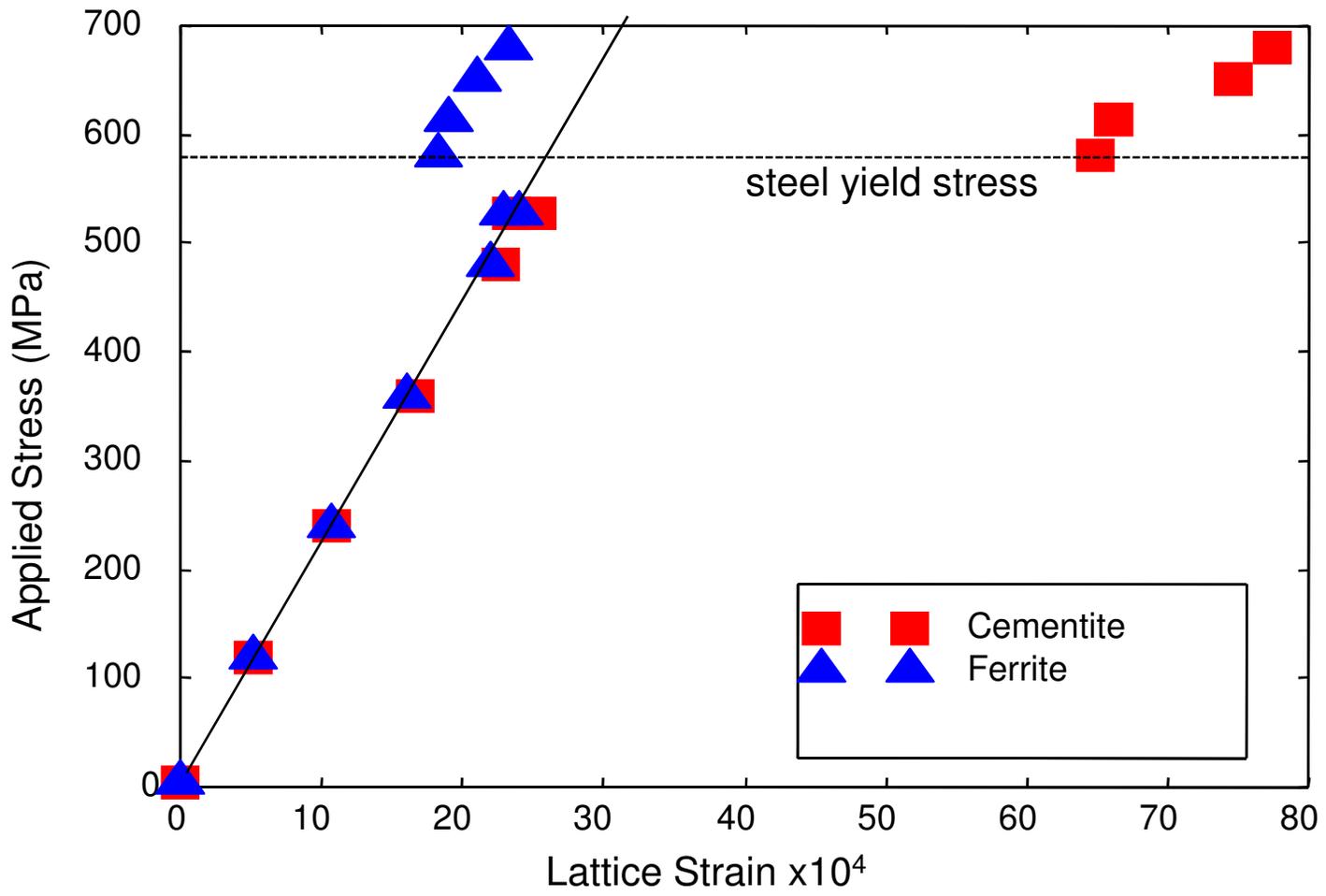


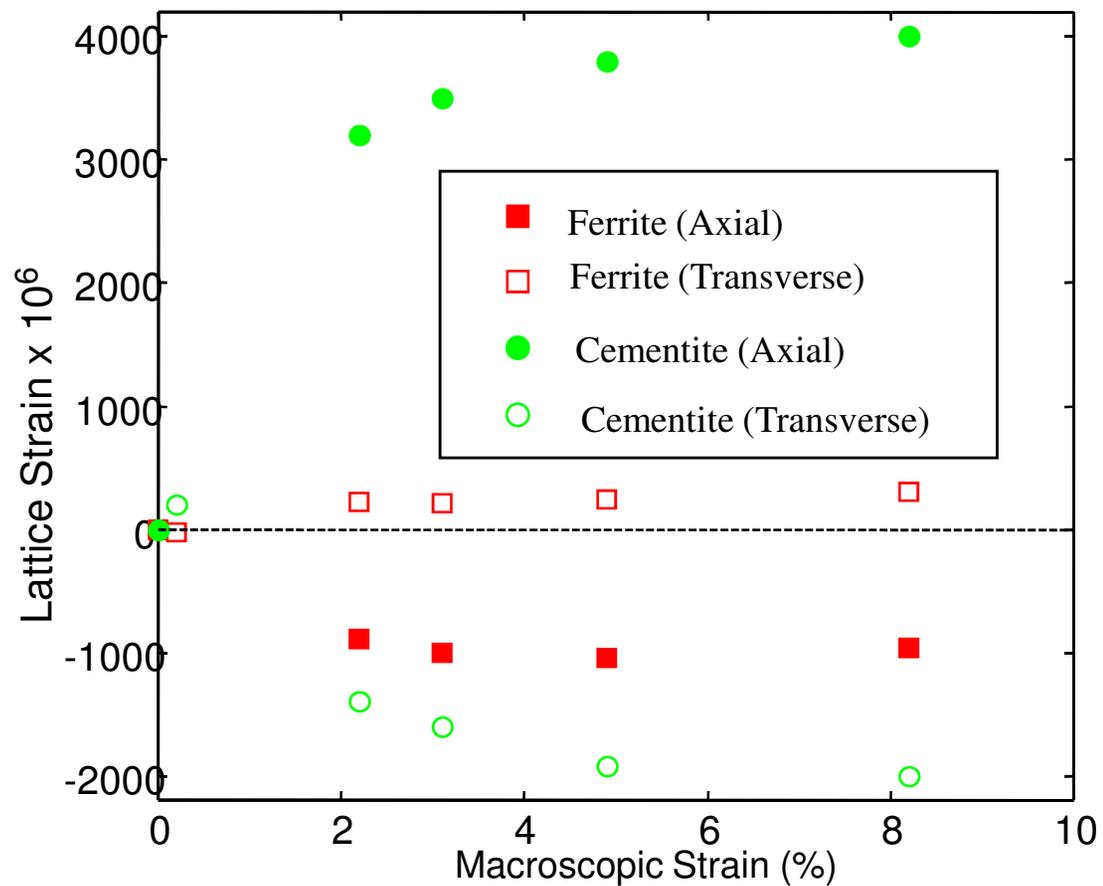
- Both 4130 steels subjected to 4-point bending loading
  - Experiment carried out at BNL, NSLS source
- ⇒ Rietveld Analysis





### Response 4-point bending stress





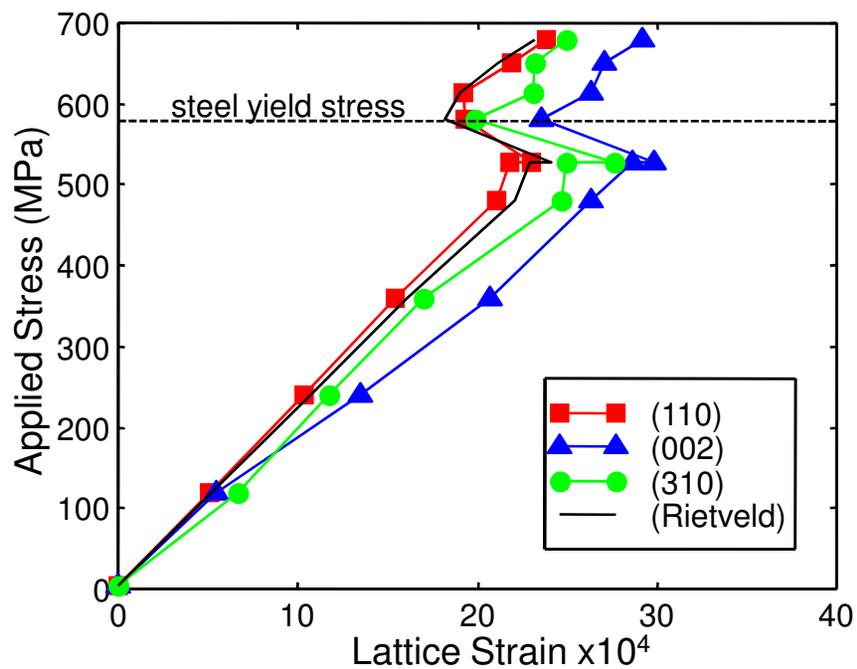
- $\sigma_{33}/\sigma_{11} = -2$  (Upper bound model)
- Vol. Fraction (Data) = 10 %
- Manufacturer specification = 9 %



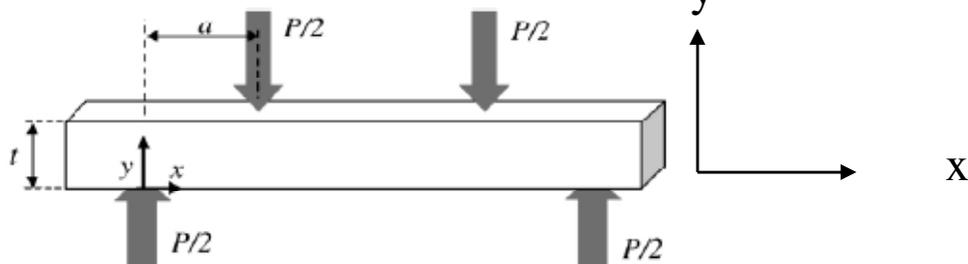
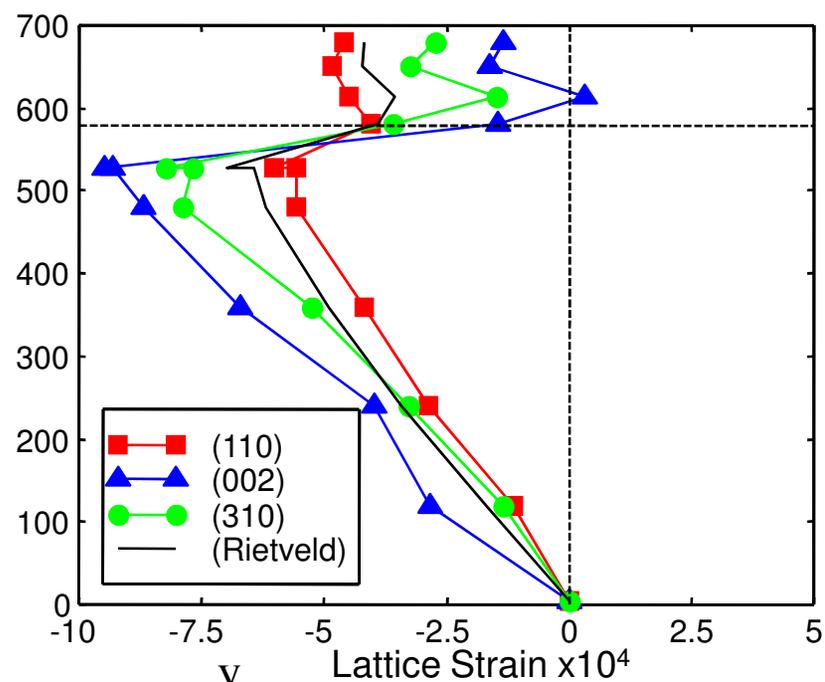


# Lattice strain response for Individual Reflections in steel

## Response // x Axis



## Response // y Axis





## Ti-6Al-4V /Substrate

### Thermal and Mechanical:

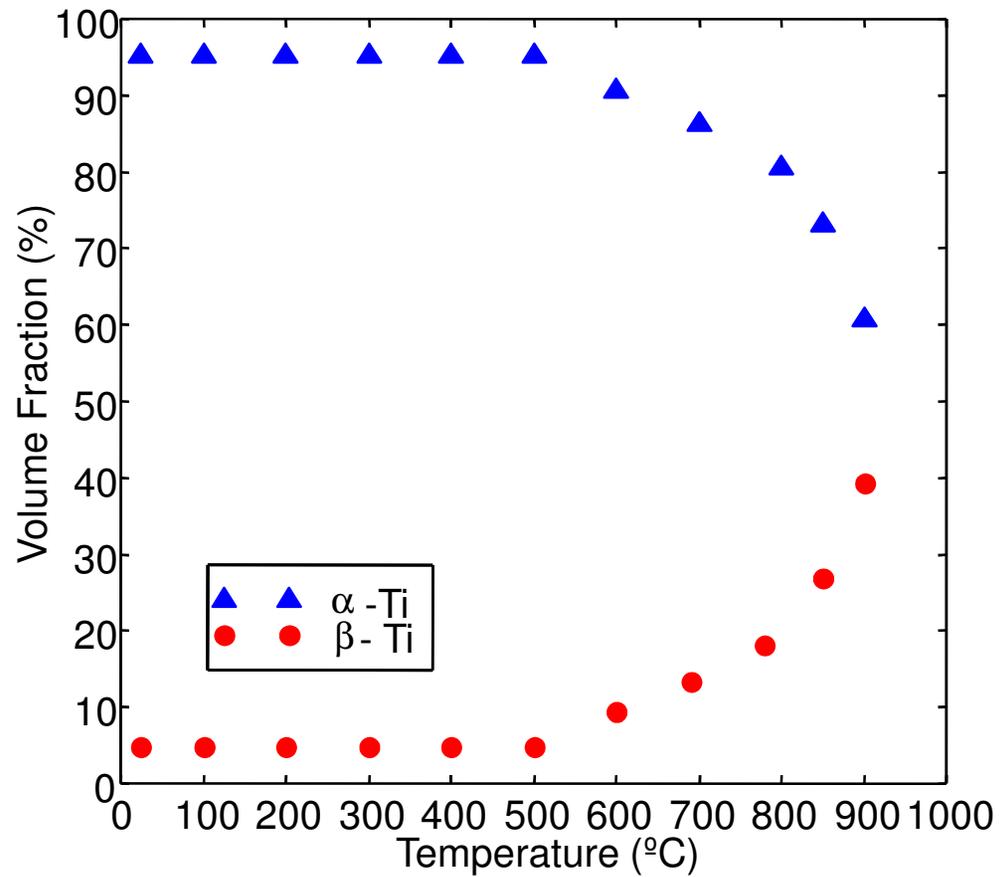
#### Ti-6-4:

- Young's Modulus = 115 GPa
- Poisson's Ratio = 0.349
- CTE =  $10 \times 10^{-6} \text{ K}^{-1}$



