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Texture and Strain Analysis of Tungsten Films Via Tilt-A-Whirl Methodology

Mark A. Rodriguez, Jamin Pillars, Nichole R. Valdez, James J. M. Griego, Matthew V. Gallegos, John A. Krukar, Andrew Polonsky and Steven L. Wolfley

Sandia National Laboratories, Albuquerque, NM

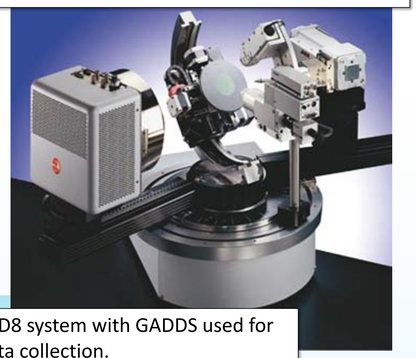
Introduction and Motivation

- Tungsten films are employed for Transition Edge Sensors (TES).
- Residual strain affects the superconducting transition temperature in Tungsten.
- We desire to visualize XRD data in 3D for intuitive analysis of strain and texture.
- A challenging aspect of pole figure visualization is peak broadening at high χ angles due to beam defocusing.
- Removal of defocusing effects improves visualization of strain on the resulting 3D pole figure (plotted as χ, ϕ, and 2θ).
- We employ a simple routine to separate the defocusing broadening from our 3D pole figure data to allow for straightforward visualization.
- We apply our method to textured Tungsten films with significant residual tensile strain.

Tungsten Film Synthesis

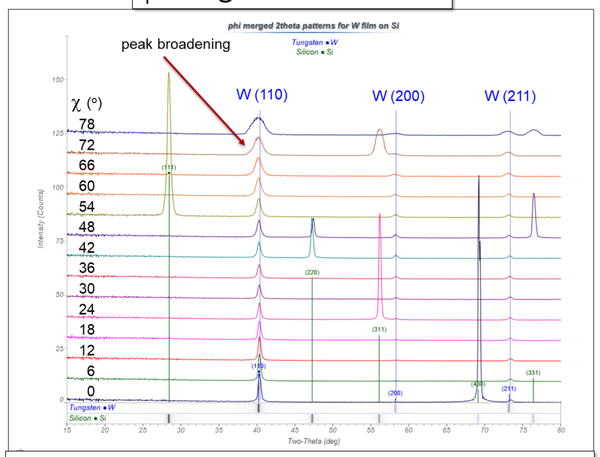
- Films were deposited on Si using a Denton 2 sputter system with a high purity tungsten target. Parameters for film were:
  - 282 watts sputter power
  - 7 mTorr Argon pressure
  - based on A. E. Lita, et al.; IEEE TRANSACTIONS ON APPLIED SUPERCONDUCTIVITY, VOL. 15, NO. 2, JUNE 2005.

Tilt-A-Whirl XRD Measurement

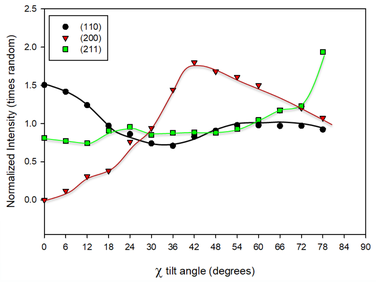


- Bruker D8 system with GADDS used for XRD data collection.
  - Vantec 2000 Area Detector
  - Eulerian-cradle
  - Cu (K_α) source with a 500µm pinhole optic
- Collect 2θ frames at varying chi (χ) and phi (ϕ)
  - 3360 frame images (10 second frames)
  - χ varied from 0 to 78° in 6° increments
  - ϕ varied from 0 to 354° in 6° increments
- Rodriguez, et al., *Powder Diffraction* **28** 81-89 (2013).

ϕ-merged XRD Patterns

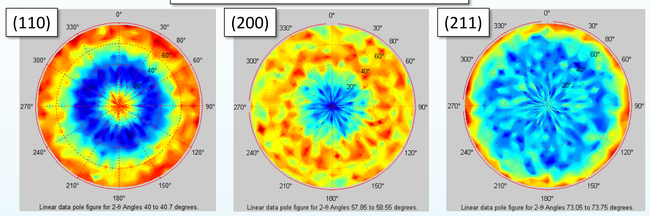


- XRD scans show mild texture for α-W (BCC) as well as peaks from the Si substrate.
- α-W peaks show detectable peak shift to lower 2θ with increased tilt angle (χ).
- There is significant peak broadening due to defocusing.



- **Normalized Intensity *vs* χ tilt angle** shows a mild texture with (110) out-of-plane preferred orientation. The (110) shows an additional broad intensity peak centered at χ = 60°.
- The (200) intensity shows the expected maximum at χ = 45° as dictated by the (110) out-of-plane preferred orientation.
- The (211) shows a nearly constant intensity from overlapping maxima of the 30°, 54.7°, 73.2° and 90° interplanar angles which are all allowed for a (110) out-of-plane preferred orientation film.

