**Presentation at University of Virginia**

# Mechanical Behavior of a Polycrystalline C-2000 and an Amorphous $Zr_{50}Cu_{40}Al_{10}$ Alloy Subjected to Surface Severe Plastic Deformation ($S^2PD$)


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- My advisor: Dr. P.K. Liaw
  Committee Members: Dr. Y.F. Gao, Dr. H. Choo, Dr. H. Bozdogan
- Collaborators: Dr. L.L. Shaw, Dr. A. Ortiz, Dr. Y. Yokoyama, Dr. D. Klarstrom, Dr. P.J. Withers, and Dr. A.L. Greer

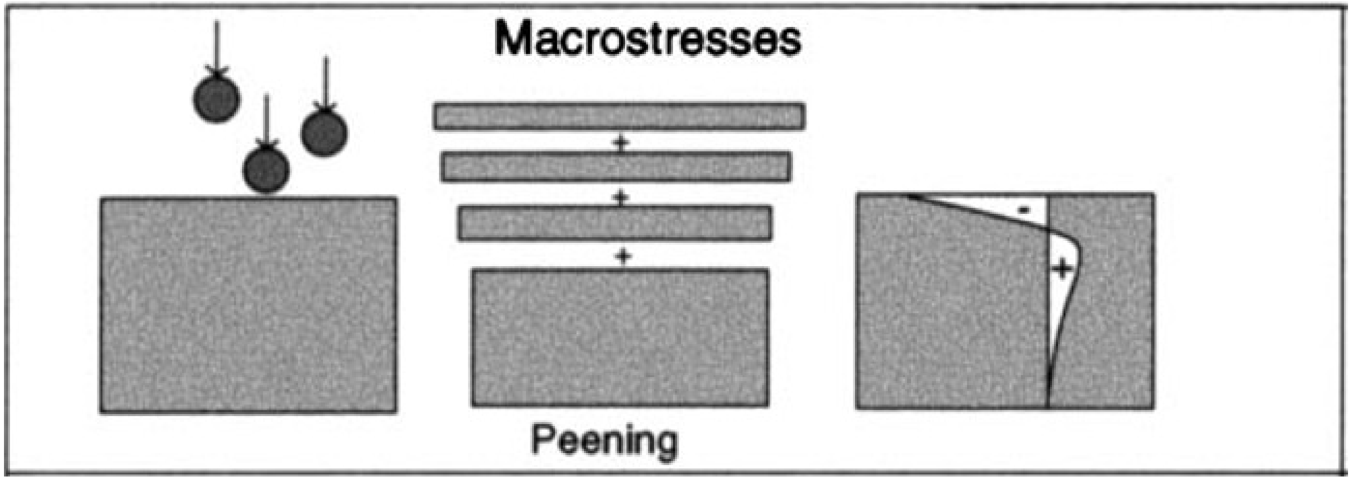
# Outline

- Background Reviews
- Experimental Procedures
- Effects of Surface Treatments on Polycrystalline Ni-based C-2000 Superalloy
- Effects of Surface Treatments on Amorphous Zr-based $Zr_{50}Cu_{40}Al_{10}$ Bulk Metallic Glass
- Summary

# Shot peening: an old process

- Shot peening is a process used to produce a compressive residual stress layer and modify mechanical properties;
- It entails impacting on a surface with shots (round metallic, glass, or ceramic particles) with force sufficient to create plastic deformation;
- Shot peening is often called for in aircraft repairs to relieve tensile stresses built up in the grinding process and replace them with beneficial compressive stresses;
- Shot peening can increase fatigue life from 0% – 1,000%, depending on the materials and peening parameters.

# Effects of shot peening




- Residual stresses originate from misfits between different regions within a component – Type-I residual stress*
- Work hardening originates from the saturated dislocation density which has resistance to new dislocation-formation
- Surface roughness is a co-effect of dents-generating and peak-removing
- The above-mentioned features are closely related to fatigue property



*P.J. Withers et al. Materials Science and Technology, 17(2001), 355-364

# Nanocrystallization: a hot topic

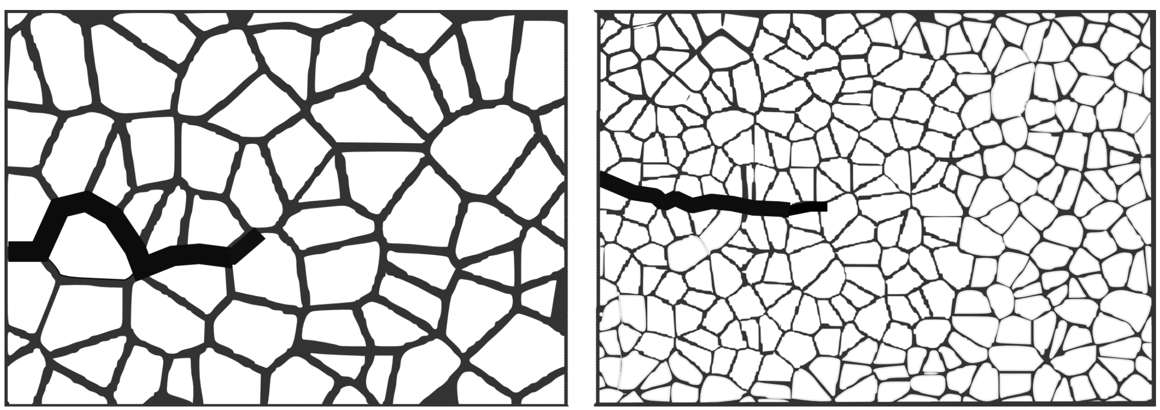
- Good global mechanical properties, such as high yield strengths and toughness, high hardness, good thermal and magnetic properties
- Generally, the nanocrystalline synthesis methods can be classified into two categories:
  - Breaking the large grain size down (a top-down method, such as equal channel angular pressing (ECAP))
  - Consolidating small clusters up (a bottom-up method, such as gas condensation, mechanical alloying, etc.)
- Difficulties:
  - High pressure required
  - Porosity/contamination problems

# Effects of fine grains

- For crack initiation along a slip band, the number of cycles for a crack to initial, $N_f$, is proportional to the reciprocal of the grain size *, *d*, i.e.,

$$N_f \propto \frac{1}{d}$$

- Crack-growth path can be deflected by the coarse grain more effectively than by fine grains:

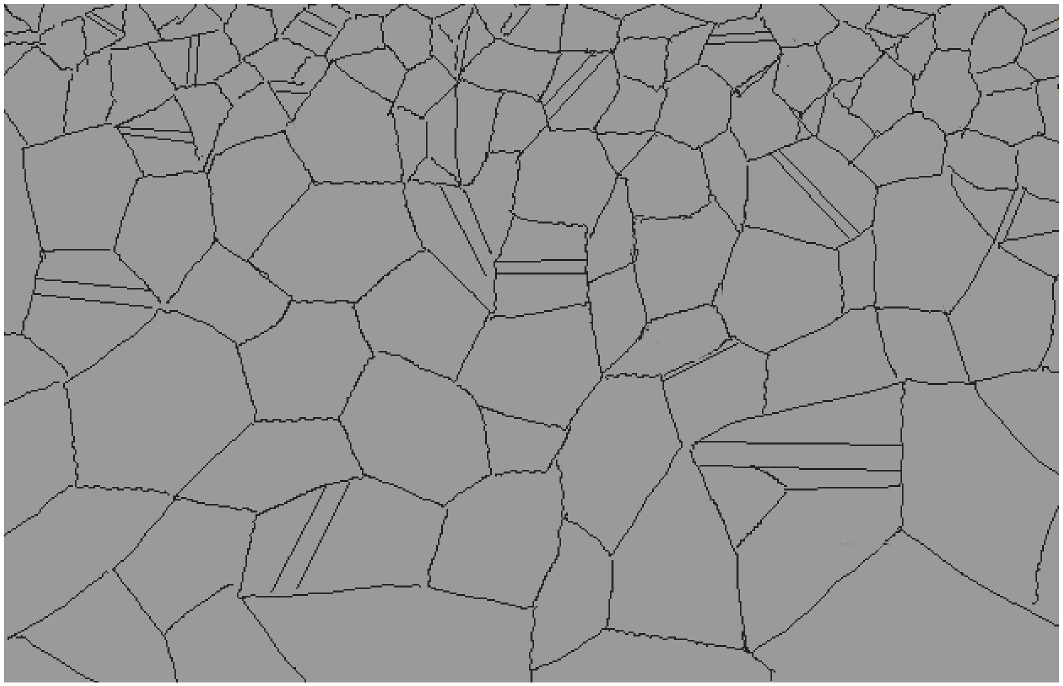


- It is usually true that cracks initiate from the surface and propagate into the interior in a fatigue test



* *K.S. Chan, Metallurgical and Materials Transactions A, 34(2003), 43-58*

# Surface severe plastic deformation ($S^2PD$)



Schematic illustration of grain size profile after the $S^2PD$ process

- Work-hardened surface layer (~ 1000 um)
- Surface region with compressive residual stresses
- Nanocrystalline (nc) surface layer (5 ~ 100 um)
- Grain-size gradient with a nc surface and a coarse-grained interior

**shot peening + surface nano-layer → $S^2PD$**

# Why surface treatment?

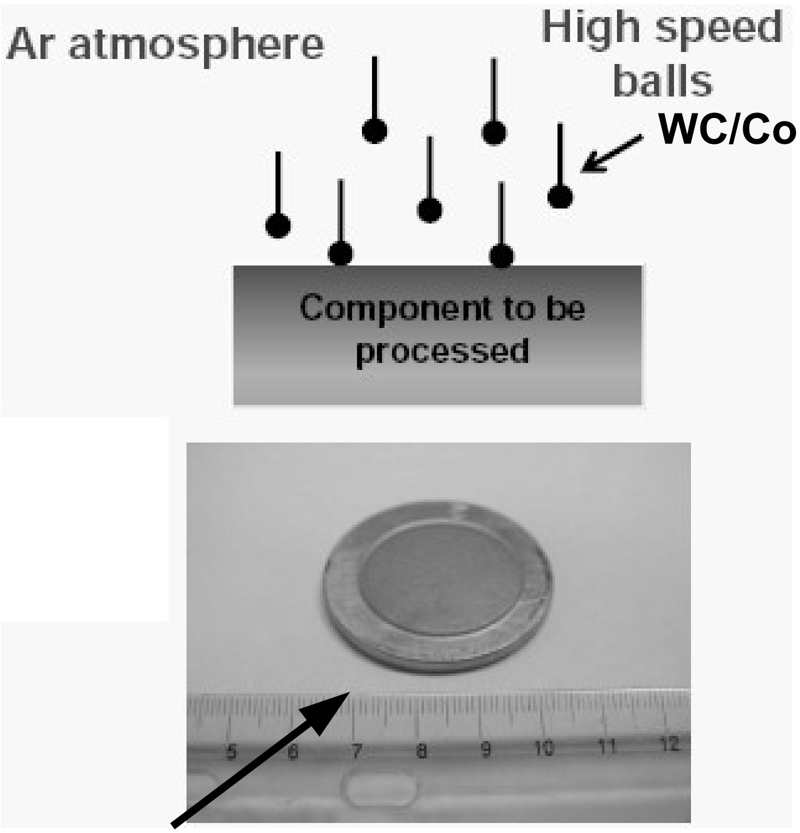
- (Many) failures starting from the component surface
- Applying on existing components
- No heterogeneous composition/interface
- Taking advantage of the advanced properties of nano-materials in a simple way
- Introducing beneficial compressive-residual stresses
- Improving the overall mechanical properties including fatigue property
- Any structural/property changes for bulk metallic glass system?

# Experiments: materials

- Polycrystalline Ni-based C-2000 superalloy
  - Composition: 59Ni – 23Cr – 16Mo – 1.6Cu – 0.01max C – 0.08max Si, wt.%)
  - Structure: Single phase
    Face Centered Cubic (fcc)
  - Main Properties:
    Yield strength = 350 ~ 400 MPa
    Elongation = 60 ~ 70 %
    Density = 8.5 g/cm$^3$
  - Geometry: Diameter = 50 mm
    Thickness = 3.2 mm

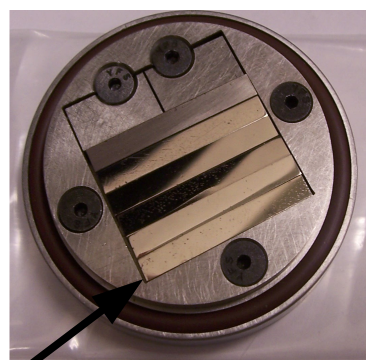
- Zr-based bulk metallic glass
  - $Zr_{50}Cu_{40}Al_{10}$ (in at.%)
  - Properties:
    Tg = 706 K; Tx = 792 K
    Tl = 1,092 K
    Yield strength = 1.86 GPa
    Hardness = HV506
    Modulus = 88 GPa
    Elongation = 2.1%
  - Geometry: 3 × 3 × 25 mm$^3$ bar
    (original as-cast: Ø8.0 × 60 mm$^2$)

# Experiments: equipment




For C-2000 treatment

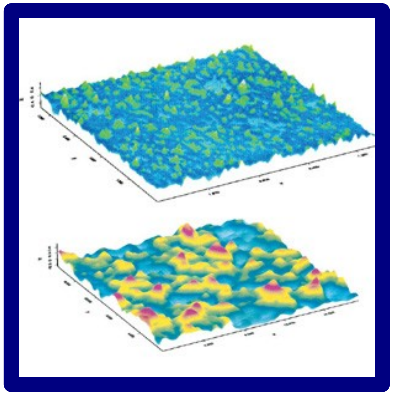

| | |
|---|---|
| Sample: | C-2000/Zr-based BMG |
| Geometry: | Diameter =50 mm |
| Balls: | Ø7.9 mm × 5 units |
| | Ø4.9 mm × 5 units |
| | Ø1.6 mm × 20 units |