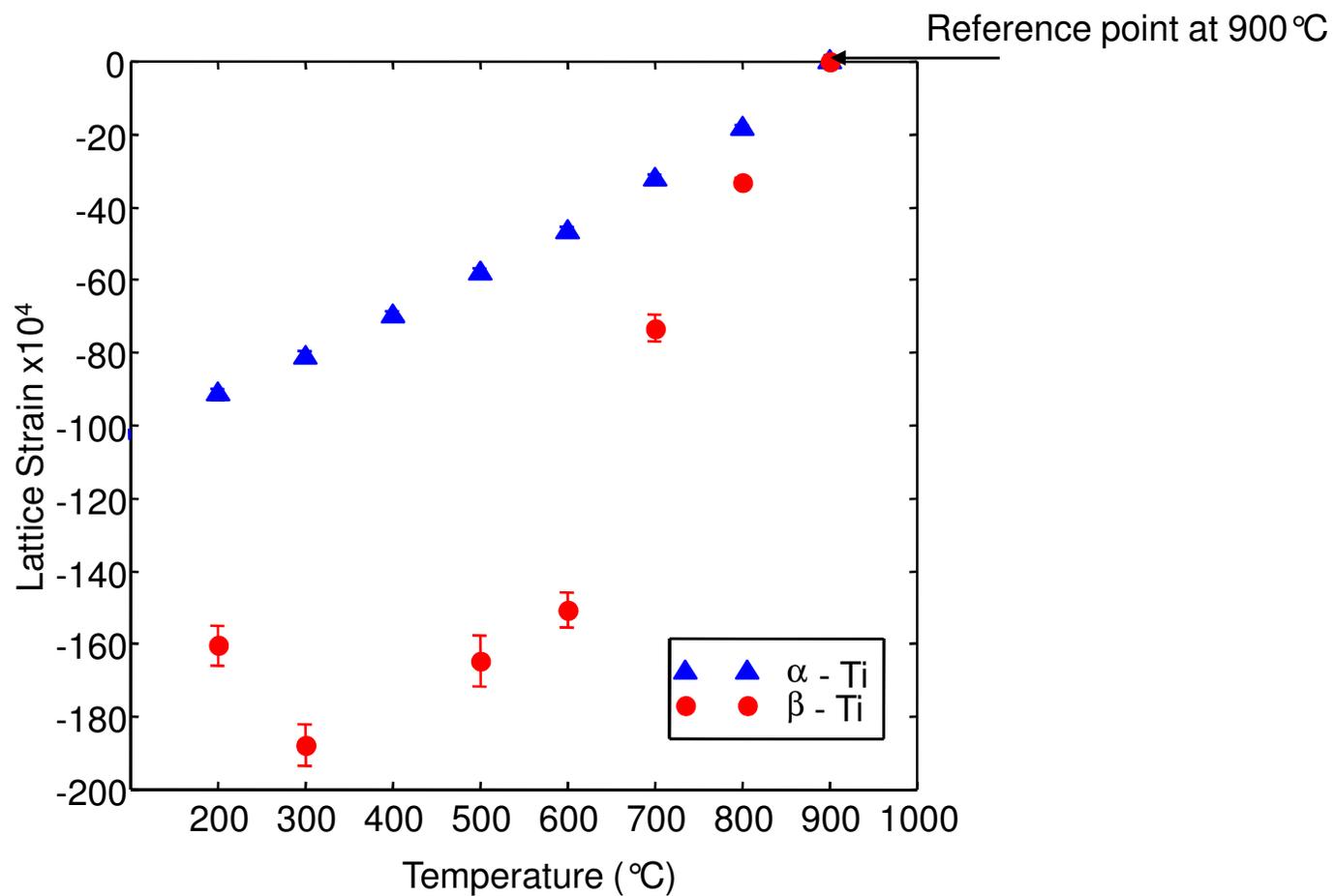


## Distribution of $\alpha$ and $\beta$ phase of Ti6Al-4V as a function of Temperature





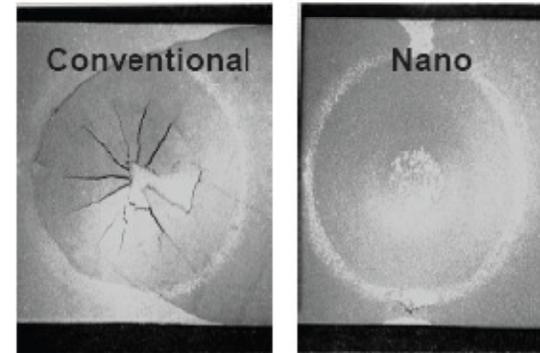
## Thermal strain in $\alpha$ and $\beta$ phase of Ti6Al-4V as a function of Temperature





# Thermal Sprayed Nanostructured Coatings

- **$n\text{-Al}_2\text{O}_3\text{-}13\text{TiO}_2$  coatings fabricated by conventional plasma spray**
- **2X the bond strength and 4X the wear resistance**
- **Extraordinary deformability without failure**
- **Direct transition to fleet and industry (fully commercial)**



***No failure even after Severe deformation***



**MCM shafts fail after 18 months service  
 Requiring dry docking for weld repair**

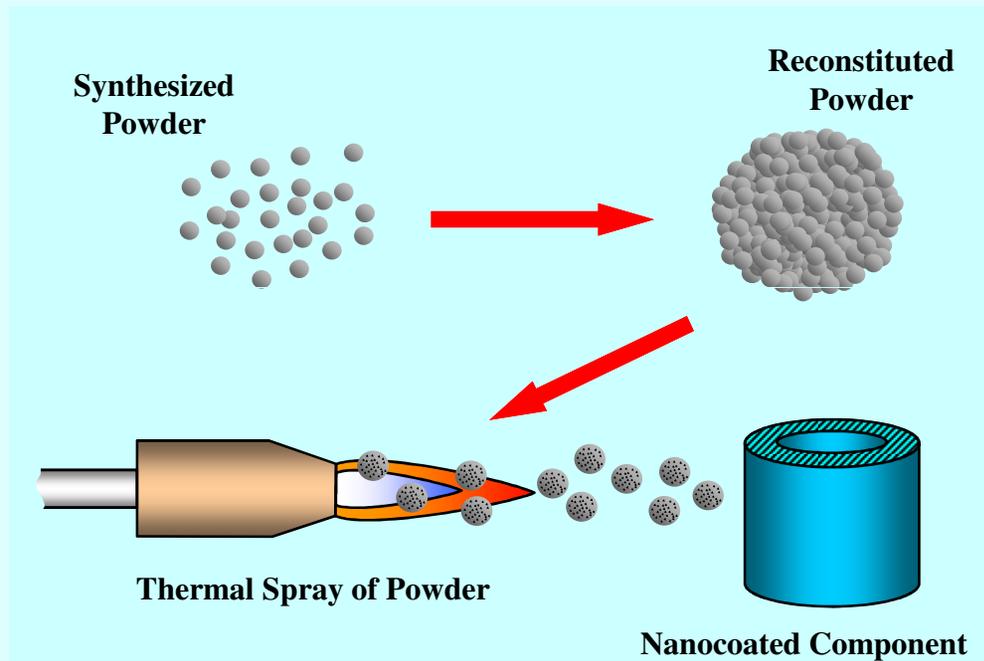


**Uncoated shaft experiences  
 Severe scoring damage**



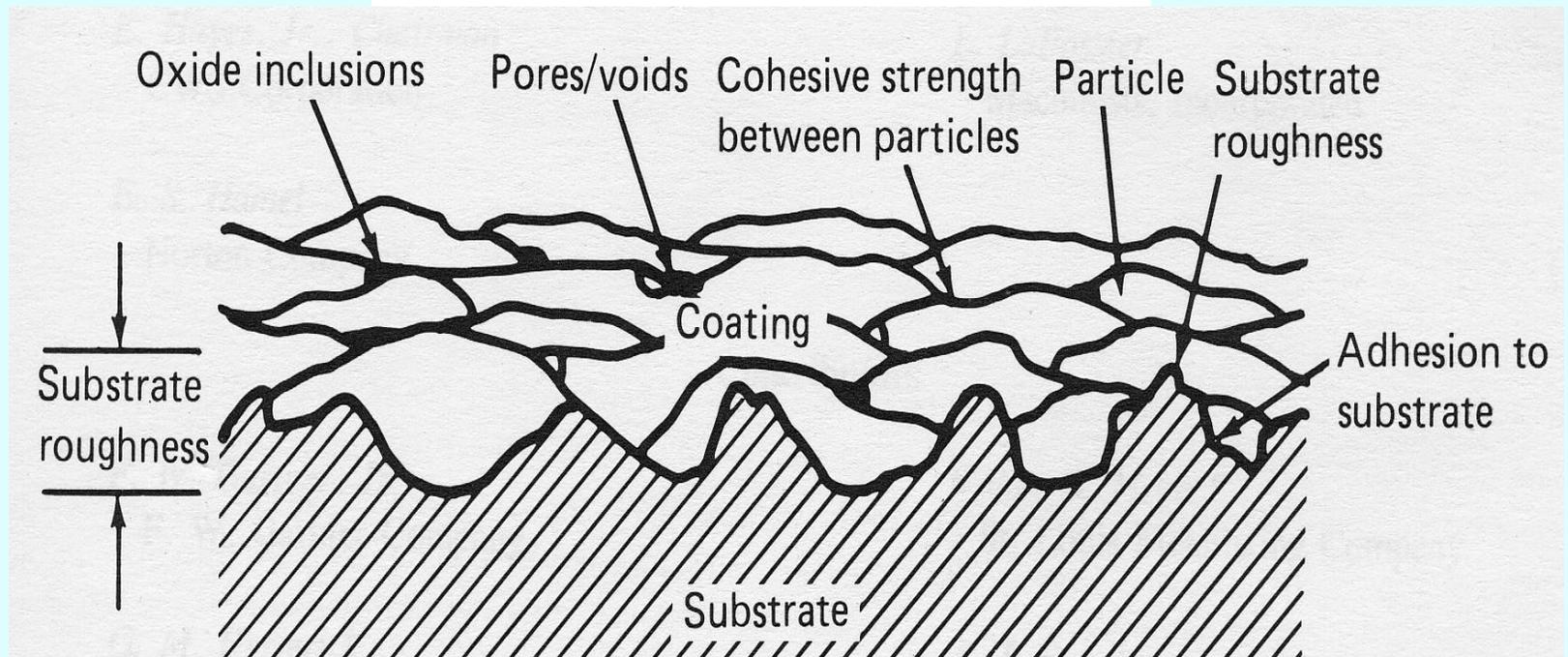
**No visible damage after  
 Four years of service**

## Advanced Coating Technology Development for Enhanced Durability and Reduced Cost in Naval Applications



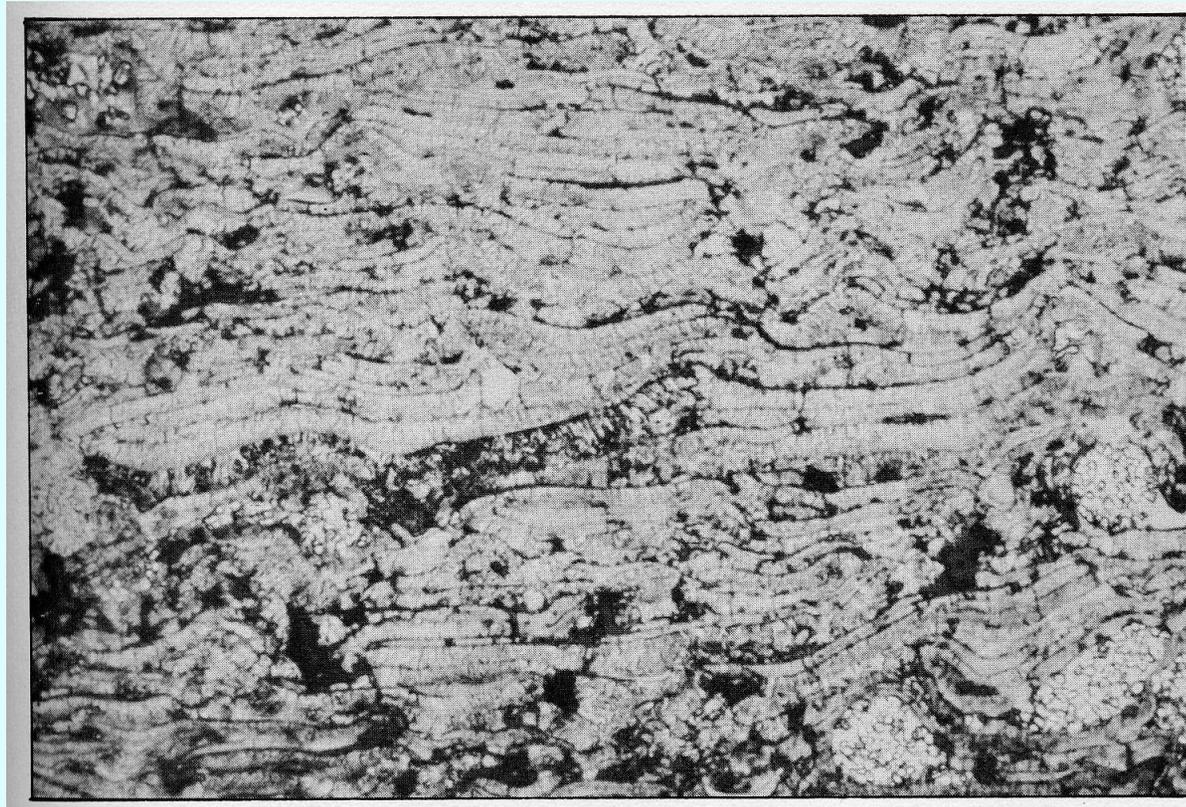


# Fundamentals of the Process





# Coating Properties





## Material Systems

**1.  $\text{Al}_2\text{O}_3$ -13wt% $\text{TiO}_2$**  coatings made from starting micron size (Metco 130) and nanosized powders (Inframmat 2613)(Particle size 20-30 nm) With Grit Blasting

In addition we compared the phases and strains as a function of coating thickness in both micro- and nano- coatings.

Substrates: 1) 1020 Steel (with a Ni bond coat)

2) Titanium (with a Ti bond coat)

Varying thickness of micro- and nano- coatings were applied.

Substrates were grit blasted (compressive stresses).

Table 1, summarizes the coating samples produced with 1020 steel. Table 2 is similar for the titanium substrate coating samples produced.

All the samples were prepared by A&A company,



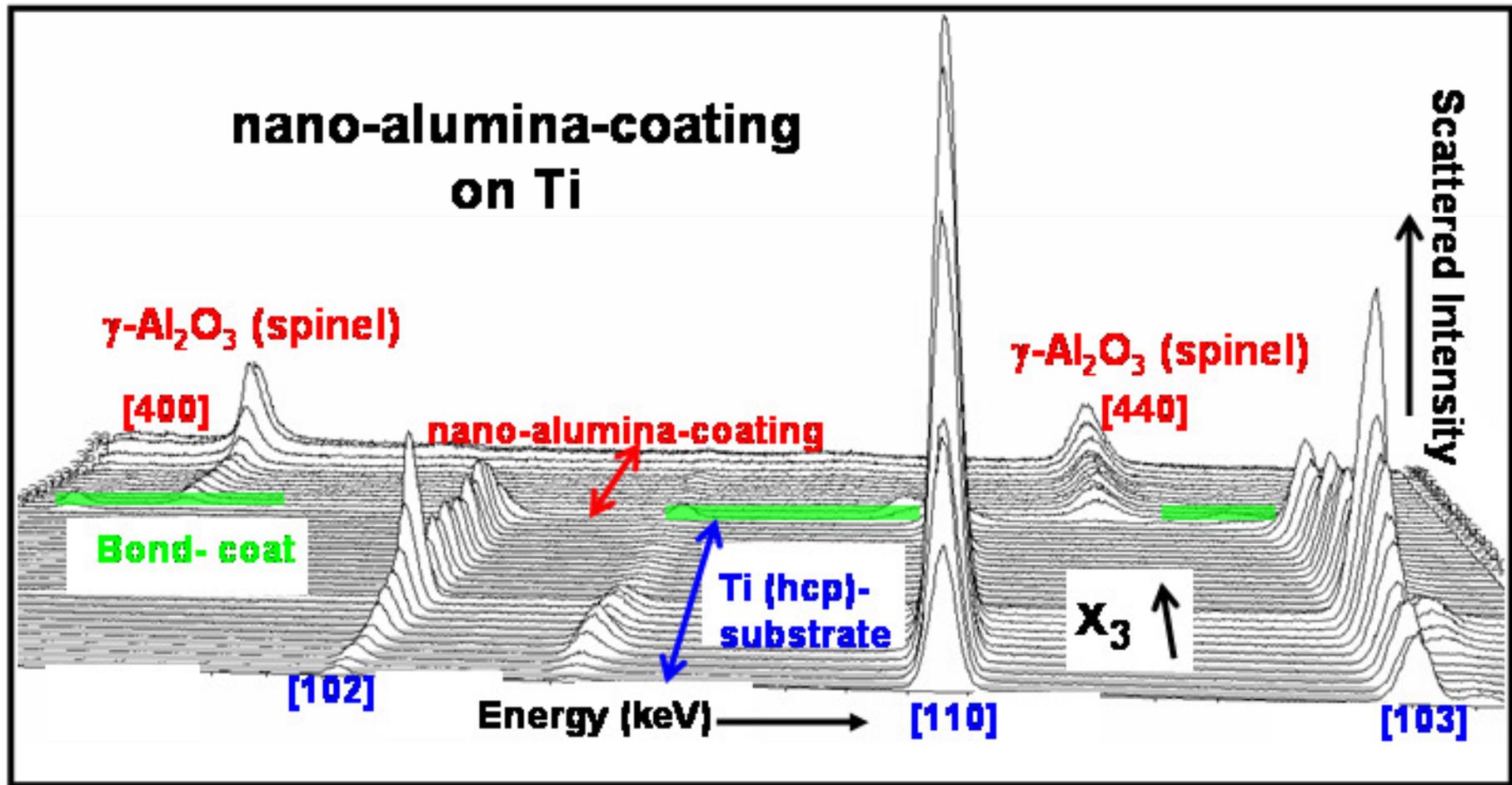
# Ti substrate samples



Starting Powder	Substrate	Coating thickness	Designation
Micron	Titanium	None	T1
		Grit blast only	T2
		Grit blast and bond coat	T3
		2 passes on T3	T4
		Typical coating on T3	T5
		Over-coating, on T3, before failure	T6
		Over-coating, on T3, after failure	T7
Nanosize	Titanium	2 passes on T3	T8
		Typical coating on T3	T9
		Over-coating, on T3, before failure	T10
		Over-coating, on T3, after failure	T11