Tungsten Pole Figures

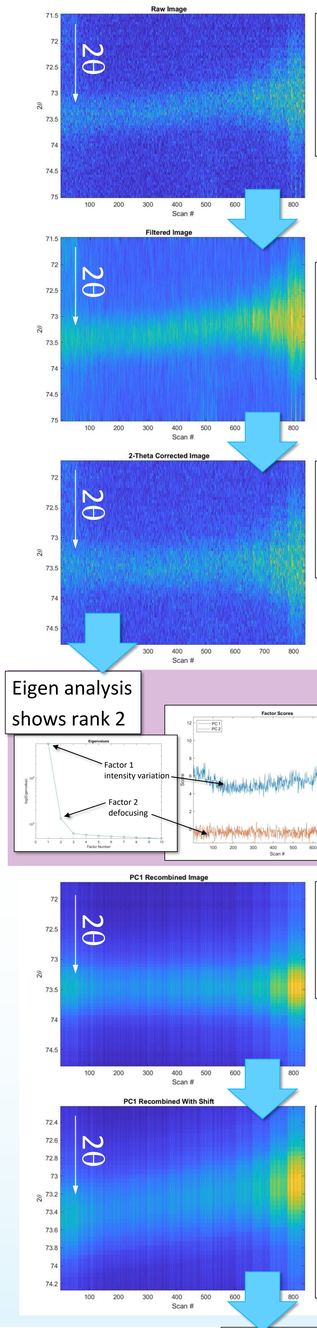


The (110) pole figure shows a mild out-of-plane texture.

The (200) pole figure shows a ring at χ = 45° confirming in-plane fiber texture.

The (211) pole figure shows only a small intensity variation until high χ angles.

Data Processing for Strain Visualization



This plot shows all 840 XRD patterns measured for the (211) peak. This series shows a clear shift to lower 2θ values with increased χ (i.e. scan #).

Scans are smoothed to enable improved fitting and determination of 2θ shift with scan number (χ).

Raw data is shifted and patterns are interpolated to maintain alignment within the data matrix.

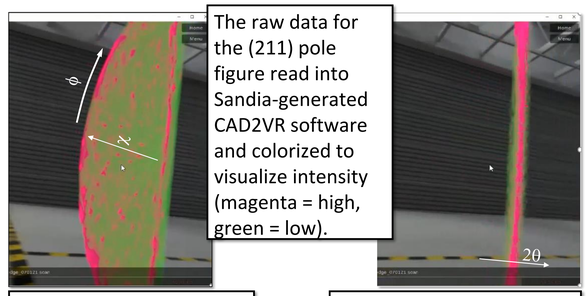
Principal Component Analysis shows Factor 1 as isolated peak for α-W (211) and Factor 2 as defocusing.

Weights and Scores from Factor 1 are recombined to build a new data matrix absent defocusing artifacts.

XRD scans in the new data matrix are then corrected back to original 2θ location based on initially derived peak maxima. The patterns are again interpolated in 2θ to maintain matrix alignment.

Raw and corrected data matrices are ported to Virtual Reality for visualization

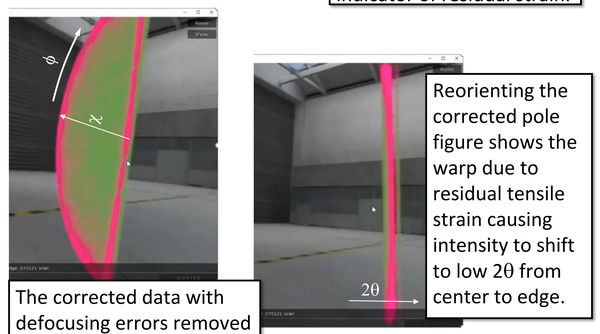
Visualizing Strain in Virtual Reality (VR)



The raw data for the (211) pole figure read into Sandia-generated CAD2VR software and colorized to visualize intensity (magenta = high, green = low).

The pole figure is cross-sectioned to view the intensity distribution in the **2θ dimension**.

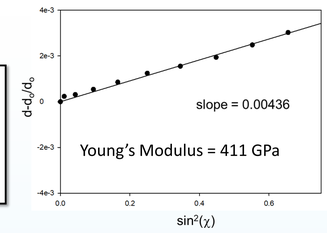
When the pole figure is viewed ***edge-on*** we see the **warp** of the intensity causing an arc from the center to the edge of the figure. This is a tell-tale indicator of residual strain.



The corrected data with defocusing errors removed shows a cleaner view of the (211) pole figure.

Reorienting the corrected pole figure shows the warp due to residual tensile strain causing intensity to shift to low 2θ from center to edge.

Conventional $\sin^2(\chi)$ analysis shows strong in-plane tensile strain with estimated stress of **1.8 GPa** for α-W film.



Summary

- We have successfully visualized residual strain effects in VR using the Sandia-developed CAD2VR software.
- The use of Principal Component Analysis enables the extraction of defocusing effects to better visualize 3D datasets.
- This sputter-deposited α-W film showed very high in-plane tensile strain as confirmed via conventional $\sin^2(\chi)$ analysis.
- 3D visualization enables rapid, intuitive detection of strain.

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