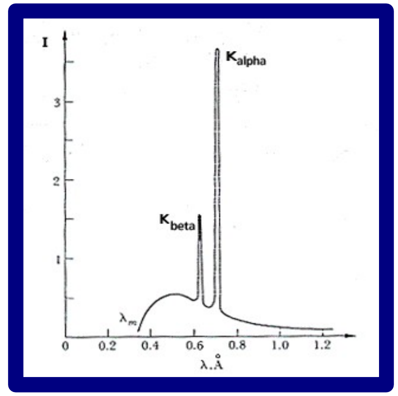
For BMG treatment

# Experiments: tests performed

OM/SEM/TEM

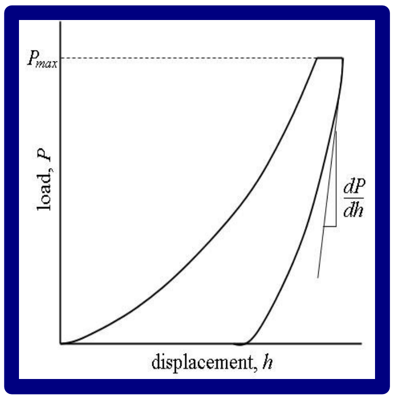


Roughness

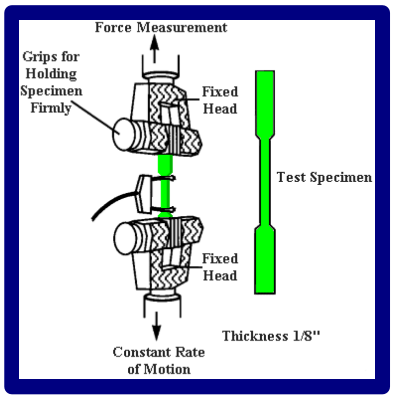



XRD/Synchrotron

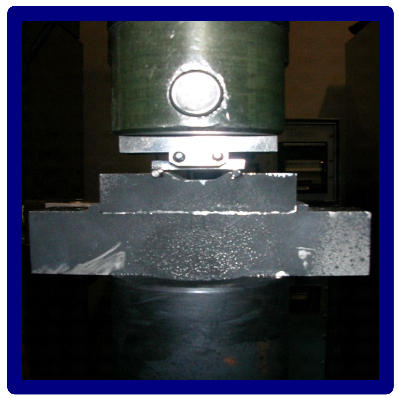
DSC



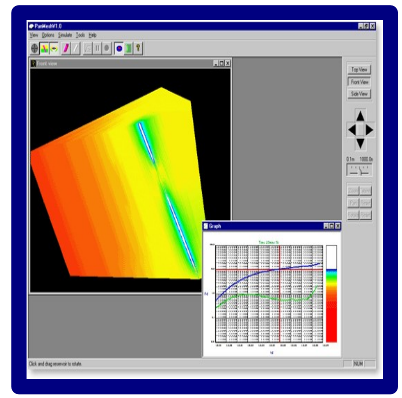

Nanoindentation




Tensile



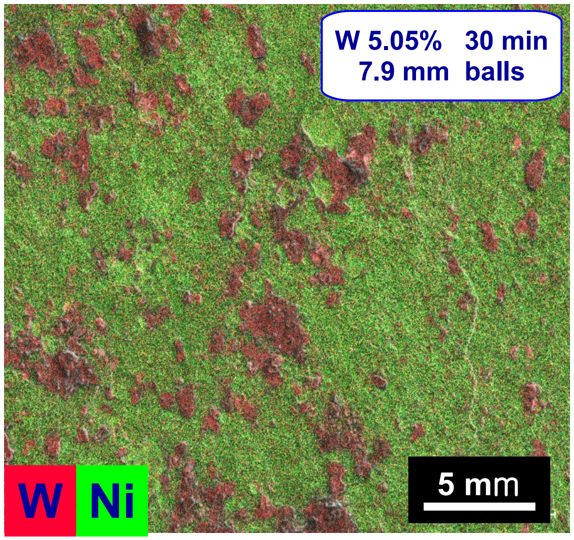
Bending Fatigue

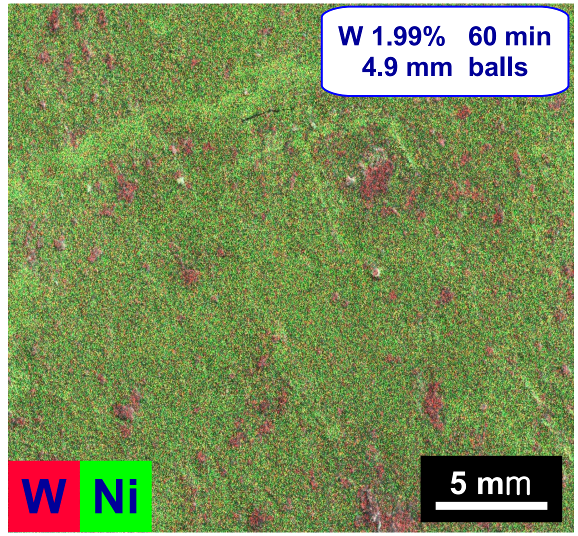


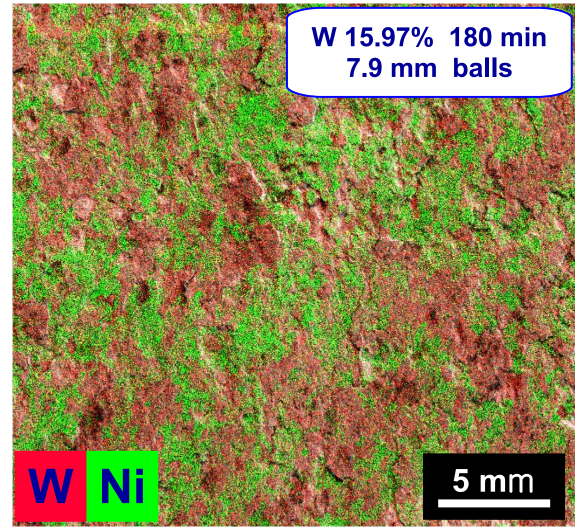
FEM

# Effects of Surface Treatments on Crystalline Ni-based C-2000 Superalloy

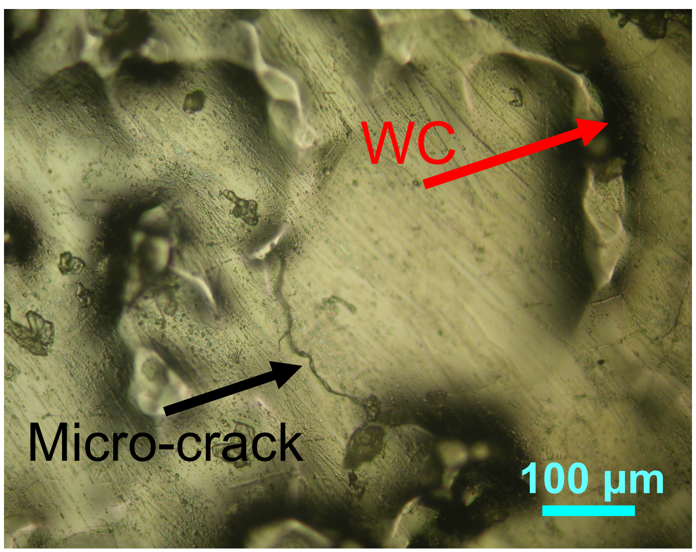
# Surface contaminations



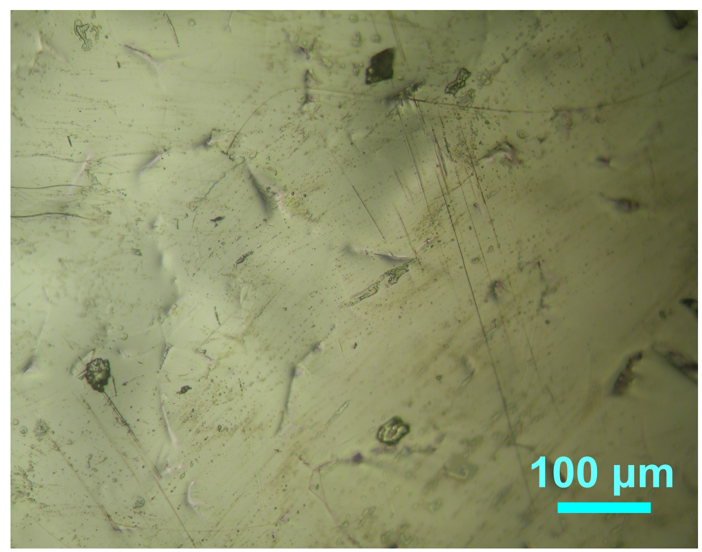




***Energy Dispersive Spectrum (EDS) mapping of the C-2000 sample surface***

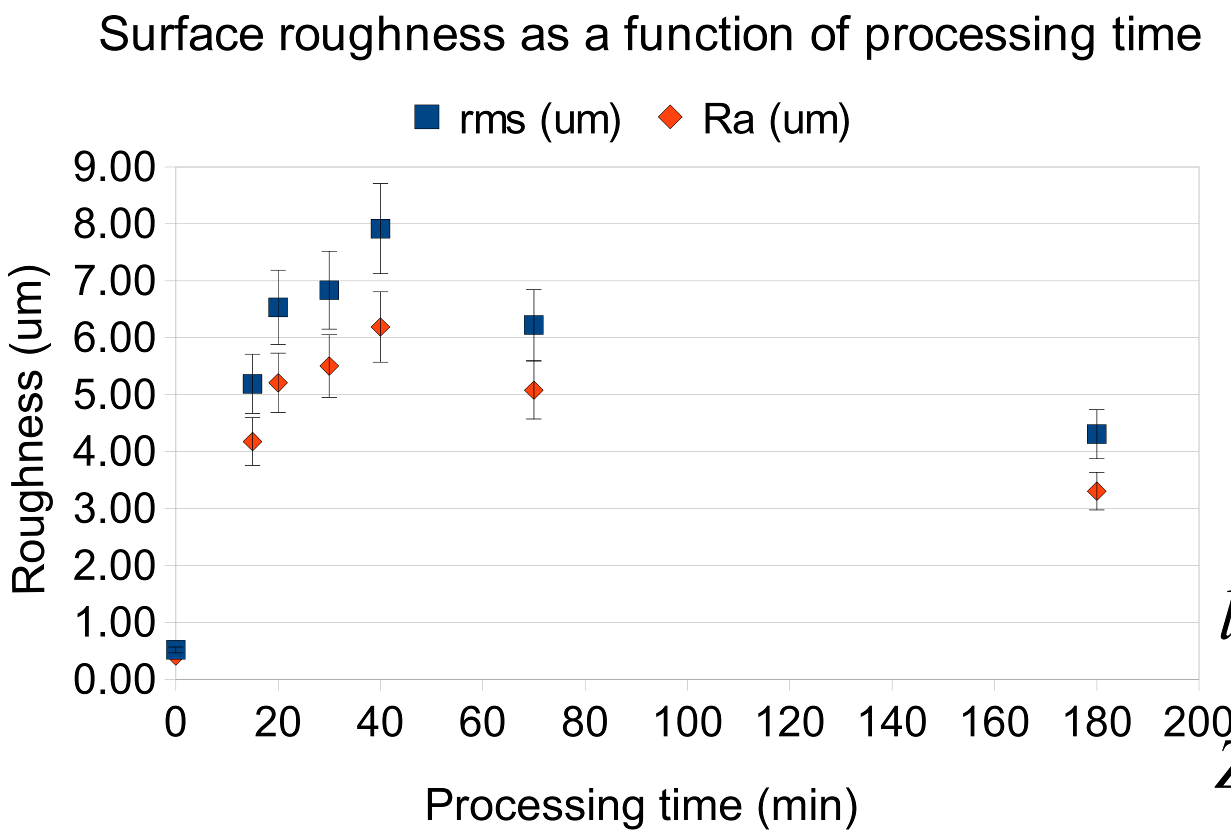



10-um electro-polished away
(micro-crack & residual WC)




25-um polished away

# Surface roughness




$$R_a = \frac{1}{l_m} \int_{x=0}^{x=l_m} |Z(x)| \, dx$$

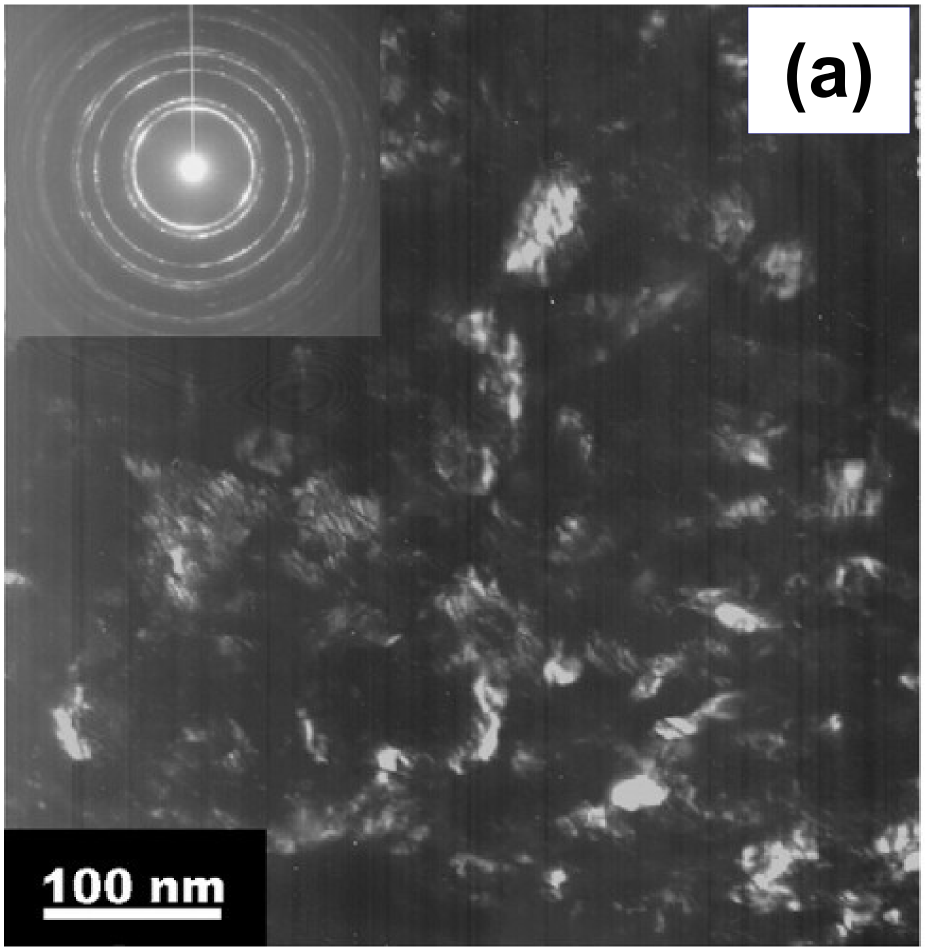
$$rms = \sqrt{\frac{1}{l_m} \int_{x=0}^{x=l_m} Z^2(x) \, dx}$$

$l_m$ : The length that measured for roughness

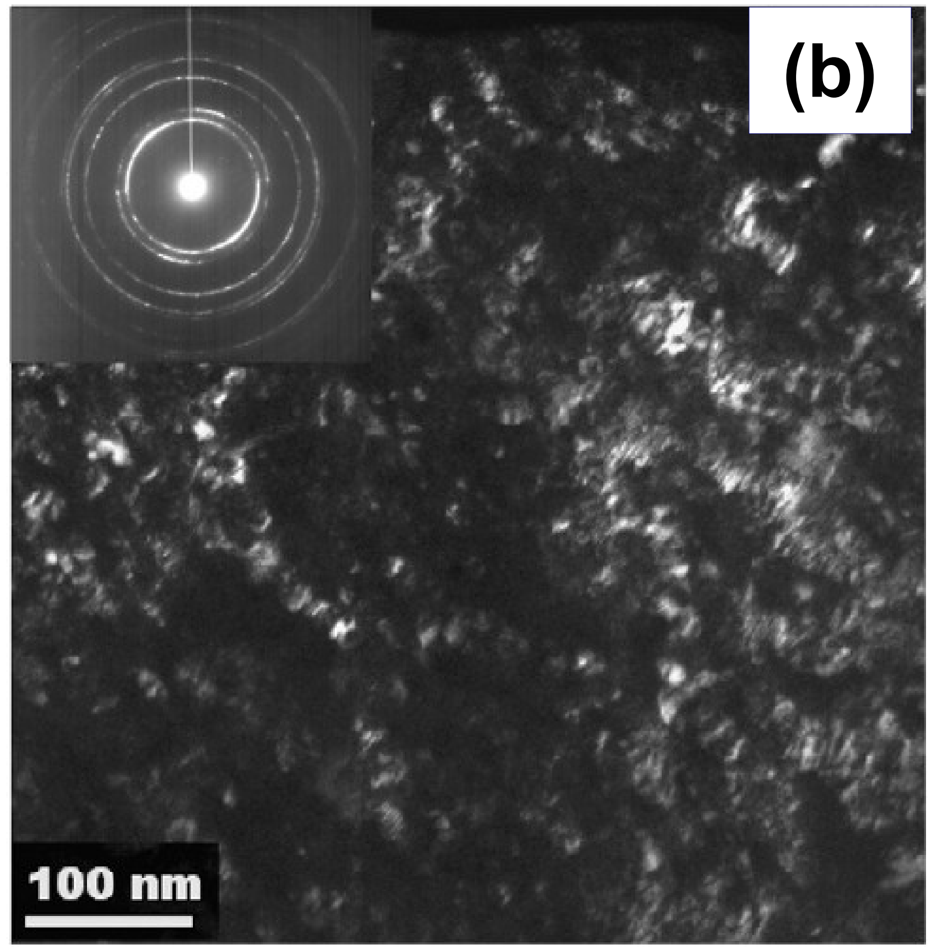
$Z_x$ : The height of a certain point on the surface to the reference point

- Large balls are easier than small balls to induce surface roughness
- Roughness increases first, and then decreases with processing time

# TEM dark-field images from surface




30-min processed

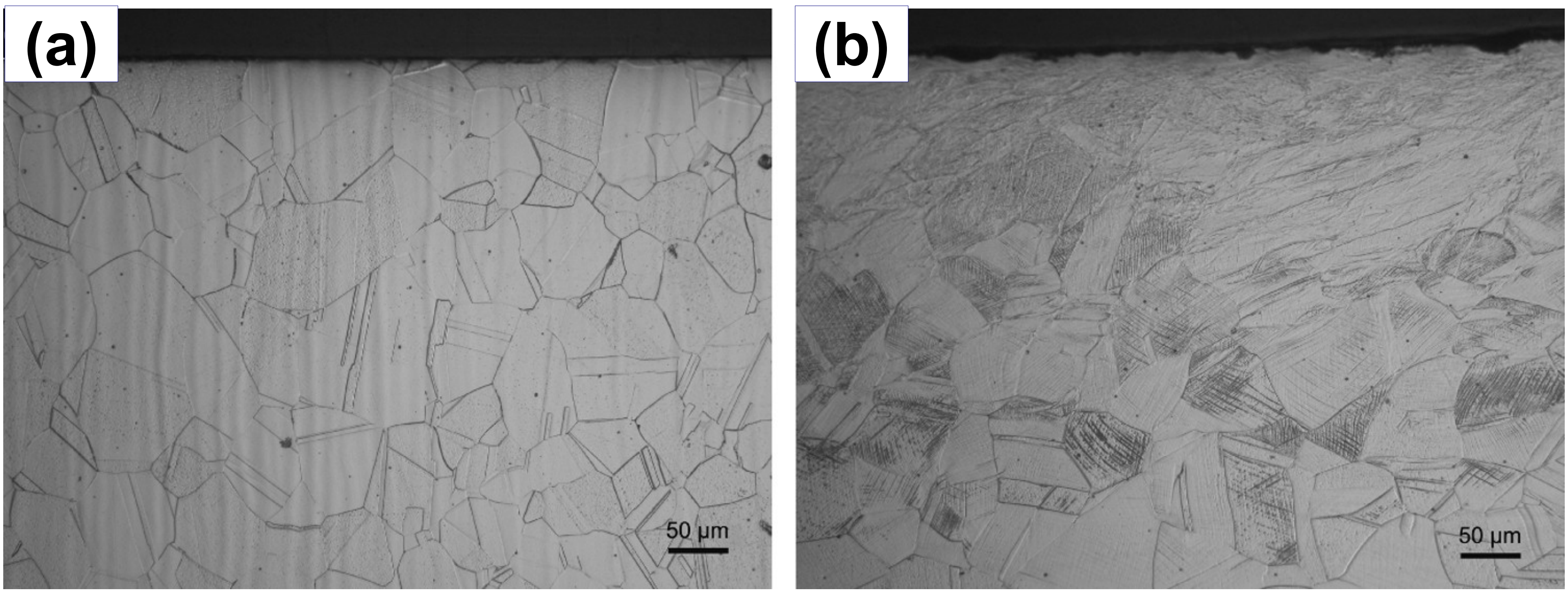



180-min processed



J.W. Tian, K. Dai, J.C. Villegas, L.L. Shaw, P.K. Liaw, D.L. Klarstrome, and A.L. Ortiz, Materials Science and Engineering: A, 493(1-2), 2008, pp.176-183