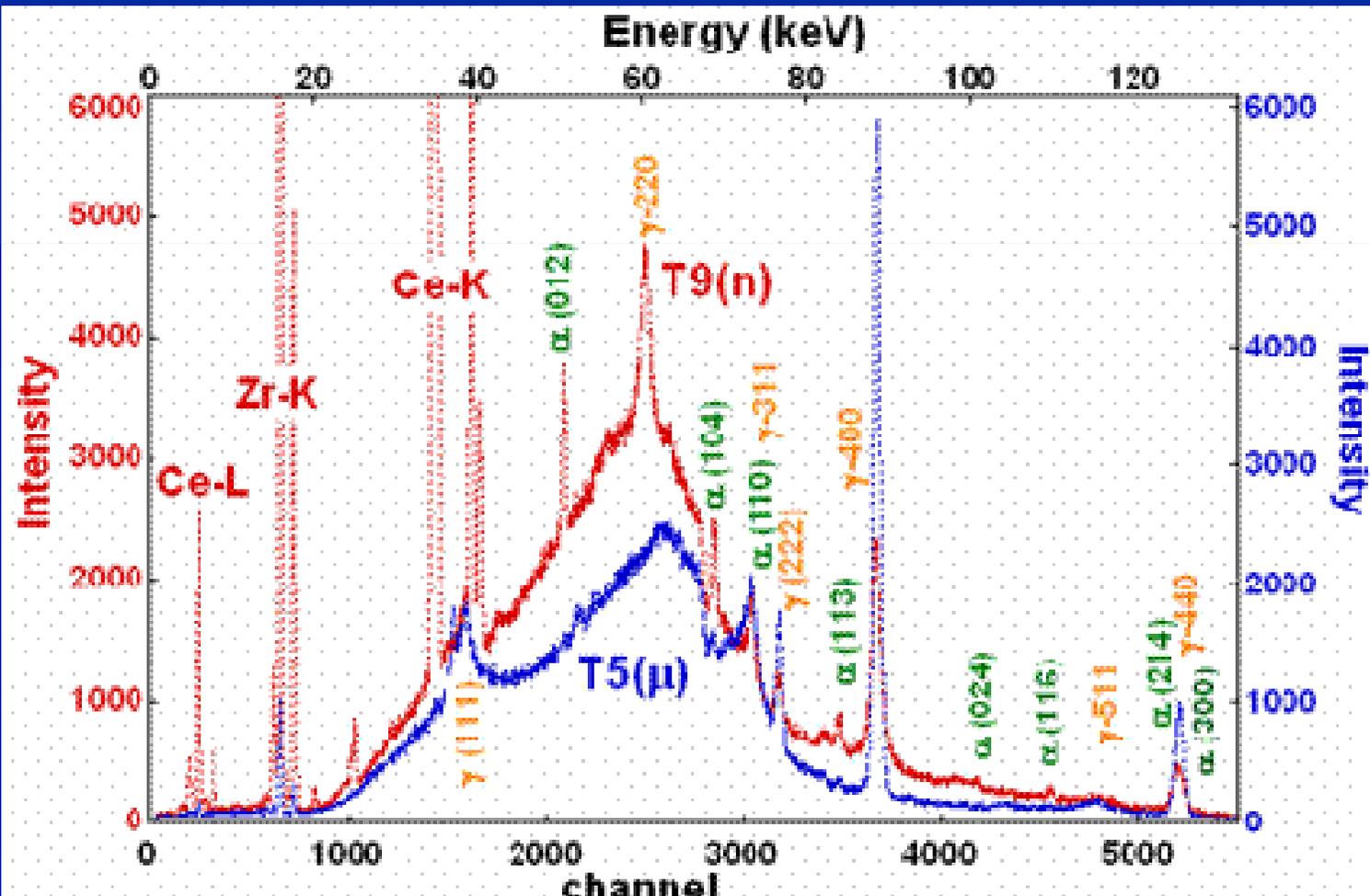
# Phase Mapping

A series of EDXRD spectra as a function of ( $x_3$ ) depth for a nano-alumina-titania coating on a Ti substrate. Note that the spectra are displaced equally so that the coding region can be seen. The Miller indices for the Ti (hcp) substrate and the spinel structure phase in the coating are indicated.



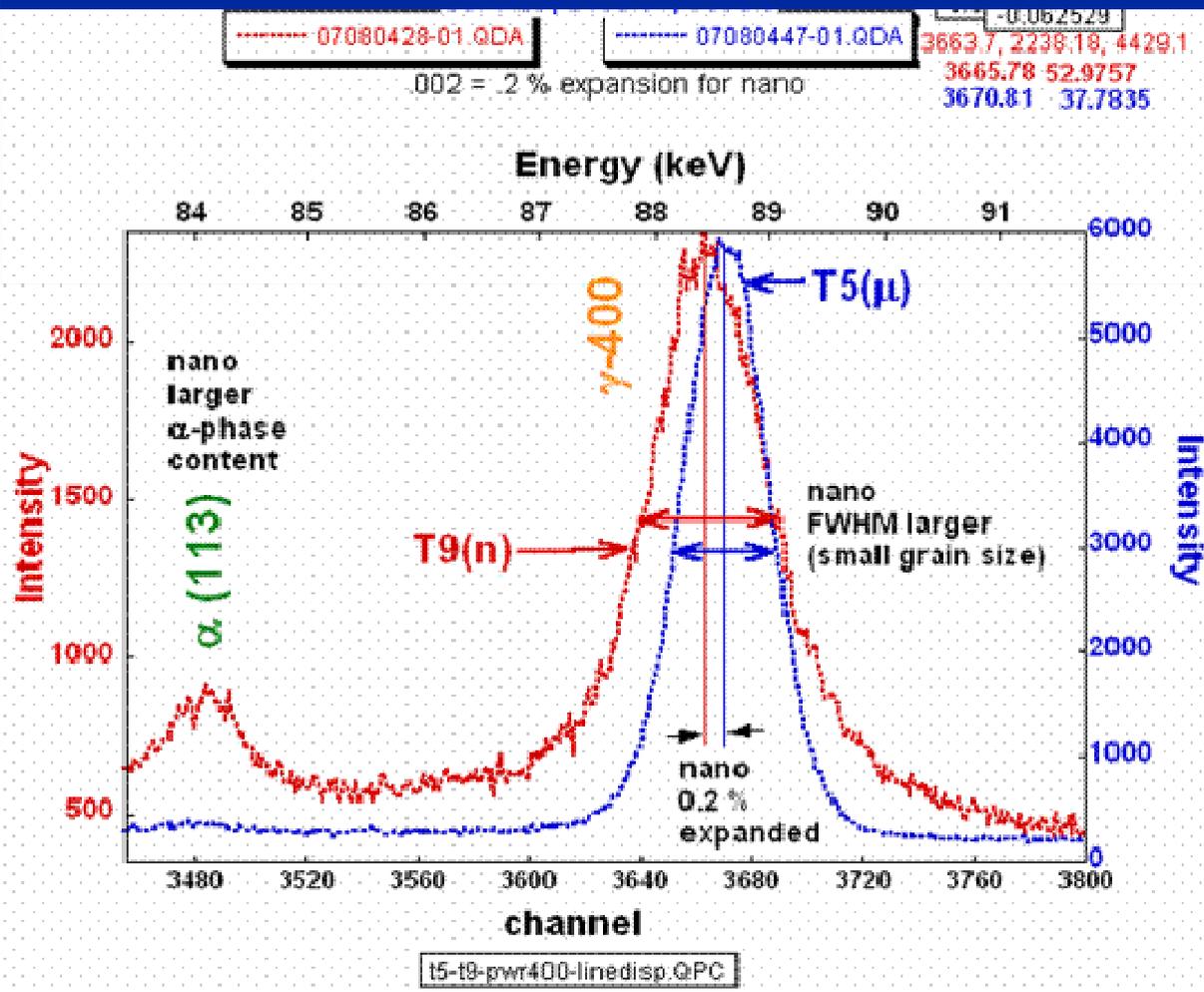
# Phase Mapping

Wide view of EDXRD patterns of powders obtained by delaminating TS titania-alumina coatings under 4-point compression and then grinding the coating into powder. The T5 is a typical thickness micro-coating and T9 is a typical thickness nano-coating. Note: the  $\gamma$ - $\text{Al}_2\text{O}_3$  spinel and  $\alpha$ - $\text{Al}_2\text{O}_3$  corundum Bragg line indexing; the expected Ce atomic fluorescence lines in the nano material; .



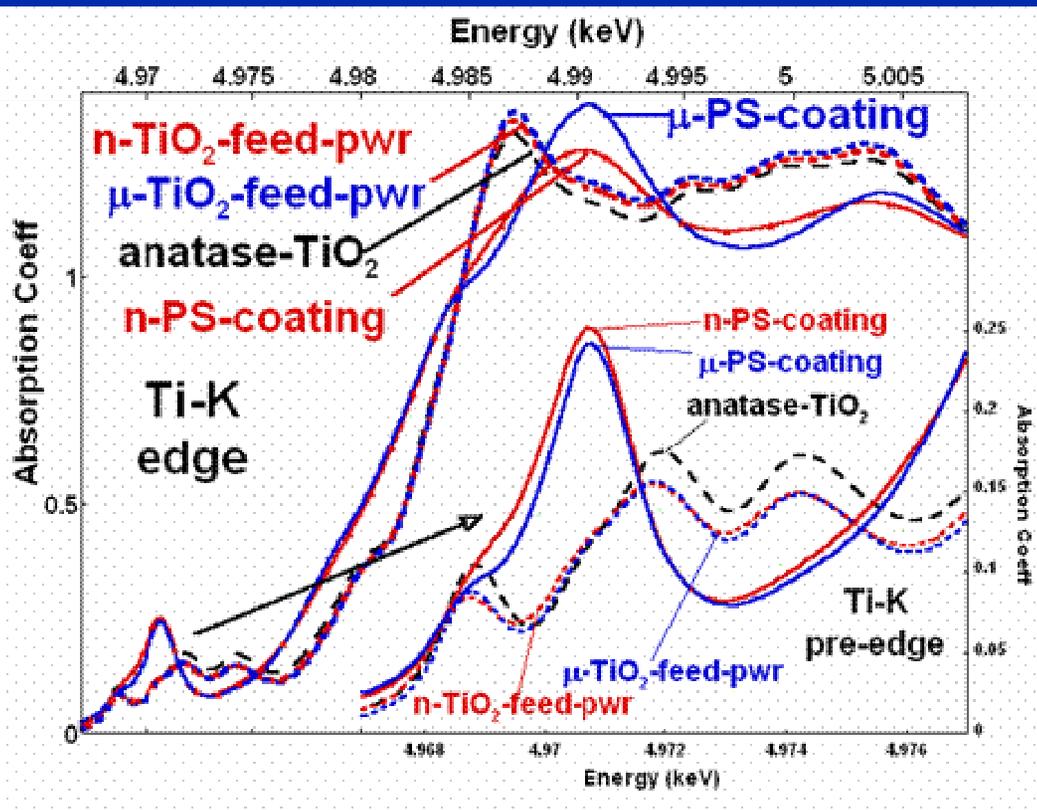
# Phase Mapping

The  $\gamma\text{-Al}_2\text{O}_3$  spinel  $\gamma$ -400 Bragg line for the T5(micro)- and T9(nano)-coating powders. This is the line used to evaluate the strain variations in the coatings. Note: the strong broadening of the small grain size nano material line relative to the micro material; the nano- material is dilatation of the lattice parameter by +0.2% relative to the micro-material; the prominent  $\alpha\text{-Al}_2\text{O}_3$  corundum phase Bragg line in the amorphous phase.



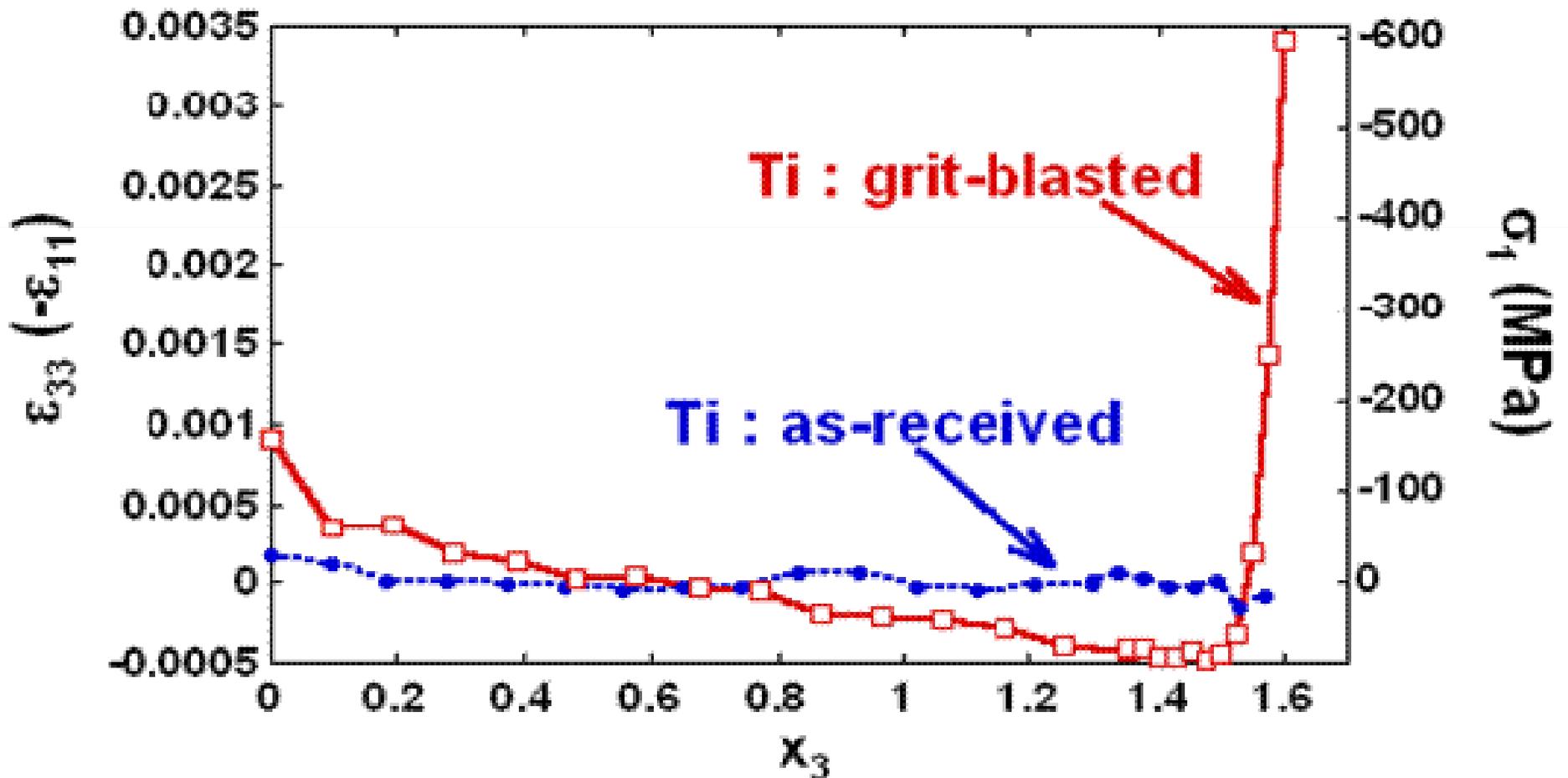
# Phase Mapping

Ti-K edge XAS near edge structures for a series of Ti compounds. The pre-edge features are shown on an expanded scale in the inset of the figure. Comparison of the n- and  $\mu$ -feed powder spectra are to the anatase phase  $\text{TiO}_2$  standard clearly confirm this as the the feed powder phase in both cases. The Ti environment in the plasma sprayed (PS) n- and  $\mu$ - coatings are dramatically different and are unambiguously not anatase phase. Note that the FS oscillations above the edge are sharper for the  $\mu$ -PS-coating (relative to the  $\mu$ -PS-coating). The broadening of the  $\mu$ -PS-coating FS features are typical of a more disordered local environment as expected for nano-phase materials.



$\text{TiO}_x$ ,  $2 > x > 1.5$   
Chemically reduced Ti.

A comparison plot of the  $\epsilon_{33}$  strain in as-received titanium substrate, and grit blasted titanium substrate. It can be noted that in the grit blasted sample surface compression introduces a bending moment in the other underlying bulk material which is clearly visible by the sloping data between  $0 < x_3 < 1.5$

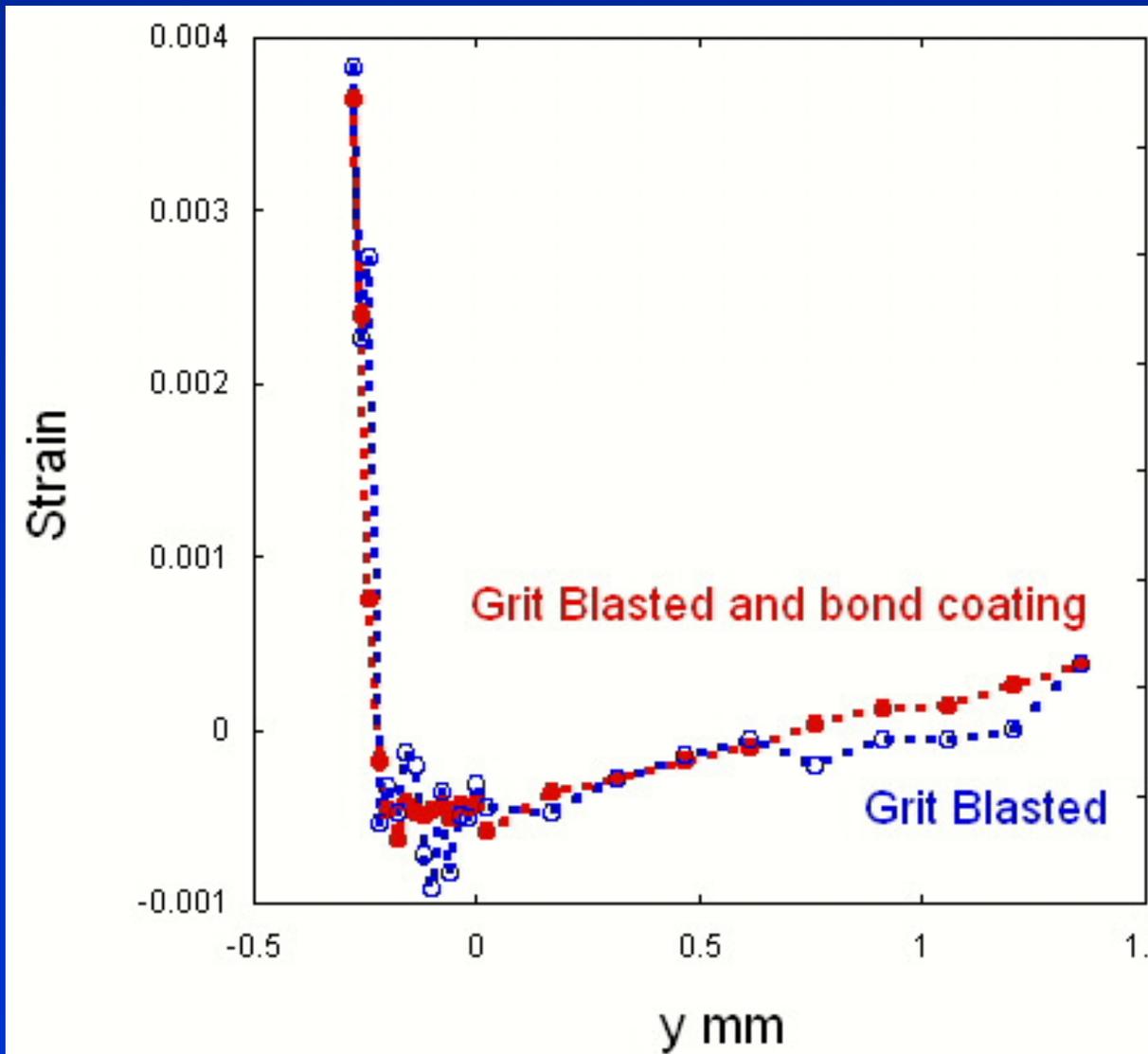




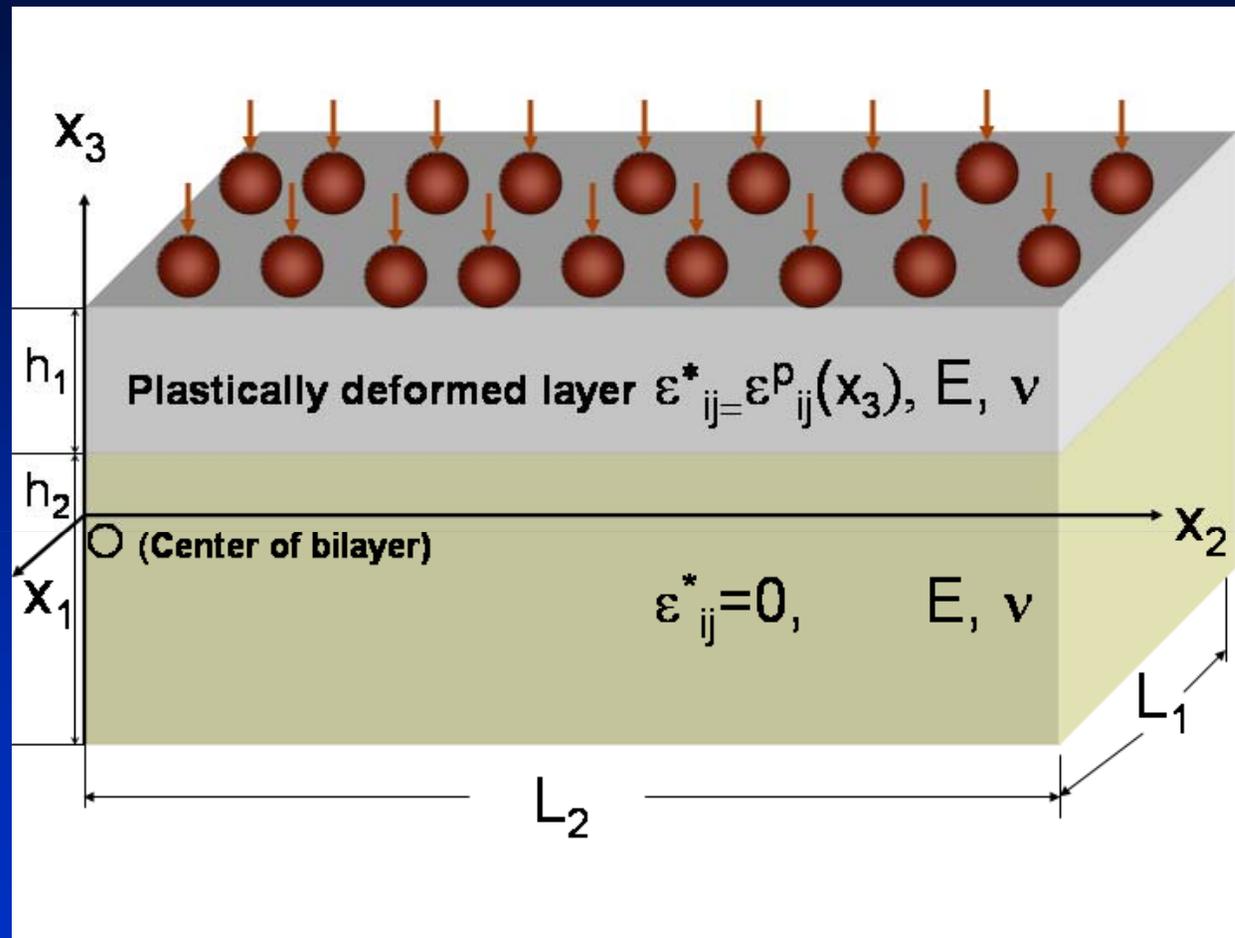
# Strain Mapping



Effect of bond coat on compressive strain  $\epsilon_{33}$  of Grit Blasted Ti

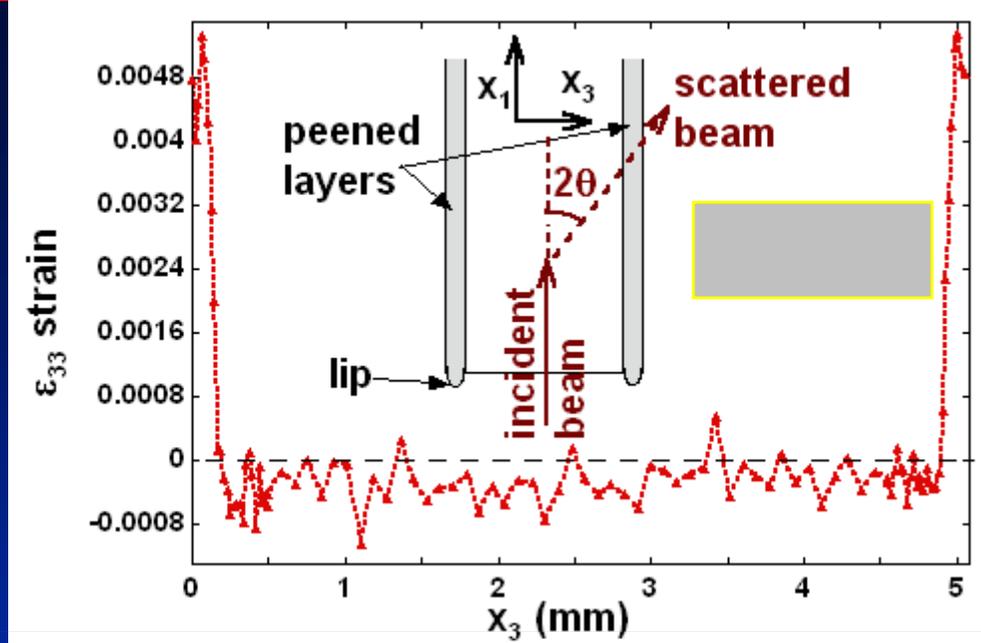


## VALIDATION 2. Grit Blasted

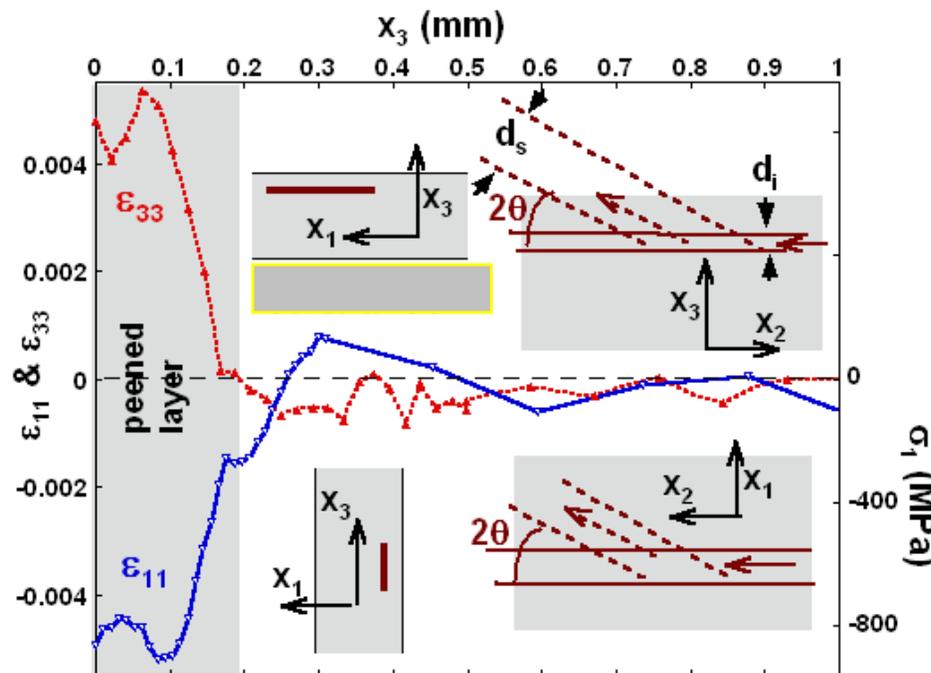


**Schematic of model for Grit Blasted  
strains/stresses**

# EDXRD Grit Blasted; Steep strain gradients; High Resolution



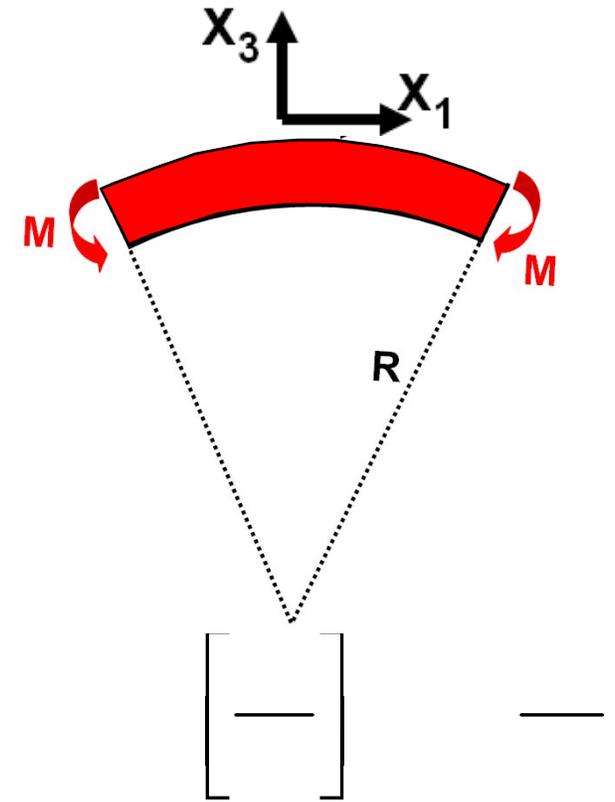
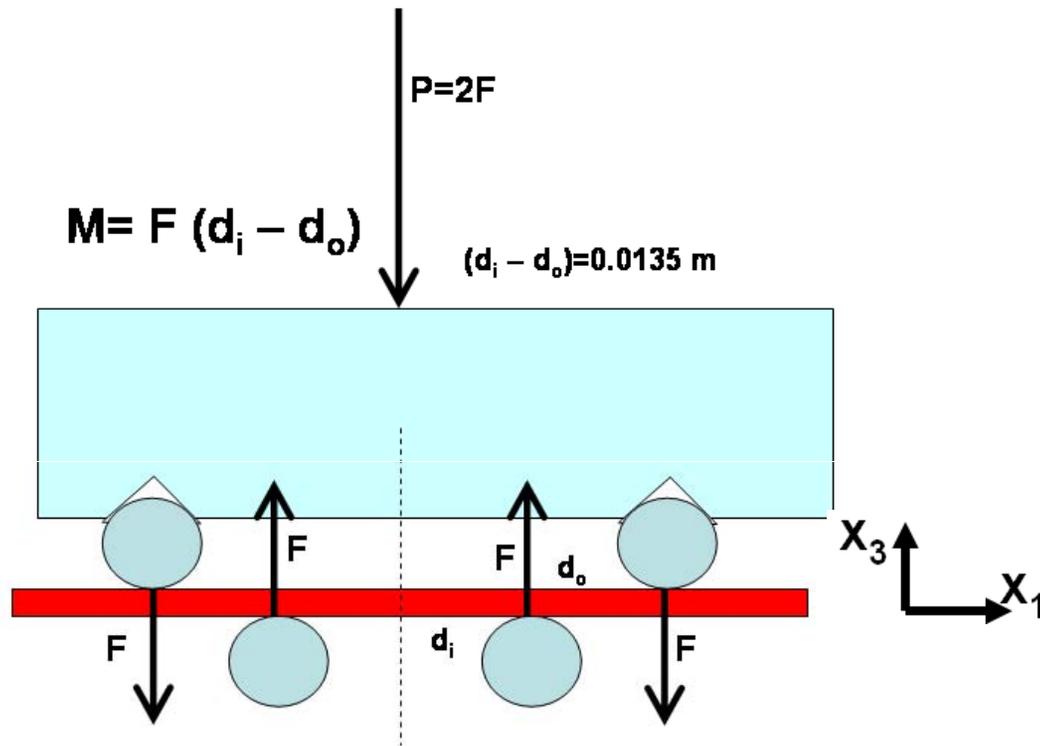
Strain profile of  $\epsilon_{33}$  across the entire thickness of a Ti double-sided shot peened specimen. The inset shows schematic of the x-ray scattering geometry along with the definition of the coordinate directions. Note the schematic representation of the lip which was optically profiled in Figure above.