# Cross-sectional microstructures



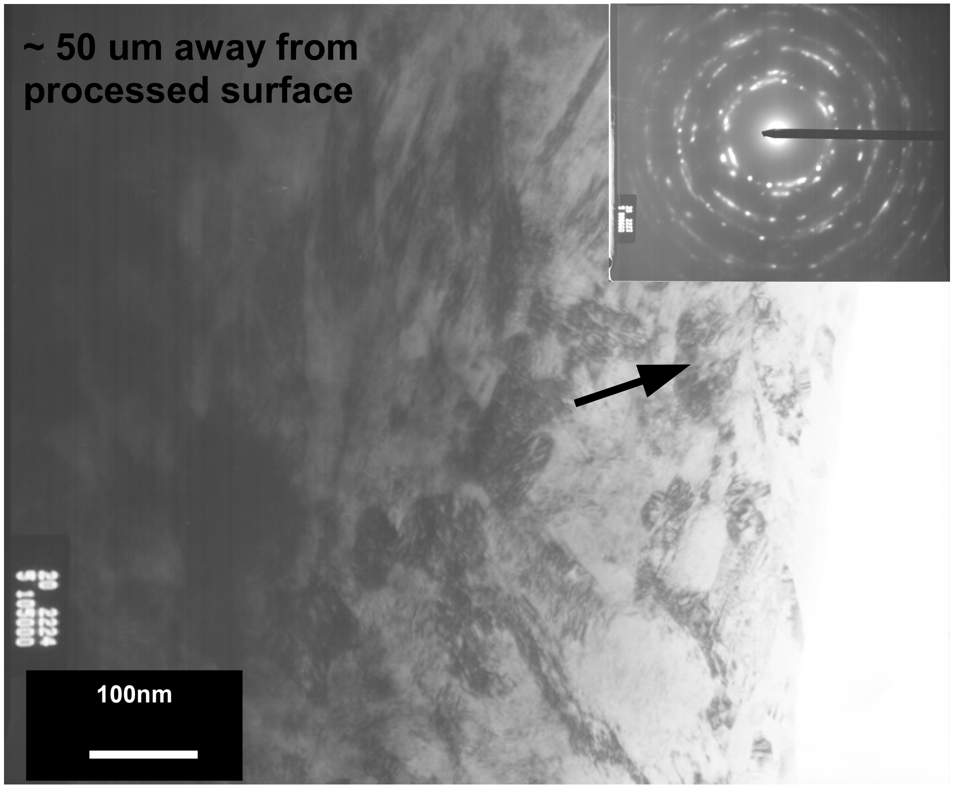

**As-received** **180-min processed**

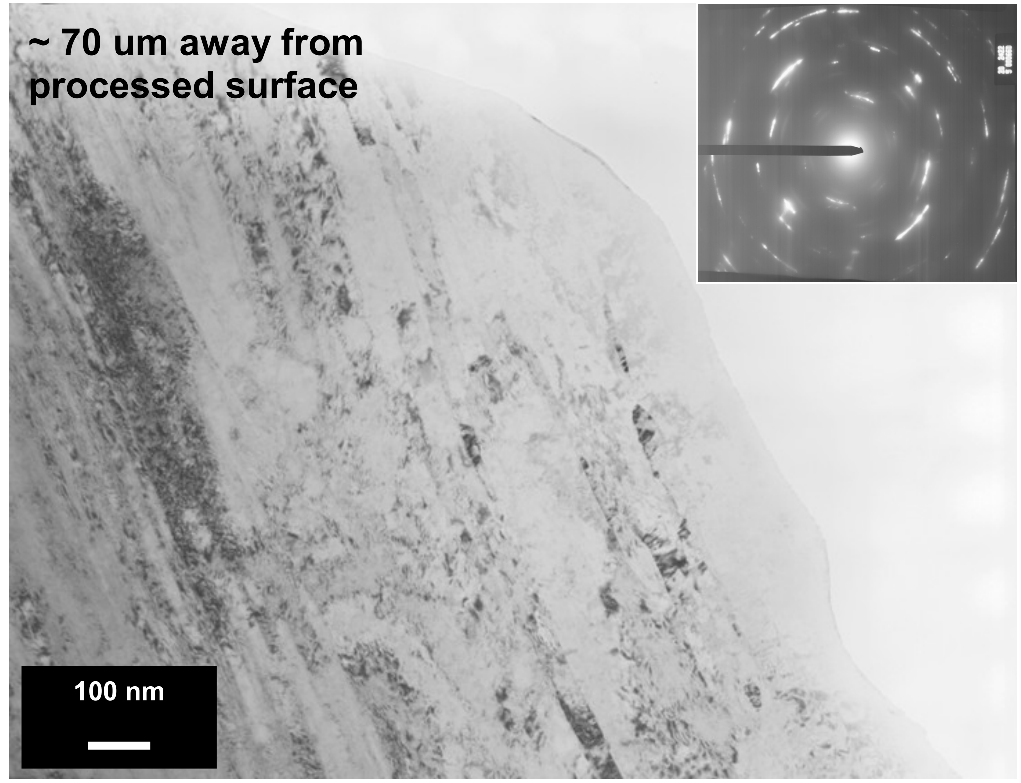
Optical images of the cross-sectional view of (a) the annealed and (b)180-min $S^2PD$ processed C-2000 samples



J.W. Tian, J.C. Villegas, et al, Materials Science and Engineering: A, 468-470(11), 2007, pp.164-170

# TEM bright-field images

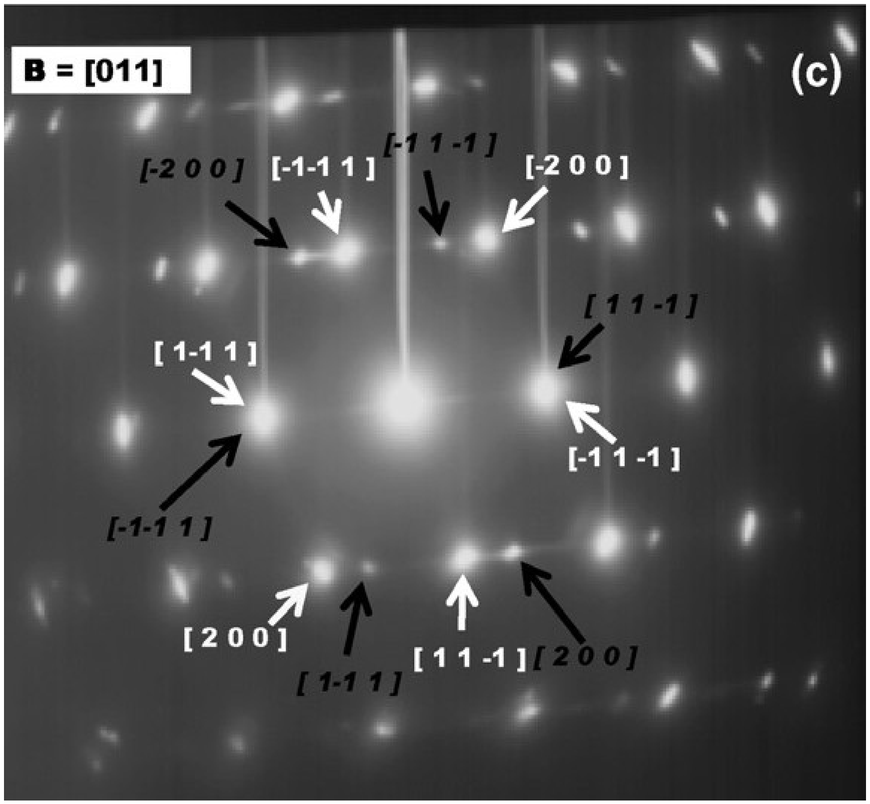


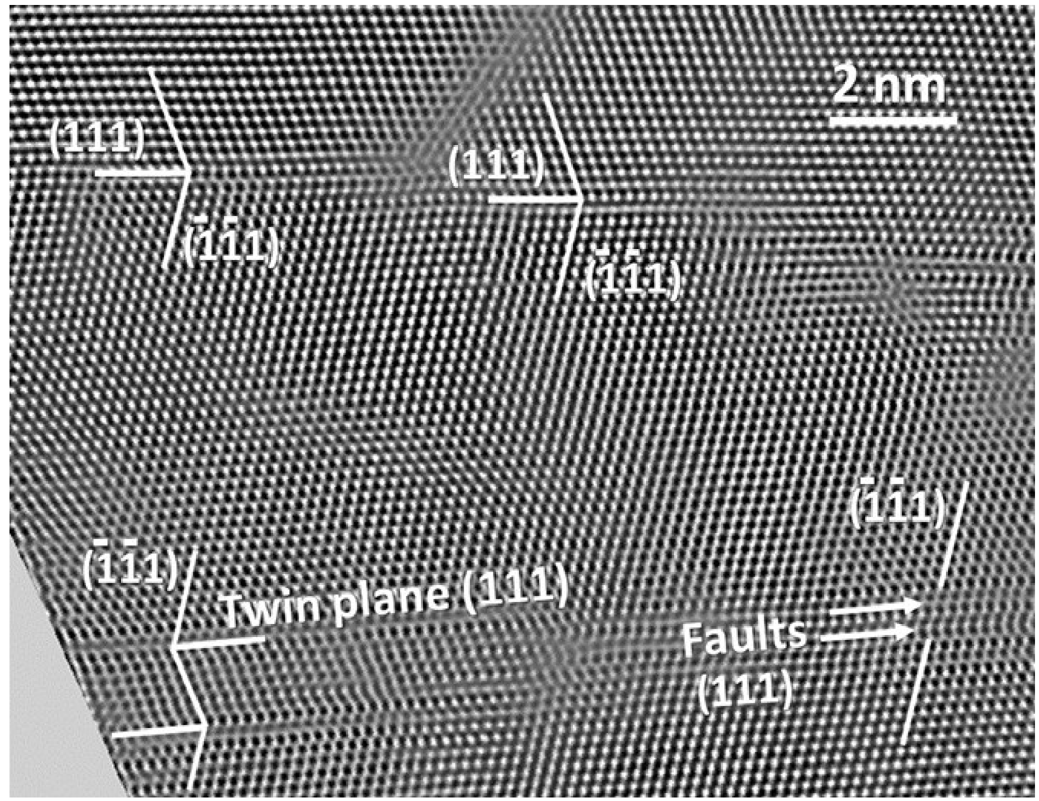




**180-min processed C-2000**

J.W. Tian, J.C. Villegas, W. Yuan, D. Fielden, L. Shaw, P.K. Liaw, and D.L. Klarstrom, Materials Science and Engineering: A, 468-470(11), 2007, pp.164-170

# Confirmation of the deformation twins via SAD and HRTEM*



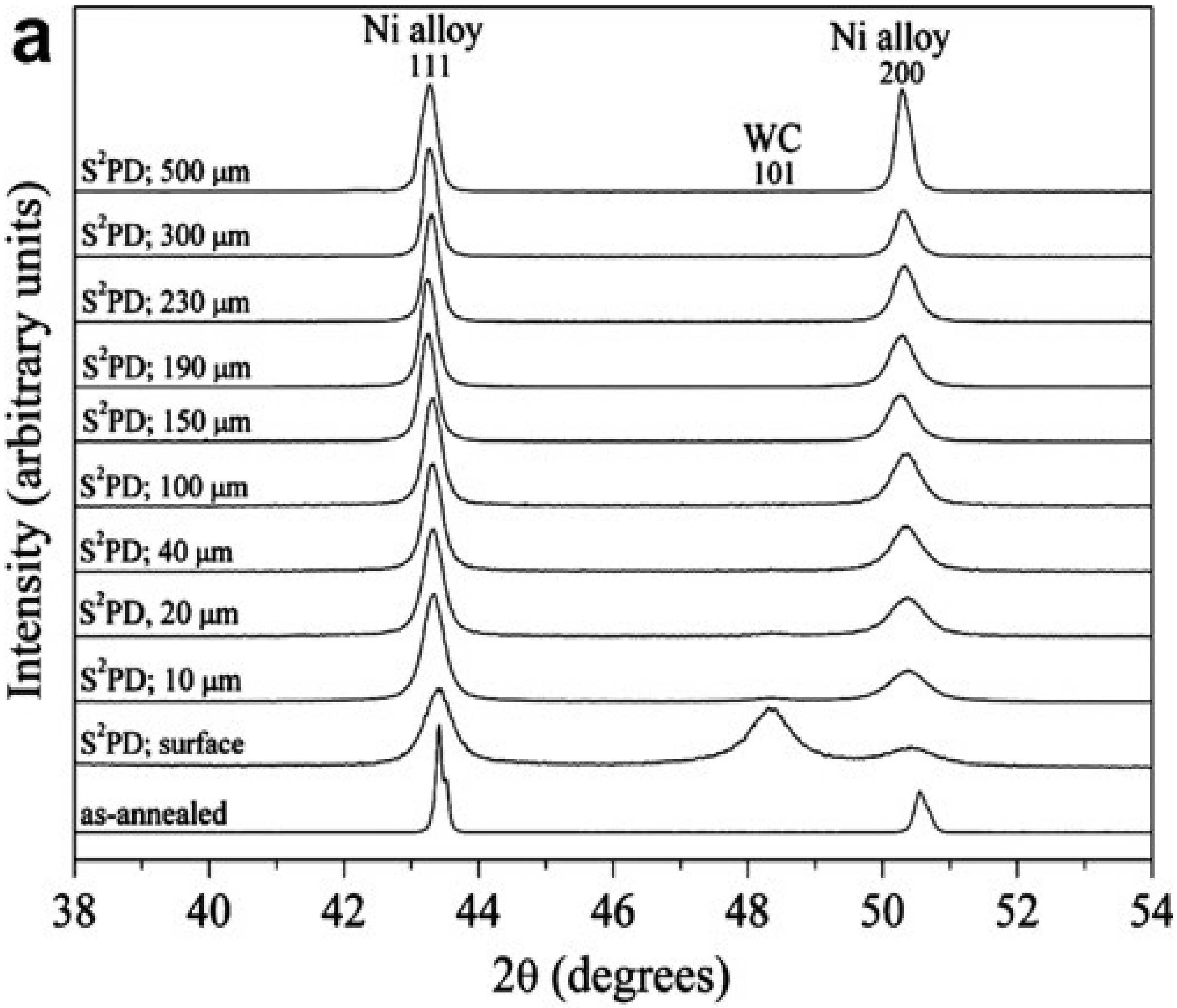




The deformation twins were identified by two symmetrical sets of diffraction patterns with respect to the {111}



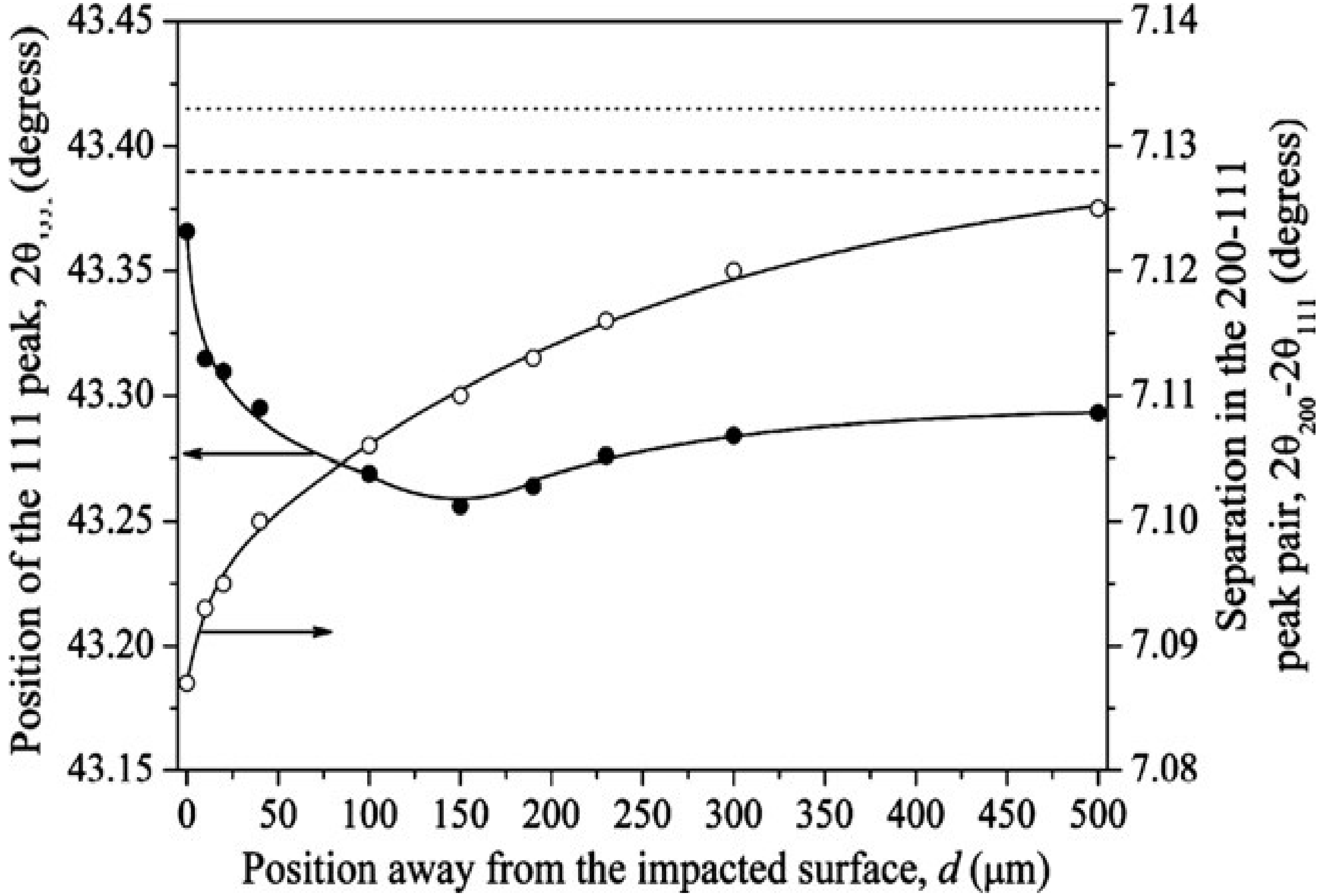
*Collaborator, Dr. Leon Shaw's work

# XRD from different positions (layer-by-layer electro-polishing)




XRD patterns for C-2000 alloy as a function of distance to the impacted surface. (Processing time: 180 min, Sample size: 20 X 20 $mm^2$)

# Peak position varies along depth






A.L. Ortiz, J.W. Tian, J.C. Villegas, L.L. Shaw, and P.K. Liaw, Acta Materialia, 56(3), 2008, pp. 413-426

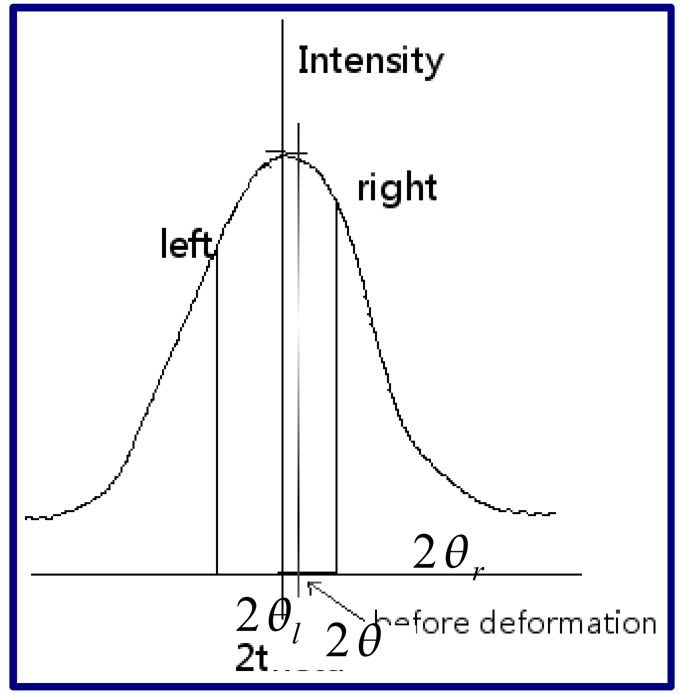
# A slide supposed to be skipped …

$$\gamma = \frac{\sqrt{3}\pi 2\theta^r \left(I^{2\theta_l} - I^{2\theta_r}\right)}{2\beta}\left(1 + \left[\frac{\lambda}{4\pi D_{\mathrm{eff},200}(\sin\theta^r - \sin\theta_{B,200})}\right]^2\right)$$

$$\Delta_f(2\theta) = 2\theta_0 - 2\theta_B = \frac{90\sqrt{3}C_f \tan\theta_0}{\pi^2}\alpha$$

$$\alpha = \frac{\pi^2[\Delta_f(2\theta_1 - 2\theta_2)]}{90\sqrt{3}(C_{f,1}\tan\theta_1 - C_{f,2}\tan\theta_2)}$$

$$a = \frac{\lambda\sqrt{h^2 + k^2 + l^2}}{2\sin(\theta_0 - \Delta_f(2\theta)/2)}$$



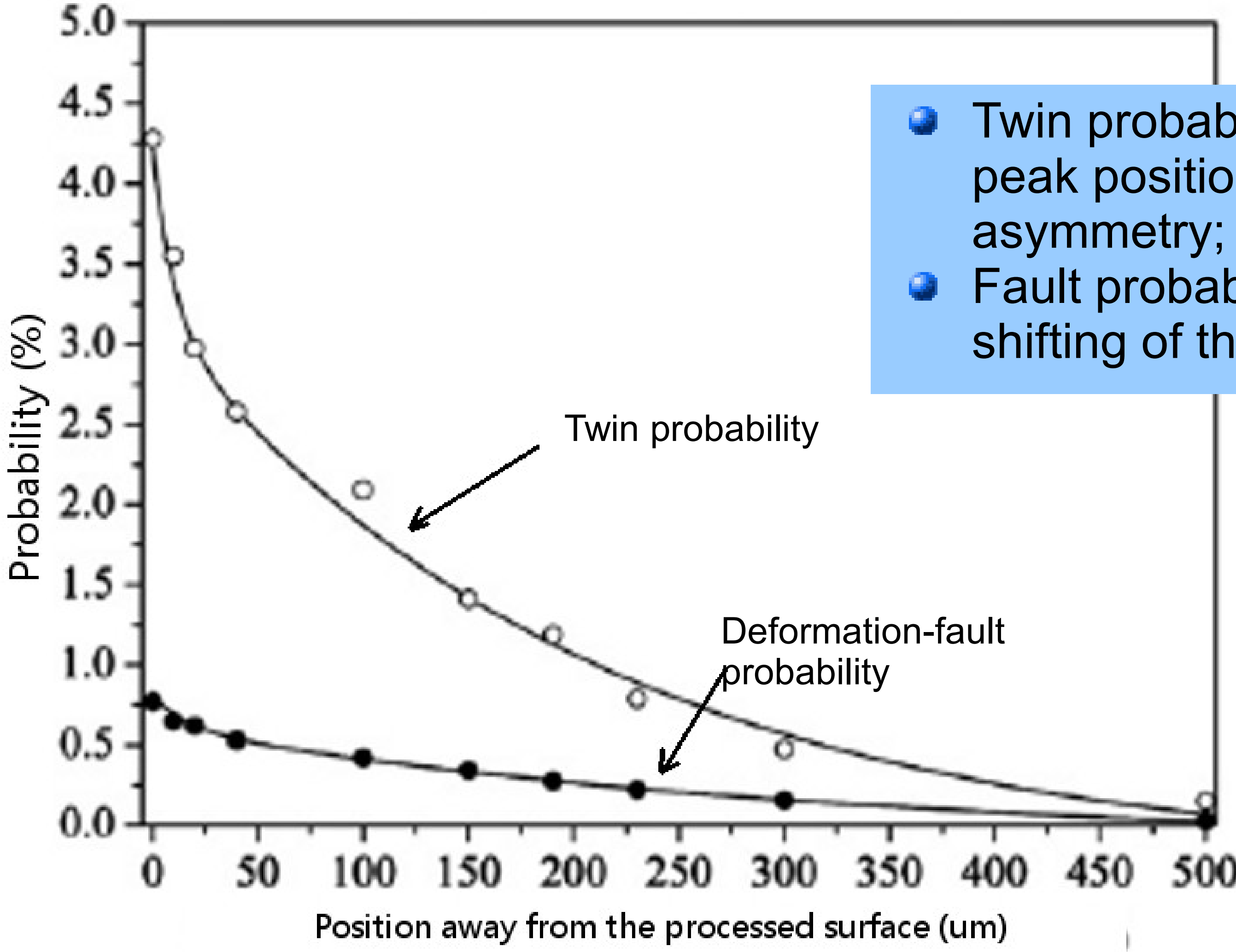

$$\varepsilon = \frac{d_0 - d_0^R}{d_0^R} = \frac{\sin\theta_0^R - \sin(\theta_0 - \Delta_f(2\theta)/2)}{\sin(\theta_0 - \Delta_f(2\theta)/2)}$$

$$\sigma_{||} = -\frac{E}{2v}\varepsilon$$

$$D_{\mathrm{eff}} = \frac{90K_{\mathrm{K}}\lambda}{\pi(\beta_{\mathrm{L}} - \beta_{\mathrm{L}}^R)\cos\theta_0}$$

$$D = \frac{K_{\mathrm{K}}D_{\mathrm{eff}}a}{K_{\mathrm{K}}a - (1.5\alpha + \gamma)C_{\mathrm{D}}D_{\mathrm{eff}}}$$

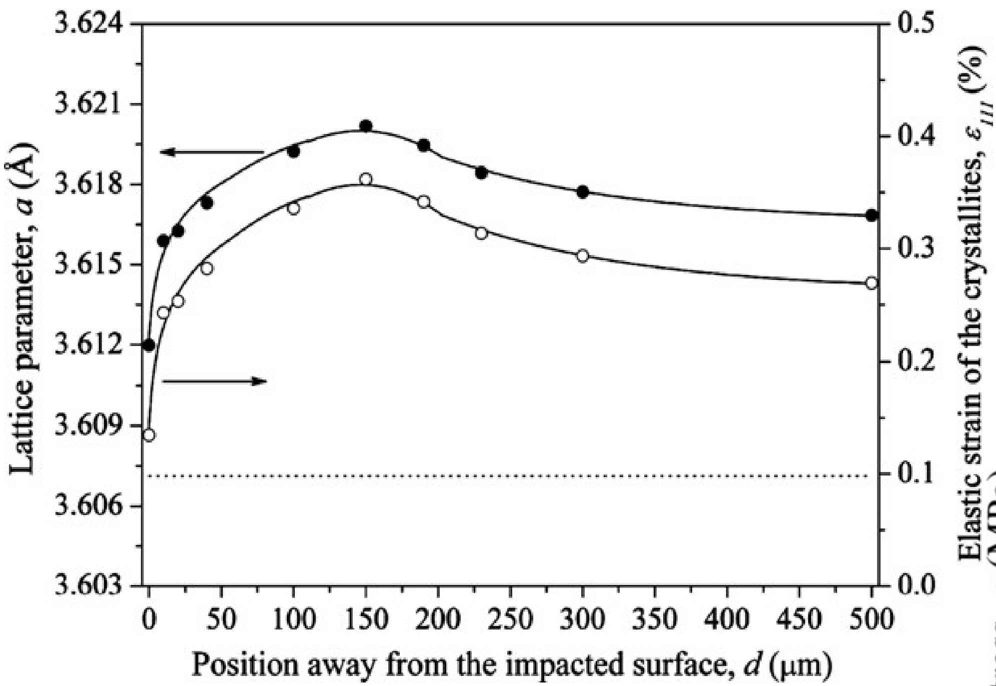
# Results from XRD: twins and faults

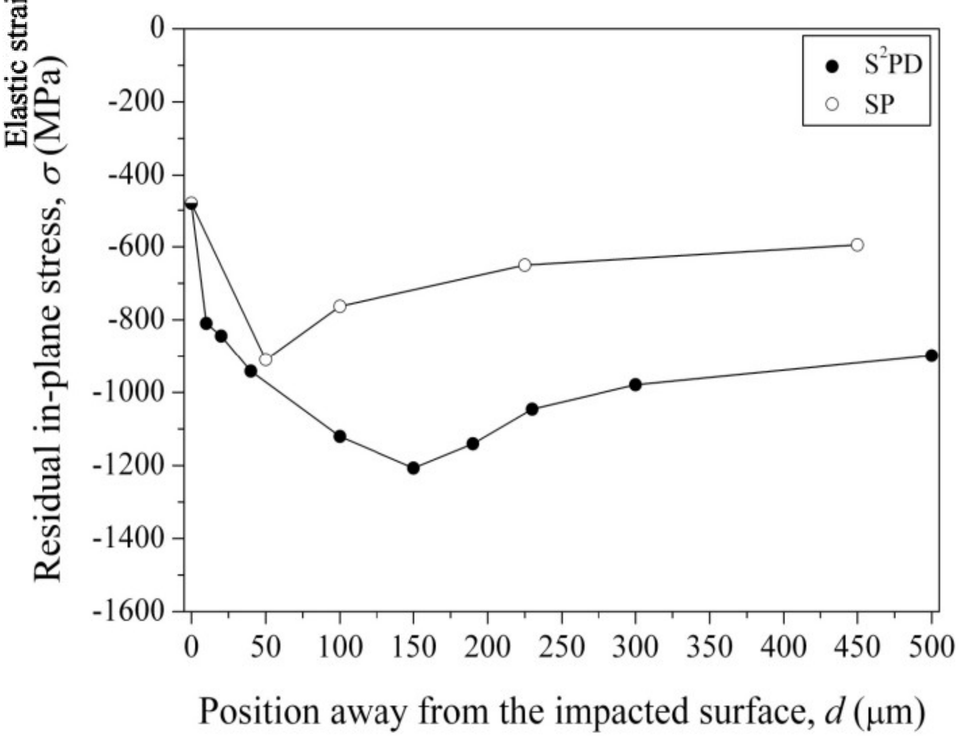



- Twin probability (γ) – determined by peak position, intensity, and the asymmetry;
- Fault probability (α) – determined by shifting of the neighboring peak pairs;

Twin and deformation fault probabilities of the 180 min processed sample as a function depth from surface

# Results from XRD: residual stresses

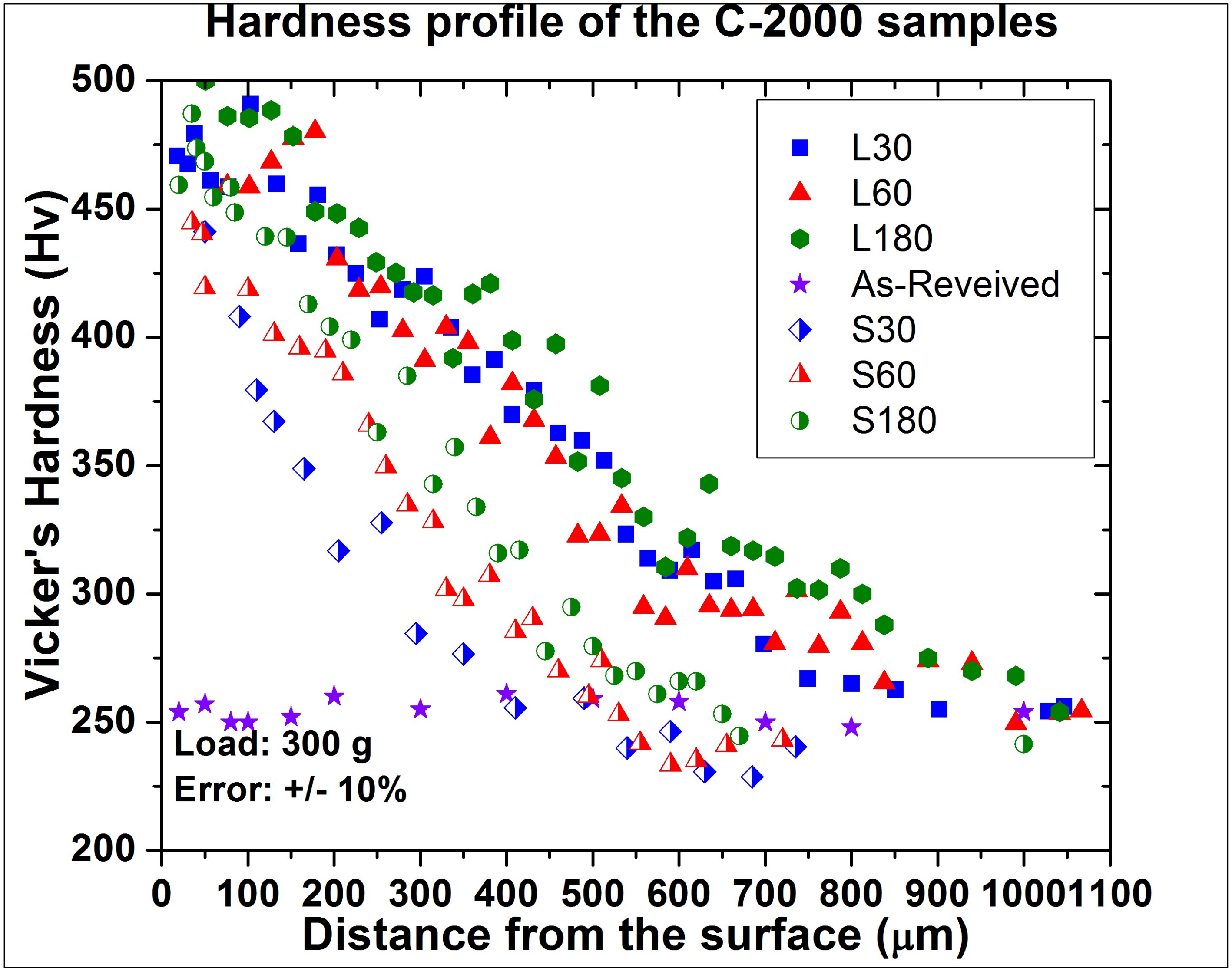



(i) Formation of solid solutions with C and/or W atoms as solutes in the C-2000 alloy matrix