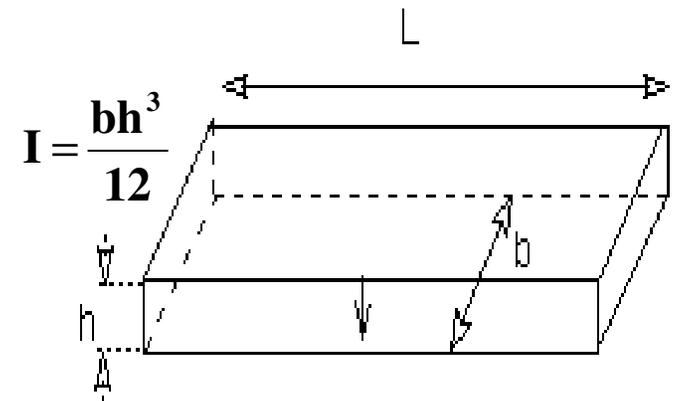
## VALIDATION 2.

The strain profiles of  $\epsilon_{33}$  and  $\epsilon_{11}$  in the vicinity of the peened surface layer and the underlying bulk material of the Ti specimen. The insets illustrate the x-ray scattering geometries for the  $\epsilon_{33}$  (top) and  $\epsilon_{11}$  (bottom) measurements. Note the stress scale (lower right) uses  $E=118$  GPa and  $\nu=0.33$ .  $\epsilon_{33} = -2\nu/(1-\nu) \sim -\epsilon_{11}$