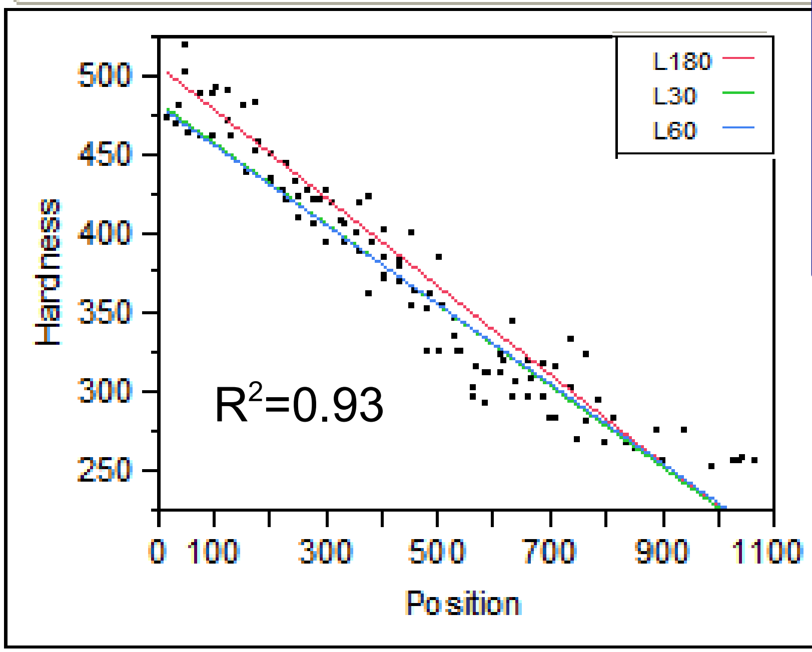
(ii) Introduction of macroscopic residual in-plane compressive stresses

# Hardness profiles



# Statistical analysis on hardness

**Regression Plot**

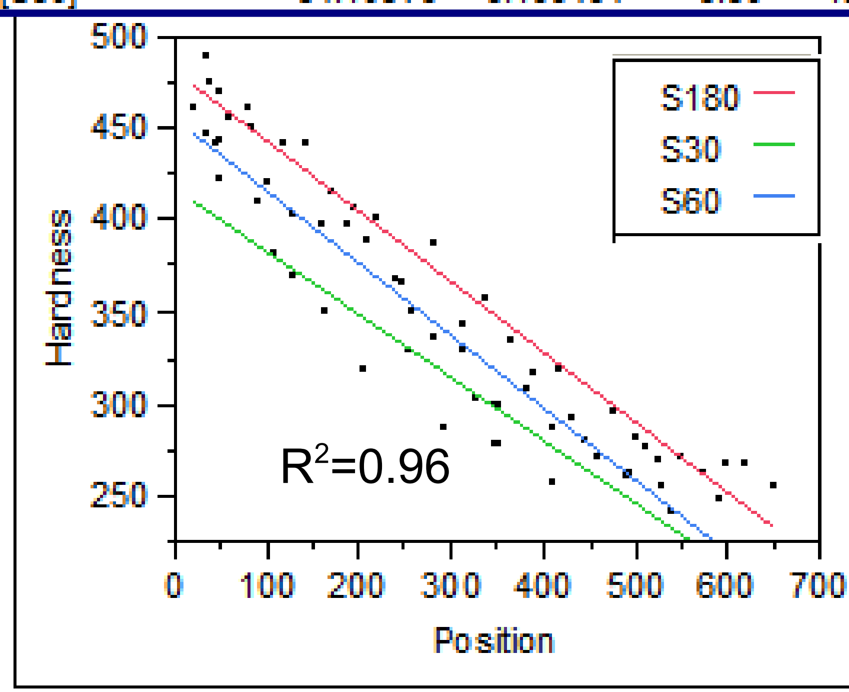



**Parameter Estimates**

| Term | Estimate | Std Error | t Ratio | Prob>\|t\| |
|---|---|---|---|---|
| Intercept | 489.80243 | 4.002434 | 122.38 | <.0001* |
| Position | -0.262353 | 0.00793 | -33.09 | <.0001* |
| Group[L180] | 17.076517 | 6.052536 | 2.82 | 0.0058* |
| Group[L30] | -7.648758 | 5.318255 | -1.44 | 0.1536 |

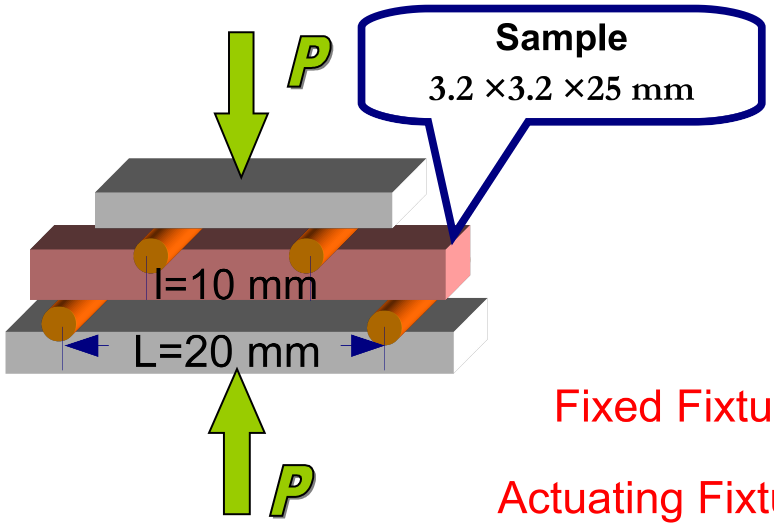
**Parameter Estimates**

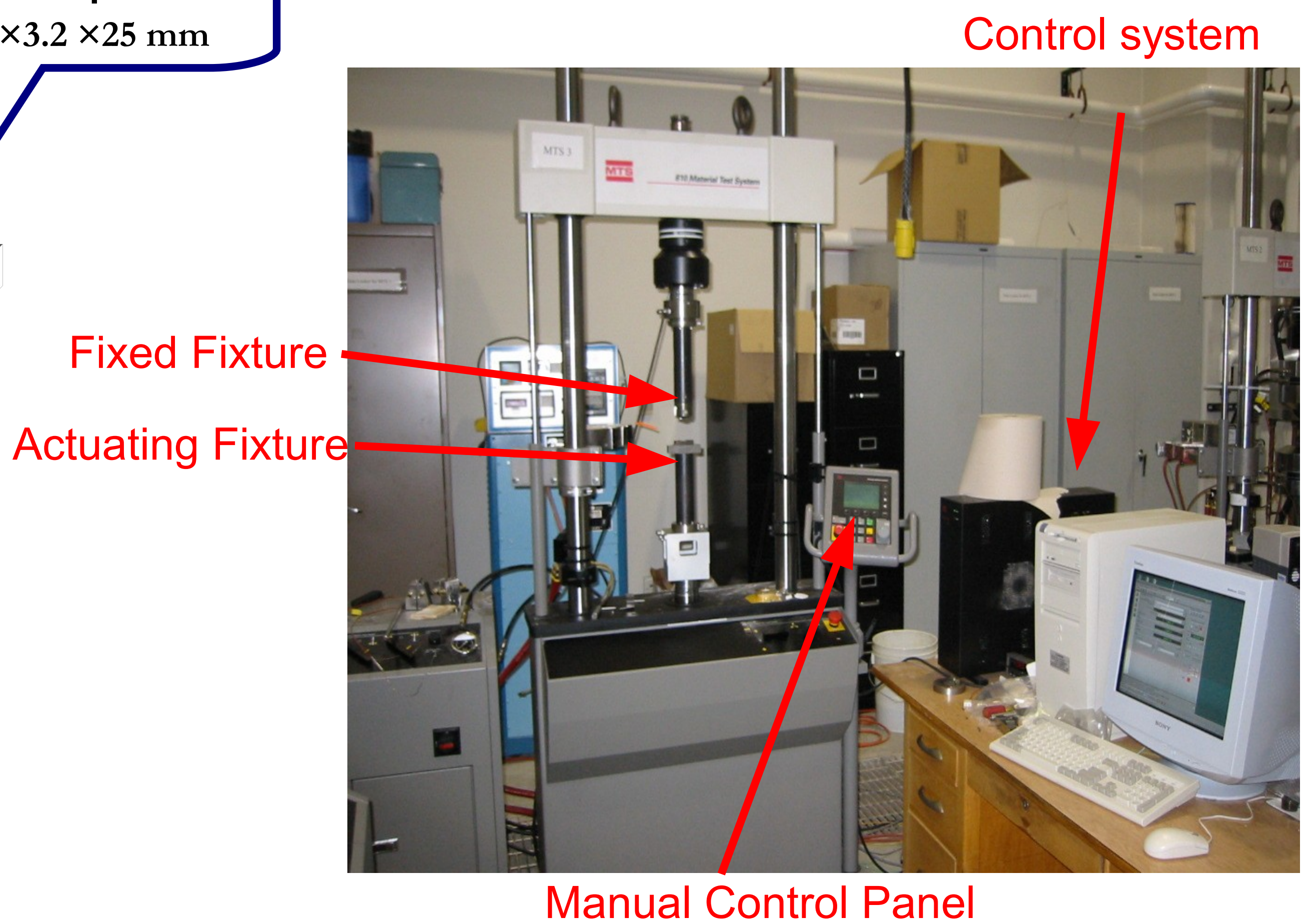
| Term | Estimate | Std Error | t Ratio | Prob>\|t\| |
|---|---|---|---|---|
| Intercept | 450.37537 | 3.999357 | 112.61 | <.0001* |
| Position | -0.372362 | 0.011982 | -31.08 | <.0001* |
| Group[S180] | 30.603856 | 5.02413 | 6.09 | <.0001* |
| Group[S30] | -34.10579 | 6.199464 | -5.50 | <.0001* |




- No significant difference in slope;
- No significant difference between intercepts of L30 and L60, though L180 has a little higher intercept
- Significant difference in intercepts for S
- Indication of saturated bombardment for 7.9 mm balls but not unsaturated for 4.9 mm balls

# Four-point-bending fatigue



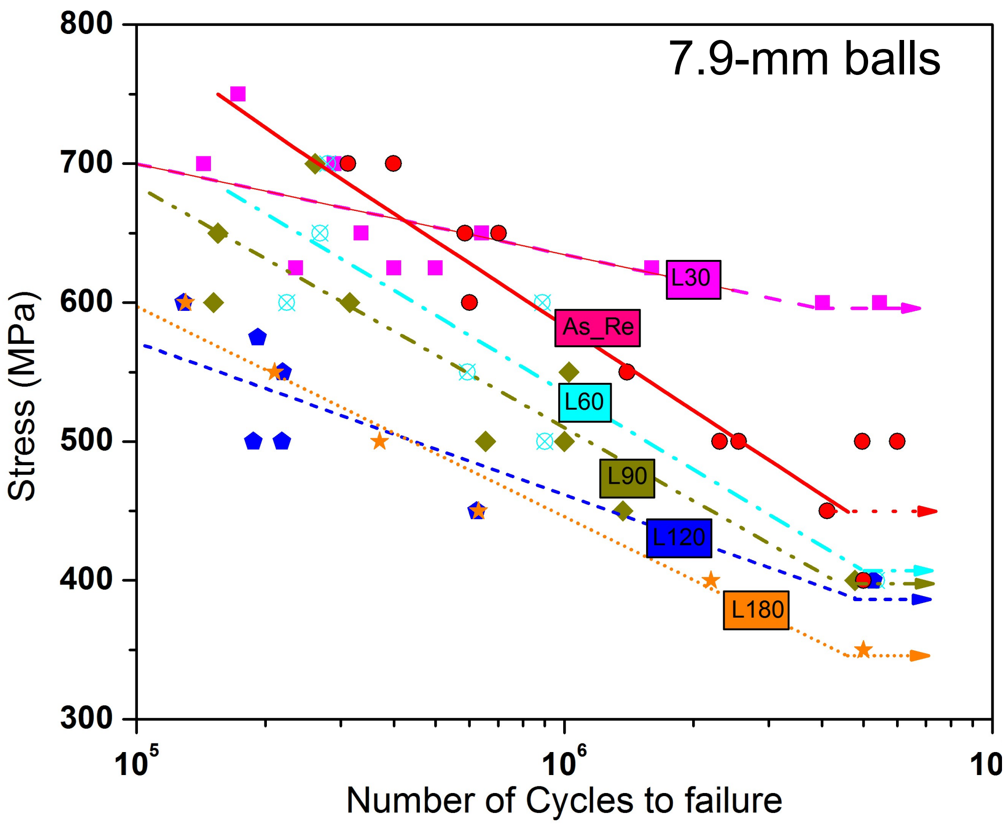

$$\sigma = \frac{3P(L-l)}{2Wh^2}$$

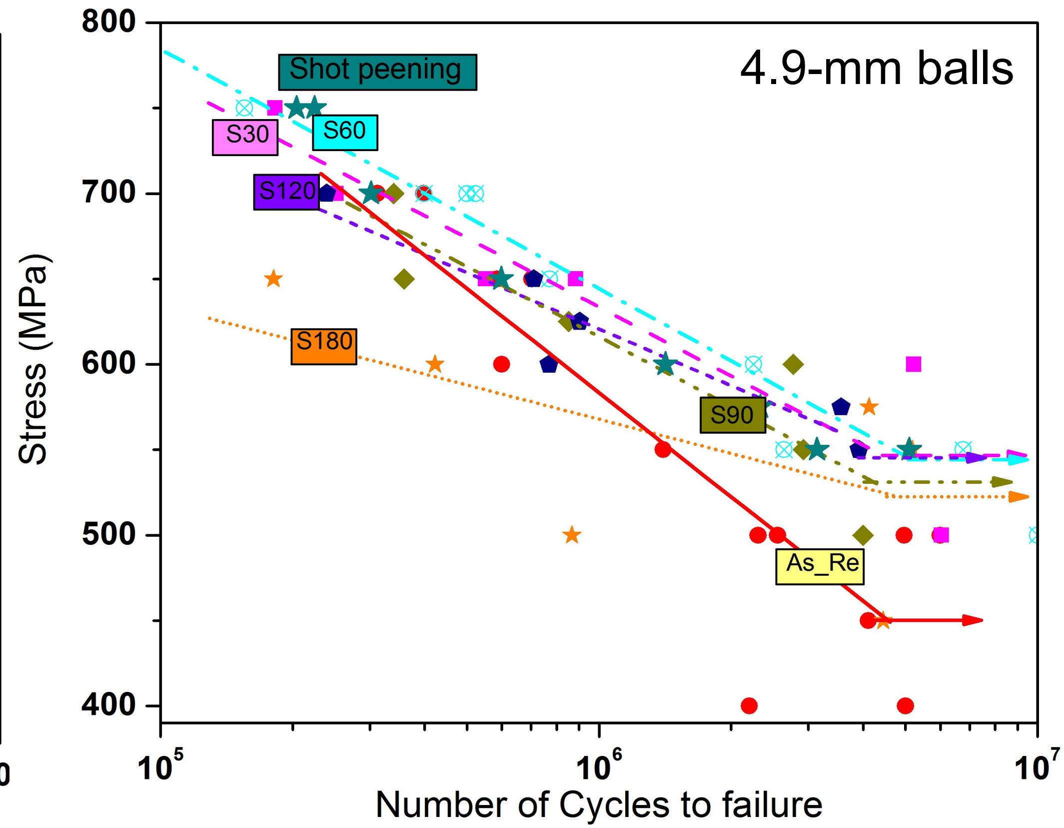
$$R = \frac{\sigma_{min}}{\sigma_{max}} = 0.1$$

P: load
W: width
h: height

$\sigma$: stress
R: stress ratio

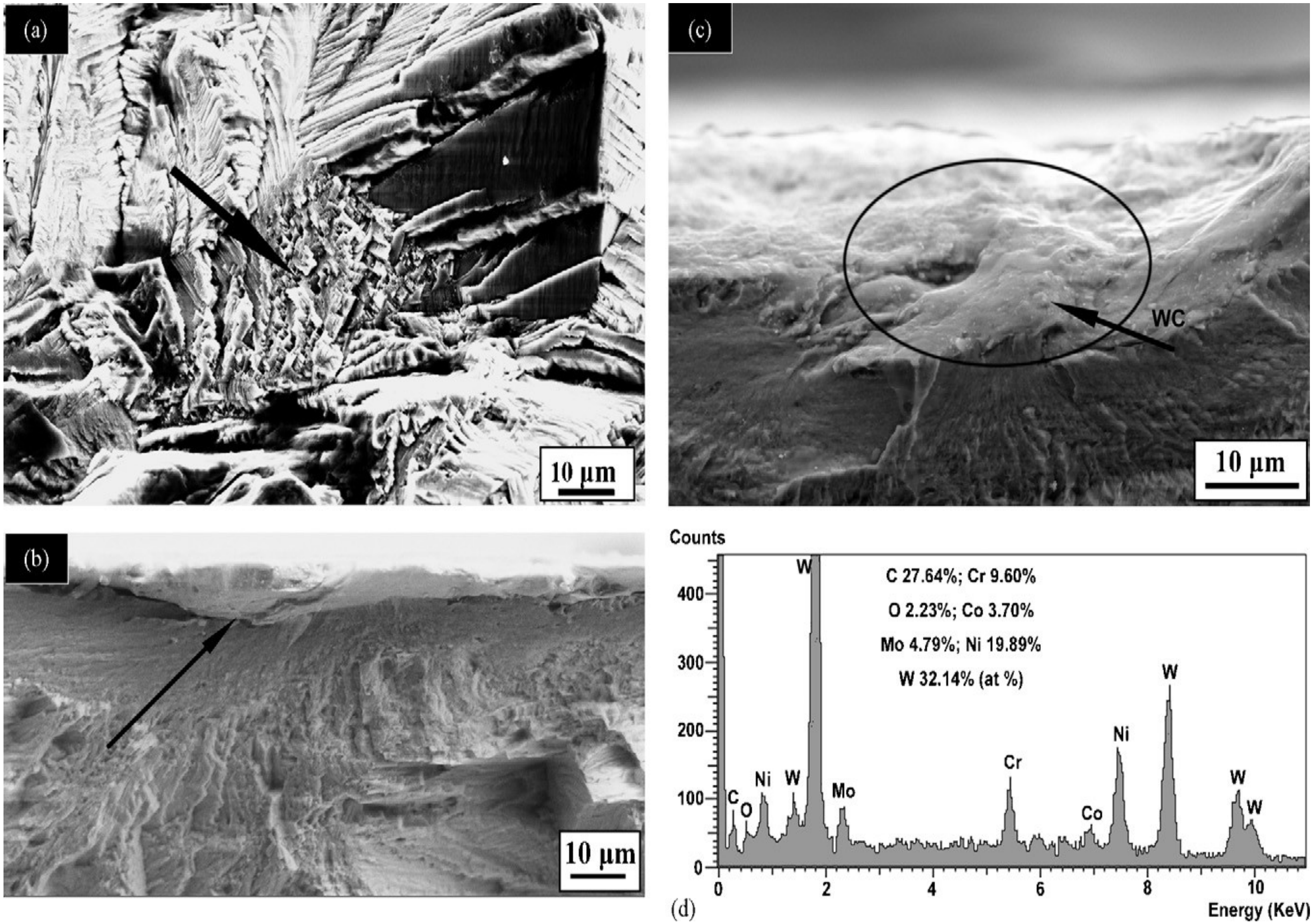
# S-N curves



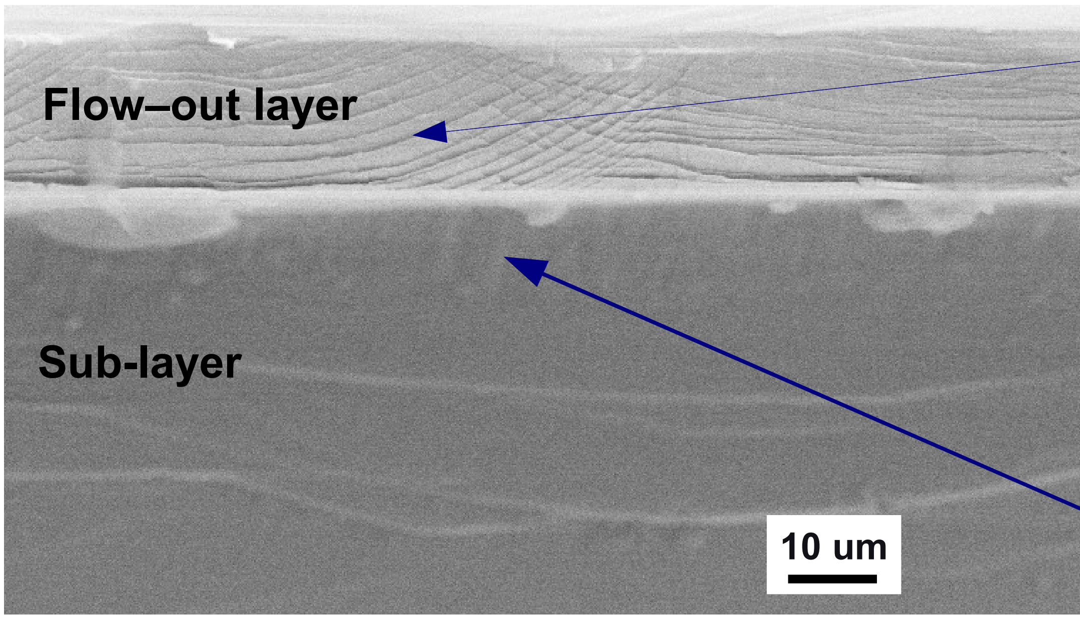

- The fatigue performance of the sample processed with 7.9-mm balls decreases with the processing time;
- The fatigue performance of the sample processed with 4.9-mm balls does not show much difference with time, and all strengths are higher than that of the as-received sample;

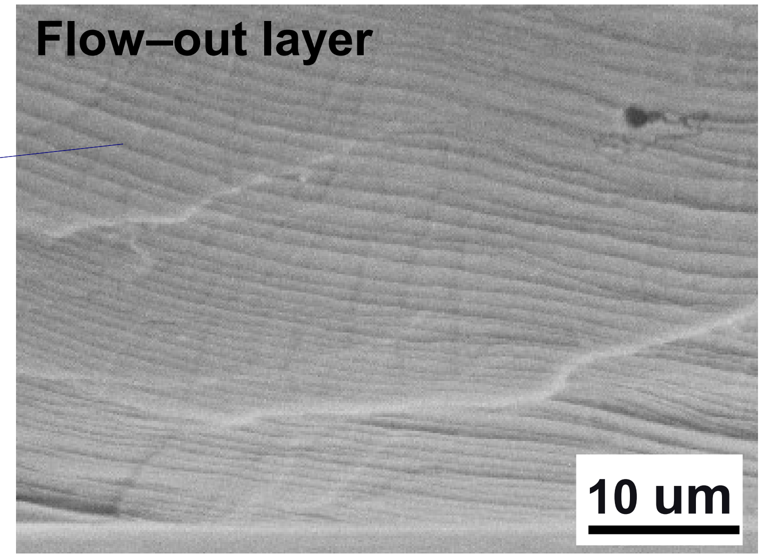
# Fatigue fractography

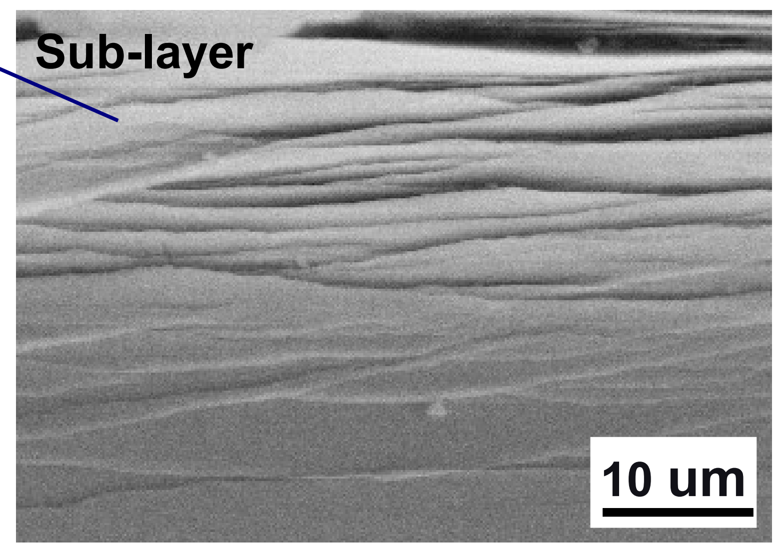


# Effects of Surface Treatment on Amorphous Zr-based $Zr_{50}Cu_{40}Al_{10}$ Bulk Metallic Glass

# Side View of the Deformed Surface Layer

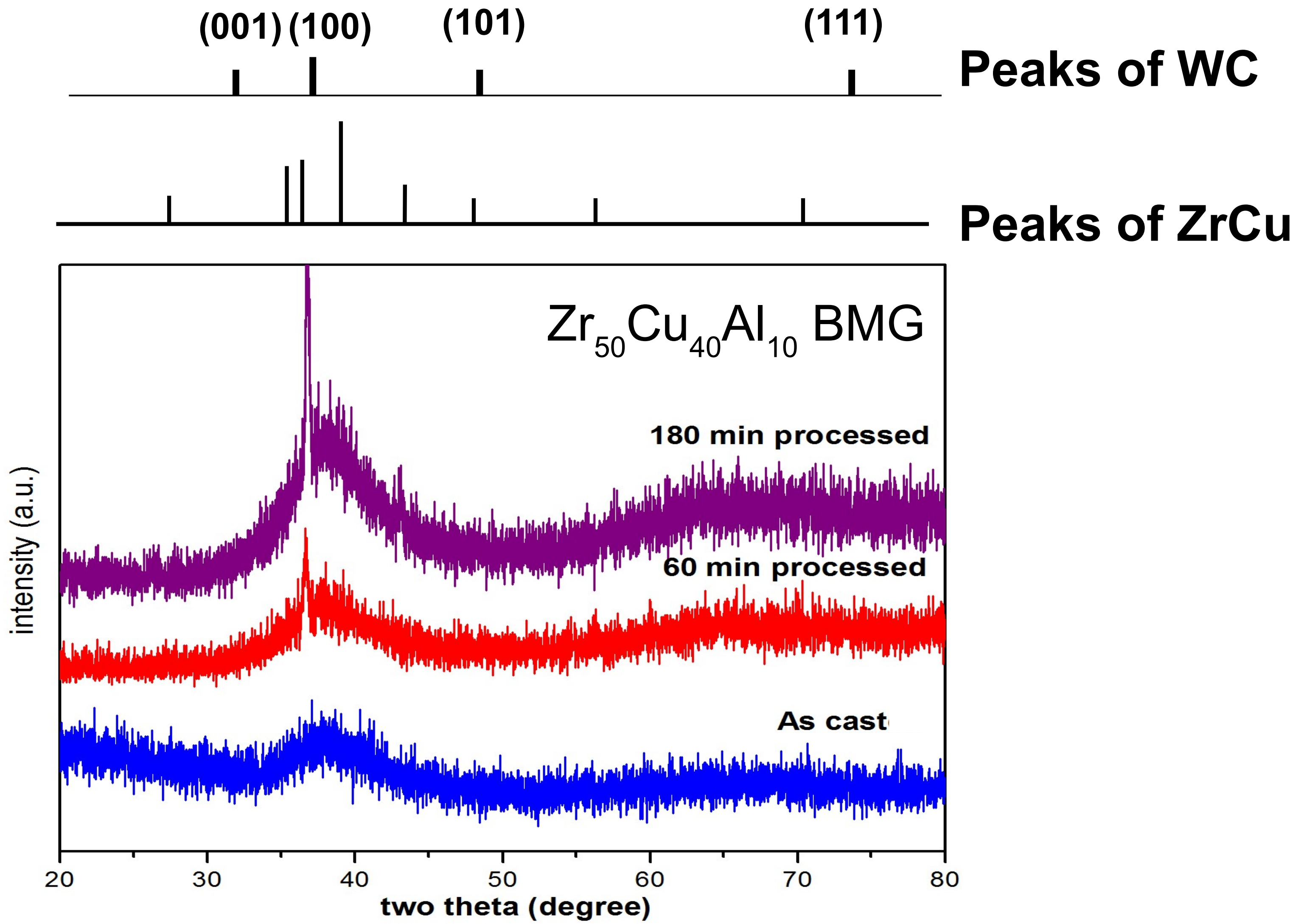


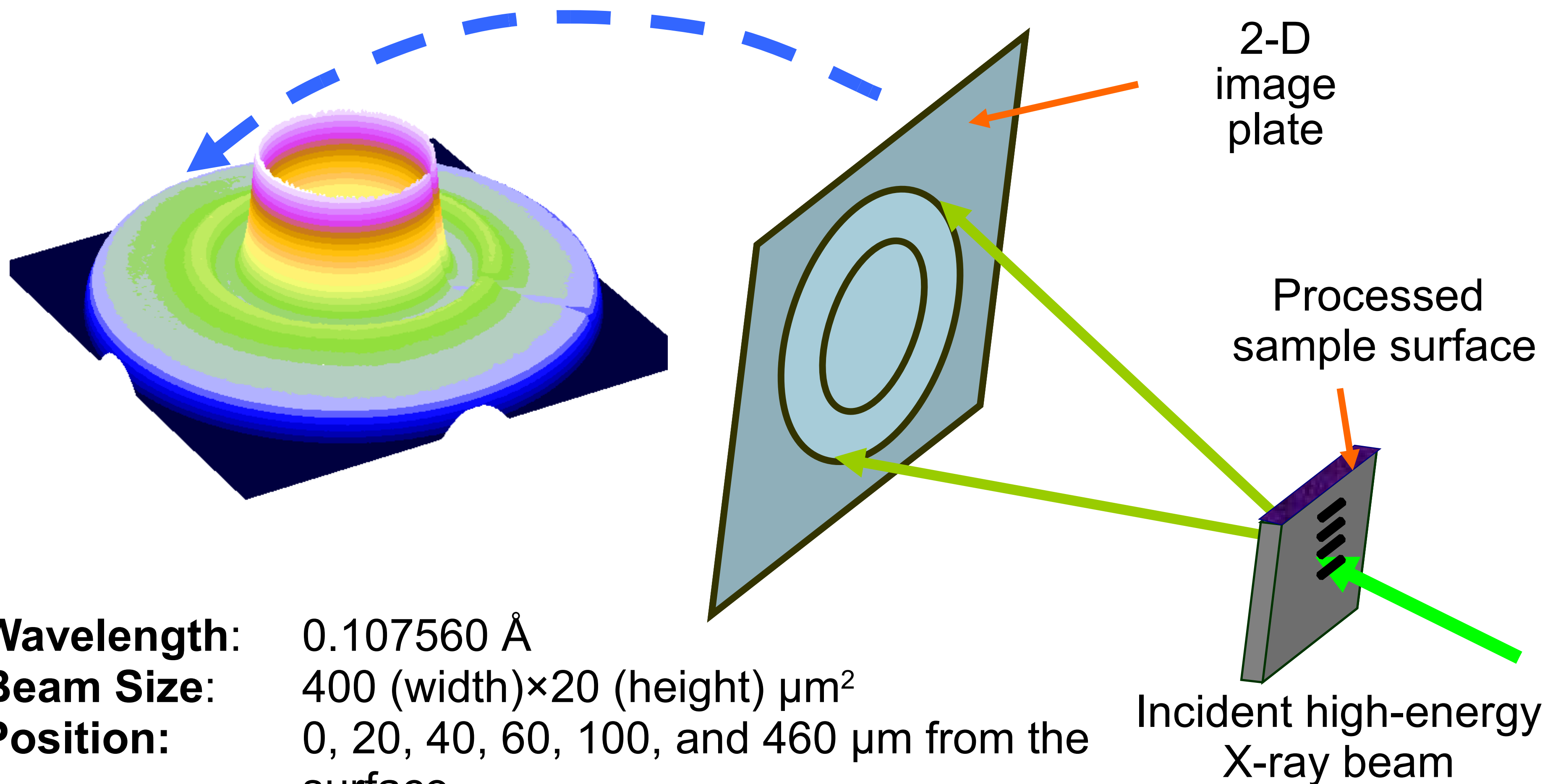

180-min-Processed $Zr_{50}Cu_{40}Al_{10}$

# XRD from the Processed Surface




Crystal phase appears after the surface treatment process

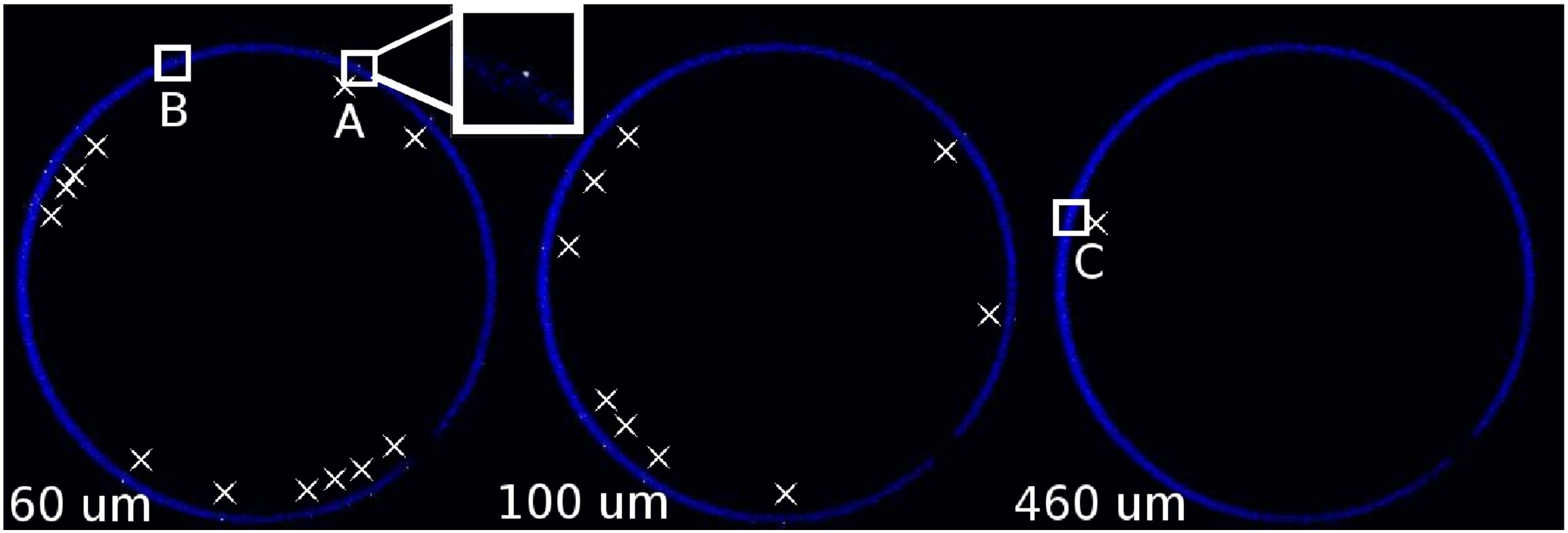
# Synchrotron high-energy XRD from the side surface*