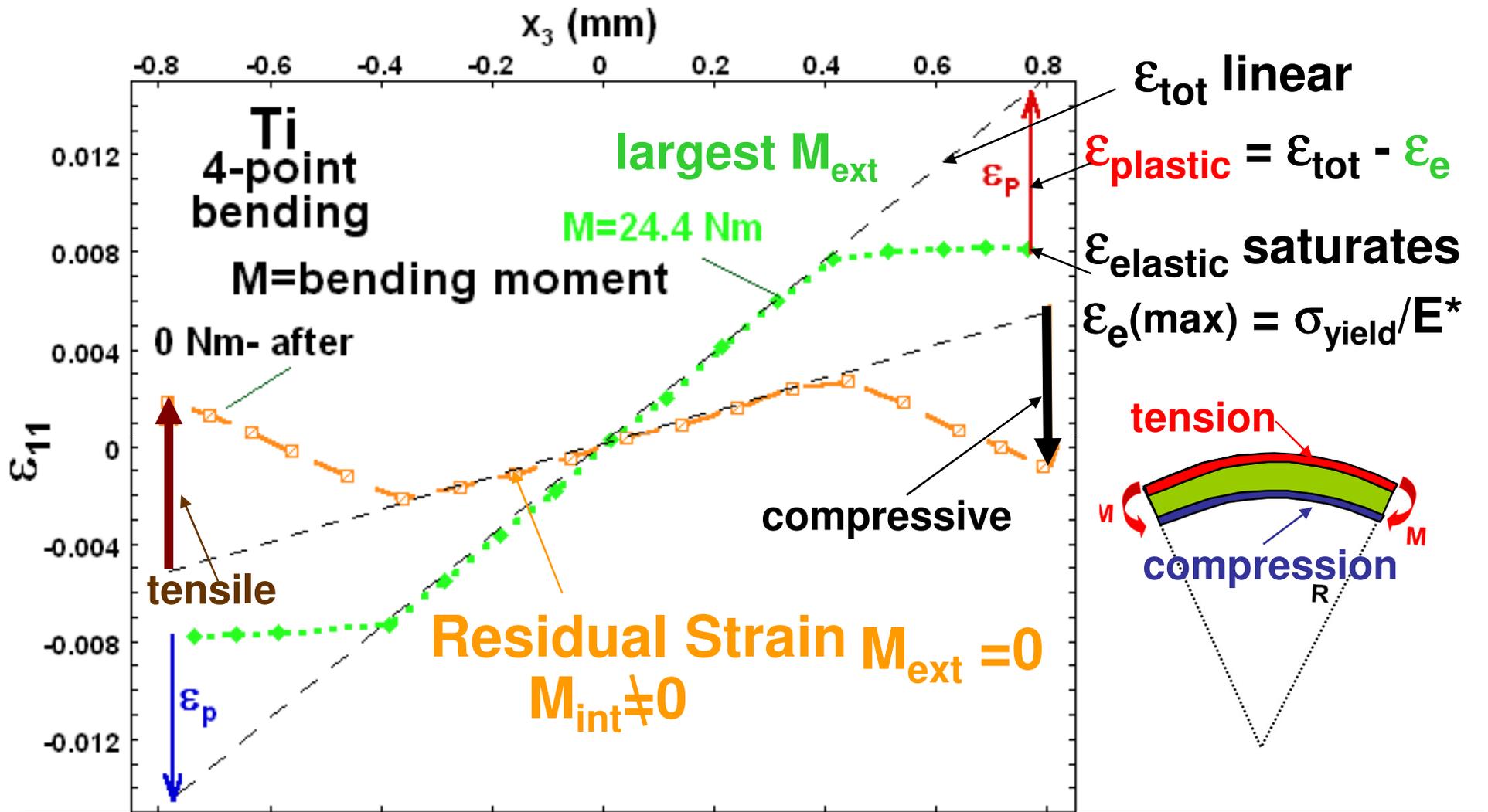
# In-situ 4-point bending experiment Ti

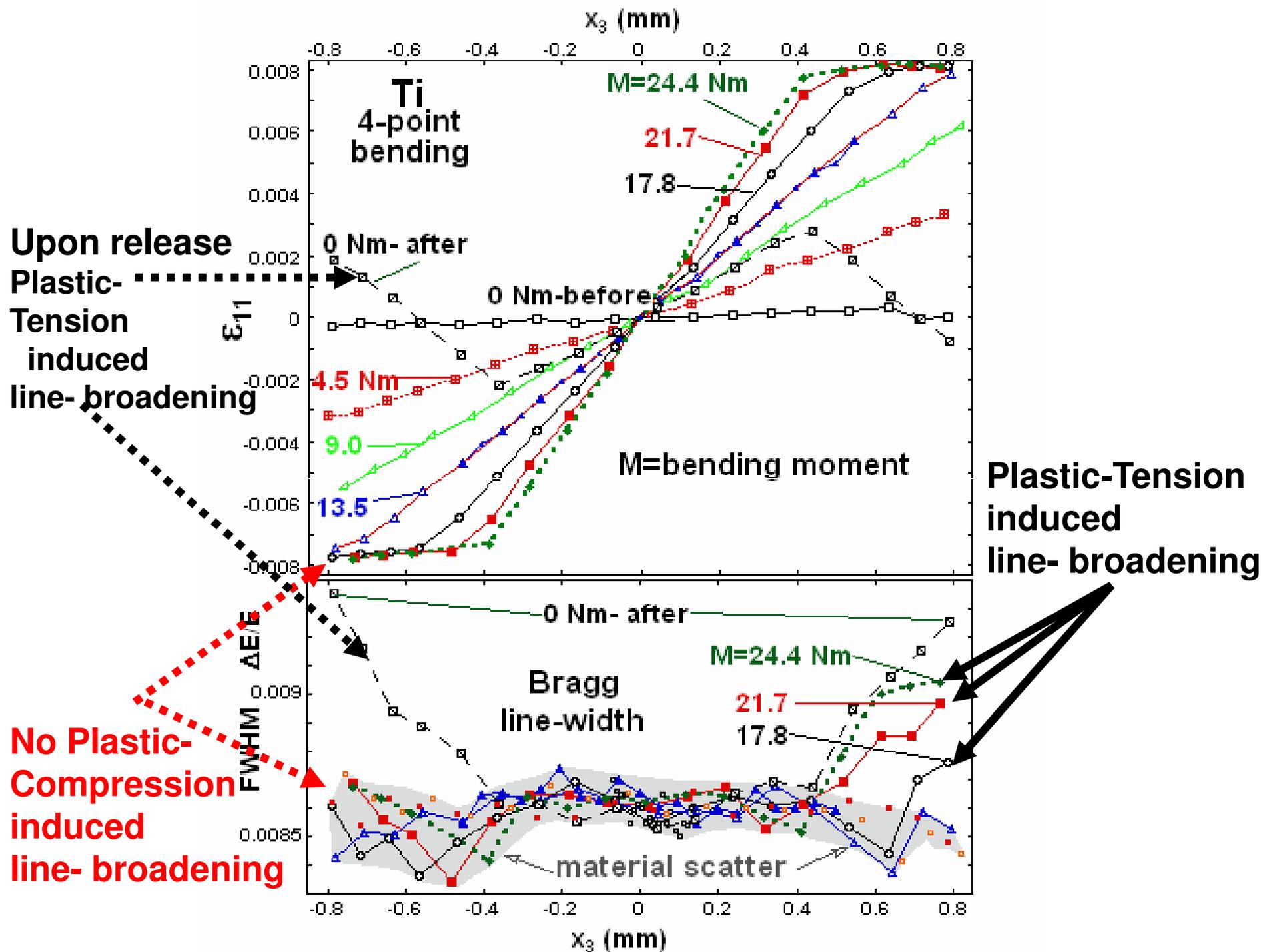


**Linear depth variation**

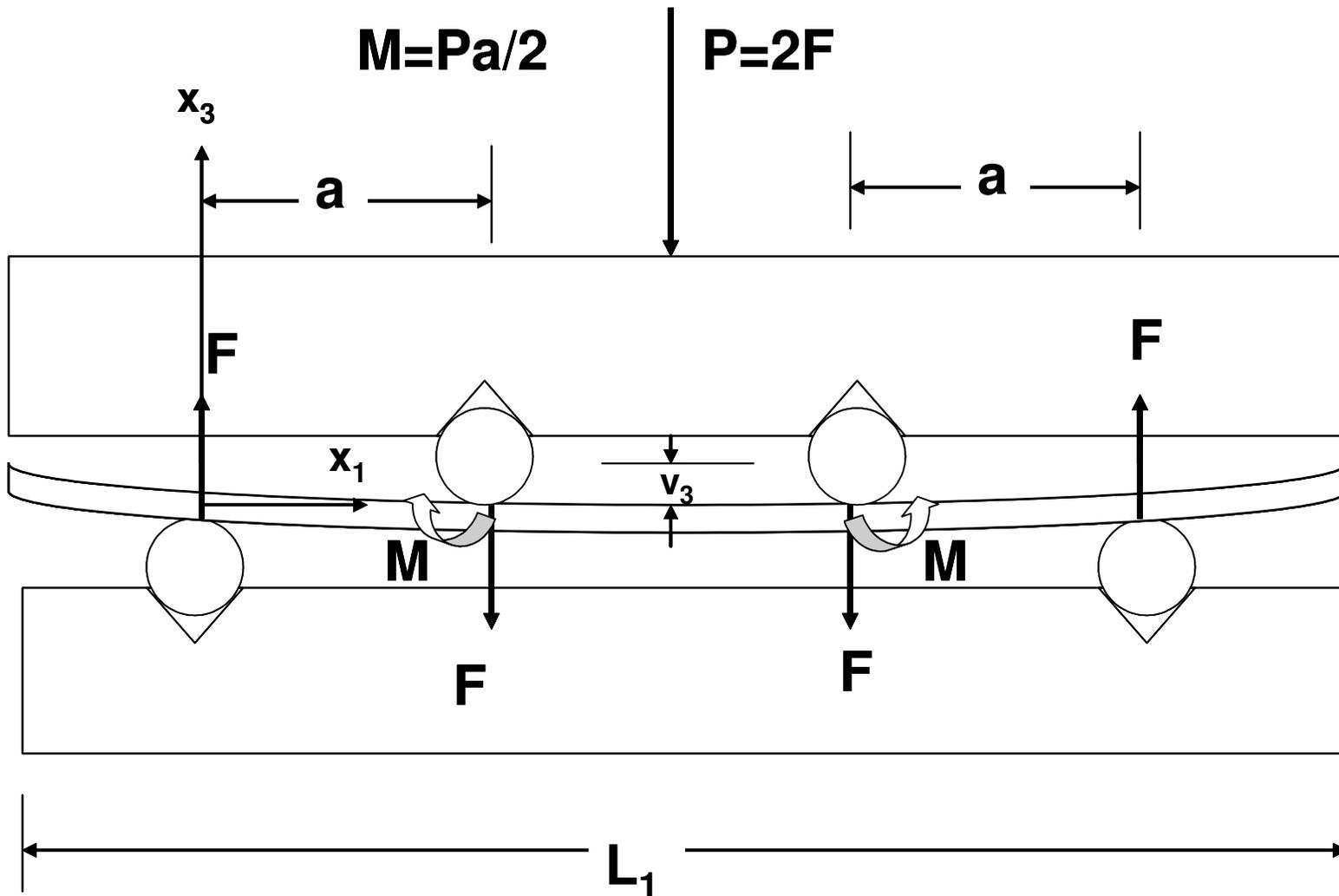


# Bending released: Residual stresses and bending





## Four-point Bending of substrate and coatings plates





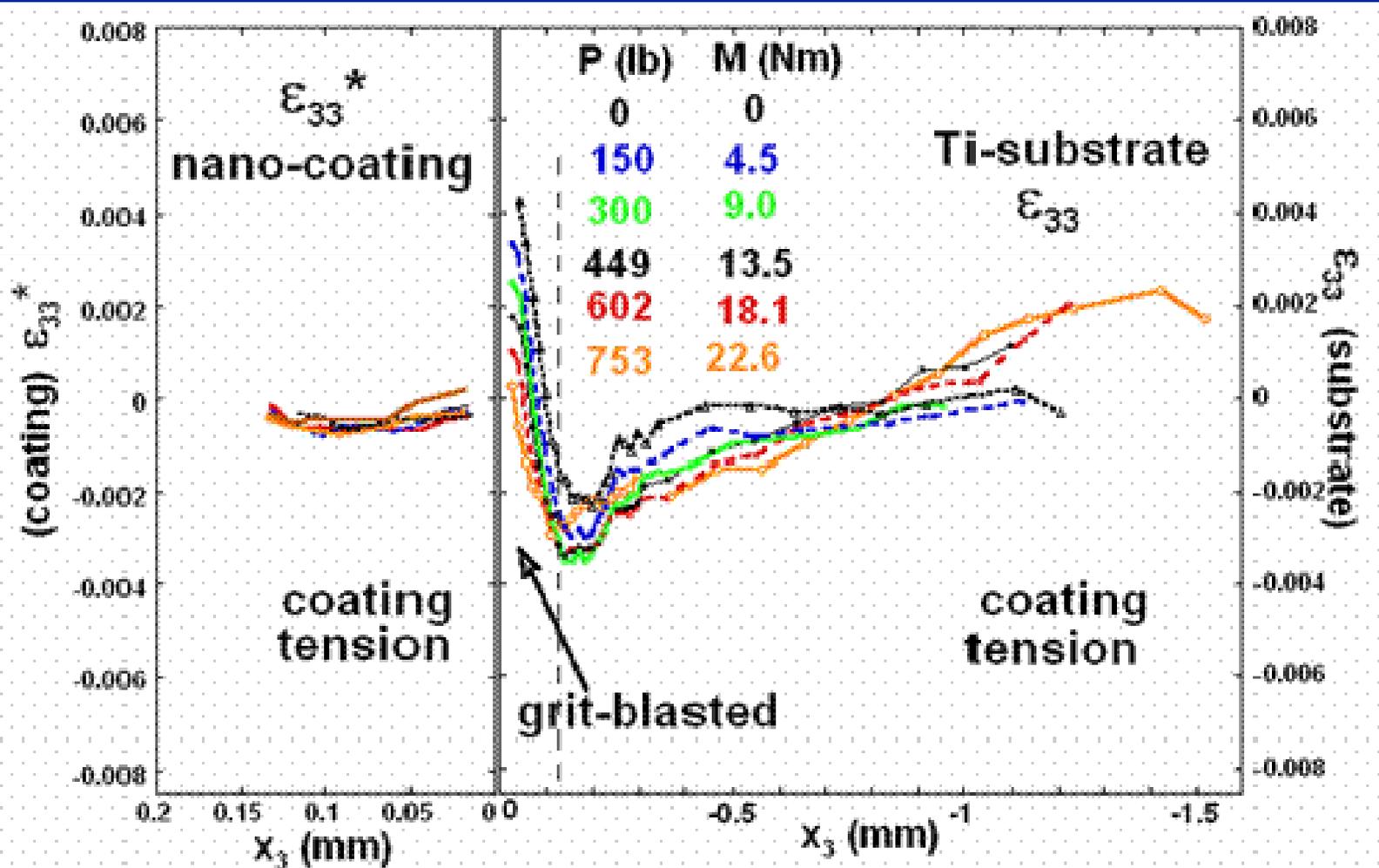
# Strain Mapping

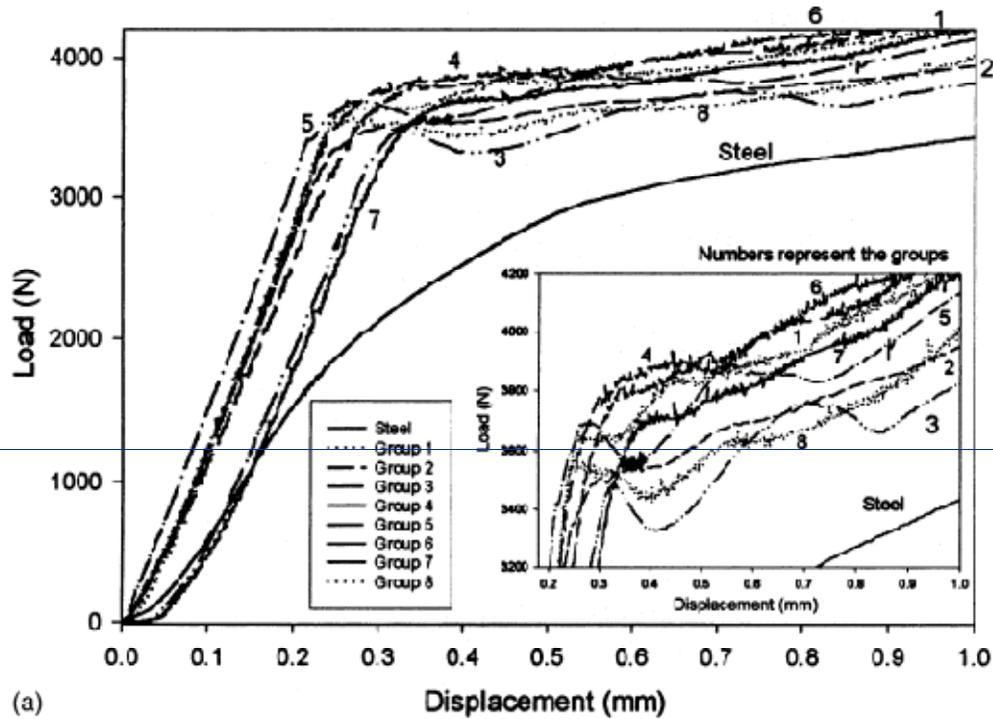


$\epsilon_{11}$  depth ( $x_3$ ) variation profiles of of DLC/4142 steel substrate test specimen at various load levels in the four point bending geometry

# Phase Mapping

$\epsilon_{33}$  depth ( $x_3$ ) variation profiles of alumina-titania-coating/Ti-substrate test specimen at various load levels in the four point bending geometry.





(a)

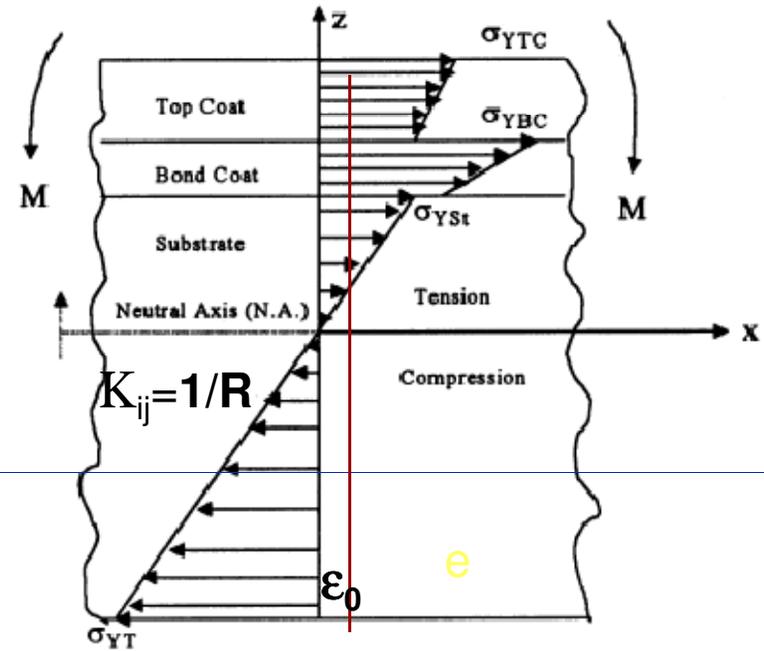
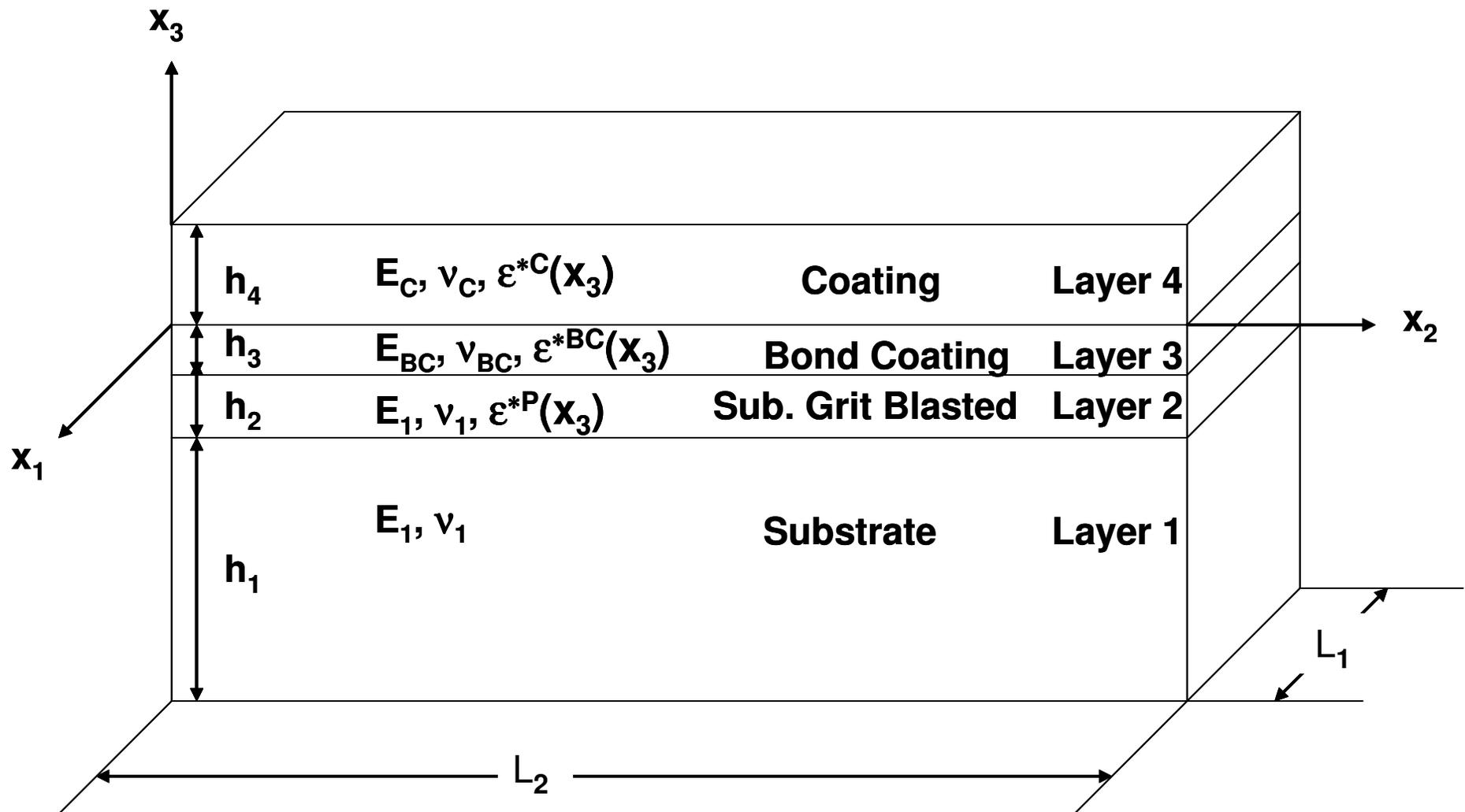
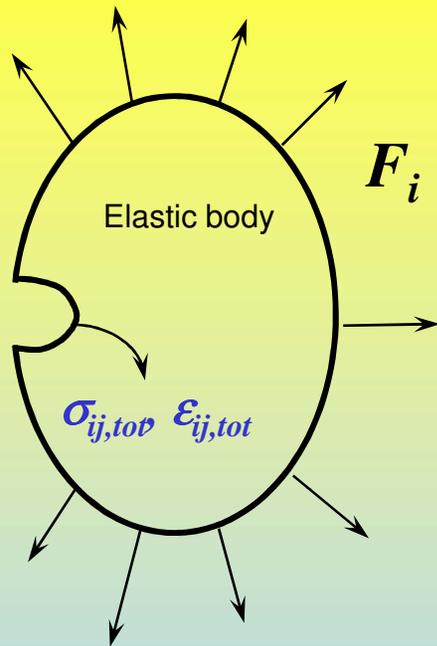


Fig. 2. Schematic illustration of the bending stress in the coating under four point bend test.



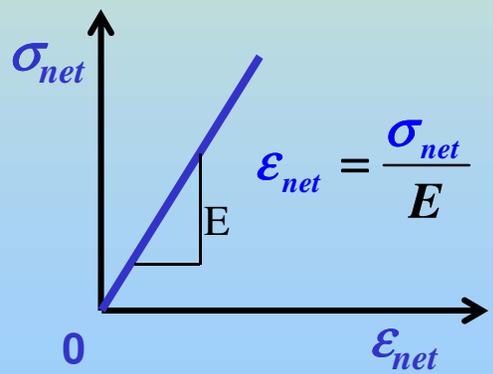
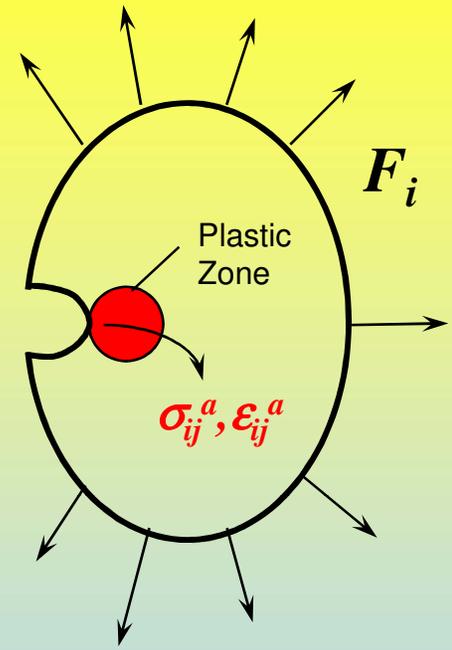
Schematic of the layered stack structure of the substrate/coating materials systems used in this study

# stresses and strain –the elasto-plastic analysis Eigenstrain



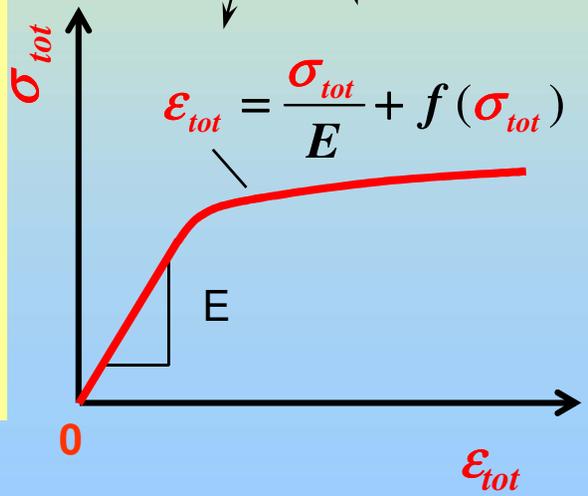
Multiaxial stress state

$$\begin{cases} \sigma_{ij,net}^e \epsilon_{ij,net}^e = \sigma_{ij,tot}^a \epsilon_{ij,tot}^a \\ \epsilon_{ij,tot}^a = f(\sigma_{ij,tot}^a) \end{cases}$$



Uniaxial stress state

$$\begin{cases} \sigma_{22,net}^e \epsilon_{22,net}^e = \sigma_{22,tot}^a \epsilon_{22,tot}^a \\ \epsilon_{22,tot}^a = \frac{\sigma_{22,tot}^a}{E} + \left( \frac{\sigma_{22,tot}^a}{K'} \right)^{\frac{1}{n'}} \end{cases}$$



## Equations of the multiaxial stresses and Hencky's equations of plasticity

$$\left\{ \begin{array}{l} \varepsilon_{11}^a = \frac{1}{E} \left[ \sigma_{11}^a - \nu (\sigma_{22}^a + \sigma_{33}^a) \right] - \frac{f(\sigma_{eq}^a)}{\sigma_{eq}^a} \left[ \sigma_{11}^a - \frac{1}{2} (\sigma_{22}^a + \sigma_{33}^a) \right] \\ \varepsilon_{22}^a = \frac{1}{E} \left[ \sigma_{22}^a - \nu (\sigma_{33}^a + \sigma_{11}^a) \right] - \frac{f(\sigma_{eq}^a)}{\sigma_{eq}^a} \left[ \sigma_{22}^a - \frac{1}{2} (\sigma_{33}^a + \sigma_{11}^a) \right] \\ \varepsilon_{33}^a = \frac{1}{E} \left[ \sigma_{33}^a - \nu (\sigma_{11}^a + \sigma_{22}^a) \right] - \frac{f(\sigma_{eq}^a)}{\sigma_{eq}^a} \left[ \sigma_{33}^a - \frac{1}{2} (\sigma_{11}^a + \sigma_{22}^a) \right] \\ \sigma_{11}^e \varepsilon_{11}^e = \sigma_{11}^a \varepsilon_{11}^a \\ \sigma_{22}^e \varepsilon_{22}^e = \sigma_{22}^a \varepsilon_{22}^a \\ \sigma_{33}^e \varepsilon_{33}^e = \sigma_{33}^a \varepsilon_{33}^a \end{array} \right.$$

where :  $f(\sigma_{eq}^a) = \left( \frac{\sigma_{eq}^a}{K'} \right)^{\frac{1}{n'}}$

$[A]$  ,  $[B]$  and  $[D]$  are the elastic stiffness matrices derived by Tsai(1988):

$$[A] = \int_h [C(x_3)] dx_3, [B] = \int_h [C(x_3)] x_3 dx_3 \text{ and } [D] = \int_h [C(x_3)] x_3^2 dx_3 \quad (10)$$

$\vec{N}^*$  and  $\vec{M}^*$  are the forces and moments respectively, generated by the eigenstrains  $\epsilon_{ij}^*(x_3)$

$$\vec{N}^* = \int_h [C(x_3)] \vec{\epsilon}^*(x_3) dx_3 \quad \text{and} \quad \vec{M}^* = \int_h [C(x_3)] \vec{\epsilon}^*(x_3) x_3 dx_3 \quad (11)$$

From (8) and (9) we obtain:

$$\begin{pmatrix} \vec{N} + \vec{N}^* \\ \vec{M} + \vec{M}^* \end{pmatrix} = \begin{bmatrix} [A] & [B] \\ [B] & [D] \end{bmatrix} \begin{pmatrix} \vec{\epsilon}^0 \\ \vec{k} \end{pmatrix} = [C'] \begin{pmatrix} \vec{\epsilon}^0 \\ \vec{k} \end{pmatrix} \quad (12)$$

Eq. (12) depicts 3 algebraic equations for  $\vec{N}$  and three for  $\vec{M}$  totaling 6 equations.