**Wavelength**: 0.107560 Å
**Beam Size**: 400 (width)×20 (height) $\mu m^2$
**Position:** 0, 20, 40, 60, 100, and 460 μm from the surface

*Conducted at the Advanced Photon Source (APS), **Argonne National Laboratory**

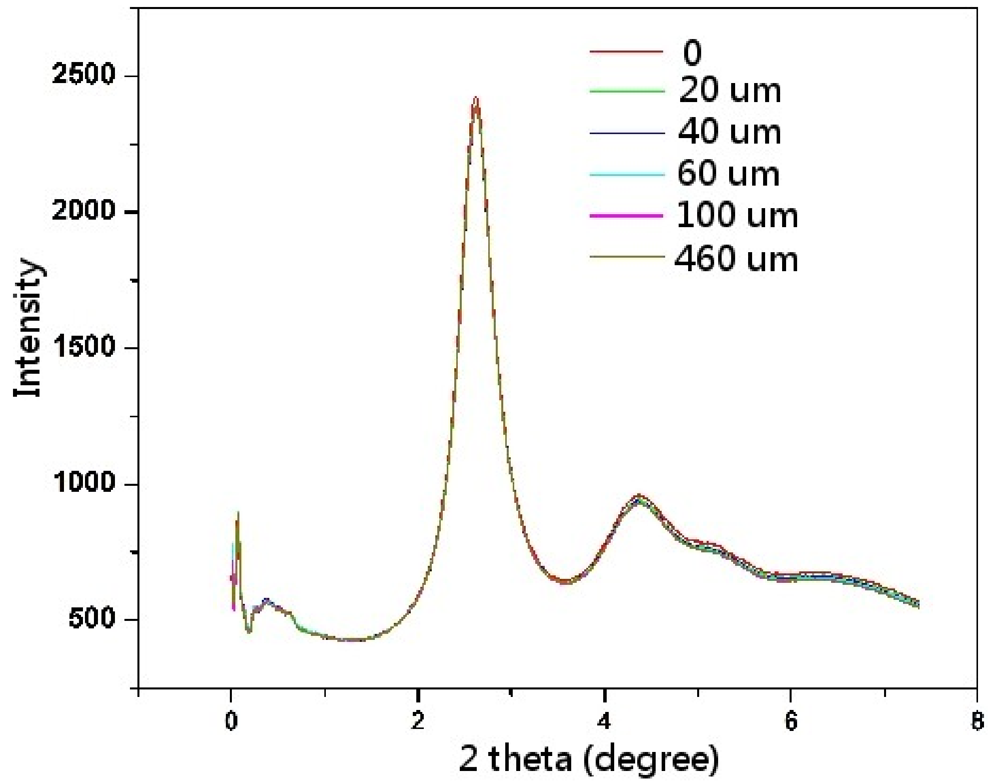
# 2-D Synchrotron high-energy x-ray patterns at different positions

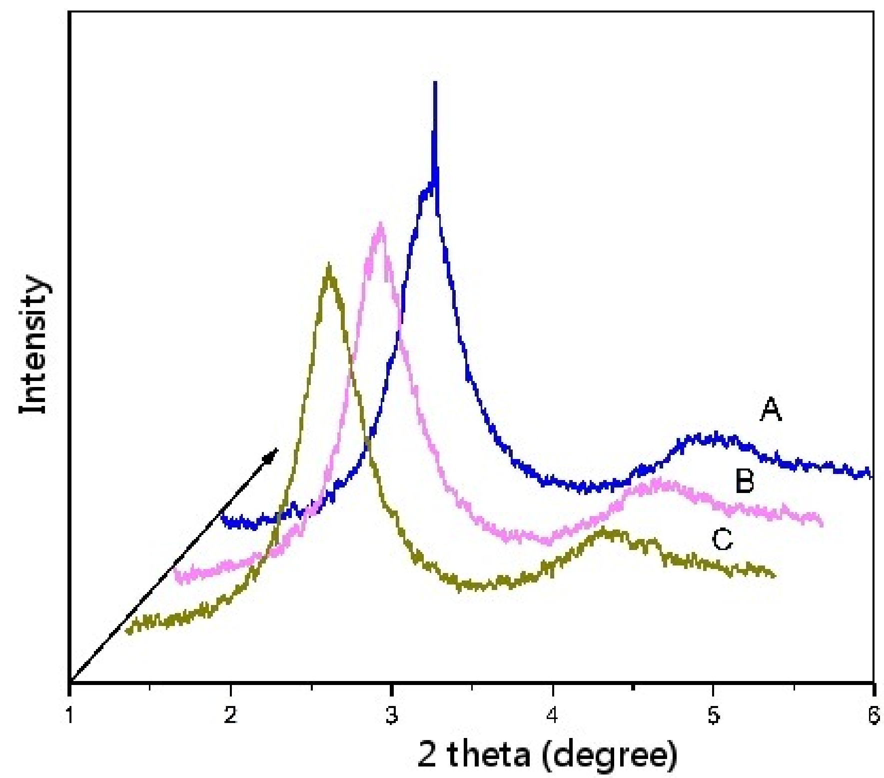



Relative amount of crystal phases becomes less and less when the position moves from processed surface to the interior

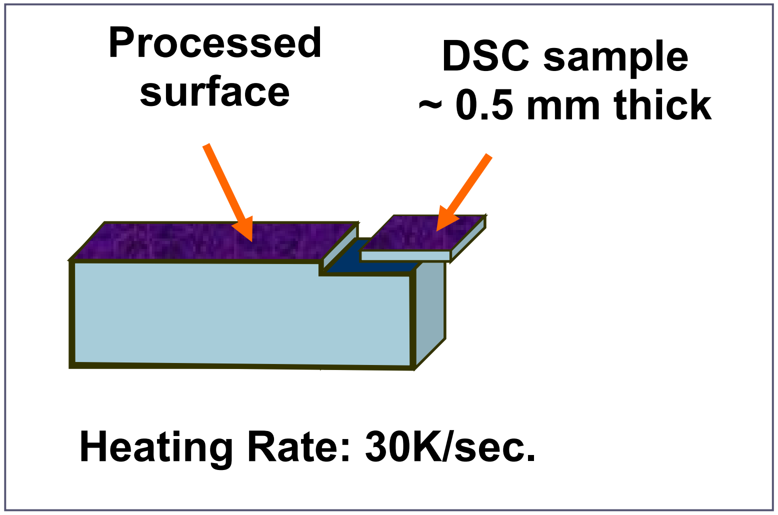
J. W. Tian, L.L. Shaw, Y.D. Wang, Y. Yokoyama, and P.K. Liaw , "A Study on the Surface-Severe-Plastic Deformation Behavior of a Zr-based Bulk-Metallic Glass (BMG)", (revised version under review)

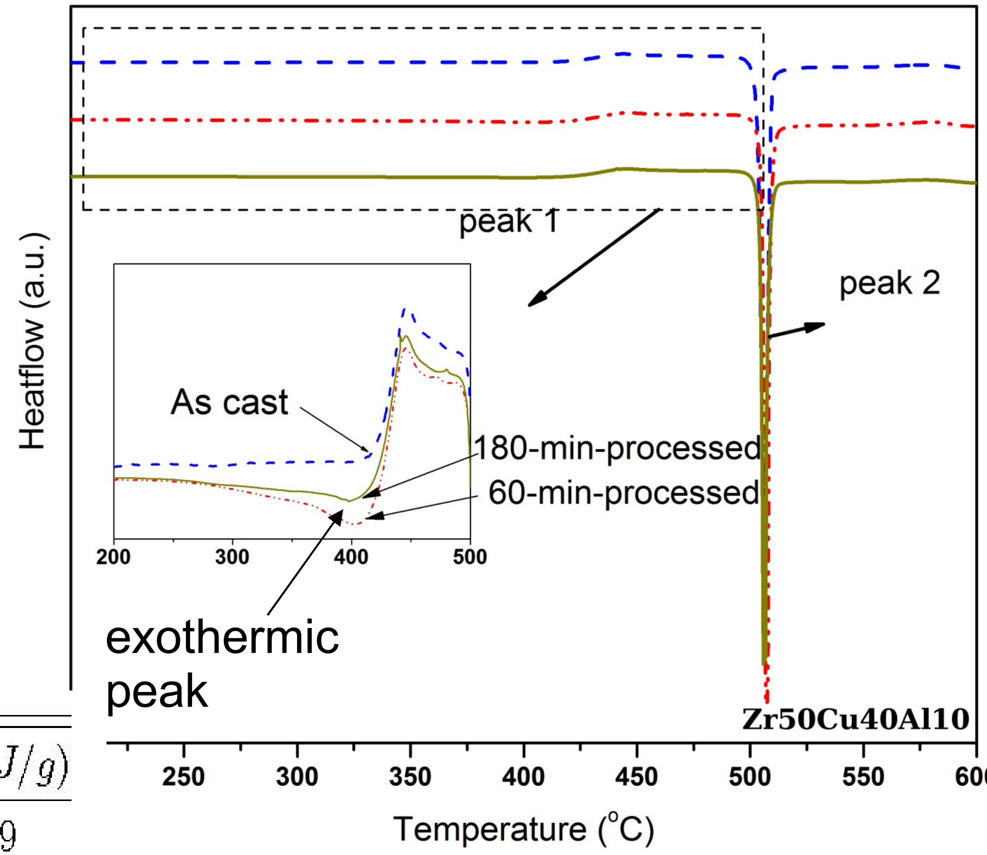
# Integrated 1-D synchrotron high-energy X-ray spectra



# Differential-Scanning Calorimetry (DSC)

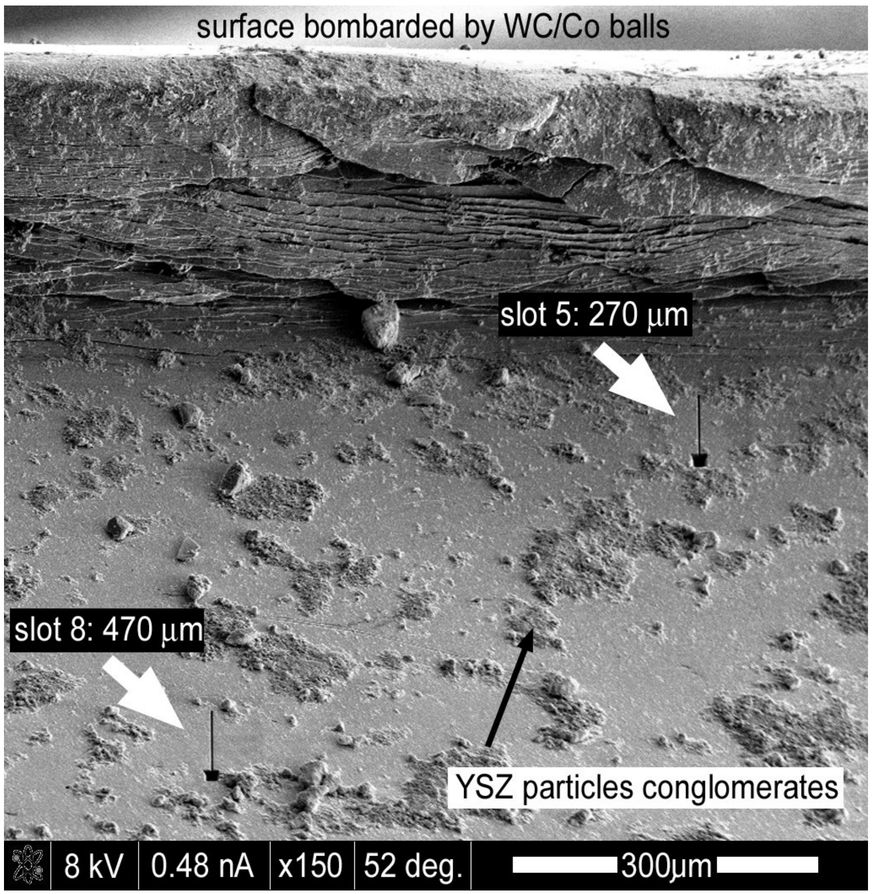


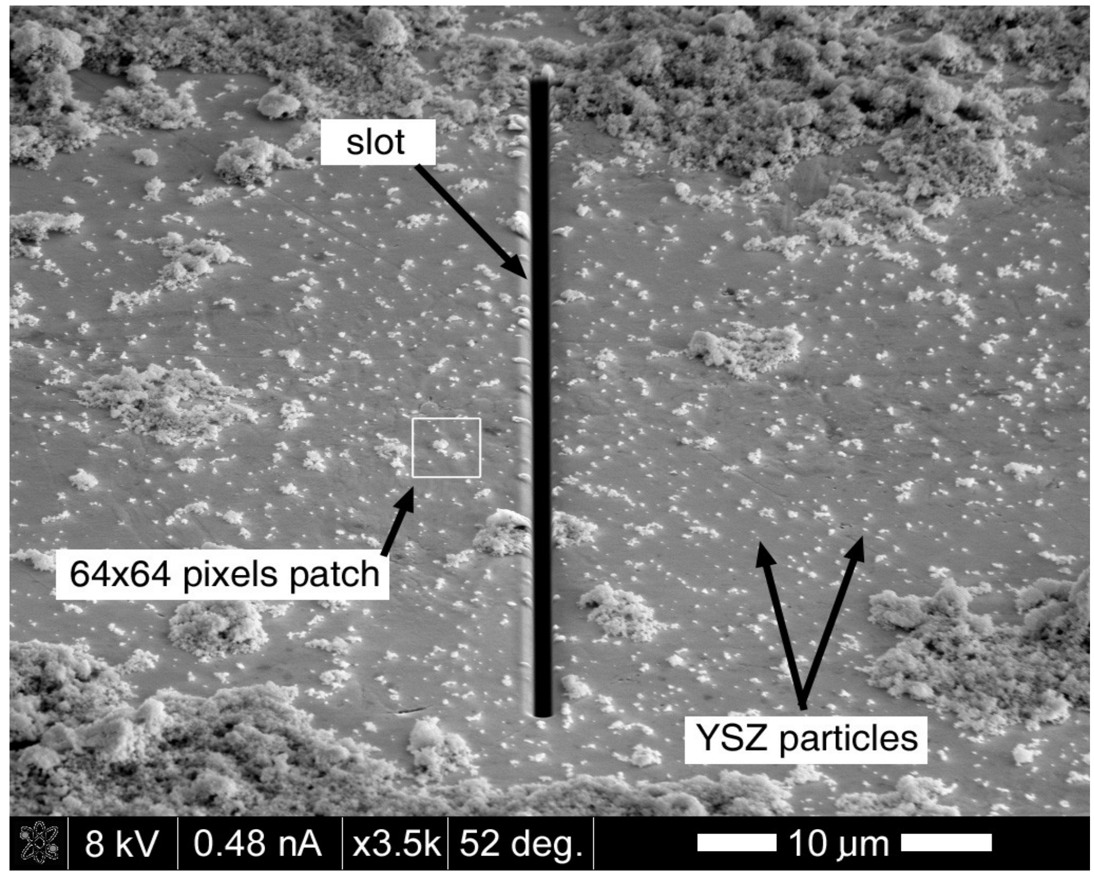




| Sample | $Tg(^\circ C)$ | $Tx(^\circ C)$ | $\Delta H_2(J/g)$ |
|---|---|---|---|
| As-Cast | 422.8 | 501.9 | 45.9 |
| 60 min | 421.7 | 502.3 | 42.7 |
| 180 min | 420.5 | 504.0 | 36.4 |

DSC shows that the crystallization enthalpy, $\Delta H_2$, decreases as the processing time increases

# Residual stress measurement through focused ion-beam (FIB)

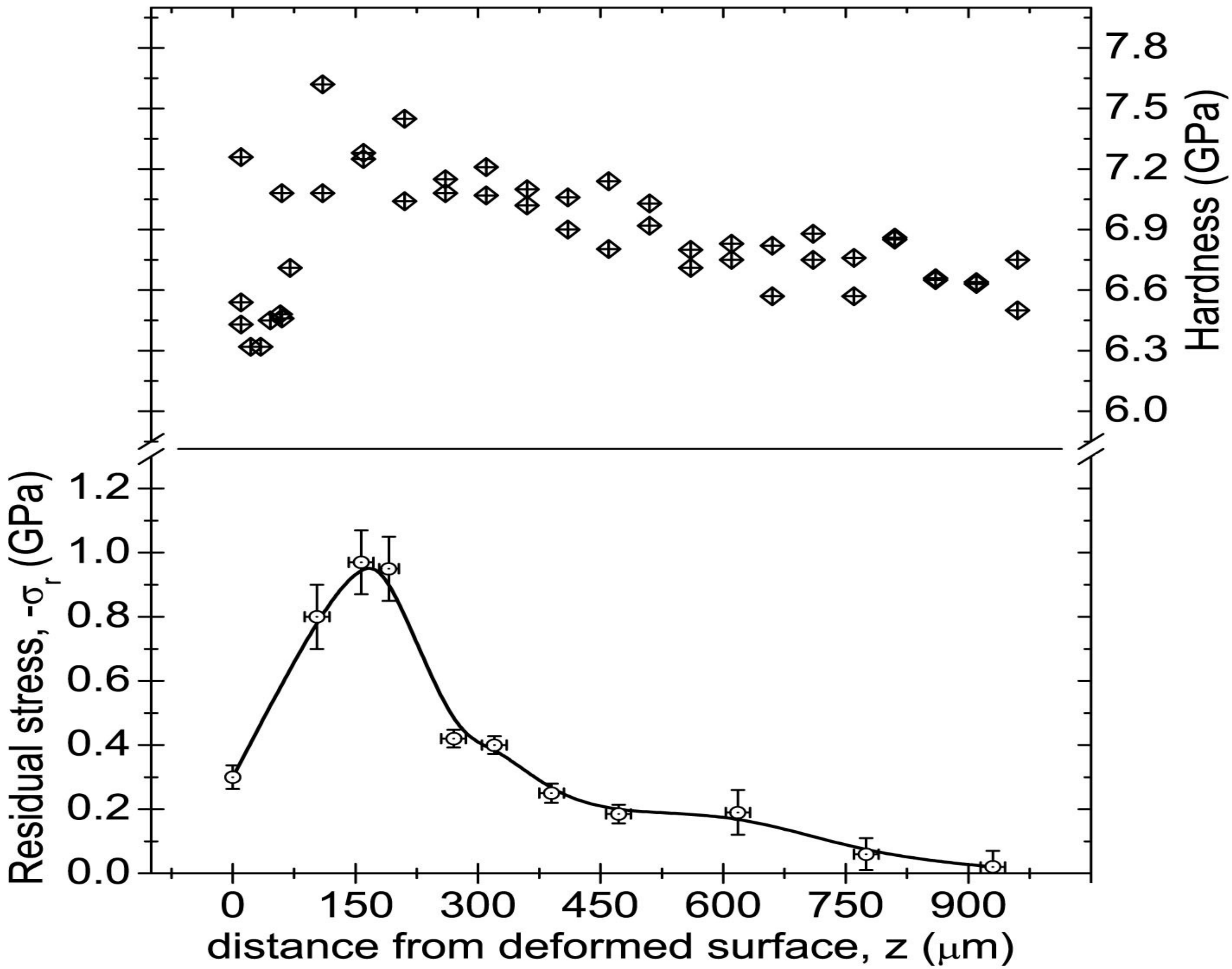


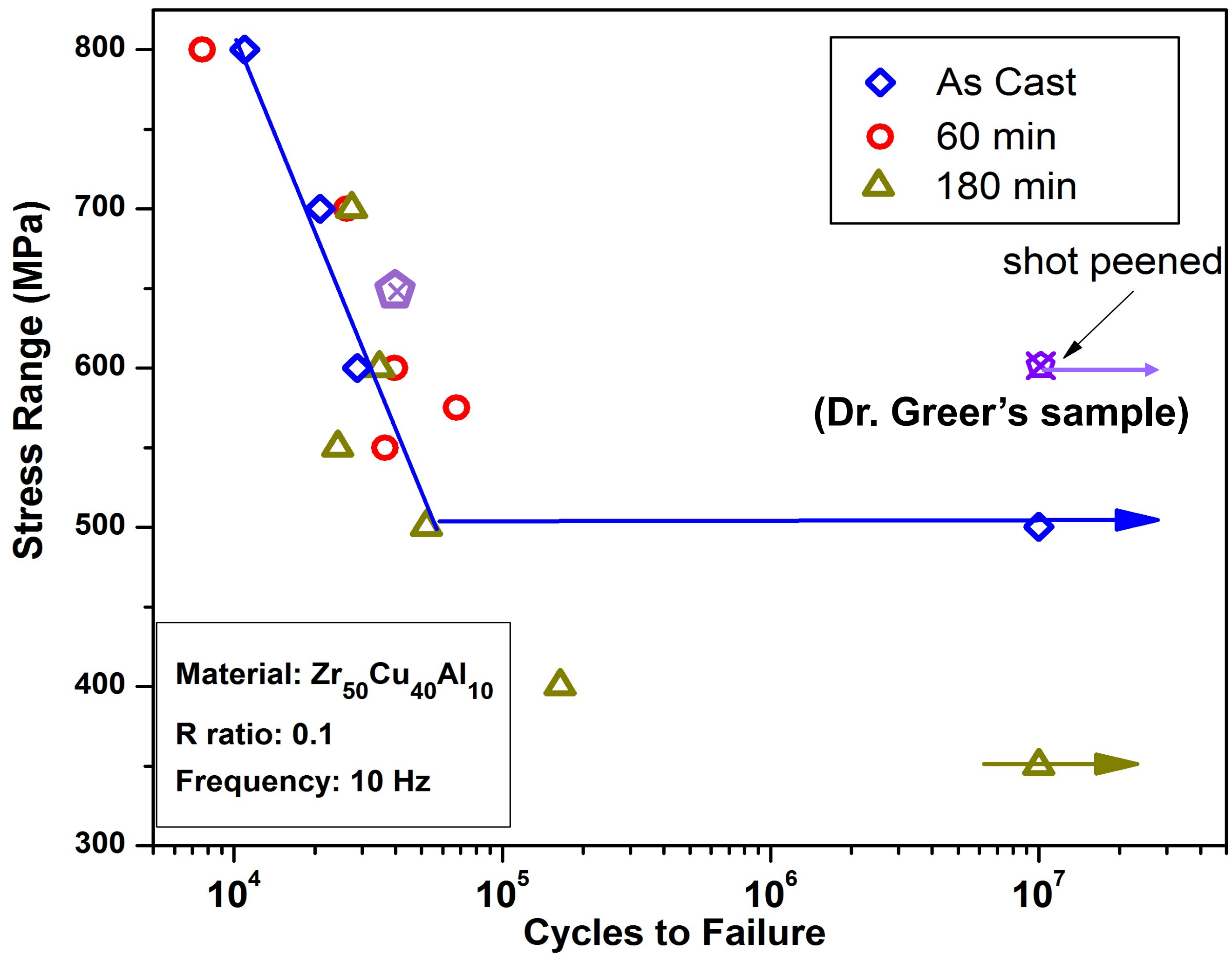



B. Winiarski, J.W. Tian, R.M. Langford, P.K. Liaw, and P.J. Withers, "Residualstress measurements of amorphous materials using a focused ion beam",(in preparation)

# Microhardness and residual stress profiles



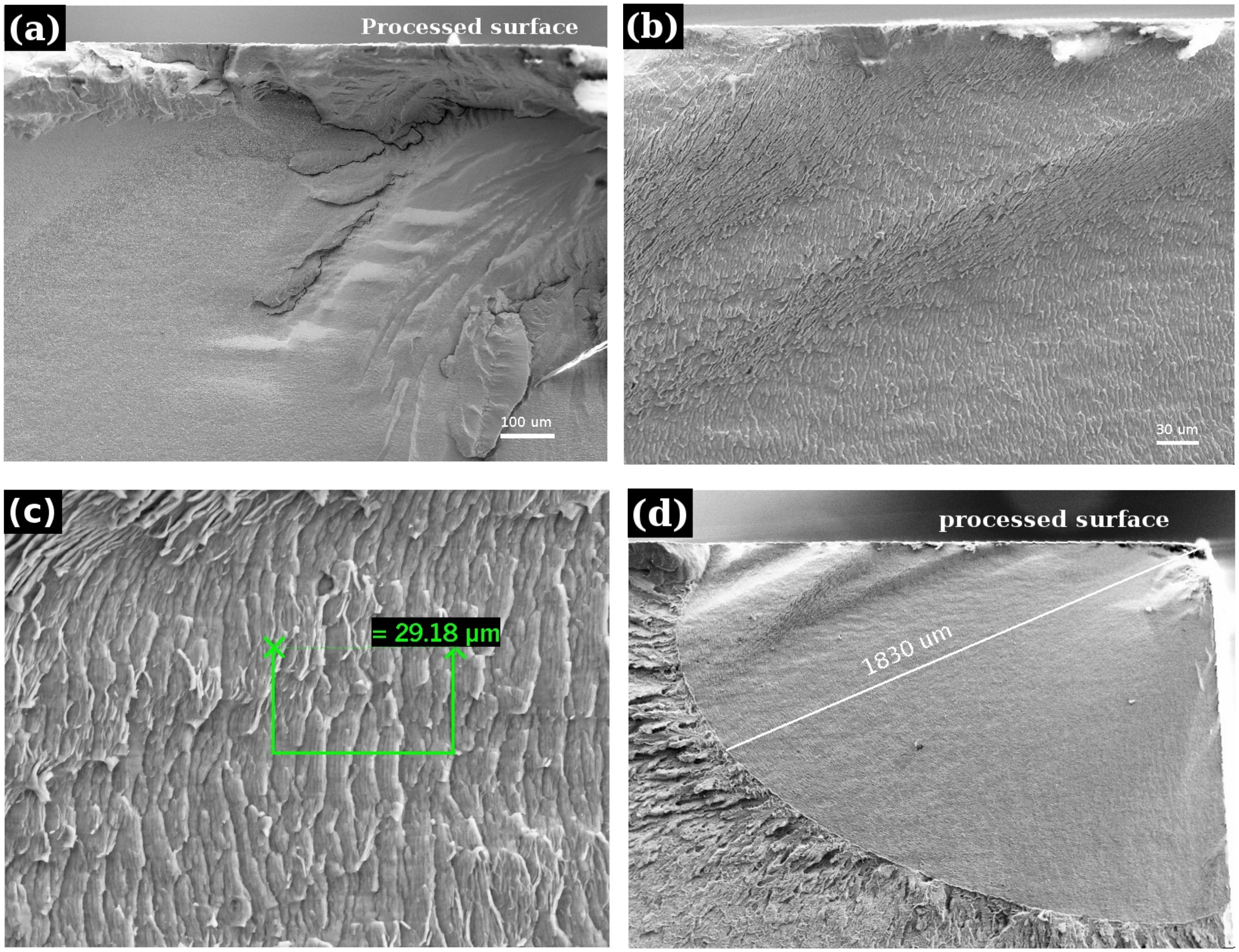
# Decreased Fatigue Strength




* The as-cast results were from Dr. G.Y. Wang

G.Y. Wang, P.K.Liaw, W.H. Peter, et al., Fatigue behavior of bulk metallic glasses, Intermetallics, 2004(12), 885-892

# Fractography of Fatigue Samples

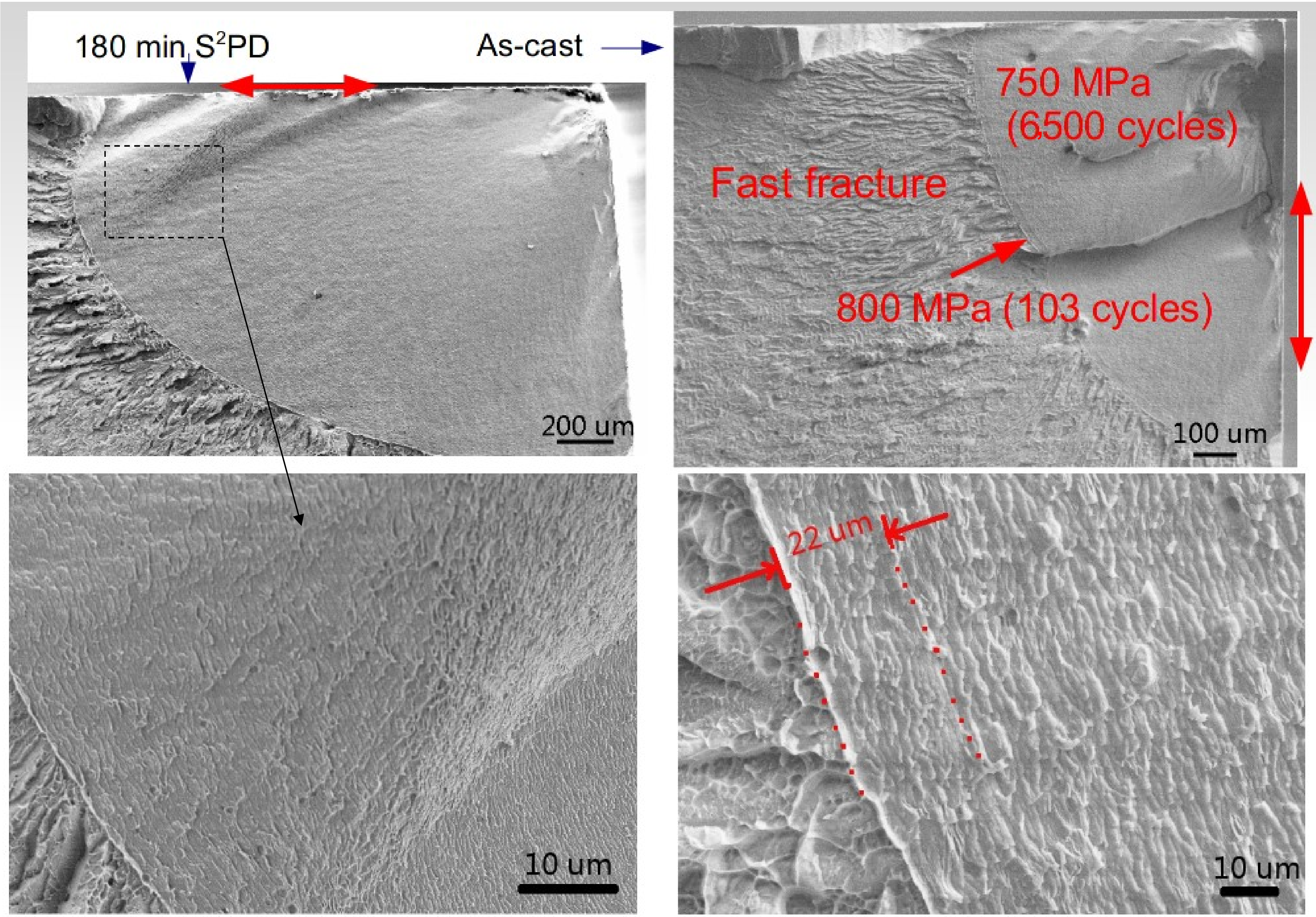



Sample: $Zr_{50}Cu_{40}Al_{10}$
Processing time: 60 min
Load: 800 MPa
Fatigue Life: 7633 cycle

Striation width: ~ 4.17 µm
# of Striations: ~ 439

% of propagation in total life:
439/7633 = 5.75%

# One cycle, One striation?






**Conclusion: Propagation life takes about 50% of the total life**

# Conclusions:

- Changes after the $S^2$PD process are:
    - the formation of a surface nanocrystalline layer;
    - the surface work hardening;
    - the presence of residual compressive in-plane stresses at the surface layer;
    - the increased surface roughness;
    - the surface contamination due to the material transfer between balls and the plate.
- Fatigue strength of the $S^2$PD-treated samples were highly improved due to the nano-layer, residual compressive and work hardening under certain processing conditions; However, excessive treatment may deteriorate the fatigue properties.

# Conclusions (cont'd)

- For the BMG material, the process could increase the free volume, and both work hardening and work softening were identified;
- Nano-crystallites were identified after the treatment, and as the distance from the processed surface increases, the amount of nano-crystallites decrease;
- Tg, Tx remain unchanged after the deformation process, and the fatigue property does not benefit from the treatment due to the possible surface damage.

# Selected Publications

- J.W. Tian, J.C. Villegas, W. Yuan, D. Fielden, L. Shaw, P.K. Liaw, and D.L. Klarstrom, "A study of the effect of nanostructured surface layers on the fatigue behaviors of a C-2000 superalloy", Materials Science and Engineering: A, 468-470(11), 2007, pp.164-170
- J.W. Tian, K. Dai, J.C. Villegas, L.L. Shaw, P.K. Liaw, D.L. Klarstrome, and A.L. Ortiz, "Tensile properties of a nickel-base alloy subjected to surface severe plastic deformation", Materials Science and Engineering: A, 493(1-2), 2008, pp.176-183
- J. W. Tian, L.L. Shaw, P. K. Liaw, and K. Dai, "On the ductility of a surface severely plastically deformed nickel alloy", Materials Science and Engineering: A, 498(1-2), 2008, pp. 216-224
- J. W. Tian, L.L. Shaw, Y.D. Wang, Y. Yokoyama, and P.K. Liaw , "A Study on the Surface-Severe-Plastic Deformation Behavior of a Zr-based Bulk-Metallic Glass (BMG)", (revised version under review)
- A.L. Ortiz, J.W. Tian, J.C. Villegas, L.L. Shaw, and P.K. Liaw, "Interrogation of the microstructure and residual stress of a nickel-base alloy subjected to surface severe plastic deformation" Acta Materialia, 56(3), 2008, pp. 413-426
- B. Winiarski, J.W. Tian, R.M. Langford, P.K. Liaw, and P.J. Withers, "Residualstress measurements of amorphous materials using a focused ion beam",(in preparation)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