By inverting the matrix of these equation we solve with respect to the 6 components of  $\epsilon_{ij}^0$  and  $\kappa_{ij}$  :

$$\begin{pmatrix} \vec{\epsilon}^0 \\ \vec{k} \end{pmatrix} = [C']^{-1} \begin{pmatrix} \vec{N} + \vec{N}^* \\ \vec{M} + \vec{M}^* \end{pmatrix} \quad \leftarrow$$



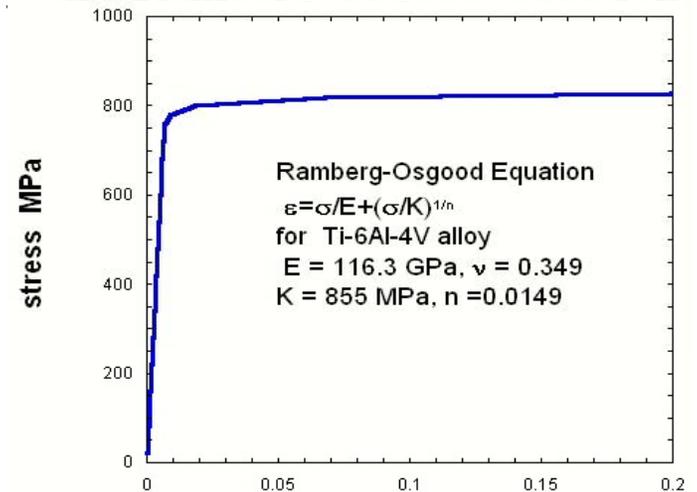
# Modulus and yield stress



The material properties used in this analysis are  $E = 116.3$  GPa,  $\nu = 0.349$ ,  $K = 855$  MPa, and  $n = 0.0149$

The stress strain curve for Ti-6Al-4V alloy expressed by the Ramberg-Osgood equation:

$$\epsilon = \frac{\sigma}{E} + \left( \frac{\sigma}{K} \right)^{\frac{1}{n}}$$
 is depicted in Figure 8.



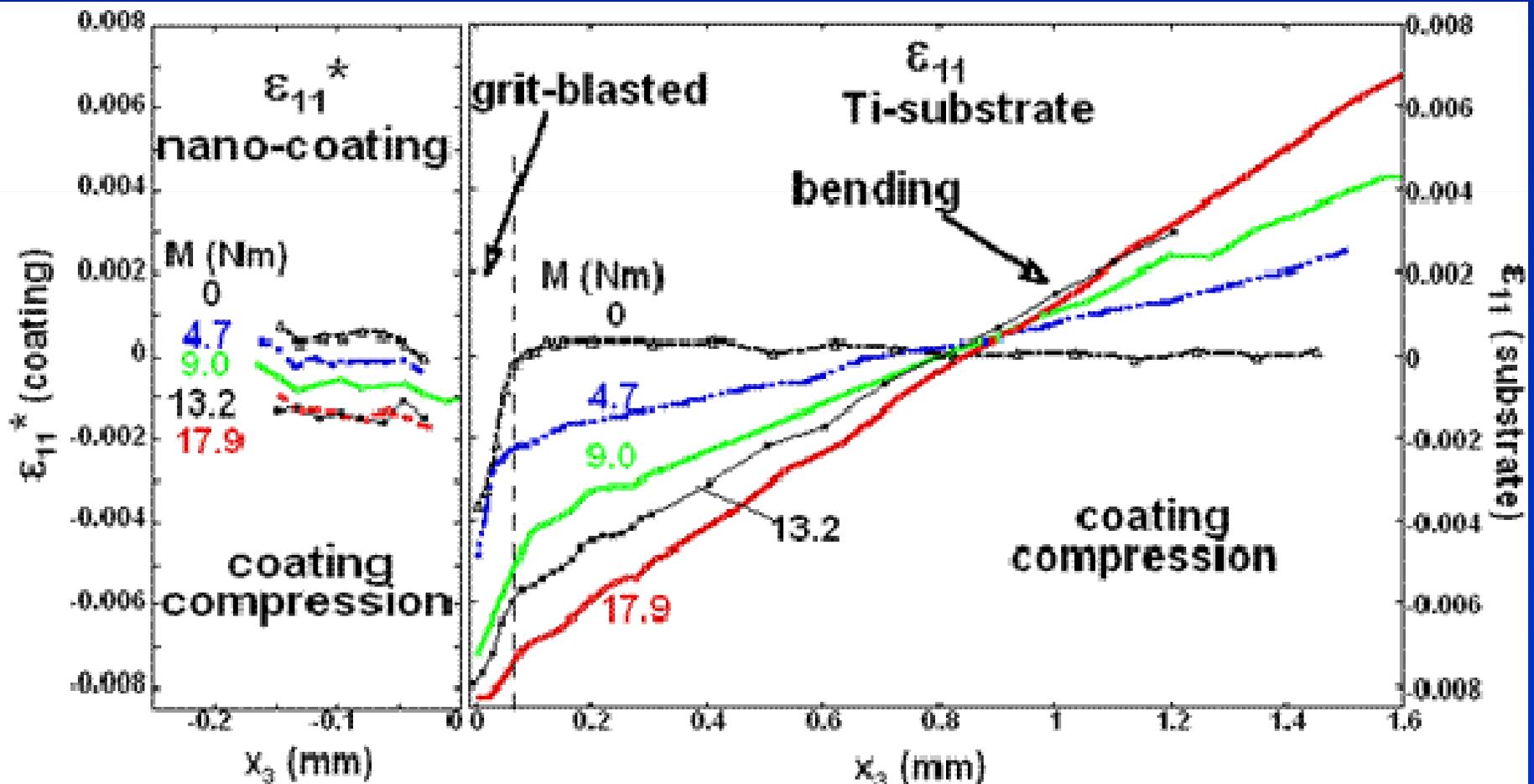
Experimental results from slopes of Bending versus strain curves 4-point bending experiments the Elastic Modulus of Ti-6Al-4V substrate.

$E' = 137$  GPa ,  $E = 117 \pm 3$  GPa and yield stress  $\sigma_y = 843$  MPa

Literature:  $E = 116.3 \pm 3$  GPa,  $\sigma_y = 830$  MPa  $E_{\text{coating}}$  and  $K_{Ic}$   
Fracture toughness, Tensile strength of Coating  $\sim 0$ ,

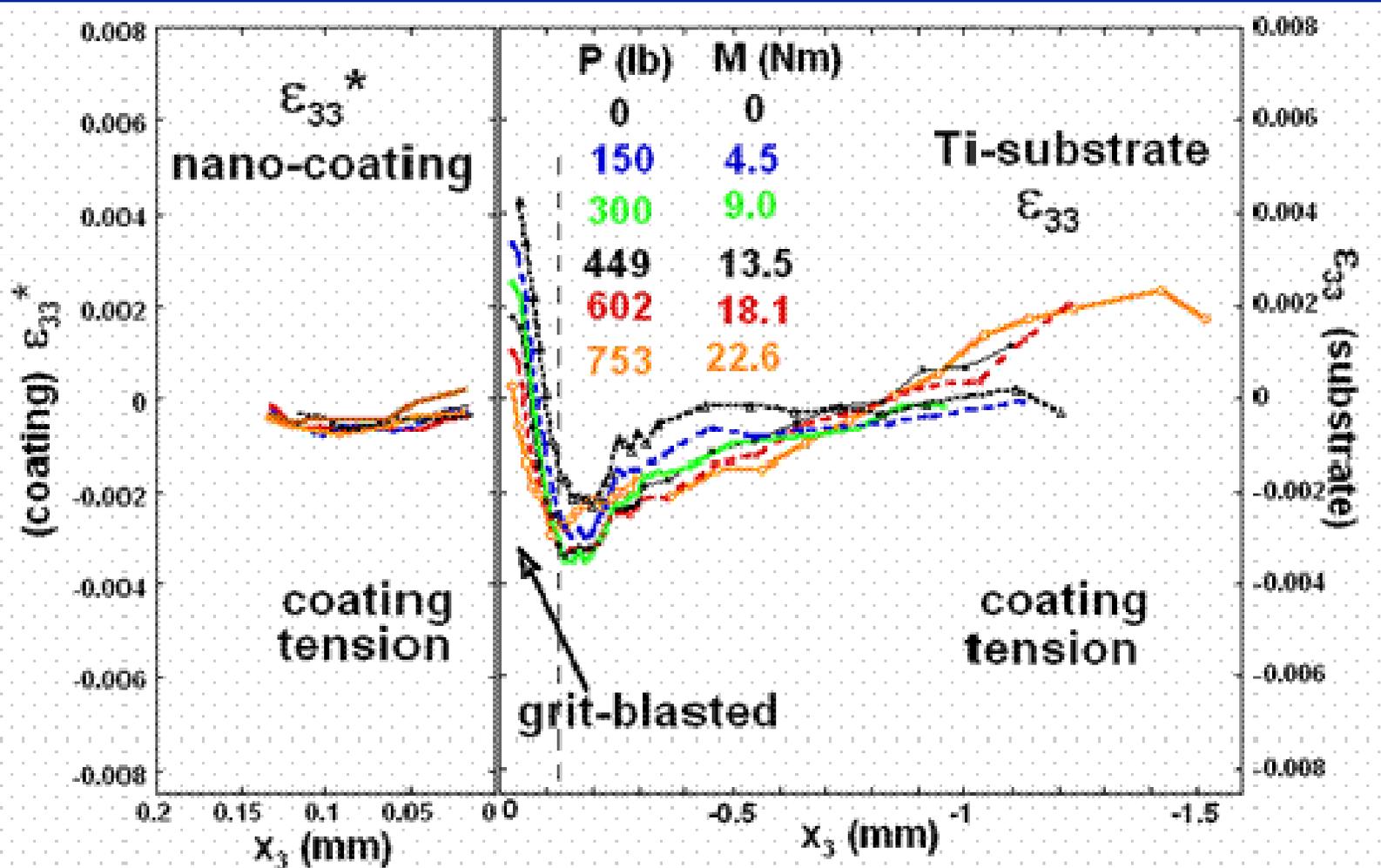
# Strain Mapping

$\epsilon_{11}$  depth ( $x_3$ ) variation profiles of alumina-titania-coating/Ti-substrate test specimen at various load levels in the four point bending geometry



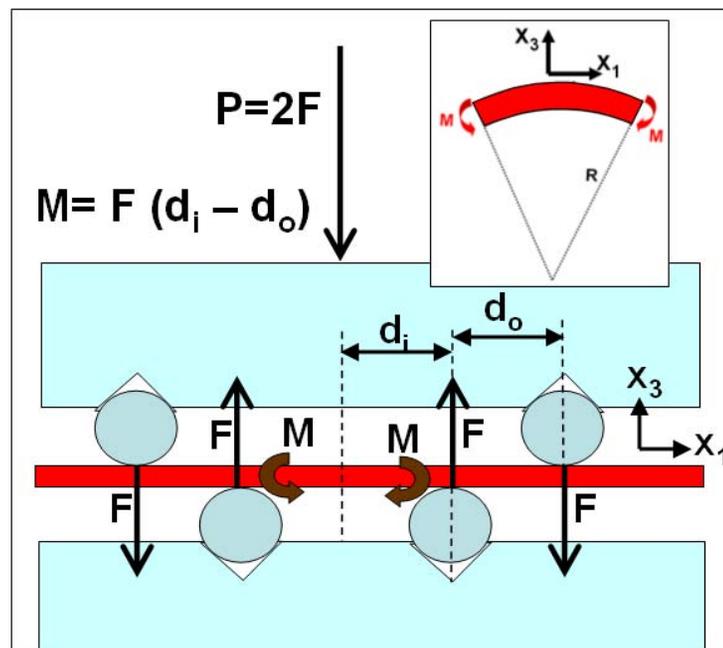
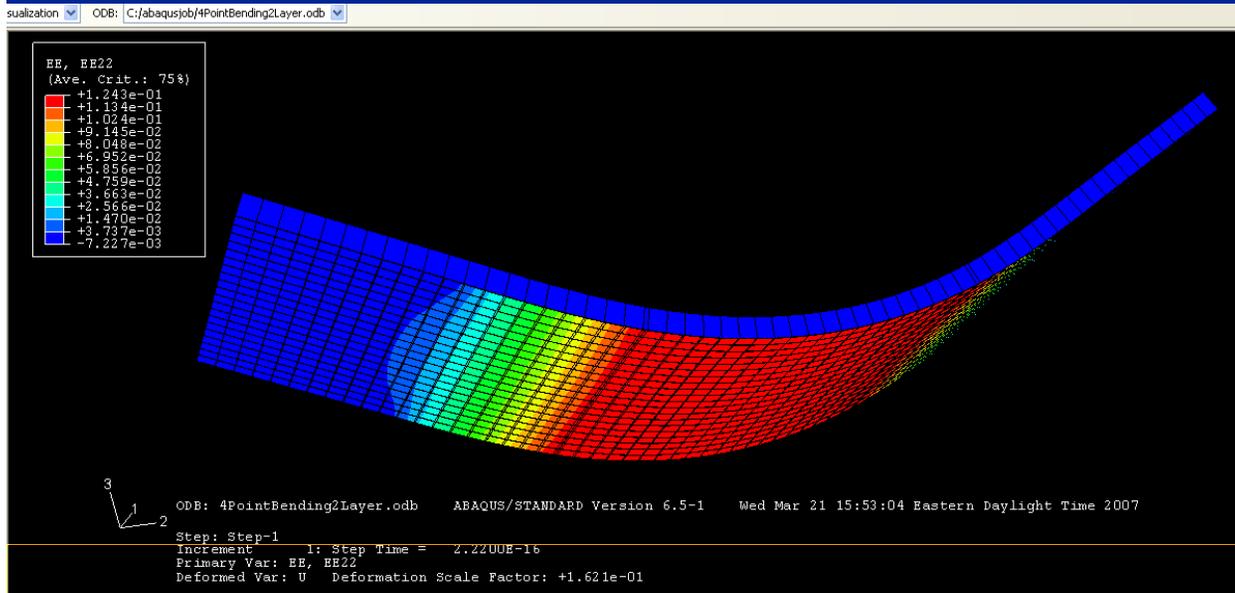
# Phase Mapping

$\epsilon_{33}$  depth ( $x_3$ ) variation profiles of alumina-titania-coating/Ti-substrate test specimen at various load levels in the four point bending geometry.





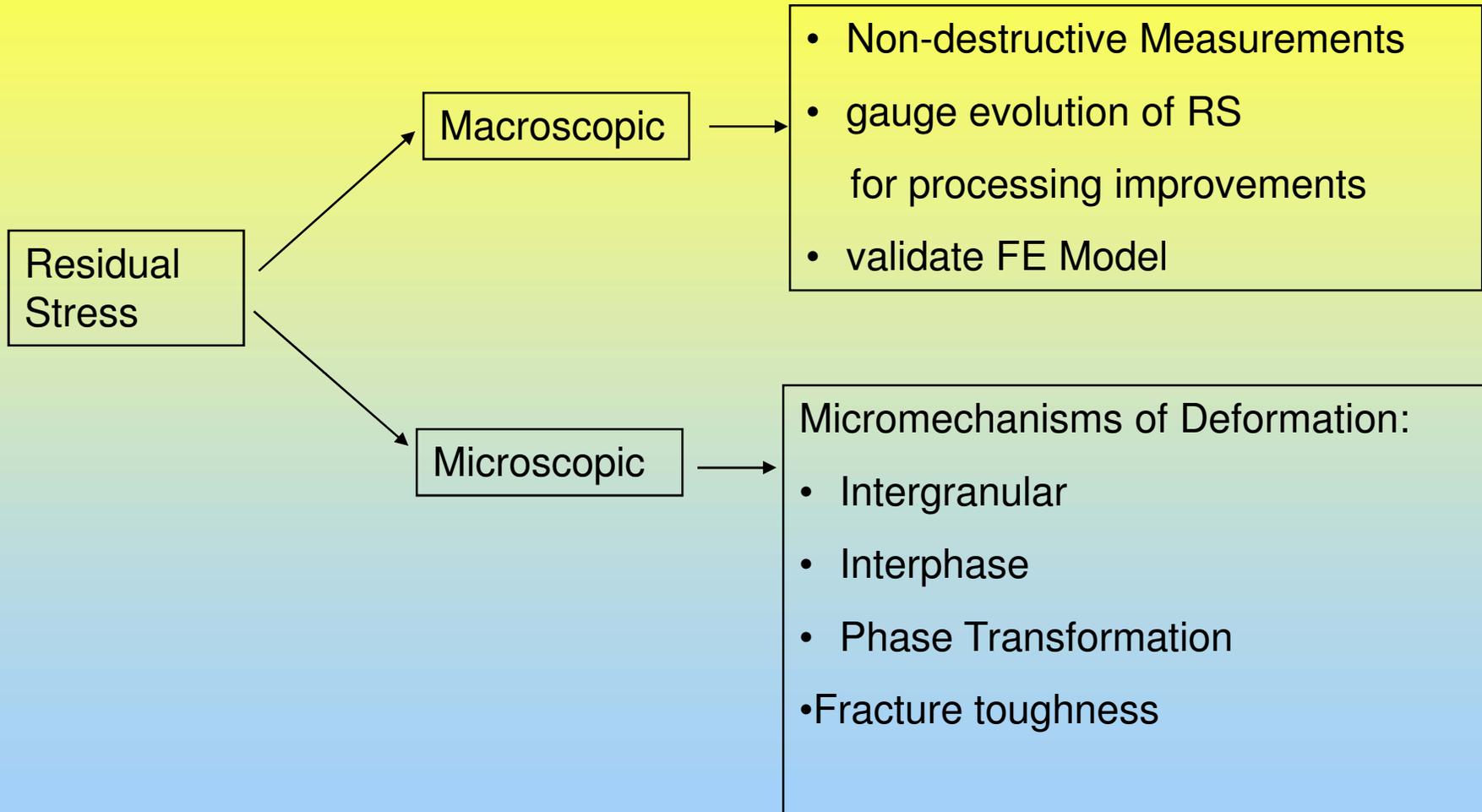
# Modeling and Theory



Result Options... Contour Options...

Willi... 4:01 PM

## Energy Dispersive X-ray Diffraction

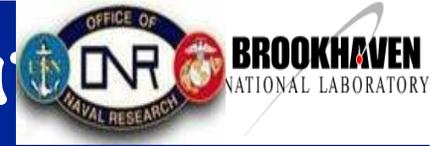


**Thank you.**

**Questions ?**



# A look to the future at EDXRD Residual Stress



- ← In-situ Synchrotron triaxial strain measurements
- ← Anisotropic materials
- ← Small strain systems
- ← Real time studies
- ← Small diffraction volumes (e.g. gradients, buried interfaces, grain boundaries,)
- ← Line Broadening studies
  
- ← Email [tsakalak@rci.rutgers.edu](mailto:tsakalak@rci.rutgers.edu)



# EDXRD Capabilities



## Two themes (0-300 KeV photons)

- a) High resolution Energy Dispersive X-ray Diffraction (EDXRD) 1  $\mu\text{m}$  to 1 cm
- b) High Energy EDXRD :Penetration depth  $\sim$  2 inches of steel and 2 ft of Al

## Two strategies

- a) Strain Mapping (Internal/Residual Stress Spatial Distributions)
- b) Phase Mapping ( Spatial Distributions of Phases Stresses in each phase)

## Three Scales:

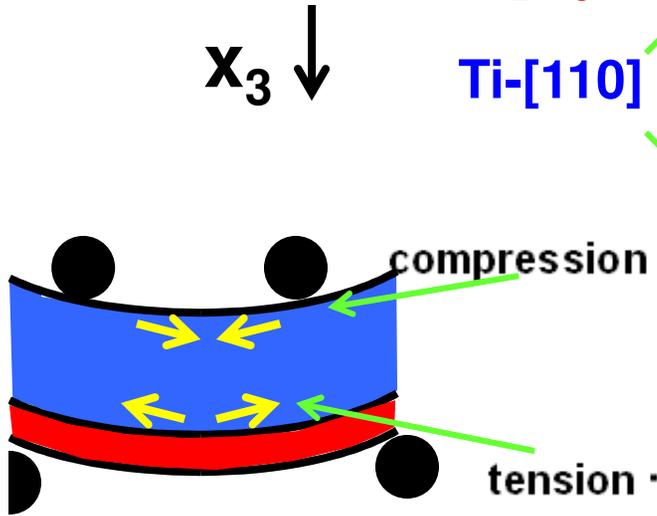
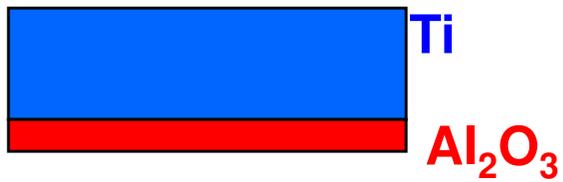
- a) Nanometer scale 0.1 to 100 nm (Line broadening EDXRD is best.
- b) Mesoscopic scale 100 nm to 5  $\mu\text{m}$  (individual grains, particles etc)
- c) Macroscopic scale 5  $\mu\text{m}$  to 10-100 mm

**In situ Load/stress application** ( tensile, compressive, three point bending, etc load during measurement)

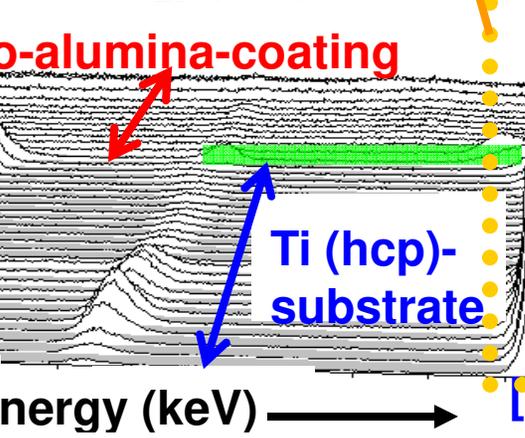
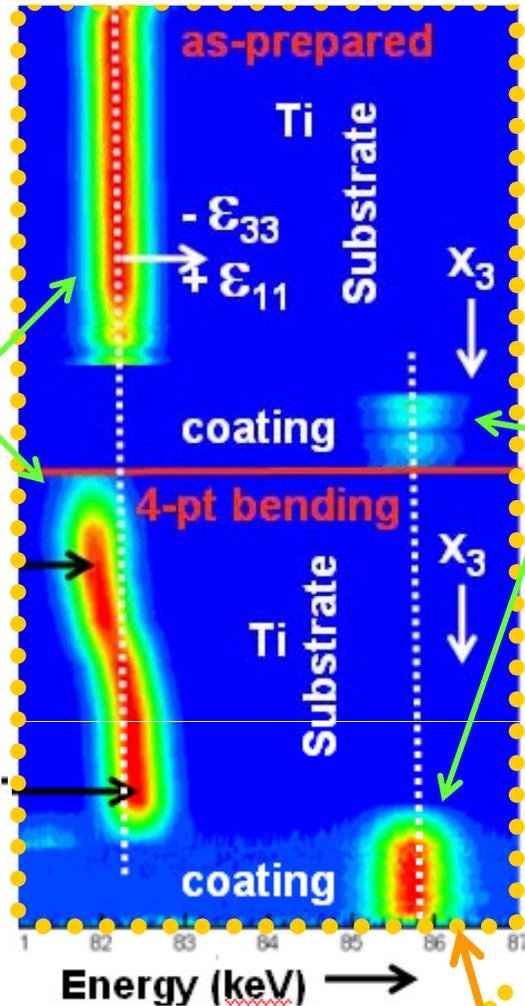
**Performance studies** in fatigue, fracture toughness, crack propagation, thermal shock stress effects and weldmends, shot & laser peening, & other.

Measurement of microscopic stresses **within each phase** due to mismatch stresses and or thermal effects.

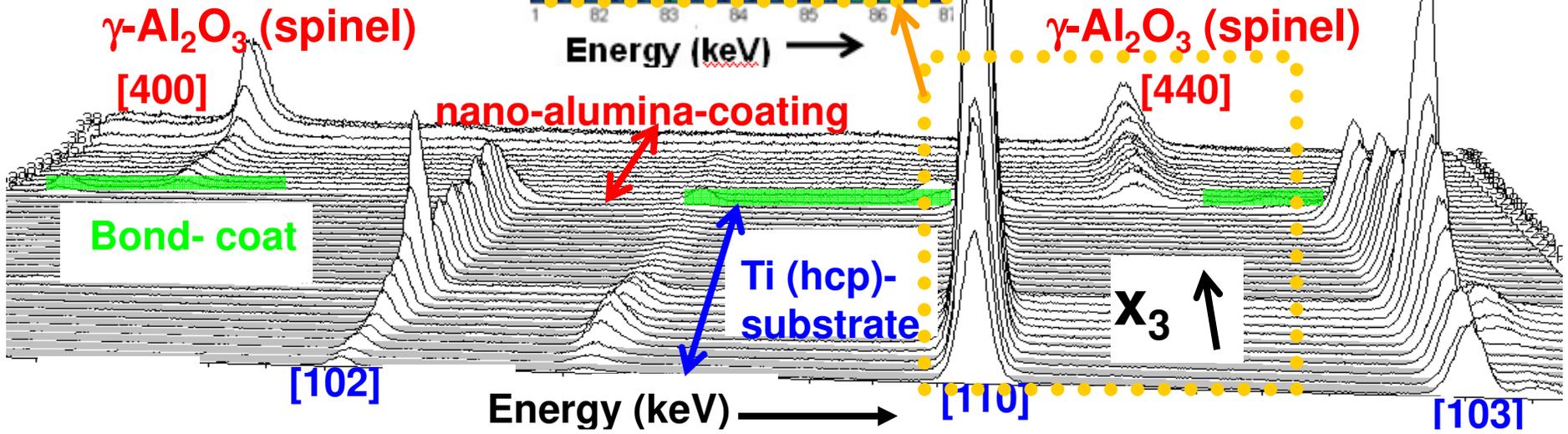
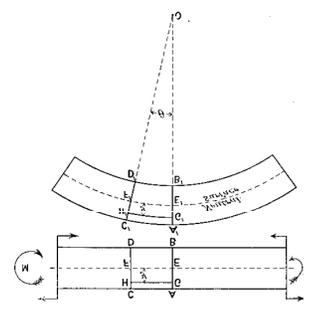
# 4-point bending



# nano-alumina-coating on Ti

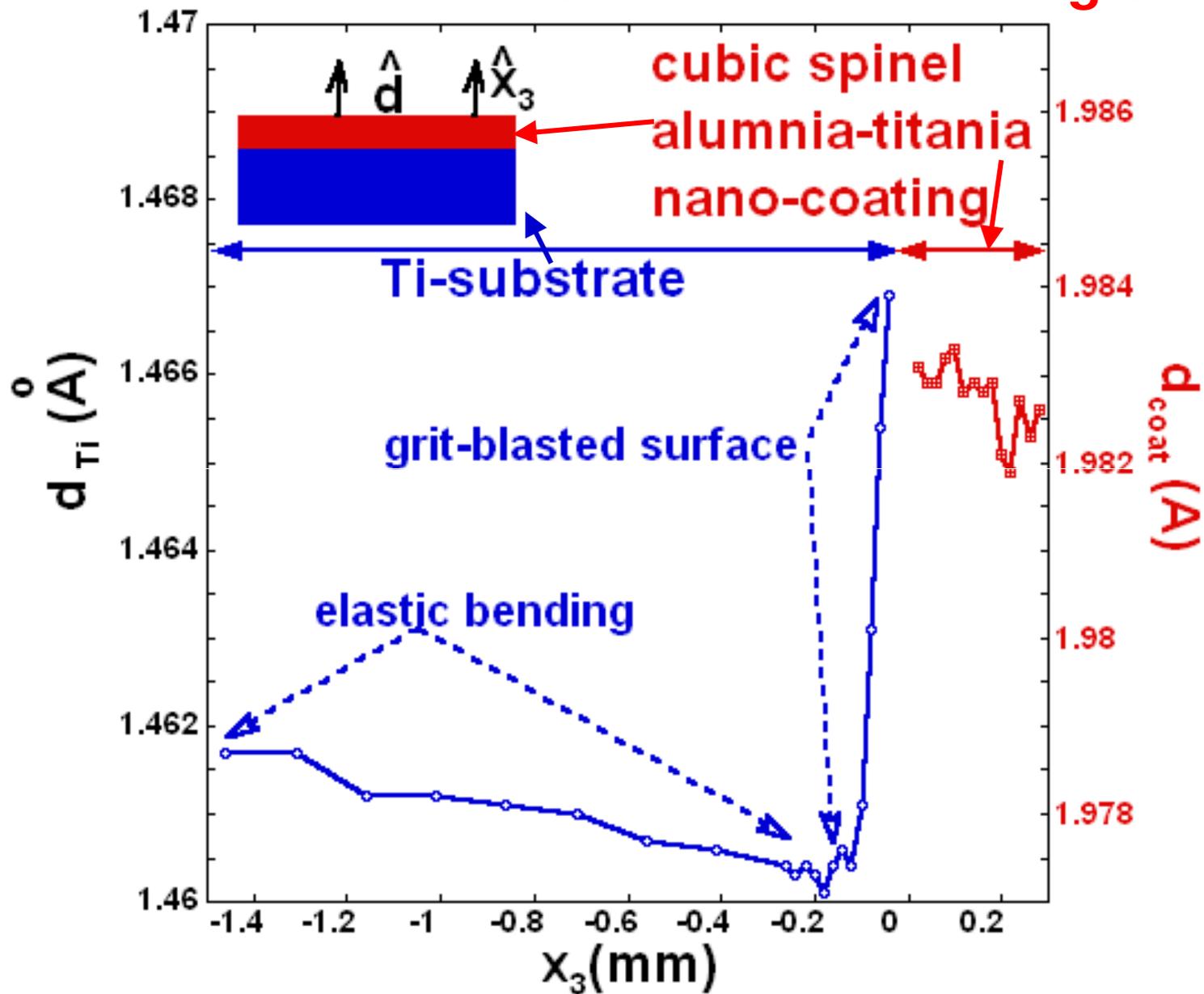


$\gamma\text{-Al}_2\text{O}_3$  (spinel) [440]



# Complex strain regimes

nano-alumina-coating on Ti





$$\sigma_{11} = \frac{E\varepsilon_{11}}{1-\nu^2} = -\frac{Ex_3}{1-\nu^2} \frac{d^2v_3}{dx_1^2} = \frac{Ex_3}{1-\nu^2} \kappa_{11}$$

Integrating over cross section area we obtain the moment

$$M = -\int_h \sigma_{11} L_2 x_3 dx_3 = \left( \frac{EI}{1-\nu^2} \right) \frac{d^2v_3}{dx_1^2} = E'I\kappa_{11}$$

Where  $E' = \frac{E}{1-\nu^2}$  and  $I = \frac{L_2 h^3}{12}$

The transverse strain  $\varepsilon_{33} = (1/E)(-\nu(\sigma_{11} + \sigma_{22})) = -\nu(1+\nu)\sigma_{11} = [-\nu/(1-\nu)] \varepsilon_{11}$

$$\varepsilon_{33} = [-\nu/(1-\nu)] \varepsilon_{11}$$

For  $\nu = 1/3$ ,  $\varepsilon_{33} = -(1/2) \varepsilon_{11}$



## Micromechanics and Kirchoff's formalism of multilayers.

For such loading, we can neglect the transverse components of the strain  $\epsilon_{3i}$ . Large deformations have not been employed here due to the small thickness. In the context of micromechanics the elastic strain tensor  $\epsilon_{ij}^{el}$  is given by:

$$\epsilon_{ij}^{el}(x_3) = \epsilon_{ij}^{tot}(x_3) - \epsilon_{ij}^*(x_3) \quad (1)$$

where the total strain from classical Kirchoff plate theory is given by:

$$\epsilon_{ij}^{tot}(x_3) = \epsilon_{ij}^0 + \kappa_{ij} x_3 \quad (2)$$

where **the curvatures  $\kappa_{ij}$  and the strains  $\epsilon_{ij}^0$  at  $x_3 = 0$  can be assumed to be constant** in the plate and  $i, j$  indices take values 1, 2 (in plane stresses case).

From micromechanics the constitutive equation can be written as:

$$\sigma_{ij}(x_3) = [C_{ijkl}] (\epsilon_{kl}^{tot} - \epsilon_{kl}^*) \quad (3)$$

where  $C_{ijkl}$  represent the general stiffness matrix for the in plane stress-strain tensor case. From Eqs. (2) and (3) we can obtain the resultant force and moment due to the stress distribution in the  $x_3$  through the thickness of our plate.

$$N_{ij} = \int_h \sigma_{ij}(x_3) dx_3 \quad \text{and} \quad M_{ij} = \int_h \sigma_{ij}(x_3) x_3 dx_3$$